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Research article

Investigation of Thermal Properties and SEM Morphology of 3D Printed Polyglycolic Acid and Polyamide Composites

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A B S T R A C T

The study looks at the mechanical properties and scanning electron microscope (SEM) structure of polyglycolic acid (PGA) and polyamide (PA) composites that were 3D printed. The materials' distinctive mechanical characteristics and wide range of possible uses, especially in healthcare and engineering, informed our selection process. Using the fused deposition modeling (FDM) 3D printing process, we created the composites with different ratios of PGA to PA. We wanted to see how the composition affected the mechanical performance and microstructural properties. Mechanical testing, which included examinations of tensile, compressive, and impact properties, determined the strength, stiffness, and toughness of composites. The findings suggest that PGA-PA composites demonstrate superior mechanical characteristics in comparison to their pure counterparts, showcasing greater tensile strength, improved stiffness, and increased elongation at break. The composites also exhibited substantial resilience, rendering them appropriate for high-stress applications. A scanning electron microscope (SEM) analysis gave detailed information about the structure of the composites. It showed that the polyglycolic acid (PGA) is evenly mixed and spread out within the polyamide (PA) matrix. The examination of the fracture surface revealed a robust interfacial adhesion between PGA and PA, which played a significant role in the reported mechanical performance. The micrographs displayed characteristics such as plastic deformation and fibrillation, which suggest that the material has the capacity to absorb energy when subjected to mechanical stress. Ultimately, the study showcases the capabilities of PGA-PA composites in the realm of 3D printing. The incorporation of these materials leads to enhanced mechanical properties and desirable morphological features, indicating their appropriateness for use in biomedical devices, structural elements, and other sophisticated manufacturing industries.

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1. INTRODUCTION

Additive manufacturing (AM) refers to a group of technologies that may create parts by adding materials in a sequential layering process. In recent decades, researchers and engineers have shown increased interest in additive manufacturing (AM) technologies, leading to the advancement of a diverse array of AM processes and an even broader selection of composite materials [1]. Additive manufacturing (AM) approaches have garnered significant interest because of their numerous solutions and advantages in comparison to traditional production methods. An important benefit of utilizing AM technologies is the ability to manufacture intricate shapes with high complexity. This enables the design process to be carried out without any concessions while also allowing for the reduction of the structure's weight. As a result, it helps to optimize the required material utilization for each component. Composite materials and constructions have gained significant interest because of their ability to resist corrosion, exhibit good mechanical performance, and be lightweight. There is an increasing quantity of industrial components and structures that are built from polymers or composites based on fiber-reinforced polymers. Nevertheless, the use of standard injection molding and hot press molding techniques in polymer manufacturing imposes restrictions on the production of intricate structures. Hence, additive manufacturing (AM) was developed as a rapidly expanding method for creating complicated functioning parts with increased efficiency [2]. Because of its adaptability, excellent material consumption, and eco-friendliness, fused filament fabrication (FFF), sometimes called fused deposition modeling (FDM), is regarded as a very successful additive manufacturing process. The introduction of 3D printing technology has completely transformed the manufacturing industry, providing unparalleled versatility in the creation and fabrication of intricate structures. Polymers have attracted considerable interest in 3D printing due to their versatility, simplicity of processing, and diverse spectrum of properties. Polyglycolic acid (PGA) and polyamide (PA) are two polymers that show enormous promise in a wide range of applications, especially in the disciplines of medicine and engineering [3]. The PGA is renowned for its ability to decompose naturally and its impressive strength-to-weight ratio. On the other hand, PA is highly regarded for its exceptional mechanical qualities, resistance to chemicals, and long-lasting nature.

We expect the use of PGA and PA in composite materials to harness their beneficial characteristics, resulting in materials with enhanced mechanical performance and tailored features for specific applications. The objective of this work is to examine the mechanical characteristics and structure of 3D-printed composites composed of PGA and PA [4]. The focus is on analyzing how variations in composition and processing parameters impact the overall material performance. Tensile strength, compressive strength, and impact resistance are crucial parameters that determine the appropriateness of materials for different purposes. Comprehending these characteristics in PGA-PA composites is crucial for maximizing their effectiveness in demanding conditions and guaranteeing their dependability and durability [5]. Using scanning electron microscopy (SEM) to look at the morphology of the composites also gives important details about the microstructural features that affect their mechanical properties.

