IARJSET

ISSN (Online) 2393-8021
ISSN (Print) 2394-1588

International Advanced Research Journal in Science, Engineering and Technology

Factura '20 - National Conference On Emerging Trends In Manufacturing

NSS College of Engineering, Palakkad, Kerala, India

Vol. 7, Special Issue 1, August 2020

Fabrication and Testing of Nylon (PA)-6 Polymer Composite Reinforced with Graphene Nano Particles

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Abstract: Polymer Nano composites have been gaining much attention in producing high strength materials due to the low cost and process ability of polymers. In this research, polymer composites with nylon 6 (Poly amide 6) as matrix and graphene Nano particles as reinforcement are fabricated. There are three specimens of 0.5%, 1%, 1.5% by weight of Graphene Nanoparticles (GNPs) reinforced polymer composites are prepared through melt and mixing method. In the experiment, GNP nanoparticles are first mixed with nylon 6 pellets to make Nano composites. The mechanical properties of specimens were characterised through toughness test, tensile test, hardness test, flexural strength test and water absorption test. It is found that the specimen with 1.5% GNPs reinforcement shows a better value for all these tests. The results indicate that the existence of GNP Nano fillers provide a noticeable contribution to the enhancement of mechanical properties. This research gives a priority for modifying the existing 3D printing material to polymer composite material with better properties.

Keywords: Nylon 6, graphene Nano particles, 3D printing

I. INTRODUCTION

A composite material is a material that consists of one or more discontinuous components (particles/fibres/reinforcement) that are placed in a continuous medium (matrix). In a fibre composite the matrix binds together the fibres, transfers loads between the fibres and protects them from the environment and external damage.

Composite materials, or shortened to composites, are microscopic or macroscopic combinations of two or more distinct engineered materials (those with different physical and/or chemical properties) with a recognizable interface between them in the finished product. For structural applications, the definition can be restricted to include those materials that consist of a reinforcing phase such as fibers or particles supported by a binder or matrix phase. Wood composites are commonly seen examples of composite materials. Other features of composites include the following: (1) the distribution of materials in the composite is controlled by mechanical means. (2) The term composite is usually reserved for materials in which distinct phases are separated on a scale larger than atomic, and in which the composite's mechanical properties are significantly altered from those of the constituent components. (3) The composite can be regarded as a combination of two or more materials that are used in combination to rectify a weakness in one material by a strength in another. (4) A recently developed concept of composites is that the composite should not only be a combination of two materials, but the combination should have its own distinctive properties. In terms of strength, heat resistance, or some other desired characteristic, the composite must be better than either component alone. Composites were developed because no single, homogeneous structural material could be found that had all of the desired characteristics for a given application. Fiber-reinforced composites were first developed to replace aluminium alloys, which provide high strength and fairly high stiffness at low weight but are subject to corrosion and fatigue. An example of a composite material is a glass-reinforced plastic fishing rod in which glass fibers are placed in an epoxy matrix. Fine individual glass fibers are characterized by their high tensile stiffness's and very