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Study on the Effect of Mold Temperature on The Mechanical and Thermal Properties of Recycled Polyphenylene Sulfide

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ABSTRACT

Polyphenylene sulfide (PPS) is a high-performance thermoplastic commonly employed in aerospace components due to its outstanding strength, heat tolerance, and resistance to chemicals. However, as the use of PPS materials continues to grow, so does the volume of resulting waste, raising concerns about long-term sustainability. Therefore, the integration of PPS recycling methods requires systematic investigations. This study examined the effect of mold temperature during hot-press forming process on the mechanical and thermal properties of the recycled PPS. The experiment focused on the behavior of recycled PPS at various temperatures, ranging from 180 °C to 220°C. Mechanical properties of the recycled PPS at the optimized temperature were evaluated through tensile, flexural and impact tests, while thermal properties were evaluated through differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The findings show that raising the mold temperature (Tm) enhances interfacial adhesion between the matrix and the carbon fibers (CF), which also improves the composite's thermal stability. At a Tm of 200 °C, the recycled PPS exhibits its optimal thermal, and mechanical performance, achieving a tensile strength of 549.92 MPa, flexural strength of 821.10 MPa, impact strength of 100.18 J/m, and the highest degree of crystallinity of 25.03%. Overall, this work highlights how mold temperature influenced key properties of recycled PPS and provided useful guidance for processing such materials in aerospace and other engineering fields.

Keywords: Recycled PPS; thermoforming; mold temperature; properties; aerospace; sustainability

INTRODUCTION

Composite materials are highly valued for their exceptional strength-to-weight and stiffness-to-weight ratios, making them increasingly common in sectors such as aerospace, transportation, sports gear, wind energy, and medical devices (Sun et al. 2022). Particularly, advanced composites, especially those with resin matrices, have gained significant traction across various industries. The

shift from traditional metals to these modern composites is now seen as a major trend shaping the future of global manufacturing. Polyphenylene sulfide (PPS), a type of thermoplastic polymer, has emerged to be one of the most used materials in aerospace industry due to its good dimensional stability, tensile strength and modulus, fatigue resistance, flame resistance and resistance to solvents, chemicals, and high temperatures. Despite the excellent performance of PPS, its recyclability has raised concerns among public, especially where aerospace industries are more focused on developing sustainable materials (Barnett et al. 2024). Therefore, the study of various mold temperatures on the properties of recycled PPS is crucial as it defines a new pathway to integrate the recycling method of recycled PPS.

According to research that has been conducted, processing parameters are essential for thermoforming process as it will affect the internal structure or reaction between the polymer chain of the matrix and the reinforcement fiber. For instance, Salehpour et al. (2018) discovered that process parameters such as temperature, pressure and heating time used in a hot-pressing process play an important role in the properties and interfacial characteristics of the nanocomposites. They came into the conclusion that excessive high temperature and pressure can cause both the natural fibers reinforcement and the polymer to degrade, the reinforcement content in the final composite may be reduced, ultimately leading to a decline in material performance. Besides, Kang et al. (2022) explored that the mechanical properties of recycled PET such as modulus, ultimate tensile strength, melting temperature and crystalline temperature were 95-99% preserved, provided optimum mold temperature. Additionally, Samoryadov et al. (2021) examined thermal residual stresses in a unidirectional thermoplastic composite laminate by contrasting heterogeneous and homogeneous material behaviors at both the micro- and macro-scales and further analyzed how temperature gradients contribute to these stresses. Besides thermal effects, the mold itself restricts the composite's ability to deform freely, which significantly affects the development of residual stresses. For flat laminate structures, the mold not only applies pressure but also interacts with the composite within the plane. This interaction arises primarily from the mismatch in coefficients of thermal expansion between the tooling and the composite, a phenomenon commonly described as tool–part interaction (Shao et al. 2022). This relevant research further emphasized the importance of mold temperature in terms of recycling PPS.

