IARJSET

ISSN (O) 2393-8021, ISSN (P) 2394-1588

International Advanced Research Journal in Science, Engineering and Technology

ISO 3297:2007 Certified ⁜ Impact Factor 7.105 ⁜ Vol. 9, Issue 6, June 2022

DOI: 10.17148/IARJSET.2022.9628

Experimental Investigation on Mechanical Behavior of Bio-Composite Material

UDHAYA. A. R[1], T. SEKAR[2], N. NANDAKUMAR[3], R. SURENDRAN[4]

PG Research scholar, Government College of Technology, Coimbatore[1]

Professor and HOD Manufacturing Engineering, Government College of Technology, Coimbatore[2]

Professor and HOD Engineering Design, Government College of Technology, Coimbatore[3]

Assistant Professor Manufacturing Engineering, Government College of Technology, Coimbatore[4]

Abstract: Polylactic acid (PLA) and hydroxyapatite (HAp) are both used in bone healing materials in different ways. Magnesium (Mg)-based biomaterials are being studied extensively for implant applications that are biodegradable. One of the approaches used to enhance bioactivity and minimize Mg degradation rate is to develop Mg-based composites. In this study, new bio-composite material was developed using Hydroxyapatite (HAp) which has been derived from fishbone waste and Magnesium (Mg) reinforced Polylactic acid (PLA) composites. This Bio-composite was prepared through a compression moulding method with different wt.%, such as PLA/HAp/Mg (84%, 15%, 1%), (82%, 15%, 3%), and (80%, 15%, 5%) respectively. The mechanical behaviour of HAp/Mg reinforced PLA was investigated through universal tensile testing. The tensile strength of PLA has been improved by incorporating the HAp/Mg. The ultimate tensile strengths of virgin PLA and PLA/HAp/Mg composites (84%, 15%, 1%), (82%, 15%, 3%), and (80%, 15%, 5%) were 37.9, 40.5, 56.7, and 70.9 MPa respectively. The ultimate tensile strength of the PLA/HA/Mg-(80%,15%,5%) composite was approximately two times greater than virgin PLA. Based on the project outcomes of mechanical strength, the fabricated HAp/Mg with PLA composite is the promising material for high-strength bone replacement applications.

Keywords: Polylactic acid (PLA), hydroxyapatite (HAp), Magnesium (Mg)

I. INTRODUCTION

Healing of bone defects has remained a difficulty in recent years, resulting in an increase in demand for bone repair materials. To overcome this limitation, adequate bone repair materials with good mechanical properties and osteogenic activity must be created. Polylactide (PLA), a biodegradable polyester, has become popular in the bone healing sector. However, its mechanical properties are poor, prohibiting it from meeting the mechanical support requirements of bone repair. Bio-nanoparticles employed as reinforcements to increase the mechanical properties of the PLA matrix have been demonstrated to be successful and widely used. More importantly, bone repair materials should be osteoinductive, permitting natural formation of new bone tissue. Because of PLA's osteogenic properties, it's a good choice for people who want to Adding bioactive chemicals to PLA has been proven in numerous studies to significantly boost its osteogenic activity. Some studies have sought to improve PLA's mechanical properties and osteogenic activity by adding magnesium oxide or halloysite nanotubes to it. Natural bone is made up of collagen fibres and hydroxyapatite (HAp), which is composed of calcium (Ca), phosphorus (P), and a few trace elements. HAp has been used to increase the mechanical features and osteogenic activity of PLA because of its exceptional rigidity and osteoinductivity. According to some investigations, HAp particles and the PLA matrix can form favourable inorganic and organic interactions due to hydrogen bonding and weak ionic bonding. During mechanical testing, HAp particles may transfer successive loads from the polymer matrix to stiff particles via the PLA and HAp interface, resulting in a mechanical reinforcing effect for the PLA matrix. Because of their high aspect ratio and fewer surface flaws, HAp whiskers have a higher elastic modulus and ultimate tensile strength than HAp powders, and are more conducive to improving the mechanical strength and modulus of the polymer matrix. Because of its various benefits, such as biocompatibility and biodegradability, magnesium and its alloys are considered new materials for orthopaedic implants.