high tensile strengths, but because of their small diameters, have very small bending stiffness's. If the rod were made only of epoxy plastic, it would have good bending stiffness, but poor tensile properties. When the fibers are placed in the epoxy plastic, however, the resultant structure has high tensile stiffness, high tensile strength, and high bending stiffness. The discontinuous filler phase in a composite is usually stiffer or stronger than the binder phase. There must be a substantial volume fraction of the reinforcing phase (about10%) present to provide reinforcement. Examples do exist, however, of composites where the discontinuous phase is more compliant and ductile than the matrix. Natural composites include wood and bone. Wood is a composite of cellulose and lignin. Cellulose fibers are strong in tension and are flexible. Lignin cements these fibers together to make them stiff. Bone is a composite of Strong but soft collagen (a protein) and hard but brittle apatite (a mineral). The different types of reinforcement being used, together with the alternative processing methods, are discussed. Depending on the initial processing method, different factors have to be taken into consideration to produce a high quality billet. With powder metallurgy processing, the composition of the matrix and the type of reinforcement are independent of one another. However, in molten metal processing they are intimately linked in terms of the different reactivities which occur between reinforcement and matrix in the molten state. The factors controlling the distribution of reinforcement are also dependent on the initial processing method. Secondary fabrication methods, such as extrusion and rolling, are essential in processing composites produced by powder metallurgy, since they are required to consolidate the composite fully. Other methods, such as spray casting, molten metal infiltration, and molten metal mixing give an essentially fully consolidated product directly, but extrusion, etc., can improve the properties by modifying the reinforcement distribution. The mechanical properties obtained in metal matrix composites are dependent on a wide range of factors, and the present understanding, and areas requiring further study, are discussed. The successful commercial production of metal matrix composites will finally depend on their cost effectiveness for different applications. This requires optimum methods of processing, machining, and recycling, and the routes being developed to achieve this are considered. Additive manufacturing (AM) (or three-dimensional (3D) printing) technologies have been widely adopted in making intriguing parts for aerospace industry, medical industry, food industry, and automotive industry. Thanks to the nature of layer wise building in AM processes, parts with complex geometry can be built with ease. Other advantages of AM technologies include reduced lead time, less material waste, shortened supply chain, and simplified manufacturing process planning. In spite of the distinct advantages, parts obtained by AM technologies are generally weaker compared with the traditional moulding processes (for polymers) or the traditional forming processes (for metal alloys). Typically, it can be attributed to the low density (or high porosity) or low degree of cross-linking in the AM materials. Since the birth of modern AM technologies, polymers have been the popular materials for part making in many AM processes such as stereo lithography and Fused Deposition Modelling (FDM). To create polymer-based products with higher mechanical properties, adding reinforcements to the polymer matrix in AM processes has become a viable option. Different reinforcement materials can be added to the polymer matrix such as nanoparticles, short fibers, and long fibers. This is a research area that has caught enormous interests in recent years.

