

Synthesizing Biodegradable Smart Composite Material Through Extrusion Technique and Characterizing for 3D Printing (FFF)

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Abstract - Biodegradable and bio-compactable characteristics of Polylactic Acid (PLA) have attracted the attention for numerous biomedical applications. Numerous tissue engineering problems, restoration of weakened or damaged tissues have been reported by using PLA and its copolymers due to their biocompatibility and distinctive mechanical and electronic properties. Similarly, biomedical application of Graphene and Carbon fiber can be exposed with significant potential. Therefore, use of graphene and carbon fiber blend to PLA can positively impact, accelerate and can provide very high influence in tissue engineering, regenerative medicine and other biomedical sectors. In this research study, a screw extruder is used to synthesis PLA-G-CF polymer composite filaments of various nano and micro particles with different particle morphology as reinforcement to PLA matrices. Polylactic Acid (PLA) has been used as matrix material and Graphene and Carbon fiber particles used as reinforcement. These polymer composite filaments are then used for Additive Manufacturing to fabricate medical components. The aim of this work was to investigate a potential method of preparing and processing biodegradable Polylactic Acid (PLA), Graphene and Carbon fiber (CF) through 3D Printing (FDM) Technology. In this research, the capability to use materials with conductive properties are also investigated which may lead to a new generation of 3D smart material. Synthesized composite filaments were characterized through Scanning Electron Microscope (SEM), Differential Scanning Calorimetry (DSC), Tensile Test and Fatigue Test. Among the various blend proportions considered in this work, 80/20% PLA-G-CF blend exhibited the highest elongation and impact strength. Differential Scanning Calorimetry (DSC) showed the influence of Graphene and Carbon fiber content on glass transition temperature (T_g), melting temperature and degree of crystalline of PLA-G-CF blends. Based on their characterization properties, PLA-G-CF Composite material can focus on various biomedical applications such as scaffolds, implants, drug delivery, cancer therapy, biological imaging etc.

Key Words: Additive manufacturing, Biodegradable polymer, PLA, Graphene (G), Carbon fiber (CF), composite, filament, FDM

1.INTRODUCTION (Size 11, Times New roman)

This Today, the environmental pollution has become a great concern and evil due to the high impact of plastic wastes in daily use. One of the possible solutions to this problem is to replace the commodity synthetic polymers with a biodegradable and ecco-polymers which are readily susceptible to microbial action. Among them, bio-based and biodegradable

polymers, Polylactic Acid (PLA) has been attracting much attention due to its mechanical properties resembling that of present day commodity plastics such as PE, PP and PS. Pure PLA is usually colorless and glossy thermoplastic polymer with similar properties as that of polystyrene. It can be processed using injection-molding, compression-molding, extrusion, thermoforming etc [1]. Polylactic acid (PLA) polymers are synthesized and obtained from renewable agricultural resources by polymerization of lactide, which is the cyclic di-ester of lactic acid [2]. PLA has high modulus, reasonable strength, excellent flavor and aroma barrier capability, good heat seal ability and can be readily fabricated,

The tensile strength gained from PLA structures ranges from 15.5 MPa to 89.1 MP [42]. There by making it one of the most promising biopolymers for varied applications.

PLA polymers have already been demonstrated as surgical implant materials, drug delivery systems [3][4], guided tissue and bone regeneration platforms, and also as porous scaffolds for the growth of neo-tissue [5],[6]. Use of PLA in biomedical applications is not based only on its biodegradability but also on its thermo-mechanical properties [7][4][8]. Hence, various medical devices have been prepared from different PLA types including degradable sutures, drug releasing micro-scale particles, nano-particles, and porous scaffolds for cellular applications [9]. Despite these desirable features, several drawbacks tend to limit its widespread applicability such as high cost, lower mechanical properties, and narrow processing windows [10]. Thus in order to broaden the applications of PLA, material properties and process ability has to be improved by blending with some higher grade biocompatible material. Even though, PLA polymers have a numerous limitations, significantly, thermal and mechanical properties, Its tensile strength and elastic modulus are comparable, but poor toughness limits its use in applications at higher stress levels. For instance, when Polylactic acid interference screws were used for graft fixation during anteriorcruciate ligament (ACL) reconstruction, the devices were reported to be mechanically weaker than their metallic counterparts and often fractured during implantation [10]. Its biodegradation rate is considered to be quite slow, which depends on its crystallinity and molecular weight of the material [11].