On the other hand, Because of its numerous advantages, such as biocompatibility and biodegradability, magnesium and its alloys are recognised as innovative materials for orthopaedic implants. Traditional metallic biomaterials like titanium alloys and stainless steels are used in orthopaedic applications. They do, however, have two issues: a high Young's modulus and the need for a second procedure to remove implants once they have healed. A stress shielding effect is caused by a Young's modulus misfit between implant 2 and human bones, which results in decreased bone density. At the same time, the need for a second surgery is a significant issue. When implants are removed, complications such as screw breakage and hidden implants can arise. Furthermore, the expense of surgery is prohibitively high.

Magnesium and its alloys are frequently touted as potential solutions to these two issues. Reduced lack of fit also has fewer chances of causing stress shielding effects. Magnesium is also a biodegradable substance, meaning it can dissolve in the human body. As a result, after the healing process, the implant does not need to be removed. Magnesium is also non-toxic because it is a necessary component of the human body. Magnesium and its alloys, on the other hand, have a number of issues, including rapid corrosion and hydrogen evolution. The materials degrade at a rate that is far faster than the rate at which human bone heals. As a result, maintaining the required mechanical integrity during the healing process is difficult. Furthermore, due of the rapid rate of degradation, the hydrogen evolution rate must be considered, as hydrogen gas might harm the surrounding tissue. As a result, slowing the rate of degradation is critical if magnesium and its alloys are to be used for orthopaedic implants. In this application, hydroxyapatite (HAp) is the optimum material for forming a composite with magnesium. Human bones are made up primarily of HAp, which has a high biocompatibility and good mechanical characteristics. Furthermore, HAP performs an unrivalled function in bone healing in humans. According to recent research, Mg-HAp composites or Mg alloys with HAP coatings are interesting prospects for orthopaedic implant applications due to improved mechanical properties. In this present work, new bio-composite material was developed using Hydroxyapatite (HAp) which has been derived from fishbone waste and Magnesium (Mg) reinforced Polylactic acid (PLA) composites. This Bio-composite was prepared through a compression moulding method with different wt.%, such as PLA/HAp/Mg (84%, 15%, 1%), (82%, 15%, 3%), and (80%, 15%, 5%) respectively. The mechanical behaviour of HAp/Mg reinforced PLA was investigated through universal tensile testing.

II. MATERIALS AND METHODOLOGY

Polymer composites are utilised in dentistry, orthopaedics, tissue engineering, and regenerative medicine, among other biomedical applications. Biomedical polymers are materials that are engineered to work in tandem with the human body. They lay the groundwork for an increasing number of artificial organs and replacement parts designed to fit inside the human body. Polymeric materials have proven to be valuable in the realm of biomedicine Table.1. This is due to their benefits, which include ease of processing, lightness and flexibility, high strength-to-weight ratio, availability, and recyclability. And the methodology of the work is shown in Fig.1.

Table 1. List of biopolymers used in medical applications

Bio polymers	**Medical applications**
Polyhydroxy Kanoates (PHA)	Medical apparel accessories, suture, galenic, vascular implant
Polyglycolides(PGA)	Suture, clip, staple and adhesive
Polylactic acid	Fixation, screw and pin, artificial ligament and tendon, tissue regeneration matrix, orthopaedic fixation
Polyglactin (PLA-PGA)	Sutures, orthopaedic fixation, screws and pins, ligaments, tendon and artificial vessels.
Cellulose	Cell implantation and drug encapsulation
Polyspartates	Suture, artificial skin, medication encapsulation
Poly-lysine	Drug encapsulation, biosensors, and bactericides