II. LITERATURE SURVEY

The AM process of polymer materials reinforced by particles is relatively straightforward and generally does not impose an additional requirement for the existing AM machines. Many works related to 3D printing of particle-reinforced polymer matrix composites have been reported.

Arnaldo D. Valino et Al [4] present, analyze and summarize trends related to advances in thermoplastic composites used in AM methods. The different types of 3D printing methods that utilize commodity polymers and thermoplastic composites are first discussed. Next, an analysis and summary of the preparation, processing, and properties of improved thermoplastic composites based on the filler types such as particle-based, fiber-based, nanomaterial-based, and polymer blends with high-performance polymers is presented. The emphasis is on outlining progress in the development of materials used in AM through recent literature. The limitations of current technologies and a perspective on materials preparation, performance requirements and applications are also discussed.Vamsi Krishna Balla et Al [13] reviewed the additive manufacturing (AM) of polymer composites demonstrated it potential in manufacturing net-shape, complex and functional parts for direct use. Further, the unique benefit of using AM to manufacture NFRCs is its ability to manufacture functionally graded composites with site-specific performance and functionality. It is also possible to design and produce structures with tailored fiber alignment by changing the deposition paths in each layer. However, AM of these composites poses significant challenges in terms of composite filament preparation for FFF, inherent agglomeration potential of nature fibers, large amount of moisture and void formation, difficulties in 3DP of NFRCs due to nozzle clogging, fiber degradation/ breakage, non-uniform curing, etc. Poor layer-to-layer and fiber-matrix interfacial bonding are two serious issues in AM of NFRCs as the pressures associated with AM are significantly lower than those in conventional processing routes such as compression or injection molding. Therefore, more focus is required to address these issues by modifying the AM process parameters, hardware and feed stock quality. It is known that increasing the fiber concentration in NFRCs is required to achieve maximized mechanical performance. However, excessive fiber content found to clog deposition heads in addition to increased brittleness of feedstock. Therefore, fundamental understanding on the influence of natural fibers, their characteristics and concentration on the rheological properties of polymer matrices, fiber-matrix interactions is essential, which is not yet clear. Such an understanding will enable addressing fiber damage during multi-step feed stock preparation followed by AM of NFRCs. Finally, the success of NFRCs processing is inherently limited by natural fibers’ characteristics and their chemical constituents as they found to have contradicting effects on mechanical, thermal, biological and moisture absorption properties. However, decreasing lignin concentration in these fibers can improve majority of properties and therefore, appropriate fiber treatment become very crucial in NFRCs processing. Overall design and processing of NFRCs using AM is very challenging but equally rewarding.SH Masood et Al [12] reviewed about The Stratasys FDM technology has established a prominent position in AM since its inception about two decades ago. The technology has attained the market leadership in terms of the largest number of units installed in plastics-based AM. The main strength of FDM technology is the wide range of high strength engineering plastics it can use to create parts not only for design verification, but also for functional testing and end-use applications in tooling and casting. The simplicity and reliability of the technology has also prompted development of several others non filament-type extrusion-based FDM systems, and it has also lead to the proliferation of several low-cost FDM-type 3D printers for domestic and hobbyist consumers. The FDM process has been extensively used by research and development community at universities and research organizations to further enhance the process and part quality, and to develop new materials for a variety of new engineering applications. The technology offers a distinct advantage in creating biomedical implants and TE scaffolds of desired characteristics and this has also led to the development and testing of several biomaterials for the FDM technology. Giovanni Postiglione et Al [11] reported a new three-dimensional (3D) printing system based on Liquid Deposition Modelling (LDM) is developed for the fabrication of conductive 3D Nano composite-based microstructures with arbitrary shapes. This technology consists in the additive multilayer deposition of polymeric Nano composite liquid dispersions based on Poly Lactic Acid (PLA) and Multi-Walled Carbon Nanotubes (MWCNTs) by means of a home-modified low-cost commercial bench top 3D printer.

