

# FABRICATION, MECHANICAL CHARACTERIZATION, AND 3D-PRINTING OF SHORT GLASS FIBRE REINFORCED POLYCARBONATE COMPOSITE FILAMENTS

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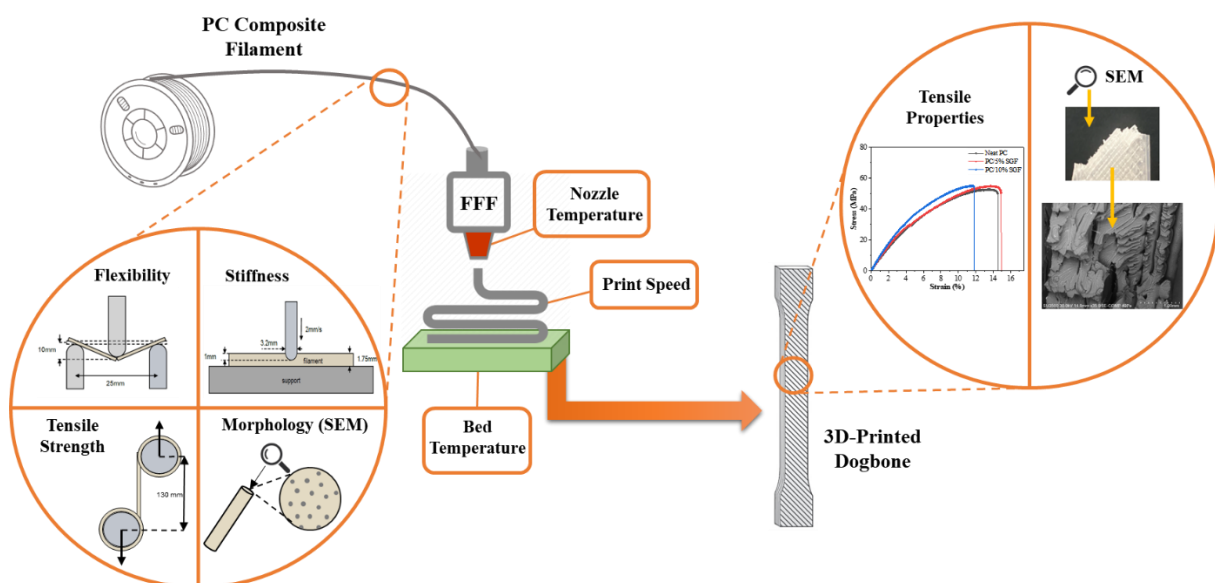
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## ABSTRACT

This study aims to enhance the mechanical properties of fused filament fabrication (FFF) filaments and 3D-printed parts by incorporating short glass fibres. The primary objective is to develop low-cost composite materials with superior mechanical performance for applications with critical requirements. To achieve this goal, the study investigates the thermal, tensile, and flexural properties of the fabricated filaments at varying reinforcement contents to explore a relationship between filament and print qualities. The thermal analysis confirms the presence of fibre content in the extruded filaments and shows that the glass fibres slightly increase the glass transition temperature of the PC matrix. The high tensile strength and stiffness of the filaments indicates their good printability, but the flexural testing results show that the glass fibres induce less flexibility, which can adversely affect the handling and printing process. The tensile testing of 3D-printed specimens revealed the high elastic modulus of the printed components compared to PC injection-moulded parts reported in the literature. However, SEM images of the fracture surface of filaments and 3D-printed parts show a transition from ductile to brittle failure behaviour. The micrographs also demonstrate a considerable enhancement of adhesion between the layers, indicating how customization of filaments can impact final print quality.



Graphical Abstract: The schematic representation of 3D-printing and characterization methods used to evaluate short glass fibre reinforced polycarbonate filaments.

## 1 INTRODUCTION

Due to their higher specific strength and stiffness, as well as more efficient processing, lightweight fibre-reinforced polymer composites are increasingly replacing metallic parts in advanced systems such as aerospace components [1]. During the initial phases of the design process, it is crucial to carefully select the appropriate material for the desired application, considering the chosen fabrication method. Recently, it was demonstrated that polycarbonate (PC) can be a cost-effective candidate with moderate properties for 3D-printing of aerospace components in comparison to the high-temperature thermoplastics like polyether imide (PEI), which provide comparatively superior mechanical properties but at a high cost [2]. The use of short fibre-reinforced composite filaments has been identified as a beneficial approach for achieving high-strength polymer composites through fused filament fabrication (FFF) [3–5]. For instance, Mohan *et al.* [6] demonstrated the effectiveness of incorporating short glass fibres (SGF) into an Acrylonitrile Butadiene Styrene (ABS) matrix for achieving significant improvements in the elastic modulus and tensile strength of 3D-printed composites. Specifically, their study showed increases of up to 26% and 57% in these properties, respectively. As a rather new material in 3D-printing applications, there is a dearth of literature on the performance of the 3D-printed glass fibre reinforced PC components. However, it has been widely reported that this composite demonstrates excellent performance when injection moulded. For example, Gamal Sadek *et al.* [7] evaluated the mechanical performance of injection moulded PC composites with different contents of SGF and found that addition of only 10 wt.% short glass fibres into the matrix results in almost 31% increment in Young's modulus of the composite. Similar conclusions were drawn by Jawali *et al.* [8] who studied mechanical and load-bearing properties of injection moulded PC/SGF composites at different reinforcement contents. Although it is well-established that the maximum achievable properties of 3D-printed parts are limited in comparison to the injection moulded ones [9,10], exploring the characteristics of SGF reinforced PC filaments for FFF process appears promising to broaden the range of available materials with superior performance for fabrication of components with critical requirements.