Graphene, on the other hand, has been at the forefront in bio-technological and medical applications [12][13][14][15]. In particular, the role of its surface chemistry, size, and ability to adsorb active bio-molecules has a huge impact on their biomedical applications [14]. Graphene is a free-standing 2D crystalline structure with one-atom thickness. It is an allotrope of carbon comprising layers of six-atom rings in a honeycombed network and can be theoretically viewed as a true planar aromatic macromolecule [16][17]. Graphene exhibits remarkable properties [18], such as unusual electronic flexibility as well as high planar surface area (~2630 m²/g),

outstanding mechanical strength (Young's modulus, ~1100 GPa) and unparalleled thermal conductivity (~5000 W/m/K). As such, it is a highly popular nano-scale additive for reinforced biopolymers [19]. Bio-safety of graphene and graphene derivatives is attracting more attention as it enters into a biological system [18][19][20]. It has been acknowledged that the biological effect of graphene is largely affected and complex by the exposure dose, methods, chemical function, lateral size, thickness, and surface properties [21][22]. Both graphene and graphene derivatives can be adapted to function as smart biomedical devices as long as their surfaces are properly functionalized [23]. Moreover, Dulet al. [24] successfully prepared the ABS/GNP composite for the FFF process using the melt compounding method. The presence of GNP led to the reduction in the co-efficient of thermal dilation and improved the stability under long lasting loads for the 3D printed parts.

Polymer blends as composites are the one of the convenient approaches to tailor the material properties at low processing cost. Polymer blending is a method for obtaining properties that the individual do not possess and has been widely used for various kind polymers. Blending of PLA with other polymers offers the possibility of improving the degradation rate, permeability characteristics, thermal and mechanical properties. The objective of this research is to produce different blends of PLA, Graphene and Carbon fiber and to investigate the mechanical and chemical characterization. The research also reports natural jute fibers with PLA matrix that results in high ductility instead of tensile strength, as compared to carbon fibers' reinforced parts. Therefore, the brittle carbon fibers and graphene provide high strength and natural fibers exhibiting low brittleness attain higher ductility. The non-treated carbon fibers and graphene with PLA matrix depicted the highest flexural strength of 335 MPa [26]. In Rangisetty and Peel's study, they investigated the mechanical properties of the composite materials produced by FDM process. As filament materials, they used PLA, PLA/CF, ABS, ABS/CF, PETG and PETG/CF filaments from commercial products. As the result of study, rigidity of all the fiber reinforced composites were increased, but surprisingly the tensile strengths were decreased probably due to very short fiber lengths [27].

Additive manufacturing (AM) or 3D printing is the next generation manufacturing technology that allows manufacturing of complex parts without requiring specialized tooling [28]. Additive manufacturing (AM) is a bottom to up process, creating objects directly from material deposition layer by layer in a controlled manner. Because of this unique approach, AM allows to manufacture complex shaped parts with minimum material wastage and energy consumption [29]. FDM/FFF is one of the most prominent and prevalent AM Technology due to its devices simplicity and wide availability of materials. In Fused Filament Fabrication, melt thermoplastic polymers are extruded to make the layers for fabricating the design provided in one of the following formats: STL (stereo lithography), AMF (additive manufacturing file), Step (standard for exchange of product model data), Voxel, 3MF (3D manufacturing format), or JT (Jupiter tessellation) [30-33]. Today, ABS, Polycarbonate (PC), Polylactic Acid (PLA), Polyamide (PA), Polystyrene (HIPS), Poly-Caprolactone (PCL), polyvinyl Alcohol (PVA), and Polyurethane (TPU) and the mixtures of different types of thermoplastics are the most common feedstock material in other words filaments in FDM technique[34-41]. However, the parts to be manufactured using these filaments lack biodegradability and

biocompatibility, due to inherent nature of the polymeric materials, to anti oxidation property, etc. This short coming restricts implementation of the FDM technology significantly and limits its applicability to industrial, medical and domestic applications. This section highlights the tensile capability of various potential FFF. Therefore, there is an urgent need for development of novel materials with enhanced properties. Biodegradable Polymer nano-composites stand out as a potential solution to overcome this limitation.