Electrical and rheological measurements on the Nano composite at increasing MWCNT and PLA concentrations are used to find the optimal processing conditions and the printability windows for these systems. In addition, examples of conductive 3D microstructures directly formed upon 3D printing of such PLA/MWCNT-based Nano composite dispersions are presented. The results of our study open the way to the direct deposition of intrinsically conductive polymer-based 3D microstructures by means of a low-cost LDM 3D printing technique. RajkumarVelu et Al [2] reported the recent development in manufacturing demonstrates that this era belongs to 3D printing technology. Currently, synthetic polymers and fibers are almost the only focus, with highly associated environmental impacts. The need for composites of more sustainable outlook is emerging. Still, there are many barriers to overcome to incorporate natural printable materials into 3D printing. Automotive and aerospace components come in complex designs and/or organic forms, and thus their prototyping is becoming costly and time consuming. As such, 3D printing techniques involve metal parts gradually replaced with polymer composites to reduce the weight in automobile and aerospace applications. Also, the engine parts made up of polymer-based composites have shown to increase the fuel efficiency in recent studies. 3D printing is also a noteworthy technique to fabricate functionally graded polymer composites based on particle reinforcement, fiber reinforcement, and nanostructures. The composite materials can be customized by delivering various volume fractions. As a result, the mechanical properties can be optimized to the specific requirements of the application. Thermal stability is another significant advantage that comes handy with 3D printing composites, if polymer composites have a wide range of material choice that can withstand elevated temperatures when developed for automobile or aerospace applications. Researchers in the 3D printing area must put efforts and focus on natural materials and processes that are compatible with a greener environment. There are little research initiatives on green composite materials for 3D printing technologies at this moment. Green polymer composites with natural fillers represent a magnificent view for future research, aiming to reduce the use of petroleum-based non-renewable sources. Zhihui Liu et Al [10] reported the extensive efforts on 3D printing of polymer composites reinforced by nanomaterial and short fibers, and limited progress on 3D printing of polymer composites reinforced by continuous fibers, the research on hybrid strengthening effect resulted from both continuous long fibers and nanoparticles is uncharted. Moreover, the fabrication of polymer matrix composite with multiple reinforcement phases carries significant scientific and practical value because the increasingly attractive additive manufacturing techniques could enable versatile materials selection and design. To fill this gap, in this study, we aim to develop polymer matrix composites with both continuous long fibers and nanoparticle reinforcements such as carbon nanotubes and graphene Nano platelets (GNPs) by using FDM-based AM technology. In particular, we intend to investigate if adding nanoparticle reinforcements can further yield meaningful improvement in material strength. The tensile performance of the 3D-printed composites is evaluated so that the effects of long fibers and nanoparticles can be quantified separately, as well as in a combined manner. Novoselov KS et al studied importance of graphineNano fillers used as reinforcement in composite. Its described Graphene, a 2D nanomaterial, possesses exceptional physical properties due to its pure sp^2 hybridization network. And have excellent mechanical properties, exceptional electrical and thermal conductivity. This makes it a promising material for various applications like the fields of aerospace, manufacture of flexible super RC capacitor electrodes, and in artificial muscle and tissue engineering. Its described Graphine is obtained by the top-down method of mechanical exfoliation and the bottom-up method of chemical vapour deposition However, due to its limited ability to scale-up and time-consuming nature, focus has been shifted to its precursor, graphene oxide (GO). GO is usually obtained by Hummers' method. It's been indicated the planar feature of the GO sheets makes it easy to assemble into paper-like structures through self-assembly and filtration techniques. Yang H et al [3] discussed problems that still affect the incorporation of nanoparticles content into polymer matrices which is tendency for them to agglomerate. In order to homogenize the system more and to achieve a highly well-dispersed Nano composite, a possible route is modification of the components before development of Nano composite. Surface modification has attracted strong attention owing to its ability to produce remarkable integration and an improved interface between the inorganic nanoparticles and the polymer matrix. Ultra sonication to improve dispersion seems to be the main step to remove agglomeration. Wanget al [1] presentsrandom dispersion of inorganic platelets in the polymer matrix where the relationship between the Nano filler loading, and corresponding structure and performance of the Nano composite was studied. It was observed that between the ranges of loading 0-30 wt% of clay nanofiller, the microstructure of the Nano composite was observed to be random in nature. As the clay Nano filler loading increased from 30 wt% to 70 wt%, an alternating layered structure of clay Nano filler and polymer phase was observed. Indicating controlling the volume fraction of inorganic Nano platelets allows for tunability of the microstructure, and consequently its physical properties.Podsiadlo P et al [6] reports polymer composite are mainly fabricated using layer by layer (LBL) assembly. Vacuum-assisted filtration (VAF), doctor-blading (DB), freeze casting. The main attration layer by layer is based on sequential adsorption of oppositely charged compounds, is one of the most popular and well-established methods for preparation of multilayered thin films. Due to its ability to form highly tuned, functional films with nanometre-level control as well as due to its simplicity, requiring a minimum of setup and inexpensive equipment it's been widely used. It is shown showed Almost any type of charged species including inorganic molecular clusters, nanoparticles, nanotubes and nanowires, Nano plates, organicdyes, dendrimer, porphyrin, polysaccharides, polypeptides, nucleic acids and DNA, proteins, and viruse can be success fully used as the components to prepare thin films.Van Lier et al [7] simulated the mechanical behaviour of the graphene and concluded that elastic modulus of graphene could be around 1.11 TPa. Reddy et al used a continuum mechanics method and modelled the deformation of graphene sheets in terms of Brenner's potential. The elastic modulus of the graphene was estimated to be 0.669 TPa in their results. In recent years researchers have developed experimental methods to reveal the true elastic modulus. These experiments are primarily based on the utilization of Atomic Force Microscopy (AFM).