Since both manufacturing time and cost of FFF can be reduced by optimization of filament quality [11], moving toward identification of important factors affecting FFF filament quality and understanding how it can influence the ultimate print quality is important. Although fabrication of short fibre reinforced filament is reported in several studies [12], the existing literature on characterization of filament properties is insufficient. For instance, Rahimzadeh *et al.* [13] investigated the fibre length distribution and fibre orientation of recycled glass fibres within PLA filaments in different reinforcement contents and concluded that the mean fibre length decreases when the higher amounts of fibre is incorporated into the polymer. Also, regarding the microstructure of the composite filaments, they showed that the fibres are highly oriented along the longitudinal axis of the filament. Concerning the mechanical properties of the filaments, Boparai *et al.* [14] examined tensile properties of the Nylon composite filaments and their printing performance and highlighted the importance of reinforcement-matrix bonding in improving the mechanical properties of the filaments. Moreover, Singh *et al.* [15] performed a parametric optimization in order to study the effect of extrusion parameters such as filament composition, barrel temperature, and die temperature, on the mechanical properties of filaments and demonstrated that composition has the most contribution on the tensile strength and Young's modulus of the filaments, although the authors did not specify their filament testing methodology. Furthermore, another key characterization method that can be conducted on FFF filaments is printability evaluation. Xu *et al.* [16] defined filament printability as “printing feasibility of filament” and proposed a new approach to quantify and predict the printability of the filaments based on Repka-Zhang test which was introduced in their previous work [17].

In this paper, the goal is to fabricate SGF reinforced polycarbonate FFF filaments at different fibre contents and to study the effects of fibre incorporation on mechanical properties and printability of the PC filaments. The optimal printing conditions were identified to print tensile specimens and to investigate how 3D-printing characteristics are affected by filament quality. To serve this purpose, the characteristic temperatures of the composites were assessed. Subsequently, the flexibility, printability and tensile properties of the filaments were studied to evaluate the filaments quality at different SGF contents. The 3D-printed samples were tested for their tensile properties and their fracture surface was captured to gain better insight regarding their failure behaviour and 3D-printed microstructure.

## 2 MATERIALS AND METHODS

Polycarbonate pellets (LUPOY GN1006FL purchased from LG Chem, Canada) were compounded with short glass fibres (ECD11-4.5-584 purchased from China Jushi co., LTD) at different contents of 0, 5 and 10 weight percent of polymer using a twin-screw extruder (Coperion, Germany) equipped with a water bath, and a filament winder. The 11 heating zones of the extruder were set to 250, 260, 270, 270, 270, 265, 255, 245, 235, 225, 215 °C and 210 °C as the die temperature. Moreover, the extrusion speed and feeding rate were 120 rpm and 1.2 kg/hr, respectively. The diameter of the filaments was controlled using the winding speed and collected on an empty spool.

The composition and degradation temperature of the filaments were measured by thermogravimetric analysis (TGA TA Instruments, Model: Q500). The degradation temperature ( $T_d$ ) was considered as the temperature at which the material loses 1% of its initial weight. For this purpose, a sample of 8-12 mg of each filament was heated under air atmosphere. The analysis was conducted at a heating rate of 10°C/min from 25°C to 850°C to determine composition of composites using the residual amount of the filaments. Moreover, differential scanning calorimetry (DSC TA Instruments, Model: Q100) was used to quantify the effect of fibre content on glass transition temperature ( $T_g$ ) of the polymer. Samples of approximately 10 mg were scanned through two successive heating and cooling cycles between 25°C and 350°C in a nitrogen flow of 50 ml/min using a heating/cooling rate of 20°C/min.

