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Thermal Properties of Wood Dust Fibre and Recycled Polypropylene (r-WoPPc) for Development of Thermoplastic Composites Filaments of Fused Deposition Modeling

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ABSTRACT

The thermal behaviour of filament materials is one of the most important characteristics to study to produce quality filament since the FDM process strongly relies on heating operations such as mixing and printing. To develop composite filament FDM using recycled polypropylene and wood dust fibre (r-WoPPc), the thermal properties of these recycled materials were investigated. Wood dust and recycled PP are by-products of the furniture and plastics industries, and their thermal characteristics may change during manufacturing. Furthermore, wood dust is typically subjected to several cleaning and treatment processes to have a cleaner and impurity-free dust as well as better adhesion with the recycled PP matrix, which both processes may affect its thermal properties. Therefore, TGA and DSC analyses are conducted to ensure its thermal properties before developing the filament. Untreated, NaOH and silane treated wood dust and recycled PP was subjected to TGA and DSC analyses. Wood dust treated with silane had the highest combustion resistance at 372°C compared to untreated wood dust at 360°C and NaOH treated at 320°C. Meanwhile, DSC analysis of recycled PP indicated that it melted at 167°C, which was used to establish the filament extrusion and printing temperatures. In a conclusion, these studies provided a point of reference for determining filament extrusion and printing temperatures.

1. Introduction

In recent years, it has become vital in contemporary life to use goods that are more environmentally friendly, cost-effective, and healthier than others. Wood dust fibre is made up of microscopic particles found in furniture, pulp and paper, and other industries. It may develop piles and burn, causing environmental damage. Natural fibres are biodegradable and may be produced in

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large quantities without displacing other crops [1]. Using bio-composites made from waste polymers and natural fibres, such as recovered polypropylene (PP) and wood dust fibre, for 3D printing. In terms of thermal expansion and contraction of wood dust fibre, these dimensional fluctuations are minor compared to shrinkages and swellings caused by moisture variations. Temperature-related expansion and contraction are usually minor and of little practical relevance. Wood is thermally inefficient (and has a high heat-insulating capacity). As a consequence, light, dry woods are superior insulators to heavy, damp woods [2,3]. With many academics publishing their findings and reviews, interest in natural fibre composite FDM has significantly increased [4,5]. The temperature and fluid flow characteristics of polymers and fibre materials are necessary to develop bespoke composites. Because the process depends on the extrusion of the heated feedstock filament, the filament's thermal and rheological qualities are crucial [6]. The very hydrophilic characteristic of lignocellulose materials, on the other hand, makes them incompatible with thermoplastics, as with wood-plastic composites (WPC) [7,8]. Thermal testing requires fibre treatment owing to temperature differences in material chemical characteristics. Treatment of natural fibres is required to improve surface properties. Poor interface area owing to polarity interfaces is a major issue for natural fibre and matrix compatibility. A robust interface with exceptional strength and stiffness may be produced despite its fragility and quick fracture propagation across the matrix and fibre [5]. For example, alkaline treatment fibres are often treated with NaOH because it is simple, affordable, and effective in enhancing filler-matrix adhesion [9]. NaOH affects the surface of the fibre by mechanically removing it, increasing surface roughness and hence matrix coverage. In addition, chemical treatment, namely silane treatment as a coupling agent to increase the wettability of natural fibres by polymer matrix and promote interfacial bonding, is another treatment [10,11]. To assess the characteristics of wood dust fibre composites, a thermal test is necessary. Materials such as wood dust fibre polymer matrix recycled polypropylene (PP) will be examined along with their thermal properties such as TGA and DSC.

This study investigated the thermal properties of wood dust fibre treated with 6% NaOH and silane. Thermal properties of recycled polypropylene are also reported in this study. This study was conducted because of a lack of thermal data for manufacturing FDM filaments from recycled Polypropylene and wood dust. Hence this study aims to find the thermal properties of wood dust fibre and recycled polypropylene to develop filament recycle wood dust PP composite (r-WoPPc).

2. Methodology

2.1 Material and Methods

2.1.1 Material

Wood waste from small and medium-sized industries (SME) furniture manufacturing in Alor Gajah, Melaka; consists of pine grove and tongue wood. The wood waste is turned into a powder with a portable grinder and then filtered using an industrial siever at the FTKMP composite lab at UTeM. The seiver mesh size utilized ranges from 300-micron meter through 200-micron meter to 125-micron meter, then the outcome of wood powder is a 125-micron meter mesh that is clean of harmful elements as Figure 1(a), portable grinder (Figure 1(b)), industrial siever (Figure 1(c)) [12].

2.1.2 Polymer matrix

Recycled PP polymer was obtained in the form of small pallets measuring 2 mm from a plastic manufacturer at Ayer Keroh, Melaka. Figure 1(d) shows the transparent milky white recycled PP employed in this research.