III. METHODOLOGY

The experiment is conducted in different stages. First stage involves preparing the composite material by reinforcing graphene Nano particles with nylon 6 polymer matrix by melt mixing method.3 specimens of 0.5%, 1%, 1.5% GNPs reinforced specimens are fabricated and tested for toughness, ensile, hardness, flexural and water absorption test.

A. Experimental setup

Melt mixing technique utilizes elevated temperature to turn the polymer into molten phase and shear to intercalate fillers in polymer matrix. The benefit of using this technique is the elimination of the use of toxic solvents. This technique is believed to be more cost effective than solution blending and is more compatible with industrial processes. Furthermore, it is said to be more environmentally friendly due to elimination of solvent use, faster and easier. However, such methods may not be able to achieve same level of filler dispersion as solvent mixing or in situ polymerization methods at high filler loadings.

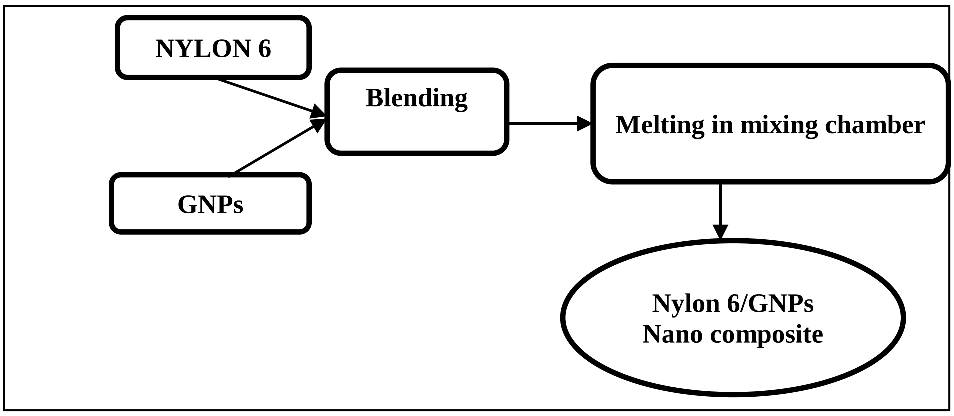

Fig.1. Experimental setup flow chart

TABLE I Properties of Nylon 6

DESCRIPTION	NYLON 6 (PA 6)
density	1.13×10^3 kg/m^3
Water absorption	1.6%
Specific heat capacity	0.29-1.67 J/g ^{0}C
Tensile strength	55.56 MPa
Hardness, shore D	80
Flexural strength	110 MPa
Impact strength	13.69J
Melting temperature	220 ^{0}C

TABLE II Properties of GNPs

DESCRIPTION	GRAPHENE NANO PARTICLES
Purity	~99%
Outer Diameter	10-30 nm
Inner Diameter	5-10 nm
Length	>10 µm
Surface Area	110 – 350 m2/g
GNT content	~95-99%
Bulk Density	0.14 g/cm3
Chemical Formula	C
Physical Form	Fluffy,VeryLight Powder
Odour	Odourless
Colour	Black Powder

B. Fabrication of polymer composite

Initially mold preparation is done, consists of cleaning the mold and applying a release agent in the surface of it to avoid sticking of specimen part. Thin metal sheets are used at the top and bottom of the mold plate to get good surface finish of the product.To prepare GNP-reinforced nylon, the nylon polymer is first mixed and melted with the Nano reinforcements. For this purpose, the PA 6 pellets are heated in the oven for 16 h at 100 ^{0}C to remove moisture. Different weight percentages of graphene are mixed with the dried nylon pellets using a beak and a stirrer.Then the mixture in liquid form is poured onto the surface of metal sheet already placed in the mold. The mixture is uniformly spread with the help of roller brush. After placing the metal sheet, release gel is sprayed on the inner surface of the top mold plate which is then kept on the stacked layers and the pressure is applied. The curing of composite was done for 30 Hr. in room temperature. The process was repeated for 1%, 1.5 % weight percentage GNPs.