Tensile, flexural, and stiffness tests were adapted according to [16] to characterize the filaments' mechanical properties, flexibility, and printability. Accordingly, the tensile properties of the filaments were obtained using Capstan grips with a diameter of 96.4 mm and a crosshead speed of 200 mm/min. Five specimens were tested for each formulation to obtain standard deviations. The diameter of the filaments was measured at 3 different points within the gauge length of 130 mm between the Capstan grips, and the average diameter was used for calculations. The 3-point bending test procedure suggested by ASTM D790-17 was modified to evaluate flexibility of the filaments. Therefore, five 50.8 mm-long pre-dried samples of each filament were tested. The support span and test speed were set to 25.6 mm and 0.62 mm/min, respectively. The crosshead motion rate ( $R$ ) was calculated using Eq. 1 considering a sample with a depth of 1.75 mm:

$$R = ZL^2/6d. \quad (1)$$

where  $L$  is the length of support span in mm,  $d$  is the depth of the sample and  $Z$  is rate of straining of the outer fibre, which shall be equal to 0.01 according to ASTM D790-17 [18]. The flexural modulus of filaments was calculated following the equation below:

$$E_B = 4mL^3/3\pi d^4. \quad (2)$$

where  $m$  is the slope of the tangent to the initial straight-line portion of the load-deflection curve, and  $L$  and  $d$  are support span and filament diameter, respectively. This equation was obtained using the second moment of area for a cylinder.

To measure the stiffness of the filaments, a filament was loaded using a metallic cylinder with a diameter of 3.2 mm. The loading continued until a penetration distance of 1 mm (57% strain) was reached at a speed of 2 mm/s and the stiffness of the filaments was determined by calculating the maximum load-bearing capacity, which is defined as the maximum load divided by the average diameter of the filament. The mechanical characterizations were performed using MTS mechanical testing machine equipped with a load cell of 500N.

Additionally, type I tensile coupons were printed using a AON3D printer (M2-2020 AON3D, Canada) following ASTM D638 with a raster angle of  $\pm 45^\circ$  in XY flat build orientation and 100% infill. The printing parameters, including nozzle temperature, bed temperature, and printing speed, were optimized using a design of experiment at three levels to obtain the best print quality (Table 1). The quality of the print for each component was assessed using a rating scale ranging from 1, representing poor print quality, to 10, indicating excellent print quality. Moreover, the chamber temperature, nozzle diameter, and layer thickness used for printing are 70°C, 0.6 mm, and 0.3 mm, respectively. The tensile testing of the 3D-printed coupons was conducted at a displacement rate of 120 mm/min using the MTS mechanical testing machine with a 5kN load cell. Finally, the fracture surface of the filaments and 3D-

printed coupons were analysed using a Hitachi SU-3500 Variable Pressure Scanning Electron Microscope (VP-SEM).

Experiment	Nozzle temperature (°C)	Bed temperature (°C)	Print speed (mm/s)
1	260	80	20
2	260	140	50
3	310	80	50
4	310	140	20
5	285	110	35
6	260	110	35

Table 1. Design of experiment for print quality optimization of neat PC filament.

### 3 RESULTS AND DISCUSSIONS

The weight loss of the filament samples under air atmosphere obtained by TGA is plotted against temperature and the results are shown in Figure 1 for the PC filaments at three different compositions. According to these results, no weight loss is detected up to 400°C which shows the high thermal stability of the materials at elevated temperatures. The main weight loss of the materials starting at approximately 450 °C can be attributed to the degradation of the chemical structure to CO, CO<sub>2</sub>, and Bisphenol A products [19]. A similar decomposition rate is observed for all the materials which shows that glass fibres do not contribute to the weight loss of PC. At temperatures above 550 °C, the rate of decomposition for neat PC filament decreased, and the sample was consumed. It can be concluded that the final residue at temperatures above 750°C is only representative of fibre content within the filaments.

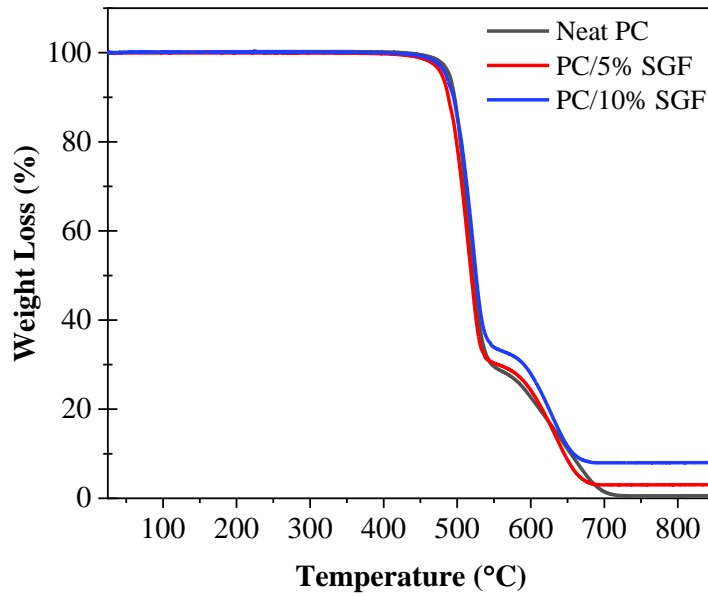


Figure 1. The results of thermogravimetric analysis on the PC filaments at different SGF contents of 0, 5, and 10 wt.% of polymer.