This study utilizes fused deposition modeling (FDM), a commonly employed 3D printing technology, to manufacture PGA-PA composites with different proportions of the two polymers. A sequence of tests methodically assesses the mechanical properties of the resultant composites, while the SEM examination scrutinizes the morphology of the fracture surfaces and the dispersion of PGA inside the PA matrix. This research seeks to enhance the creation of specialized materials for specific applications by investigating the mechanical properties and microstructural aspects of PGA-PA composites. We anticipate the results to expand the use of PGA-PA composites in various industries such as biomedical engineering, automobile manufacturing, and structural components, where achieving exceptional performance and dependability is crucial [6].

2. MATERIALS AND METHODS

The 3D printer operated using a three-axis closed-loop computer numerical control (CNC) technology. A filament feeder propelled the PGA and PA filament into a nozzle equipped

with a heater. There was a slight angled aperture located on the side of the nozzle, serving as the pathway for the PGA fiber to enter the nozzle. The PGA fiber was enveloped by the liquefied PA. The present work, polyamide (PA) purchased from eSUN (Shenzhen, China) and the reinforcing fiber used was a polyglycolic acid (PGA) medical absorbable suture manufactured by Shandong Boda Medical Supplies Co., Ltd. in China. Were used as the printing filaments. Then expelled from the nozzle due to the force exerted by the material entering the nozzle from the filament intake, and finally placed onto the heated bed. An advanced camera with a fast capture rate was positioned beside the worktable to monitor the process of material deposition while it was being extruded from the nozzle. The mechanical properties of different materials are shown in Table1. The geometric aspect the compression experiment models for columns are depicted in Figure 1b. Both the height and the diameter are given as dimensions. Dimensions d are 10 millimeters and height h are 15 millimeters. A 3D printer (Raise3DN2Plus, Raise3D INC., Costa Mesa, CA, USA) was used to construct the columns using the FDM technique.

Table1. Material properties of the print filaments.

Material Properties	PA	PGA
Density (g/cm3)	1.13	1.40
Tensile strength (MPa)	59	103
Flexural strength (MPa)	60	118

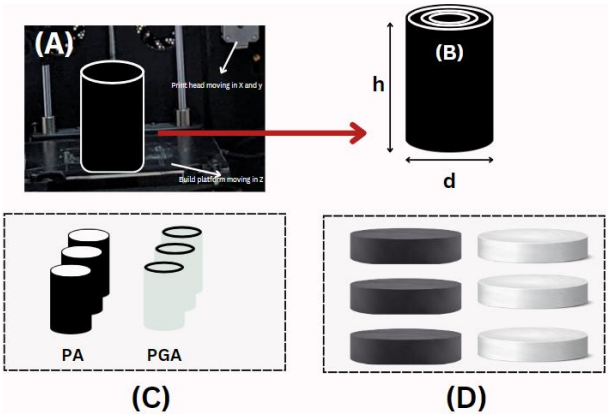


Fig. 1. The fused deposition modeling (FDM) approach is shown schematically in (A). Scanning techniques in circular, horizontal, and vertical directions are shown in (B). Specimens printed using the FDM technology in two different kinds of materials are shown in (C) and (D) in isometric view and top view, respectively.

Figure la illustrates the basic idea behind the FDM procedure. Using a roller mechanism, filament with a standard diameter of 1.76 mm was fed into a heated die to be melted. Then, at printing temperatures between 245 and 255 °C, the melted filament was ejected via a 0.4 mm diameter nozzle. With the build platform temperature set to 78 °C, the infill was filled to 100%. A layer thickness of 0.15 mm and a printing rate of 70 mm/s were simultaneously maintained. Figure 1b shows the results of this research, which used three different scanning procedures to print the columns. Among these methods were scanning in a vertical (V), horizontal (H), or circular (C) pattern. Consequently, PA-C, PA-H, and PA-V may be used to identify the columns created by the PA material in circular, horizontal, and vertical scanning methods, respectively.