2. Material and Methods

2.1. Preparation of PLA Composite Filament

PLA is mixed mechanically with reinforcement powder and then is melt compounded using the screw. This method is widely used for preparing thermoplastic nano-composite. For the filament extrusion process, the PLA composite with 1.5 to 2 wt.% of reinforcement content was mixed to 98 to 98.5 wt.% of pure PLA granules. This mixture was then melted and extruded using the screw extruder. The extrusion temperatures ranged from 180°C to 220°C from the hopper to the die. Thus, the 1.75mm diameter filament was produced and collected for the 3D printing test. Figure 1 demonstrates the equipment setup for preparing the PLA Composite filament by the melt extrusion process.

2.2. Fabrication of PLA-TPU/ABS/G/CF Composite Filaments

The process starts feeding the hopper with the PLA and TPU/ABS/Graphene/Carbon fiber granules or pellets in a proportionate condition. The screw extrusion pushes and breaks the granules due to the thermal condition provided by the induction coils. The extrusion process continues until the material comes out of the nozzle in the form of wire. The screw extrusion is utilized for the filament composite production.

The main part of the extruder is a barrel containing a screw (also sometimes referred to as an "auger", which is connected to a heater towards its far end. On the other end, the screw is connected to an electric motor which will, via mechanical action, transport the so-called resin pellets through the barrel towards the heater. Pellets are gravity-fed continuously from a hopper (a kind of feeding funnel). As the motor is continuously driving the auger, the resin pellets are pushed into the heater. The thermoplastic granules will soften and melt because of the heat generated and are then pushed mechanically through a die. Pushing the soft thermoplastics through the die will cause it to form a continuous filament strand with the diameter of the diameter (1.75mm). This process is called extrusion, hence the term "extruder".

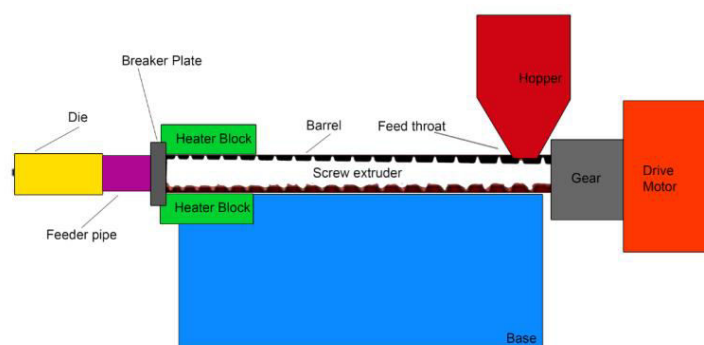

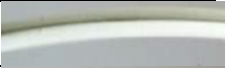




Fig -1: Screw Extrusion Equipment

Table -1: Weight ratio of the particles added into the PLA matrix and produced filament images.

S.No	PLA (wt%)	Additive (wt%)	Additives	Extruded Filament
1.	98.5	1.5	PLA + TPU	
2.	98.5	1.5	PLA + ABS	
3.	98	2 (1% G and 1% CF)	PLA + Graphene + Carbon fiber	
4.	100	0	PLA	

3. RESULTS AND DISCUSSION

3.1. Characterization of PLA Composite Filaments

The composite filaments were characterized via different methods, X-Ray Diffraction (XRD) Analysis, DSC, TGA, tensile test, surface roughness, and SEM-EDS.

3.1.1. X-Ray Diffraction (XRD) Analysis.

The X-ray diffraction (XRD) of PLA-G-CF materials was performed using an X-ray diffractometer. All samples were scanned over the range 5–40° (2θ) with a step size of 0.04, and time per step of 1 s.