The thermal characteristics of each filament including degradation temperature ( $T_d$ ) and glass transition temperature ( $T_g$ ) are summarized in Table 2 together with their residual amounts at 850 °C. It was found that the addition of 5wt.% short glass fibres to the PC matrix decreased its thermal stability. As the glass fibres are highly stable at elevated temperatures, this reduction in thermal stability of the composite can be attributed to the active sites introduced to the composite system at the fibre-matrix interface, accelerating the decomposition initiation [20]. However, this effect was alleviated at higher composition which can be due to the improved interactions between fibres and matrix and increased

contribution of glass fibres in the ultimate thermal stability of the composite. The  $T_g$  values calculated from DSC demonstrated minor improvement when short fibres were introduced to the system which can be due to the hindrance caused for polymer molecular movements. Also, the final residue amounts indicated the weight percentage of glass fibres present in each composite filament. The fibre content measured for 5 wt.% SGF reinforced filament was found to be below the intended composition which can affect the performance of the material for the succeeding analyses.

Filament	Glass transition temperature (°C)	Degradation temperature (°C)	Residue at 850 °C (wt.%)
Neat PC	148.8	454.1	0.0
PC/5% SGF	149.6	446.2	3.6
PC/10% SGF	149.6	457.0	9.5

Table 2. The results of thermal characterization of PC filaments using TGA and DSC.

The tensile properties of the filaments are shown in Figure 2. The Load-Displacement curves of the specimens clearly exhibit the improvement in tensile properties in presence of short fibres. The tensile strength of the filaments can be calculated using the average diameter of the filaments.

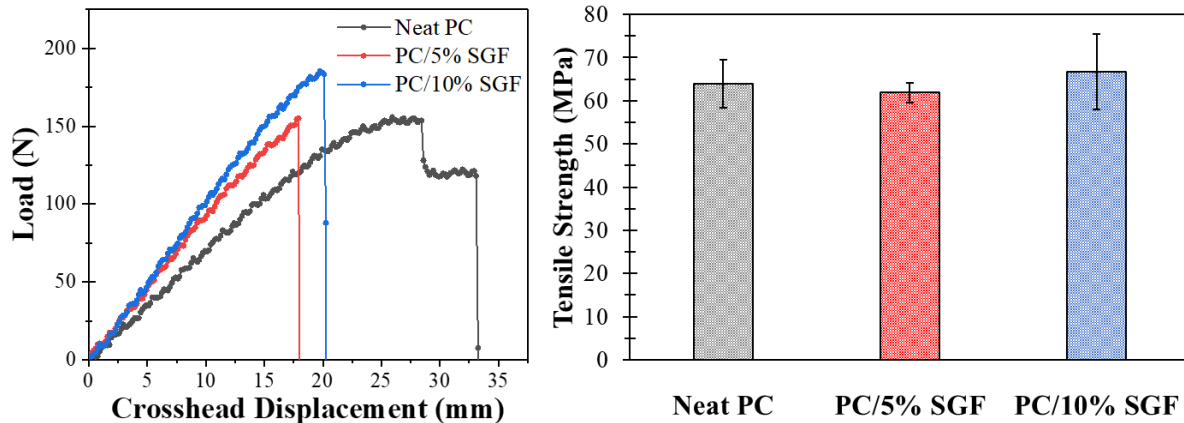


Figure 2. Tensile properties of the PC filaments at different SGF contents of 0, 5, and 10 wt.% of polymer, showing: a) Load-Displacement curves b) Tensile strength of filaments

Although PC/10 wt.% SGF revealed better performance in comparison to the neat filament, the composite filaments showed an overall comparable performance to the average tensile strength of the neat PC filament which was very close to the value reported for this property in the material's datasheet (62 MPa). This can be explained by weaker fibre-matrix interactions in these composites, indicating that a better interfacial bonding is required to allow a more efficient stress transfer and consequently, tensile strength improvement. Also, it is known that addition of reinforcements to a polymeric resin can create voids within the composite which might affect the performance of the material. This was studied using the SEM micrographs of fracture surface of the filaments shown in Figure 3. The higher void content is evident when the fibres are introduced to the system. Also, there are noticeable fibre-rich regions in the PC/5 wt.% SGF composite filament which can explain the weaker performance of this filament compared to the filament with 10wt% reinforcement. The failure observed for the neat PC filament was accompanied by necking behaviour while the composite filaments showed an abrupt failure. This indicates the higher toughness of the PC before addition of short fibres which can also be realized by comparing the fracture surfaces.