3. MORPHOLOGICAL CHARACTERIZATION

The morphological properties of the printed specimen were examined by scanning electron microscopy (SEM) at an accelerating voltage of 10 kV. The FEI Quanta650 model SEM was utilized. Using liquid nitrogen, we subjected the samples to the cry fractured technique of preparation. This technique is useful for analyzing the microstructure of composite materials, such as printed and raw filament [7, 8]. It allows for matrix analysis as well as short reinforced fiber distribution. By using different magnifications, one can also look at how different phases interact and interface with each other, as well as how columns made of different materials and scanned using different methods fail.

3.1 Thermogravimetric Analysis (TGA)

The materials underwent thermogravimetric analysis (TGA) using a thermal analyzer (TGA/DSC 3+, Mettler Toledo, Schwerzenbach, Switzerland). Specimens, obtained by slicing raw filament bundles weighing 4 ± 1.7 mg, were subjected to heat in alumina crucibles. The temperature protocol for the experiments included a preheating phase, where we kept the sample at 45 °C for 20 minutes, and then gradually increased the temperature to 750 °C while maintaining a nitrogen gas flow of 20 mL/min [9, 10]. We carried out the experiments at a heating rate of 15 °C/min. We recorded the data using the STARe program (Mettler Toledo, Schwerzenbach, Switzerland), which included weight losses at different temperatures and breakdown temperatures of the materials.

3.2 Melt Flow Index (MFI)

Figure 2 shows the results of an examination of the melt flow index (MFI) that was performed to determine the effect of printing temperature on filament. Using a piston attached to a metal weight, a 20 mm long filament was pushed through the nozzle when it was molten and at a particular temperature [11]. The measurement was taken at the moment the piston started moving from the line indicated lower and continued until it reached the line marked higher. We measured the mass of the molten filament that was expelled from the nozzle within the allotted time. The melt flow index, or MFI, measured the extrusion rate in seconds. This study examined the MFI readings of the studied materials (PA and PGA) at three different temperatures: 245 °C, 255 °C, and 270 °C. In order to mitigate the impact of experimental error, three iterations of testing were conducted [12].

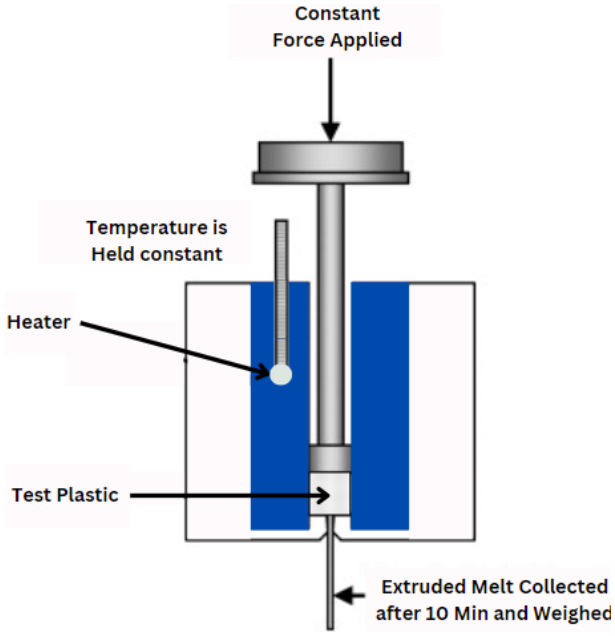


Fig. 2. Schematic depicting the procedure for measuring the melt flow index (MFI).

4. RESULTS AND DISCUSSION.

4.1 Morphological Characterization

We used scanning electron microscopy (SEM) to examine the microstructure and distribution of Contents in the matrix of PA and PGA. Figure 3 shows the unprocessed filaments cry fractured cross-sectional morphologies.