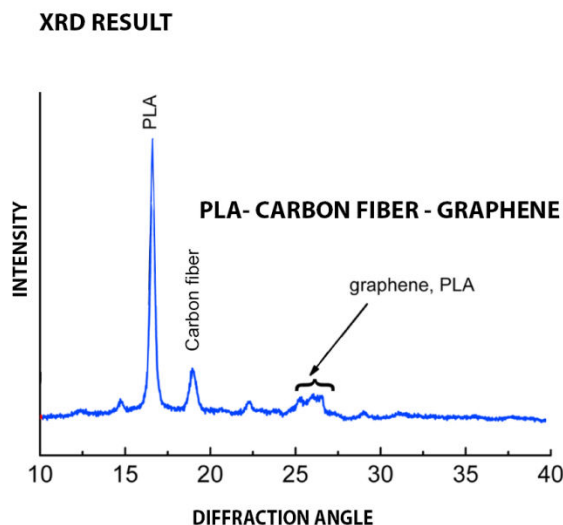


Fig -2: XRD diagram of PLA- Graphene

In this study, The XRD patterns of the PLA, Graphene and Carbon fiber are obtained from PLA-G-CF. According to Bragg's law ($n\lambda = 2d \sin \theta$), the value of D spacing depended on the θ value. The diffraction peak of graphene was observed at $2\theta = 26.7^\circ$ and carbon fiber at $2\theta = 18.3^\circ$. XRD analysis can be concluded that addition of graphene and carbon fiber into PLA results in higher crystallinity.

3.1.2. Tensile Test of the Filaments

The tensile properties comparison of PLA and PLA-G-CF were investigated at the room temperature and presented in Figure 3. From the typical stress-strain tensile curves and

corresponding statistical data, the elongation at the break value decreased with the Graphene and Carbon fiber loading. The elongation at the break value of pure PLA was determined as 5.8%, while the value of PLA-G- CF composite was 2.9%. However, by adding 2% Graphene and Carbon fiber, the tensile strength and Young's modulus of PLA were enhanced. This might be due to the interlayer cross-links of Graphene particles under loads, leading to the ordering of the hierarchical structures which results in the great significant enhancement of the mechanical properties.

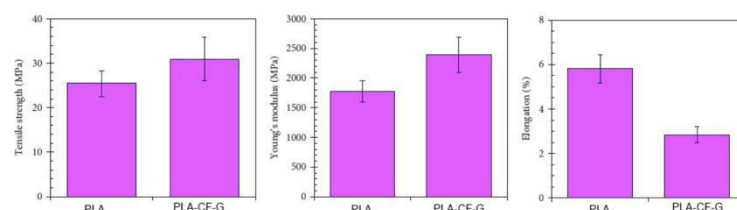


Fig -3: Stress-strain graph of the produced ABS composite filaments.

3.1.3. Comparison of the Filament Diameters

In the Figure 4, cross section surfaces of both of PLA-filament and the filament reinforced with Carbon Fiber and Graphene were compared. Today, commercial FDM filaments, mostly, have a diameter of 1.75 mm. Following the extrusion process, produced filaments were used in an FDM printer for trials. Too thin or too thick filaments caused printing errors, interrupted the process, and made impossible to print fully integrated parts. Trial processes showed that the importance of the consistency of the filament diameter. In this study, pure PLA and Graphene reinforced filament were compared and their diameter measured as 1.77 mm and 1.756 mm, respectively. Results show that, by arranging the extrusion process parameters properly, achieving suitable filaments compatible with FDM printing can be provided either using reinforcement particles or not using.

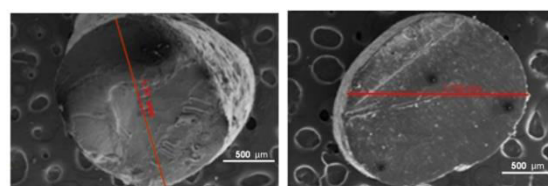


Fig -4: SEM microstructure of a) PLA FILAMENT, b) PLA-G-CF FILAMENT

3.1.4. SEM Microstructure of Fracture Surface

The alternative performance of graphene based nano filler-reinforced composites depends on the dispersion and distribution of graphene-based materials in the polymer matrix and the interfacial bonding between these two phases. The microstructures of the fractured sections of PLA-G-CF (dry mixing) on cross section are shown in Figure. PLA-G-CF demonstrated a higher-porous surface with the presence of dispersed Graphene flakes embedded in the PLA matrix. The smaller size and the more uniform distribution of Graphene and Carbon fiber were the critical keys for enhancing the

mechanical behavior and for the thermal diffusivity of the composite.