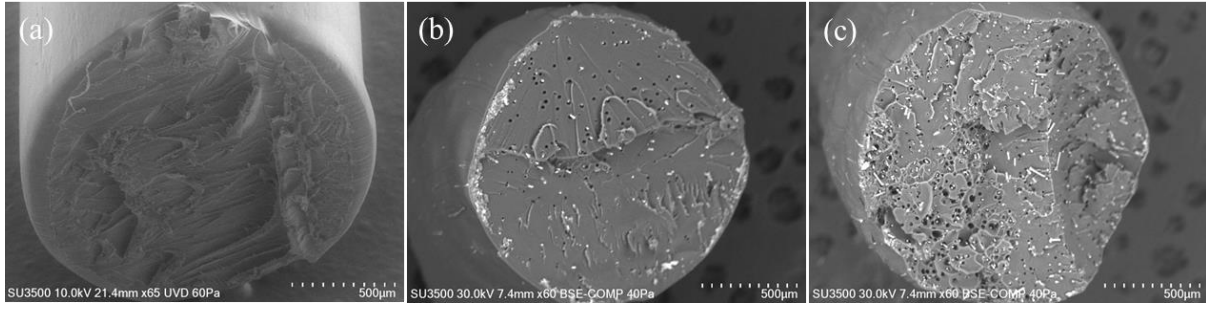


Figure 3. SEM micrographs showing the fracture surface of a) Neat PC, b) PC/5% SGF, and c) PC/10% SGF filaments.

Moreover, Ghanizadeh *et al.* [21] reported that a printable filament would demonstrate high maximum tensile stress as well as high rigidity. They showed that filaments with a tensile strength lower than 20 MPa were not printable. Comparing the results of this study to those reported by the authors, it could be expected that the fabricated PC filaments will exhibit good printability for fabrication of 3D-printed specimens. The PC filament with the highest amount of SGF showed the greatest slope in the linear region which can be an indication of higher elastic modulus and rigidity. On the other hand, the authors also analyzed the elongation at break to further investigate the flexibility and printability of the filaments, but the strain measurement technique was not specified in their methodology description. Since precise strain measurement cannot be realized for tensile testing of filament-shaped specimen with the common extensometers, it would not be practical to analyze elastic modulus and strain at break using the crosshead displacement data. The technique adopted in this study to evaluate printability is similar to the methodology employed in [16], which involved a texture analyzer - a relatively less accessible equipment as compared to a mechanical testing machine. Here, we solely compared the stiffness of the materials as it is a more easily attainable characteristic compared to the area under the curve that represents the previously stated toughness. Figure 4 demonstrates the corresponding results for all the PC filaments compared to a known unprintable filament which was fabricated using a blend of the same PC grade and an incompatible polymer. The attempts to print this filament were unsuccessful and it was tested using the same procedure as the other filaments. Here, the crucial point is that a filament must withstand a specific amount of load to be able to pass through the gears of the filament feeding system and to be viable for printing. As a result, this load-bearing capacity can be inferred as the value beyond which the filament is printable. Since the dataset available was limited for this study, the exact printability threshold could not be determined, only an estimated range is indicated in grey. Accordingly, it can be concluded that all the fabricated filaments fabricated in this study are printable.

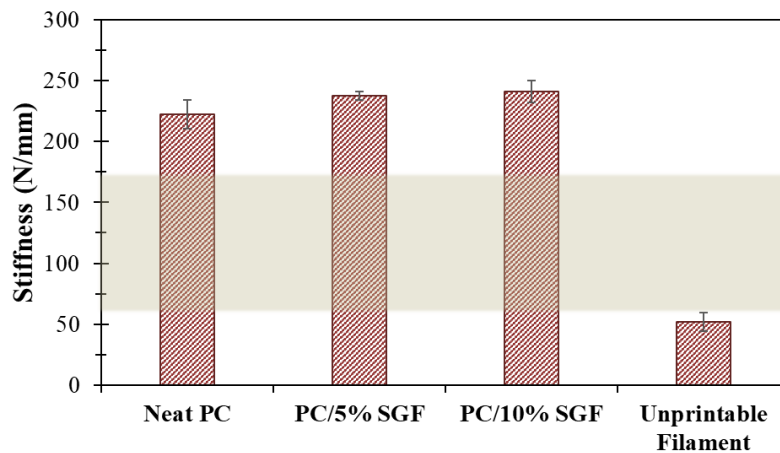


Figure 4. Comparison of the stiffness of PC filaments at different SGF contents (0%, 5%, and 10% wt. of polymer) with a known unprintable filament. The gray region highlights the possible range of load-bearing capacity threshold for a filament to be printable.