Building on recent advancements, this work focuses on evaluating how the mold temperature influences the mechanical and thermal behavior of recycled PPS composites. Components were fabricated from used PCL-based CF/PPS recycled sheets using a hot-pressing method, with mold temperatures varied between 180 °C and 220 °C. Thermal behavior was analyzed through differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Mechanical performance was determined using tensile testing and flexural testing on a universal testing machine (UTM). The study also examines the interconnection between tensile strength, flexural strength, impact energy, DSC and TGA results relate to one another.

METHODOLOGY

MATERIAL

Post-used CF/PPS composites supplied as polycaprolactone (PCL) sheets measuring 300 × 300 mm were utilized in this investigation. The resin volume content (RC, fiber mass fraction (W_f), and key thermal properties, including the glass transition temperature (T_g) and melting temperature (T_m) were characterized, with the results presented in Table 1.

TABLE 1. Details of post-used CF/PPS PCL

Material	Thickness	RC	W_f	T_g	T_m
Post-used 5 hardness (5H) CF/PPS	1.90mm	43%	58%	93°C	282°C

FABRICATION PROCESS

The testing samples were produced using a hot-press system equipped with an infrared oven for preheating. This setup included two heated plates, each fitted with aluminum tooling, and their alignment was maintained using an alignment block. The process cycle programmed into the machine is summarized in Table 2.

TABLE 2. Process parameters

Sub-processes	Parameter	Values
Preheat	Preheat Temperature	320 °C
Hot-press	Mold Temperature	180°C, 200°C and 220°C
	Mold Closing Speed	24mm/s
	Pressing Time	135 s
Cooling	Environment	Natural

Prior to pressing, the tools were heated to target temperatures ranging from 180 °C to 220 °C. The composite sheets were secured in spring-loaded frames,

preheated in the oven, and then moved to the pressing area. Once positioned, the upper tool compressed the heated material for 135 seconds. After pressing, the panels were left to cool naturally to room temperature before demolding. An overall schematic of the procedure is shown in Figure 1.

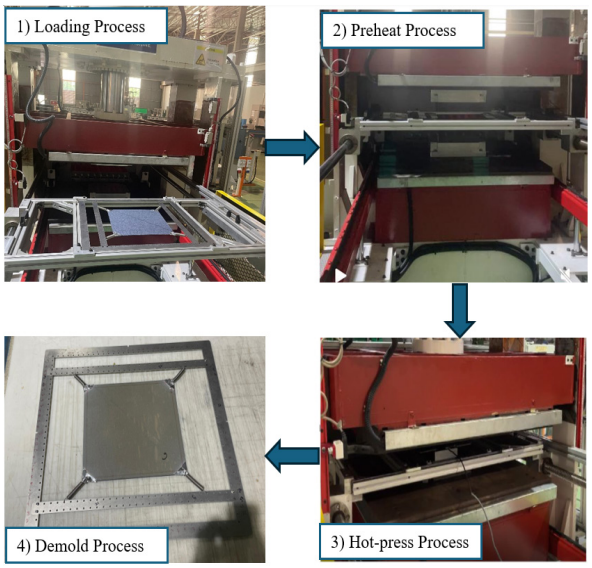


FIGURE 1. Overview of thermoforming process

IDENTIFICATION AND CHARACTERIZATION

The recycled CF/PPS samples produced by the press-forming process were labeled as shown in Table 3. The recorded pressure values for samples Pure 180, T_m180, T_m200, and T_m220 were consistent, falling within a narrow range of 4.10 to 4.50 bar.

TABLE 3. Identification of recycled CF/PPS composite

Mold Temperature (°C)	Samples Identification (ID)	Pressure (bar)
180	Pure180	4.20
180	Tm180	4.10
200	Tm200	4.40
220	Tm220	4.50

MECHANICAL PROPERTIES CHARACTERIZATION

The tensile test followed the ASTM D3039 standard and was performed at a crosshead speed of 2 mm/min using an Instron® 5585 Universal Testing Machine. As shown in Figure 2, the specimen was prepared and cut using a water jet machine to obtain a “dog-bone” shape.