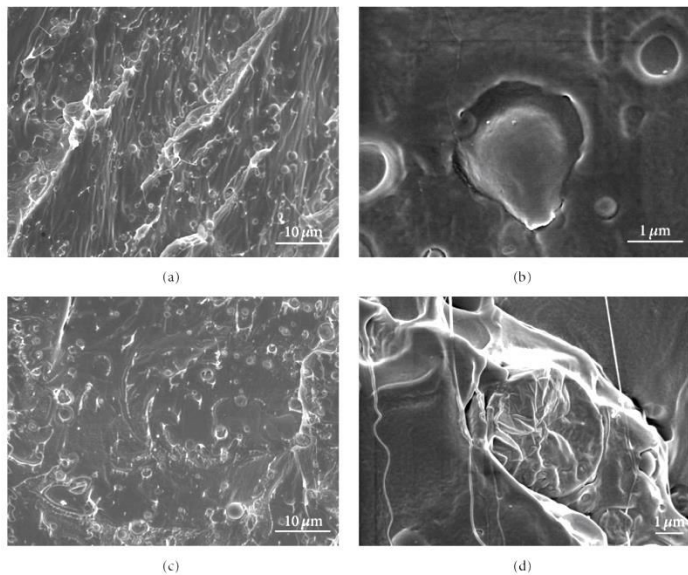


Fig -5: SEM images of PLA-G-CF dry mixing composite filaments.

3. CONCLUSIONS

The Fused filament fabrication (FFF) is a promising additive manufacturing (AM) technology due to its ability to build thermoplastics parts with advantages in the design and optimization of models with complex geometries, great design flexibility, recyclability and low material waste. This technique has been extensively used for the manufacturing of conceptual prototypes rather than functional components due to the limited mechanical properties of pure thermoplastics parts. A new 3D printing filament from PLA-Graphene-Carbon fiber composite was successfully prepared by solution mixing and followed by melted extrusion using a screw extruder. Characterization results show that these novel composites can be used as filament material in commercial FDM devices without requiring modification. According to the results;

1- The graphene and Carbon fiber reinforced PLA composite filaments for FDM type 3D printer were produced through a twin screw extruder between 190°C - 220°C without degradation of the matrix.

2- Thermal properties of neat PLA and PLA-CF-G blends were obtained from their DSC thermographs. DSC studies showed that produced filaments were compatible with commercial 3D printers because of having lower T_g values. However, a Melt Flow Index (MFI) should be applied on the composite polymers for determining exact processing temperature.

3- The solvent system improved the Graphene and Carbon fiber dispersion capability in the PLA matrix.

4- Graphene and Carbon fiber dispersion improved the thermal properties of the PLA-CF-G composite.

5- SEM microstructure of fracture surfaces indicated PLA-CF-G demonstrated a higher-porous surface with the presence of dispersed Carbon fiber and Graphene flakes embedded in the PLA matrix. The smaller size and the more uniform distribution of Carbon fiber and Graphene were the critical keys for enhancing the mechanical behavior and for the thermal diffusivity of the composite.

6- During the extrusion process, the nano composite filaments need to be fabricated-granulated for a couple cycles to achieve well dispersion of the reinforcement particles in the PLA matrix.

7- Improving mechanical properties verified the feasibility of 3D printed PLA-CF-G for potential use in engineering applications. The tensile strength and Young's modulus of PLA were enhanced naturally by adding Carbon fiber and Graphene. Besides, the elongation at break decreased and needed to be further improved.

Our study demonstrated one of the active attempts to print PLA-CF-G based composite using FFF 3D printing process directly. However, the printed part quality, functionalities, and applications based on the printed PLA-CF-G composite needed to be further exploited.

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The heading should be treated as a 3rd level heading and should not be assigned a number.

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BIOGRAPHIES



MOHAMMED BASHEER E.P, Post Graduate in Mechanical Engineering, specialization in ‘Advanced Manufacturing Technology’. I was the topper of University for Both Bachelors and Masters. I had ample of opportunity to work with different 3D Printing technologies and Projects. I have initiated a startup company “INSTA3D TECHNOLOGIES” which operates on 3D Printing technology.