Assessing the flexural characteristics of filaments is a viable method to determine their flexibility. A high bending modulus could be a disadvantageous property for FFF filaments as it implies a lesser ability of the material to deform when subjected to force or load. On the other hand, more flexible filaments are easier to handle and wind during the 3D-printing process. Hence, the three-point bending properties of a filament, as presented in Figure 5, can offer significant insights regarding its feedability and flexibility. The neat PC filament did not break during the test and showed a tough behaviour, a property that PC is known for. Nevertheless, when reinforced with SGF, 3 out of 5 samples displayed brittle fracturing, signifying the stiffening effect of reinforcement. It is worth noting that the bending strength of the filaments saw an improvement from 76 MPa for neat PC to 79 and 98 MPa for fibre contents of 5 wt.% and 10 wt.%, respectively. The PC material's bending modulus as specified in its datasheet is 2.25 GPa. Comparing this to the outcomes of this study, it can be inferred that incorporation of SGF in the composite system raised the bending modulus by around 1 GPa compared to the pure PC filament, rendering the filament more vulnerable to breakage.

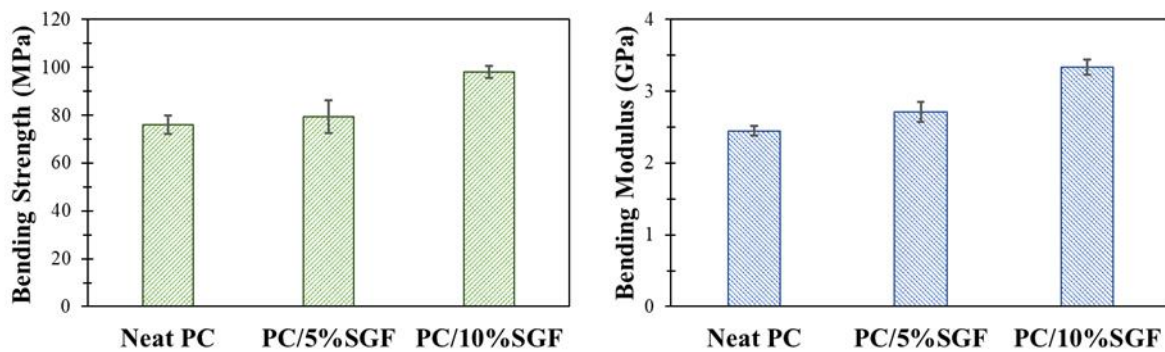


Figure 5. Three-point bending properties of PC filaments reinforced with different SGF contents of 0, 5, and 10wt.% of polymer.

As mentioned previously, the printing parameters, including nozzle temperature, bed temperature, and print speed, were optimized according to Table 1 in order to find the best print quality for the fabricated filaments. The results of the print quality assessment are shown in Table 3. The three different shades of blue represent different levels of the parameters in each experiment. Additionally, the print quality column displays red, orange, and green colors indicating poor, average, and excellent print quality, respectively. Experiment 5 exhibited the best print quality, and as such, its parameter values were employed to print the 3D-printed coupons for mechanical testing.

n°	Nozzle temp. (°C)	Bed temp. (°C)	Print speed (mm/s)	Print quality (1 to 10)
1	260	80	20	1
2	260	140	50	6
3	310	80	50	6
4	310	140	20	4
5	285	110	35	9
6	260	110	35	5

Table 3. The results of print quality optimization for 3D-printing of PC filament.

The mechanical properties of the 3D-printed coupons were investigated, and stress-strain curves were generated for 3 filaments to assess the impact of fibre addition on their properties (Figure 6). Essential tensile properties like elastic modulus, tensile strength at yield, stress at break, and strain at break were then calculated and presented in Figure 7. It was found that the introduction of glass fibres to the PC matrix resulted in an enhancement of all the mentioned mechanical properties excluding the strain at break.

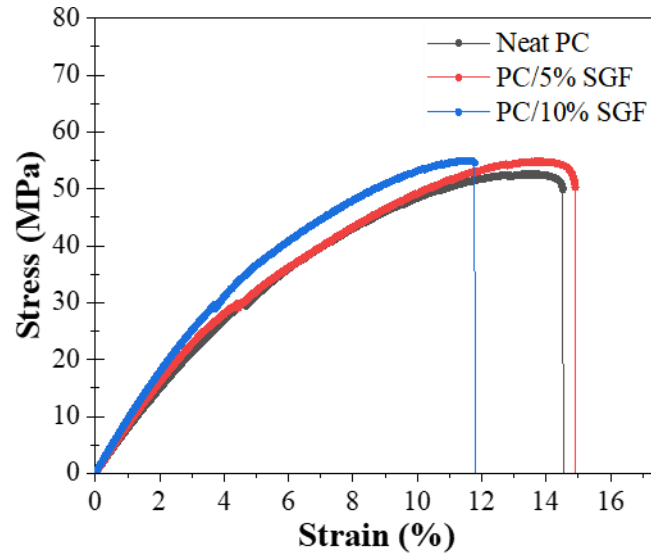


Figure 6. Comparison of Stress-Strain curve of 3D-printed coupons fabricated using the PC filaments at different SGF contents.