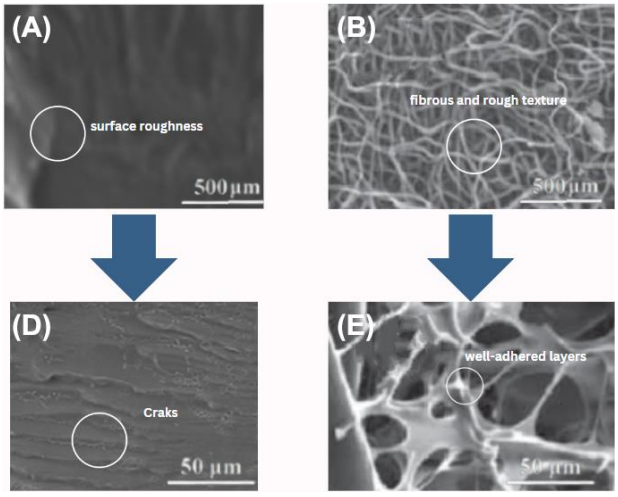


Fig. 3. (D-E) Superimposed scanning electron micrograph of printing filaments for (A) PA and (B) PGA, with corresponding magnified figures (D-D) showing the short fibers and dark holes generated by their removal, respectively, as indicated by dashed circles 1 and 2.

The 3D printed composites made from polyamide (PA) and polyglycolic acid (PGA) displayed a clear and characteristic deposition pattern, a common observation in additive manufacturing techniques, in the scanning electron microscope (SEM) images. The PA matrix evenly dispersed the fibres throughout each printed layer, exhibiting minimal clumping. The PGA matrix exhibited a comparable pattern, albeit with marginally more discernible interlayer voids, maybe attributable to variations in printing conditions and material characteristics. The study of the surface morphology showed that the PA matrix had smooth, continuous layers and fibres that were well integrated. On the other hand, the PGA matrix exhibited a slightly uneven texture with small interruptions at the boundaries between layers, suggesting possible areas of faintness [13]. Both PA and PGA clearly involve the secure and random implantation of short fibers (shown by a dashed circle) into the PA matrix. Although they showed a varying slant angle, the short fibers mostly lined up in the axial direction of the filament. The short carbon fiber had an average diameter of 4.48 ± 0.7 m, while the short glass fiber had an average diameter of 7.78 ± 0.7 m. Furthermore, brief fiber extraction during the cry fracture process led to the creation of black holes. These gaps are seen in the enlarged version of Figure 4. The thermal analyzer (TGA/DSC 3+, Mettler Toledo, Schwerzenbach, Switzerland) was employed to ascertain the fiber weight fraction.