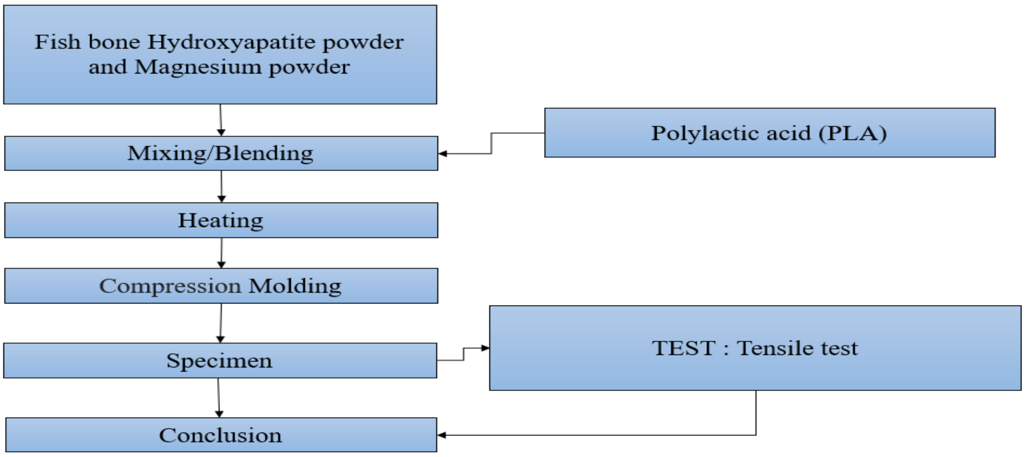

Fig 1. Work process

Materials and PLA/Hap/Mg polymer Bio-composite

PLA (poly (lactic acid)) was purchased from a supplier (Medical grade in pellet form, Polymers Ind Pvt Ltd). The Bio-polymer composite was developed using a matrix material from Coimbatore, India, with a melting point temperature of 200 °C. HAp and Mg (Nano Technologies Pvt Ltd, Coimbatore, India) were used as bio-materials for reinforcement into the polymer matrix. Melting temperatures for HAp and Mg were 1670°C and 630°C, respectively.
A vertical compression moulding procedure was used to create a functionalized HAp and Mg particle reinforced PLA polymer. The PLA polymer, HAp, and Mg particle weight percent were chosen as follows:(84%, 15%, 1%), (82%, 15%, 3%), and (80%, 15%, 5%) for fabricating the composite. The PLA polymer was mixed with preheated HAp and Mg in the proper proportions before being fed into the moulding machine with the mould die indicated in Fig 2.1. Fig .2.2 shows the compression moulding technique for making bio-composite specimens. The closed mould was heated until the temperature range of 200°C to 210°C was reached. The preheating cavity and the loaded mould were both heated without pressure for 5 to 10 minutes to allow the PLA Matrix to begin melting and percolating through the HAp/Mg composite. A consolidation pressure of 1- 2 bar was applied at this time, as indicated in Fig 3.3, and remained steady for 5 minutes. Pressure was applied during the impregnation stage to compel the molten metal to flow.

Fig 2. Compression Moulding Machine

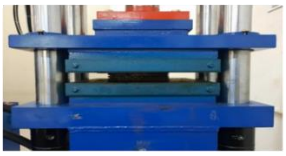

Fig 2.1 Compression Moulding Machine Applied pressure

Fig 2.3 Moulding Die

Tensile Testing is a destructive engineering and materials science test in which a sample is subjected to controlled tension until it fails completely. This is one of the most often used mechanical testing methods. Shown in Fig 3. and table 2 shown as specification of Universal Testing Machine

Table 2. Fully Automatic Micro Universal Testing Machine Of 100KN

Make / Model	FSA, M-100
Maximum Capacity	100 KN
Flat Holders	Wide: 35 mm Thickness: 0-20 mm
Round Holders	5 – 25 mm
Bend Test Attachment	Span variable: 40 mm – 250 mm

Fig 3. Universal Testing Machine

Mechanical Test

The bio-composite sample shown in Fig 4 was made using the ASTM D638 standard, and The tensile tests were carried out on a universal testing equipment. (Government College of Technology, Coimbatore, India.), with a 10 kN static load cell, 50 mm gauge length, and 5 mm/min strain rate. Tensile tests were used to measure the yield and tensile strength of the PLA/HAp/Mg bio-composites. Each sample in Fig 4.1 was put to the test. The reported values and representative samples were plotted. The width and thickness of the test specimens were measured using a Mitutoyo digital calliper prior to testing. The tests were carried out at room temperature with a 54% relative humidity.