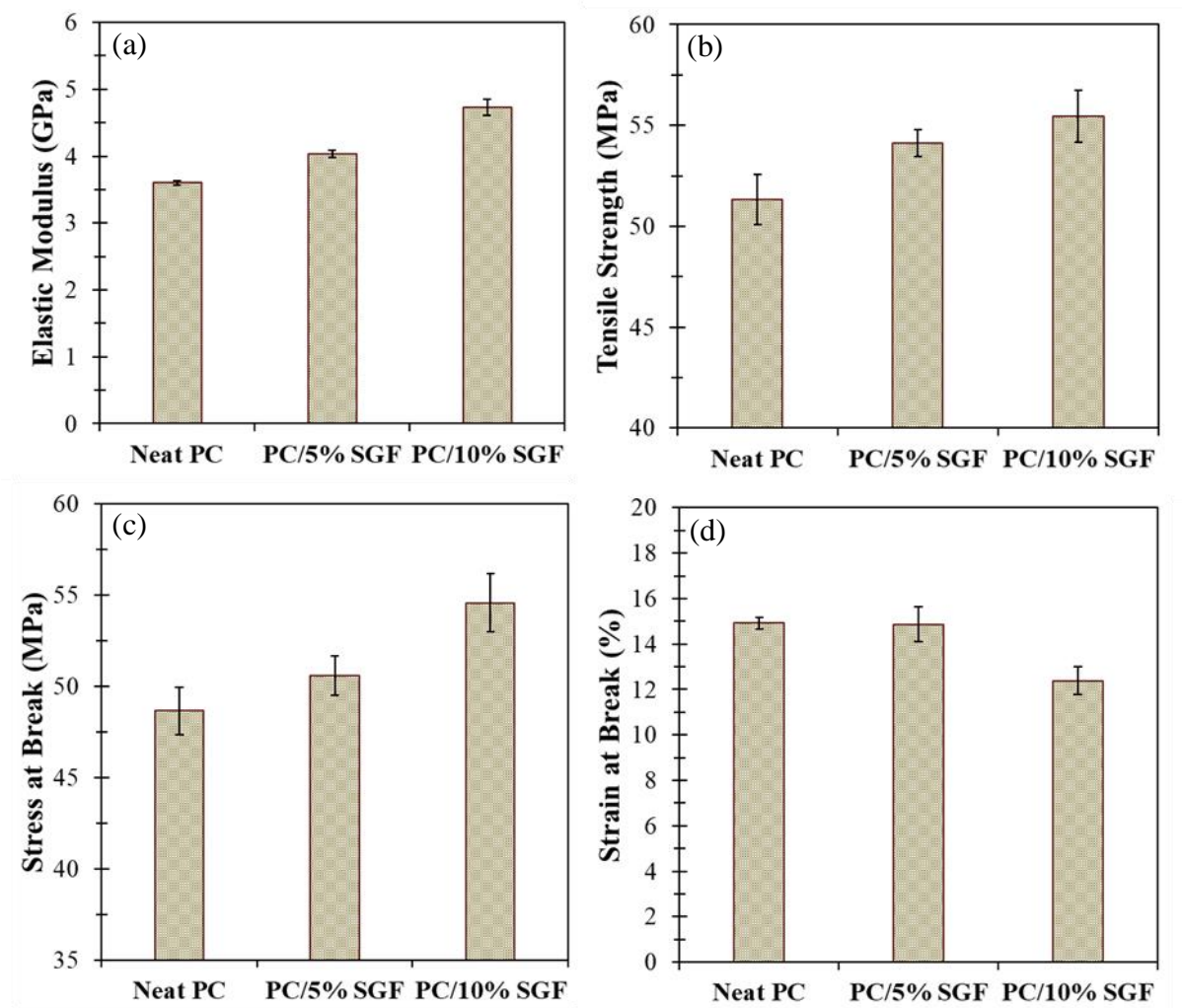


Figure 7. Tensile properties results, showing a) Elastic Modulus b) Tensile Strength, c) Stress at Break, and d) Strain at Break, of the 3D-printed PC coupons reinforced with at different SGF contents of 0, 5, and 10 wt. %



The addition of 5 and 10 wt.% SGF to the PC matrix resulted in an improvement of the elastic modulus from 3.6 GPa (neat PC) to 4 and 4.7 GPa, respectively. These values were found to be comparatively significant when compared to the ones mentioned in [22,23] for injection moulded PC parts. For instance, Gamal *et al.* [7] reported an elastic modulus of 2.6 GPa for a similar PC composite containing 15 wt.% of short glass fibres. In addition, there was a slight increase in both tensile strength and stress at break when compared to the neat PC. This outcome could be attributed to the enhanced fibre alignment in the printing direction and the processing measures that were implemented to prevent excessive fibre breakage during the extrusion of the filament. However, to verify this, it is necessary to investigate the length distribution and orientation of fibres within the composite.