IV. RESULTS AND DISCUSSION

A. Impact test result

Impact tests are designed to measure the resistance to failure of a material to a suddenly applied force such as collision, falling object or instantaneous blow. The test measures the impact energy, or the energy absorbed prior to fracture. From the test, it is found that the impact strength of polymer composite reinforced with 0.5% GNPs is 16.5 J. While the polymer composite reinforced with 1% GNPs is 20 J. But the polymer composite with 1.5% GNPs reinforcement is 25 J. This indicates that the impact strength is more in the third specimen. It is also noticeable that the impact strength gets increased as the percentage of GNPs reinforcement gets increased. The figure below represents the impact strength of three specimen under three different percentage of reinforcement.

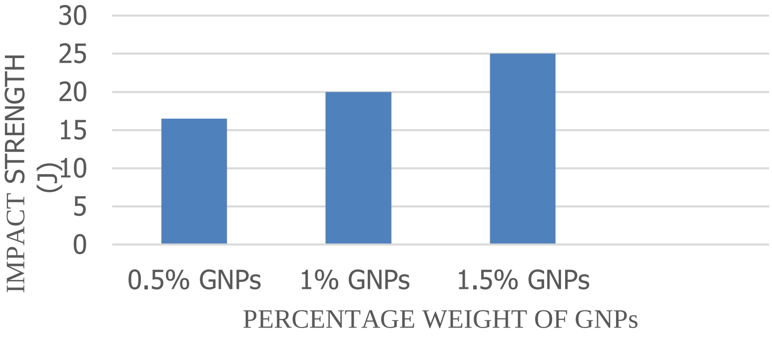

Fig.2. Impact strength of 3 specimens

B. Tensile test result

Generally, a tensile test is designed to be run until the sample fails or breaks under the load. The test result shows that 0.5 % GNPs reinforced polymer composite shows a tensile strength of 62.13 MPa, while 1% GNPs reinforced polymer composite shows a tensile strength of 68.8 MPa.it is found that the polymer composite reinforced with 1.5% GNPs gives a better tensile strength of 71.78 MPa. The figure below shows the tensile strength of three specimen under different percentage of reinforcement.

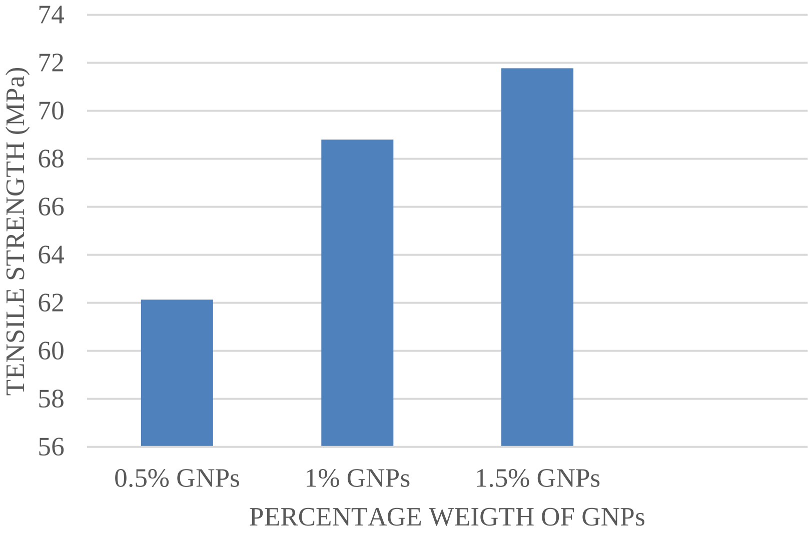

Fig.3. Tensile strength of 3 specimens

The addition of graphene filler in to composite directly influence the overall tensile strength of the composite. The common GFRP without any Nano fillers have the least amount of tensile strength of the three specimen produced. With direct increase of Nano filler content produce a rise in tensile strength. The highest Nano filler content seems to produce the highest tensile strength. This definite increase of strength can be attributed to the high strength of graphene and increase in interfacial area due to high specific surface area of graphene, resulting in better stress distribution for effective load transfer through the interface.