Fig 4. Before Tensile test specimen

Fig 4.1. After Tensile test specimen

III. RESULTS AND DISCUSSION

Tensile test of composite specimens output data

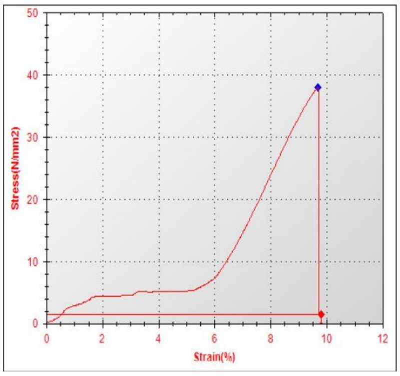

Figure 5. Tensile Test Graph Virgin PLA

Fig 5. virgin PLA shows the relationship between the strain x-axis and stress on the y-axis for the tensile test.
Table 3. Tensile test results Virgin PLA

INPUT DATA	VERTICAL	OUTPUT DATA	
Specimen shape	Flat	Load at yield	0.018 KN
Specimen type	BIO -COMPOSITE	Yield stress	1.496 N/mm^2
Specimen description	Virgin PLA	Load at peak	0.455 KN
Specimen width	04 mm	Tensile strength	37.958 N/mm^2
Specimen thickness	03mm	Load At Break	0.018 KN
Initial gauge length	28 mm	Elongation At Break	1.750 mm
Final specimen width	4 mm	Breaking Strength	1.496 N/mm2
Final specimen thickness	3 mm		
Final gauge length	28 mm		
Final area	12 mm^2		
Specimen CS area	12.000 mm^2		

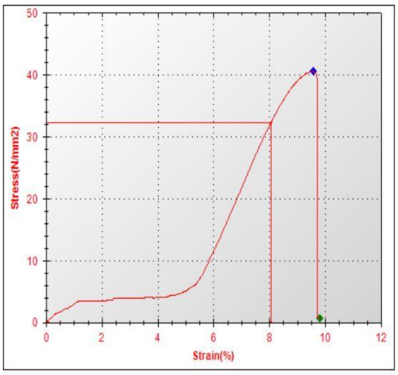

Figure 5.1. Tensile Test Graph PLA-Hap-Mg (84%,15%,1%)

Fig 5.1. PLA*HAp*Mg shows the relationship between the strain x-axis and stress in the y-axis for the tensile test.

Table 3.1. Tensile output data PLA-HAp-Mg (84%,15%,1%)

INPUT DATA	VERTICAL	OUTPUT DATA	
Specimen shape	Flat	Load at yield	0.388 KN
Specimen type	BIO-COMPOSITE	Yield stress	32.326 N/mm^2
Specimen description	PLA-HAp-Mg (84%,15%,1%)	Load at peak	0.486 kN
Specimen width	04 mm	Tensile strength	40.509 N/mm^2
Specimen thickness	03mm	Load At Break	0.007 KN
Initial gauge length	28 mm	Elongation At Break	2.01 mm
Final specimen width	4 mm	Breaking Strength	0.597 N/mm2
Final specimen thickness	3 mm		
Final gauge length	28 mm		
Final area	12 mm^2		
Specimen CS area	12.000 mm^2		

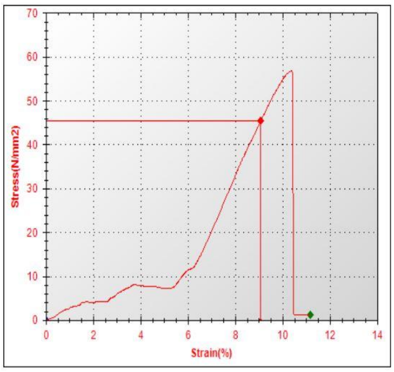

Figure 5.2. Tensile Test Graph PLA-HAp-Mg (82%,15%,3%)

Fig 5.2. PLA*HAp*Mg shows the relationship between the strain x-axis and stress in the y-axis for the tensile test.