FIGURE 2. Water jet machine operation

Later, the selected specimen measuring 150 mm in length and 22 mm in width as shown in Figure 3, was placed in the test fixture and aligned with the machine’s loading axis. The specimen length was oriented parallel to the fiber direction.

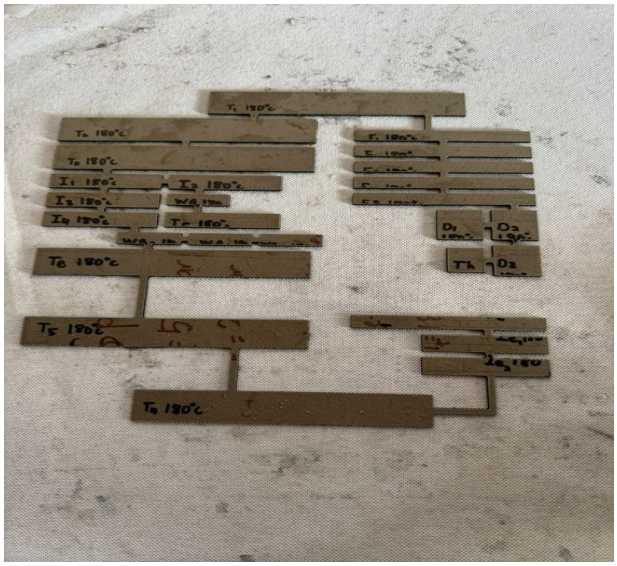


FIGURE 3. Tensile test specimen

In addition, the flexural properties were evaluated following the ASTM D790 standard. Each specimen measured 150 mm × 25.4 mm × 6 mm (length × width × thickness). The bending tests were conducted on the same machine used for the tensile experiments, employing a three-point bending setup with a strain limit of 5.0%. The span-to-thickness ratio was maintained at 16:1. For each mold temperature (T_m), three specimens with the required dimensions were prepared.

Furthermore, Izod impact testing was done to determine the amount of energy required to fracture a specimen when it is struck by a pendulum. The notch which must be machined rather than molded, served as a stress concentrator to ensure the specimen consistently broke at the intended location. According to ASTM D256 and ISO 180:1993 standards, a notch of specified size and geometry was cut into one side of the bar at a defined distance from its end. Standard Izod specimens measured approximately 3.2 × 12.8 × 63.5 mm, with the notch machined along the 3.2-mm face and extending into the 0.50-in. (12.8-mm) width. The notch shape and radius followed the exact specifications given in the ASTM and ISO test methods.

THERMAL PROPERTIES CHARACTERIZATION

The degree of crystallinity (DoC) was measured using a DSC analyzer from TA Instruments. Samples weighing between 7 and 10 mg were placed in pans, ensuring proper thermal contact. Testing was carried out under a dry inert atmosphere at a heating rate of 20 °C/min, beginning at room temperature (RT) and increasing to 320 °C, followed by a 5-minute isothermal hold and a subsequent cooling cycle. The resulting thermal curves were then used to calculate the DoC according to Eq. (1).

$$\theta = \frac{T - T_{cool}}{T_{initial} - T_{cool}} \quad (1)$$

where ΔHm represents the melting enthalpy (J/g) extracted from the DSC thermogram, W_f is the fiber mass fraction obtained from the acid digestion test (0.58), and ΔH100% is the enthalpy value for fully crystalline PPS, taken as 150.4 J/g according to recent literature (Saenz-Castillo et al. 2019).