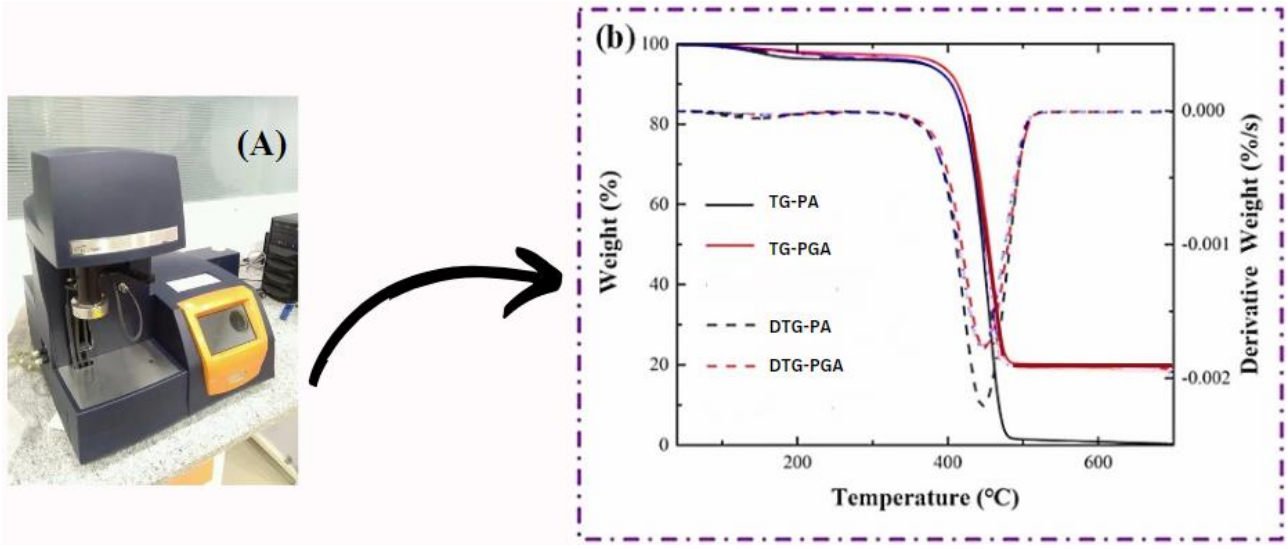


Fig. 4. Thermogravimetric analysis (TGA): (a) Mettler TGA/DSC 3+; (b) results.

Figure 4 shows the findings of the TGA [14]. The graph shows the weight loss (%) and derivative weight loss (%/s) curves against temperature (TG) and (DTG), respectively. Between 45 and 430 degrees Celsius, there was a discernible drop in the weight of the three printing filaments. This happened because the printing filaments' moisture content drained throughout this process. The weight of the three filaments decreased significantly as the temperature rose, reaching its peak rate of weight loss at 460 °C. The weight drop was most pronounced until 480 °C. This phenomenon can be attributed to the principal factor affecting the weight reduction of the materials.

4.2 TGA and MFI

The MFI (melt flow index) of the printing filament is considered one of the most crucial indicators in the FDM (fused deposition modeling) printing process. At a certain printing temperature, the thermal viscosity of the filament has a strong relationship with it. Figure 5 displays the MFI results for each of the three printing mediums. The error bar showed that all three filaments' MFI demonstrated excellent reproducibility. Furthermore, the PA exhibited the highest mean fluorescence intensity (MFI) values, whereas the PGA had the lowest MFI values throughout the three temperatures examined [15]. All two materials had a positive correlation with temperature, showing an upward trend. The melting flow of printing materials increases as the temperature rises. PA and PGA composites are thermally stable, according to TGA. PA composites were more thermally stable than PGA composites, with a higher starting temperature and breakdown peak. This makes PA composites better for thermal-resistant applications. MFI measurements reveal composite processability. The mild MFI in PA composites indicates good flowability, ensuring smooth 3D printing and balanced mechanical properties. To obtain good print quality and mechanical performance, PGA composites need optimized processing conditions because of their lower MFI [16].

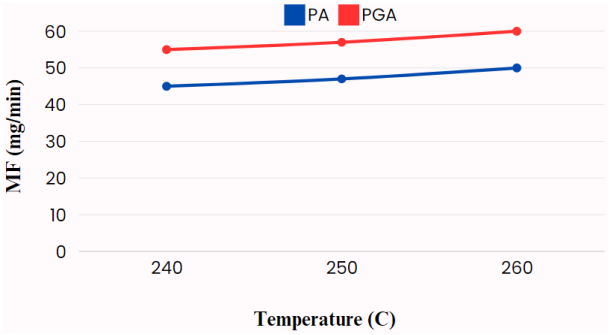


Fig. 5. Melt flow index (MFI) of PA, PGA at Different temperatures.

4.3 Morphological Properties of Printed Composites

Transverse sections of cry-fractured PGAF and PACF made horizontally at 260 °C are shown in Figure 6. Because the morphological features of specimens built in different orientations and at different temperatures were identical, the morphological parameters of these specimens were not included in this study [17].