The reduction in strain at break suggests that incorporating SGF altered the failure mechanism of the polymer matrix, transitioning it from ductile to brittle. This was corroborated by SEM microscopy analysis of the fracture surface, as depicted in Figure 8. The SEM images also revealed a significant enhancement in inter-layer adhesion as the SGF content increased. This can be ascribed to the increased surface area provided by the fibres, which creates more contact points between adjacent layers and promotes stronger bonding. However, further investigation is needed to understand the underlying mechanism by examining the polymer flow and rheological behaviour at various fibre contents. The SEM images also showed that the neat PC composite undergo a ductile failure due to the localized deformation visible on its fracture surface while brittle behaviour was detected for the composites containing SGF through a relatively smooth and flat fracture surface with little plastic deformation. Moreover, the composites exhibited extensive fibre pullout on the fracture surface, indicating that the bonding strength between the fibres and the matrix was suboptimal. Future research could explore practices to enhance the compatibility between the components of the composite and evaluate the resulting impact on its overall performance.

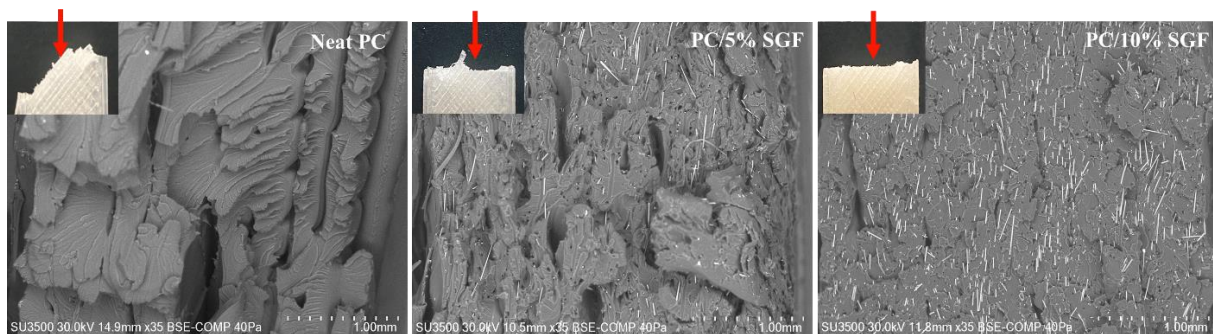


Figure 8. The SEM images showing the fracture surface of 3D-printed PC tensile coupons at different fibre contents, with the red arrow indicating the viewpoint.

#### 4 CONCLUSIONS

The objective of this study was to fabricate glass fibre reinforced PC composite filaments suitable for use in advanced applications of fused filament fabrication (FFF), and to investigate the impact of fibre content on the mechanical properties of both the filaments and 3D-printed polycarbonate composites. In addition, the printability of the filaments was evaluated by considering their stiffness. A design of experiment was utilized to identify the optimal 3D-printing parameters, which were then implemented to print tensile testing specimens. The results showed that the addition of fibres improved the tensile and flexural properties of the filaments. Although elastic modulus of the filaments was not reported due to the limitations of strain measurement methods for filament-shaped samples, a significant increase was observed in the slope of the load-displacement curve. Also, the increased flexural properties of the filaments may suggest reduced flexibility of the SGF-reinforced filaments, potentially impacting their handling and feasibility for printing. The conducted stiffness test is a promising method to evaluate printability of the filaments and the results demonstrated that there exists a stress tolerance threshold for the filaments below which their quality is inadequate for printing process. Moreover, the mechanical analysis of the 3D-printed coupons showed that the increase in fibre content led to an improvement in elastic modulus and tensile strength of the composites as well as a reduction in strain at break. The enhancement in the mechanical performance of the 3D-printed coupons were significant in

comparison to the results reported in the literature for both PC 3D-printed components and injection moulded ones. An increase in inter-layer adhesion and transition from ductile to brittle failure behaviour was observed through SEM microscopy by increase of the SGF content in the composite. These findings are important for the design and optimization of 3D-printed polymer composites, as they suggest that the mechanical properties of the material can be tailored by adjusting the fibre content.

Future research in this area could focus on understanding the flow and rheological behaviour of the material at different fibre contents, as well as exploring the possible characterization methods for FFF filaments that can benefit final printing quality and inter-layer adhesion of 3D-printed composites. Overall, this study highlights the potential of fibre-reinforced polymer composites for 3D printing applications and provides a foundation for further research in this area.

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