Moreover, thermogravimetric analysis (TGA) was performed using a Perkin Elmer STA 6000 instrument following the ASTM E1131 standard. Samples were heated under a dry inert atmosphere at a rate of 10 °C/min from 30 °C to 990 °C until the mass stabilized. After reaching the plateau, the atmosphere was changed from inert to reactive (oxygen or air). The remaining ash content was then determined using Eq. (2).

$$\theta = \frac{T - T_{cool}}{T_{initial} - T_{cool}} \quad (2)$$

where W_r is the sample mass recorded at the plateau temperature, and W_i is the initial mass of the specimen.

RESULTS AND DISCUSSION

TENSILE STRENGTH

Tensile strength represents the highest tensile stress a material can withstand before failing. This property is vital for evaluating the structural performance and load-carrying ability of recycled CF/PPS composites. Examining how mold temperature influences tensile strength is important for understanding how the material behaves under various thermal conditions and for refining its use in practical applications.

Tensile strength reflects how well a material can withstand stretching forces without deforming or failing. As shown in Figure 4, the ultimate tensile strength (UTS) of the recycled CF/PPS composites increased with higher tool temperatures. The lowest strength was recorded at Tm180 at 537.11 MPa, while Tm200 reached the highest value at 549.92 MPa. The samples processed at Tm220 showed comparable strength of 540.80 MPa. These results demonstrate that elevated mold temperatures can improve the tensile performance of the composite. The superior strength observed for Tm200 aligns with the study mentioned in the introduction section, which suggests that higher temperature enhances wettability and improves stress transfer within the composite. Figure 4 also highlights Pure 180 (Virgin PPS) exhibits higher tensile strength than that of Tm180. This occurs because when PPS is exposed to high temperatures and shear forces of melt processing for a second time, its polymer chains can undergo thermal and oxidative degradation (Tatsuno et al. 2017). This results in chain scission, meaning the long molecular chains break into shorter ones. Shorter polymer chains cannot entangle as effectively as longer ones, which directly reduces tensile strength. In contrast, virgin PPS retains its full molecular weight and structural integrity, giving it higher strength (Sathees Kumar, 2020).

Furthermore, tensile strength of Tm200 recorded the highest value because it exhibits stronger matrix–fiber adhesion, with PPS coating and bonding firmly to the carbon fibers. In contrast, Tm180 shows relatively low tensile strength among others because of fiber–matrix separation. Similar findings were reported by Zhang et al. (S. Zhang et al. 2013), who noted that higher molding temperature with saturated surface energy corresponds to improved interfacial strength. (Gao & Kim, 2000) also emphasized that cooling rate affects the crystallinity of CF/PEEK composites, which in turn influences mechanical performance. Tatsuno et al. (2018) observed that a slower cooling rate can result in higher ultimate strength. Since cooling rate affects the extent of fiber–matrix bonding, it consequently determines the final mechanical behavior of composite components (Allaoui et al. 2014).

Additionally, temperature fluctuations can influence the quality of the recycled CF–PPS interfacial bond. Interfacial debonding tends to decrease when crystallinity increases, leading to stronger fiber–matrix interactions (Weber & Schlimbach, 2019). At extreme high temperatures, however, differences in thermal expansion between the fibers and the polymer matrix may generate interfacial stresses that can weaken adhesion and reduce tensile strength. The carbon fibers themselves may also experience temperature-dependent changes, potentially losing stiffness or strength at elevated temperatures (Oshima et al. 2023). Nevertheless, Chadwick et al. (2021) reported that CF/PPS laminates formed with heated tooling exhibited significant improvements in interlaminar shear and through-thickness tensile properties as the processing temperature increased. This phenomenon matches the result shown in Figure 4, which suggests that adequate mold temperature at 200 °C will most likely enhance the mechanical properties of recycled CF/PPS.