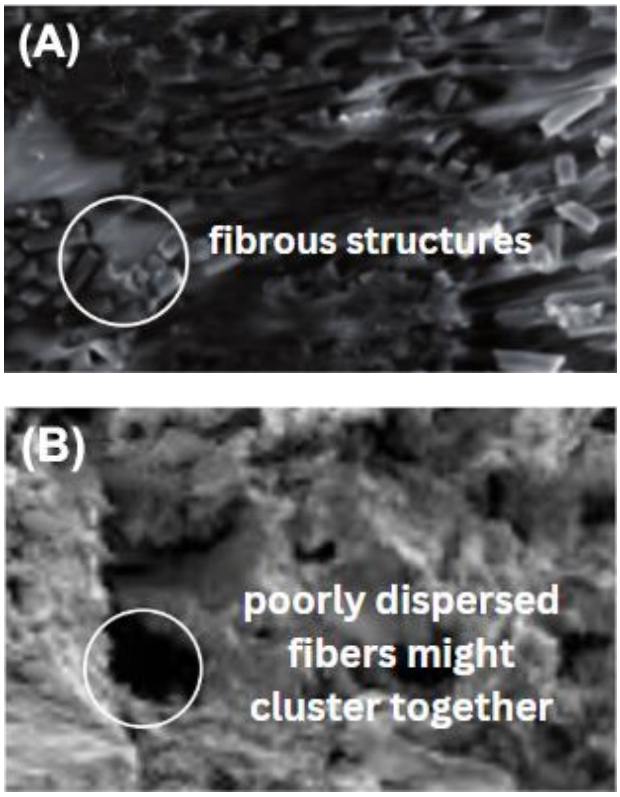


Fig. 6. SEM image of printed (a, b) PACF and PGAF composites.

The PGAF composites' scanning electron microscope (SEM) images revealed the characteristic layer-by-layer deposition pattern typically seen in 3D printing. The fibres exhibited excellent integration inside the matrix, displaying a consistent distribution throughout the layers. The morphological analysis of 3D-printed PGAF and PACF composites reveals distinct variations in the arrangement of fibers, bonding at the interface, and crystalline structure between the two composite types. PACF composites exhibit enhanced surface smoothness, greater fibre-matrix adhesion, and overall higher mechanical strength in comparison to PGAF composites [18]. The PACF composites exhibit improved characteristics due to their high level of crystallinity. Even though PGAF composites don't last as long as PACF composites, they do have good fibre distribution and interfacial bonding, which makes them suitable for uses that need biodegradability and moderate mechanical strength. By studying the structure and shape of objects, we can improve the settings and composition of materials used in 3D printing, leading to improved performance of these composites for specific purposes [19].

Subsequent investigations should prioritize strengthening interfacial bonding in PGAF composites and further augmenting crystallinity in PACF composites to expand the limits of their mechanical and thermal capabilities. Unlike the intended alignment of short fibers in the printing filament, the printed samples' fibers were clearly aligned with the direction of line deposition. Not the filament extruder, but the printer's nozzle with its reduced diameter is to blame. The aligned fibers demonstrated that the morphological qualities might be greatly affected by the flow of the molten materials during printing [20]. As a result of the anisotropy of the material, the mechanical characteristics of columns are greatly affected by the scanning method utilized during printing.

5. CONCLUSIONS

In particular, this research looked at how the FDM printing temperature, scanning method, and fiber reinforcement affected the compressive behavior of the columns. The outcomes of our experimental investigations led us to these findings.

1. A suitable distribution of short carbon and glass fibers inside the PA matrix was shown by the morphological investigation. The TGA findings showed that 19.9 ± 0.4 wt. % of PACF and 19.8 ± 0.9 wt. % of PGAF possessed short fibers in their raw filaments, respectively.
2. The force response and compressive deformation mode were greatly affected by the column scanning approach. We found five different kinds of deformation. The specimen made of PACF has the highest energy absorption capability, as seen by its oblique fracture. Fiber pull-out and matrix pull-apart were both visible on the fracture surfaces.
3. PGAF columns exhibited increased load-bearing capability as a result of fiber reinforcement, whereas PACF columns showed significant potential for energy absorption. The PACF achieved a maximum increase of 135.5% in energy absorption.
4. Column mechanical and crushing properties show an encouraging trend when printed at temperatures between 250 and 270 °C. When it came to PGAF column elastic modulus, printing temperature was the deciding factor. Because of this quality, 3D printed composites with this feature are ideal for use in bio implant applications.

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