C. Hardness test result

The principal purpose of the hardness test is to determine the suitability of a material for a given application, or the particular treatment to which the material has been subjected. The ease with which the hardness test can be made has made it the most common method of inspection for metals, alloys and composites.

The hardness test using Shore durometer for the given specimens results that hardness value for the polymer composite with 0.5% GNPs reinforcement is 86.While the hardness value for the polymer composite with 1% GNPs reinforcement is 90.But the polymer composite reinforced with 1.5% GNPs shows a hardness value of 92,which is higher than the other two values. This shows that the hardness value increases with increase in percentage of reinforcement of GNPs. The figure below shows the hardness value for the three different percentage composition specimens.

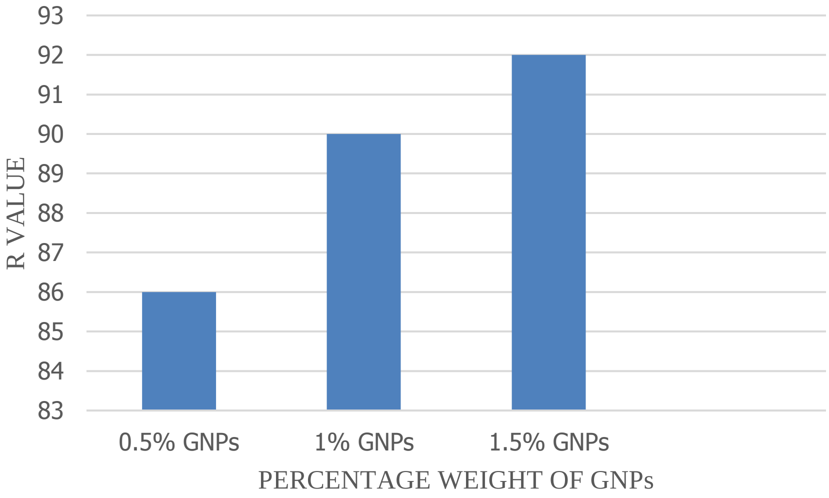

Fig.4. Hardness value of 3 specimens

D. Flexural strength test result

Flexural strength is defined as the maximum stress at the outermost fiber on either the compression or tension side of the specimen. The flexural strength value of the polymer composite with 0.5% GNPs reinforcement is 170.5 MPa. On other hand the flexural strength value for 1%GNPs reinforced polymer composite is 185.9 MPa. But the polymer composite with 1.5% reinforcement of GNPs gives a value of 207.9 MPa as flexural strength value. This indicates that the reinforcement of GNPs results a higher value of flexural strength. The figure below indicates the flexural strength value for three different specimen composition.