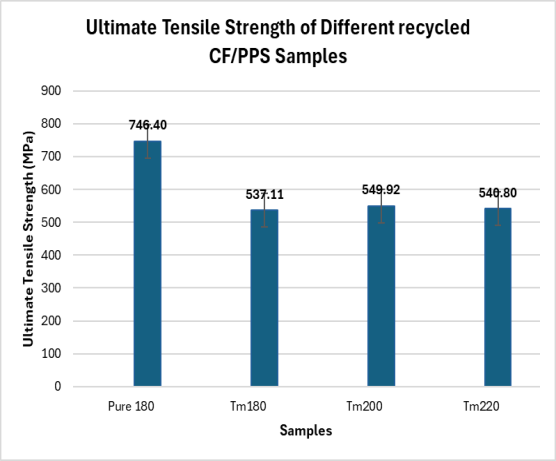


FIGURE 4. Ultimate tensile strength of different recycled CF/PPS samples

FLEXURAL STRENGTH

Flexural strength, also known as bend strength or modulus of rupture, is a material property that describes how much stress a material can withstand before breaking when subjected to bending. Flexural strength is also useful for identifying brittleness or manufacturing defects, since bending tests are sensitive to cracks, voids, fiber alignment issues in composites, and other processing-related imperfections.

Based on the results as shown in Figure 5, Tm200 shows the highest flexural strength of 821.1 MPa among others, followed by Tm220 of 810.86 MPa and Tm180 of 799.18 MPa respectively. When the mold temperature increases, reinforcement fibers act as a barrier to overcome the heat transformation during polymer chain cross-linking. Even when the fibers in the recycled material have degraded, the remaining oriented molecular chains can still contribute to reinforcement. As a result of the fiber content retained in the recycled material, both the flexural strength and modulus show substantial improvement. This notable improvement indicates that the toughness of the recycled material has increased. However, the flexural strength of Tm220 drops after excessive high temperature is applied. This situation occurs when PPS continues to undergo cross-linking process and encounters polymer degradation. Due to PPS is a semicrystalline polymer, higher temperature might prevent crystallization with numerous crosslinking processes (M. Zhang et al. 2023).

Besides, it is notable that Pure 180 (virgin PPS) demonstrates higher flexural strength as compared to Tm180. This is because of the recycling process that degrades both the polymer matrix and the reinforcing fibers. During reprocessing, PPS is exposed to high temperatures and shear forces that cause thermal and oxidative degradation, leading to chain scission and a reduction in molecular weight. Shorter polymer chains result in a weaker, less stiff matrix that cannot resist bending stresses as effectively as virgin material (Yan et al. 2022).

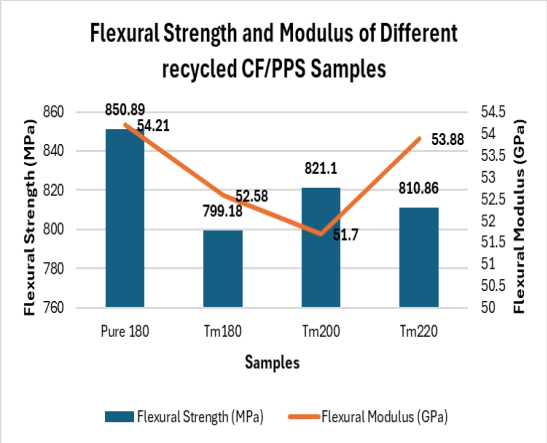


FIGURE 5. Flexural Strength and Modulus of Different recycled CF/PPS Samples

IMPACT STRENGTH

Impact strength is a measure of a material's ability to absorb energy and resist fracture when subjected to sudden or dynamic loading, such as a sharp blow or collision. It indicates how well recycled PPS can withstand impact forces without cracking or breaking. Studying the impact strength of PPS is important because this property directly reflects the material's toughness and reliability in real-world applications (Vidakis et al. 2020).