Table 3.2. Tensile output data PLA-HAp-Mg (82%,15%,3%)

INPUT DATA	VERTICAL	OUTPUT DATA	
Specimen shape	Flat	Load at yield	0.544 KN
Specimen type	BIO-COMPOSITE	Yield stress	45.332 N/mm^2
Specimen description	PLA-HAp-Mg (82%,15%,3%)	Load at peak	0.681 kN
Specimen width	04 mm	Tensile strength	56.738 N/mm^2
Specimen thickness	03mm	Load At Break	0.014 KN
Initial gauge length	28 mm	Elongation At Break	2.45 mm
Final specimen width	4 mm	Breaking Strength	1.133 N/mm2
Final specimen thickness	3 mm		
Final gauge length	28 mm		
Final area	12 mm^2		
Specimen CS area	12.000 mm^2		

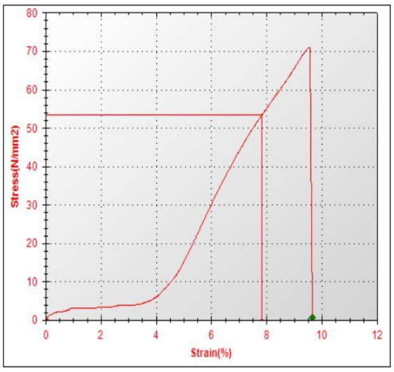

Figure 5.3. Tensile Test Graph PLA-HAp-Mg (80%,15%,5%)

Fig 5.3. PLA*HAp*Mg shows the relationship between the strain x-axis and stress in the y-axis for the tensile test.

Table 3.3. Tensile output data PLA-HAp-Mg (80%,15%,5%)

INPUT DATA	VERTICAL	OUTPUT DATA	
Specimen shape	Flat	Load at yield	0.64 KN
Specimen type	BIO-COMPOSITE	Yield stress	53.298 N/mm^2
Specimen description	PLA-HAp-Mg (80%,15%,5%)	Load at peak	0.851 kN
Specimen width	04 mm	Tensile strength	70.923 N/mm^2
Specimen thickness	03mm	Load At Break	0.007 KN

Initial gauge length	28 mm	Elongation At Break	2.705 mm
Final specimen width	4 mm	Breaking Strength	0.573 N/mm^2
Final specimen thickness	3 mm		
Final gauge length	28 mm		
Final area	12 mm^2		
Specimen CS area	12.000 mm^2		

Tensile test results provided mechanical properties for the materials

Table 4. Comparisons of composite specimen tensile test result

S.NO	Specimen	Composite	UTS (MPa)	Yield strength (MPa)	% of elongation
1	Virgin PLA	100%	37.958	25.496	1.750
2	PLA/HAp/Mg	(84%,15%,1%)	40.509	32.326	2.01
3	PLA/HAp/Mg	(82%,15%,3%)	56.738	45.332	2.45
4	PLA/HAp/Mg	(80%,15%,5%)	70.923	53.298	2.705

Table 4. PLA*HAp*Mg shows the Values of ultimate strength, Yield strength, and elongation at break obtained from mechanical tensile tests for Virgin PLA, PLA/HAp/Mg (84%,15%,1%), PLA/HAp/Mg (82%,15%,3%), and PLA/HAp/Mg (80%,15%,5%) respectively.