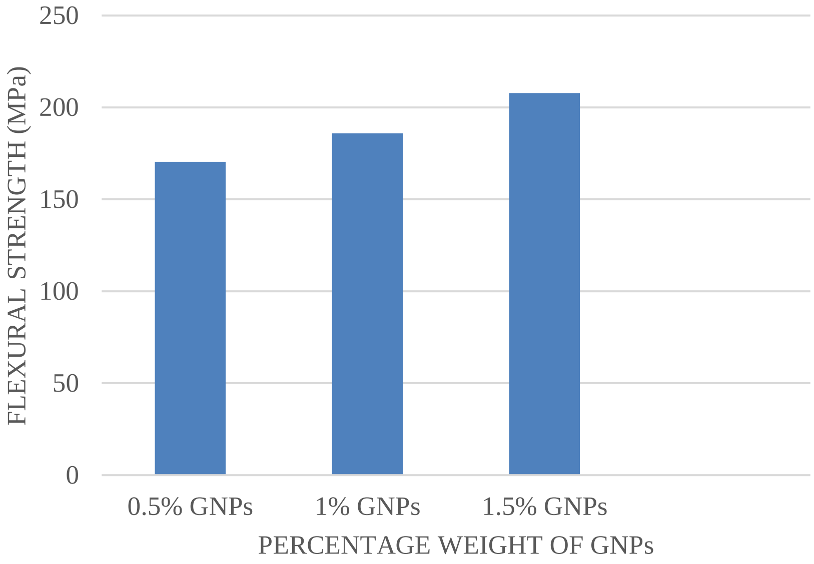

Fig.5. Flexural strength of 3 specimens

E. Water absorption test result

The water absorption test was performed under room temperature until the weight of the samples achieved constant values. The weight gained by the Nano composites is determined by Equation

$$\frac{W_2 - W_1}{W_1} \times 100$$

Where W_1 and W_2 being the weight of the dry and wet specimen

TABLE III Percentage increment in weight of Nylon-6/GNPs Nano composites

Day	**Increment in weight of 3 nylon 6 Nano composites (gram_)**		
	0.5% GNPs	1% GNPs	1.5% GNPs
1	0.2373	0.2665	0.2591
2	0.2407	0.2732	0.2604
3	0.2539	0.2909	0.2805
4	0.2617	0.2929	0.2825
5	0.2620	0.2931	0.2828
6	0.2621	0.2931	0.2829
FRACTIONAL INCREMENT OF WEIGHT	0.1045	0.1001	0.0917

According to Table III, there was no further increment in the weight of nylon-6 Nano composites from day 6 onwards. This indicated that the Nano composites had absorbed the maximum amount of water within a week. The amount of water absorbed was calculated by deriving the percentage increase in the weight of the Nano composites. Comparing the water absorption capability of all samples, increasing percentage of GNPs has contributed to better water resistance of nylon-6. The filler layers in the polymer increase the diffusion path length of water through the polymer. This attributes to the better barrier properties of Nano composites. This finding proves that the GNPs filler had dispersed well in the nylon-6 and there was no agglomeration.

V. CONCLUSION

Nano materials have attracted much attention due to the excellent properties that they possess, as well as their promising applications. The combination of 3D printing and composite materials has redefined the mechanical properties of 3D-printed products. In this research, nylon (PA) 6 Nano composites filled with GNPs with different composition of 0.5 %, 1%, 1.5% into specimens for mechanical tests. Three weight percentages Nano reinforcement are used to produce the Nano composites, in order to understand the effect of Nano filler content. The major findings are summarized as follows:

- The manufacturing of polymer matrix composites with the reinforcement of GNPs is much easier and economically better.
- Grapheme Nanoparticle reinforcement improves the mechanical properties of the specimen.
- Impact tests, tensile test, hardness test, flexural test, water absorption test reveal that the GNPs filled PA 6 Nano composites show more significant improvements in mechanical properties as compared to nylon 6 only. The specimen with 1.5% GNPs reinforcements shows a higher values.
- As noted, filler addition can result in the unpredictable formation of defects. To control and reduce defect formation, it is important to understand the nature of compatibilization, miscibility, and surface migration to improve the properties.

In summary, the extension of AM to filled polymers holds the promise of novel applications and improved properties of AM fabricated parts. However, the required optimization of process parameters depending on materials system and AM techniques presents a challenge for the AM of filled polymers and defines the need for future research. It is hoped that this research will help advance the general applicability of thermoplastic composites and Nano composites in AM.

VI. FUTURE SCOPE

This study only presents the preliminary effort of an exciting research area for additive manufacturing. It can be extended in a variety of directions. From the materials perspective, future research can address the choices of other fiber materials, such as carbon fiber, and polymer matrix materials, such as PLA. From the mechanical property perspective, future research can include other static properties such as bending, as well as dynamic mechanical properties such as fatigue. From the process perspective, new methods can be tested for alleviating the aggregation of Nano fillers so that their effectiveness can be improved. In addition, further research can be directed at the reinforcing mechanisms behind the mechanical behaviours of the 3Dprinted materials.

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