According to Figure 6, the impact strength of Tm200 marks the highest value of 100.18J/m. Tm 220 and Tm180 marks the consecutive impact strength of 97.05 J/m and 96.50 J/m respectively. The results indicate that the increase in mold temperature leads to a slight improvement in impact strength. For Tm220, a slight decline in impact strength is observed. This minor reduction can be attributed to the formation of interlayer voids within the bulk and internal polymeric structure. Consequently, reduced polymer chain interdiffusion between layers and adjacent filaments during melt deposition causing reduced mobility among the matrix and fiber particles. These are likely caused by the increase in melt viscosity at higher particle loadings, which limits the movement and interaction of the polymer chains (Vidakis et al. 2022).

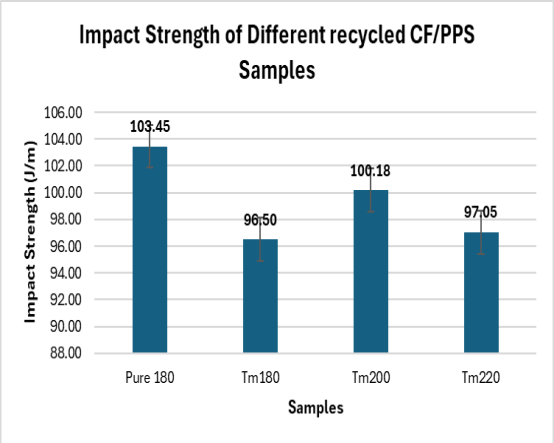


FIGURE 6. Impact strength of different recycled CF/PPS Samples

THERMOGRAVIMETRY ANALYSIS

Thermogravimetric analysis (TGA) measures how a material's mass changes as temperature increases, offering valuable information on thermal stability, decomposition behavior, and overall weight-loss patterns under controlled heating. It is widely applied to evaluate the thermal response of recycled CF/PPS composites, particularly for determining their suitability in high-temperature environments. The TGA curve reveals the composite's degradation characteristics, often showing multiple stages of weight loss that correspond to the breakdown of different constituents in the material system (Atiqah et al. 2018). Changes in temperature during the test influence both the rate and magnitude of decomposition, allowing for a clearer understanding of the composite's thermal performance.

In TGA testing, the percentage of residue remaining after thermal decomposition is measured to represent the thermally stable or non-volatile portion of the recycled CF/PPS composite. As shown in Figure 7, the highest residue content was observed at the highest mold temperature. The lowest value of residue content was recorded at Tm200 at 72.39%, whereas Tm220 reached the highest level at 73.95%. Tm180 and Pure 180 produced intermediate values of 72.54% and 73.09%, respectively. These findings suggest that raising the mold temperature generally enhances the thermal stability of the composite material.

The results are consistent with the simulation work of Yu et al. (2019), who reported that chemical reactions at the co-cured interface significantly contribute to improving composite performance. They also noted that interface strength increases with the number of chemical interactions occurring during contact. Thermal stability factors, such as heat transfer through the material, load carrying ability at elevated temperatures, high temperature dimensional stability, and general thermal behaviour, are equally important for structural applications. Sathees Kumar (2020) further emphasized that strong fiber–matrix adhesion is critical for effective resistance under loading. In addition, the thermal characteristics, including stability, showed a direct relationship with the tensile properties of the composite.