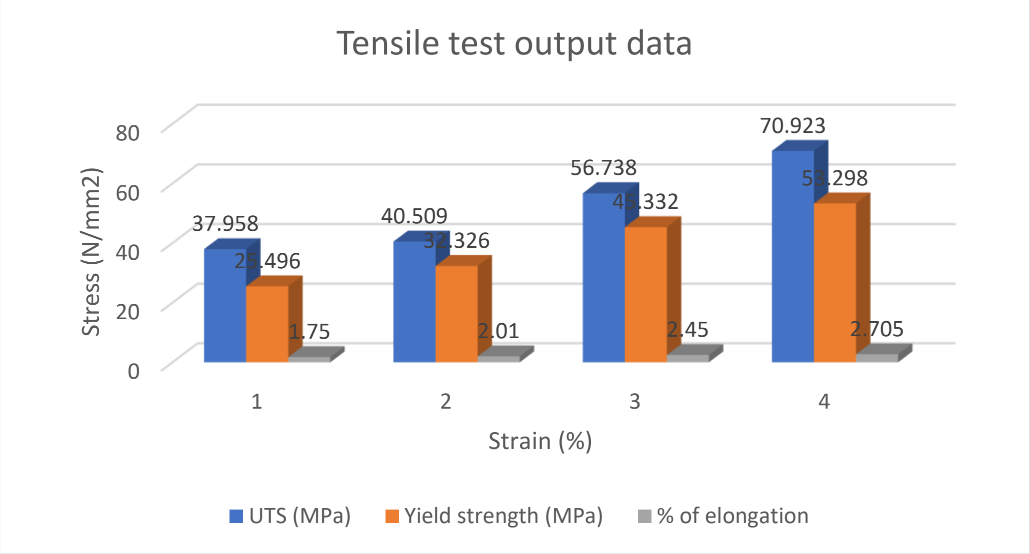

Fig 6. Tensile Test Output Data Comparison

The engineering stress-strain curves of the specimens are shown in fig 5, 5.1, 5.2 & 5.3, and the mechanical properties are listed in table 4. During the remodelling or disintegration process, an ideal biomaterial should give enough structural support to regenerated tissues. The mechanical properties of the samples were also investigated in this investigation Fig 6. The ultimate tensile strength and yield strength of virgin PLA, PLA/HAp/Mg (84%,15%,1), PLA/HAp/Mg (82%,15%,3) and PLA/HAp/Mg (80%,15%,5) respectively. As the concentration of hydroxyapatite (HAp)/Magnesium (Mg) in the polymer matrix was increased, the ultimate tensile strength and yield strength of the samples were increased. The ultimate tensile strength and yield strength of the PLA/HAp/Mg (84%,15%,1) composite specimen was 5.2% and 48% greater than virgin PLA, the ultimate tensile strength and yield strength of the PLA/HAp/Mg (82%,15%,3) composite specimen was 47% and 67% greater than virgin PLA, and the PLA/HAp/Mg (80%, 15%, 5) composite specimen had 86% higher ultimate tensile strength and 81% higher yield strength than virgin PLA. This is due to the fact that HAp/Mg has a higher ultimate tensile strength than PLA. Furthermore, the value of elongation at breaking was noticeably lower. Because polymers are flexible, they have a higher elongation at break value, whereas ceramics have a lower elongation at break value due to their brittle nature. The flexibility of the composite was diminished as a result of the presence of HAp/Mg in the PLA matrix. Because the composite has a lesser tensile strength than bone, implants made of it can be employed in unloaded areas of the body, such as the craniofacial area.

IV. CONCLUSION

The purpose of this research was to explore how the PLA polymer's matrix interacted with FHAp powder created from fish bones waste as a dispersion phase to develop Mg-based composites with a focus on mechanical qualities. These composites have a high strength-to-weight ratio and are biocompatible and biodegradable, making them ideal for usage as bone replacements and body implants. The Bio-composite was successfully prepared utilising a compression moulding machine with various weight percentages. The results of the ultimate tensile strength and yield strength of the PLA/HAp/Mg-(80%,15%,5%) composite was approximately 86% and 81% greater than virgin PLA, as a result, mechanical characteristics have improved. The improved properties of the composite could be attributable to improved interphase attractive contact and attachment of the HAp/Mg particles in the polymer matrix, which corrects the Stress shielding effect and reduced bone density problem produced by pure mg implantation.

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