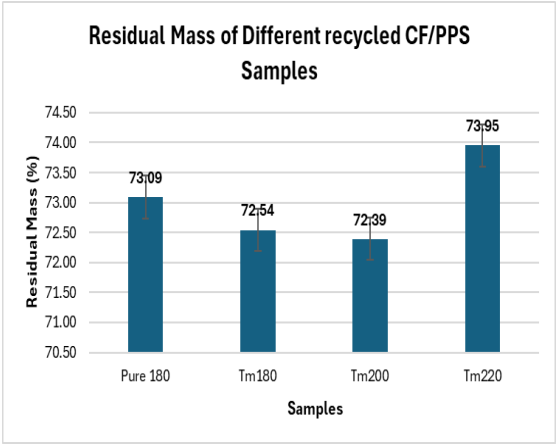


FIGURE 7. Residual mass of different recycled CF/PPS Samples

DEGREE OF CRYSTALLINITY

Temperature plays a crucial role in the melting and crystallization behavior of CF/PPS composites. As the temperature increases, the polymer chains in the PPS matrix experience increased molecular mobility, allowing for enhanced chain rearrangement and crystallization (J. Zhang et al. 2023). Higher temperatures promote the melting of crystalline regions, leading to a decrease in the DoC. The mold temperature during the processing of CF/PPS composites can significantly influence the DoC. Different temperature profiles can result in variations in

cooling rates, which affect the rate of crystallization and subsequent DoC (Sacchetti et al. 2018).

From the obtained results, the DoC represents the extent to which a material's structure consists of ordered, crystalline regions. The results of DoC in Figure 8 exhibited minimal variations among the different components. Tm200 had the highest value of 25.03%, while Tm180 had the lowest value of 21.25% with a range value of 3.78%. Overall, the DoC remains relatively consistent across the different mold temperatures in this study. The CF/PPS composite component likely has reached a saturation point in terms of crystalline regions, and further temperature increase has minimal impact on the crystalline structure. G. Sun et al. (2021) studied the effect of different processing temperatures of composite and reported that higher processing temperatures enhanced the crystallinity of the polypropylene (PP) matrix and improved the interfacial adhesion between the fibre and resin, which consequently increased the tensile strength. These findings correlate with the tensile strength and TGA obtained in this study. The DoC in almost every component was directly proportional to the tensile strength proving that strong correlation between DoC and mechanical properties as well as physical properties of the CF/PPS composite component. The CF/PPS composites have a crystallization window of 87-270°C (Yu et al. 2019). When the mold temperature in the range of the crystallization window, the CF/PPS composite undergoes the isothermal crystallization, which could increase crystallinity even further. The mechanical characteristics improved as the mold temperature increased due to improved matrix self-adhesion, whereas the fracture toughness declined due to lower matrix ductility (Zhao et al. 2023).

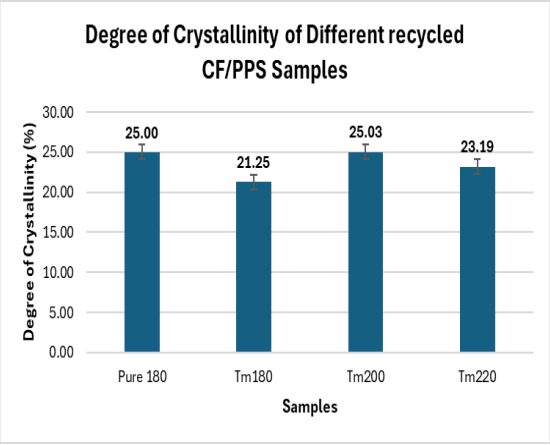


FIGURE 8. Degree of crystallinity of different recycled CF/PPS Samples

CONCLUSION

This study demonstrated that mold temperature plays a significant role in defining the mechanical and thermal performance of recycled CF/PPS composites. Among the tested conditions, 200 °C consistently provided the most favorable balance of properties, yielding the highest tensile strength (549.92 MPa), flexural strength (821.10 MPa), impact strength (100.18 J/m), and the highest degree of crystallinity (25.03%). Although higher temperatures improved thermal stability, excessive heating at 220 °C led to slight reductions in tensile, flexural, and impact performance due to potential void formation and polymer degradation. The findings confirm that an optimized mold temperature of 200 °C enhances fiber–matrix adhesion, crystallinity, and overall structural integrity, making it the most effective processing condition for recycled PPS composites. This optimization improves the material's suitability for high-performance applications while supporting sustainable reuse of PPS-based waste.

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DECLARATION OF COMPETING INTEREST

None.

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