2.1.3 Alkaline treatment of wood dust fibre

This treatment will include the use of wood dust fibre in combination with a natrium hydroxide (NaOH) solution. The NaOH solution that has been prepared in the beaker will be used to immerse the wood dust fibre filler in the beaker. After soaking for 2 hours at room temperature in the NaOH solution, after complete soaking time, the wood dust fibre was washed thoroughly with distilled water and dried in an oven at a temperature of 60°C for 24h [13]. The mixing process has been shown in Figure 1(f). The preparation of solution NaOH will be based on the usage ratio that was required to make it, which was 6% sodium hydroxide and 94% distilled water, both of which will contribute to a 100% concentration of the solution [1,14].

2.1.4 Chemical treatment of wood dust fibre

One of the chemical treatments in combination with other chemicals. 2% silane was soluble in this procedure, and the chemical compound APS (aminopropyltriethoxy silane) was dissolved in a solution comprising 70% methanol (CH3OH) and 30% distilled water, respectively. Following that, the solution was agitated for another 30 minutes. Then, the wood dust fibre was soaked in silane solution for 3 hours and dried in an oven at 60°C for 72hours to remove all the moisture content of the fibres [15,16]. Figure 1(g) are shown the silane treatment conducted and Figure 1(h) material in an oven.

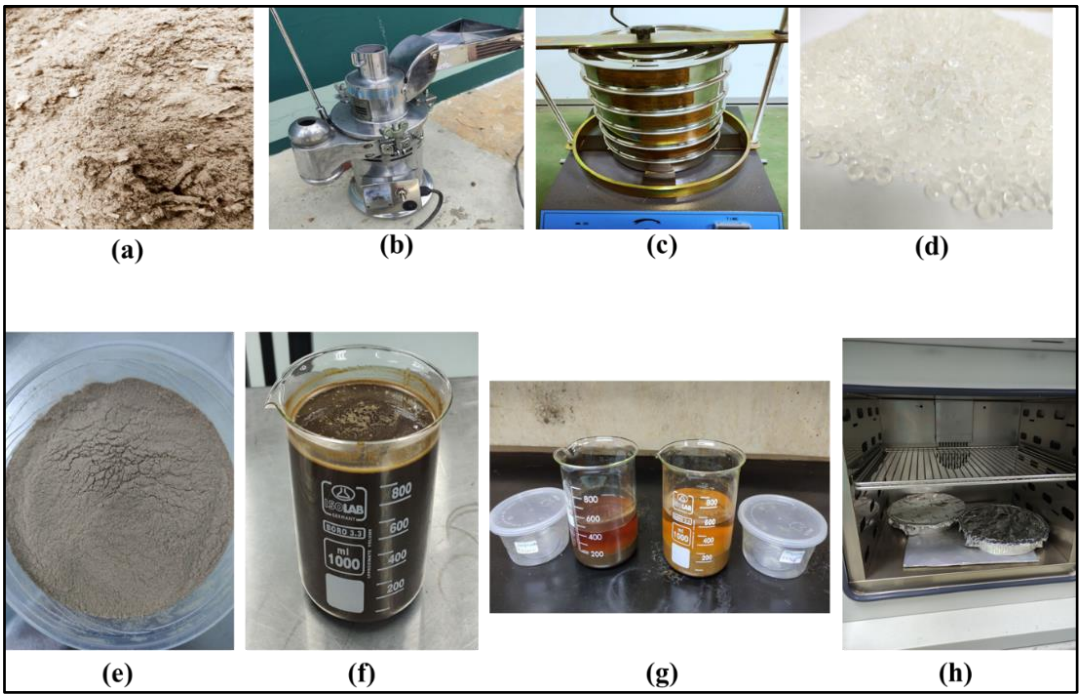


Fig. 1. (a) Raw wood dust fibre from the workshop (b) grinder machine for grind into dust (c) Industrial siever for getting result 125 µm (d) recycled PP pallet form (e) the result of 125 µm wood dust fibre (f) NaOH treatment represent (g) NaOH-Silane and Silane Treatment (h) material after treatment dry on the oven

2.2 Thermal Test on a Sample

2.2.1 Thermogravimetric analysis

A thermogravimetric analysis (TGA) analyser was used to investigate the thermal stability of wood dust fibre concerning weight loss due to an increase in temperature. TGA was carried out with the help of machines from TA instruments and filament samples according to the ASTM D3850 standard. Temperature rate between 0°C and 800°C with a heating rate of 20 °C/min. TGA was obtained using Mettler-Toledo brand Thermogravimetric Analyzer TGA 2 - Thermogravimetric Analyzer with small furnace (SF) specification; Temperature ranges up to 1100 °C, heating rate 0.02 to 250K/min and crucible volume up to 100µL. The sample will perform on 4 samples which are recycled PP, treated NaOH, treated silane, and untreated wood dust.

2.2.2 Differential Scanning Calorimetry (DSC)

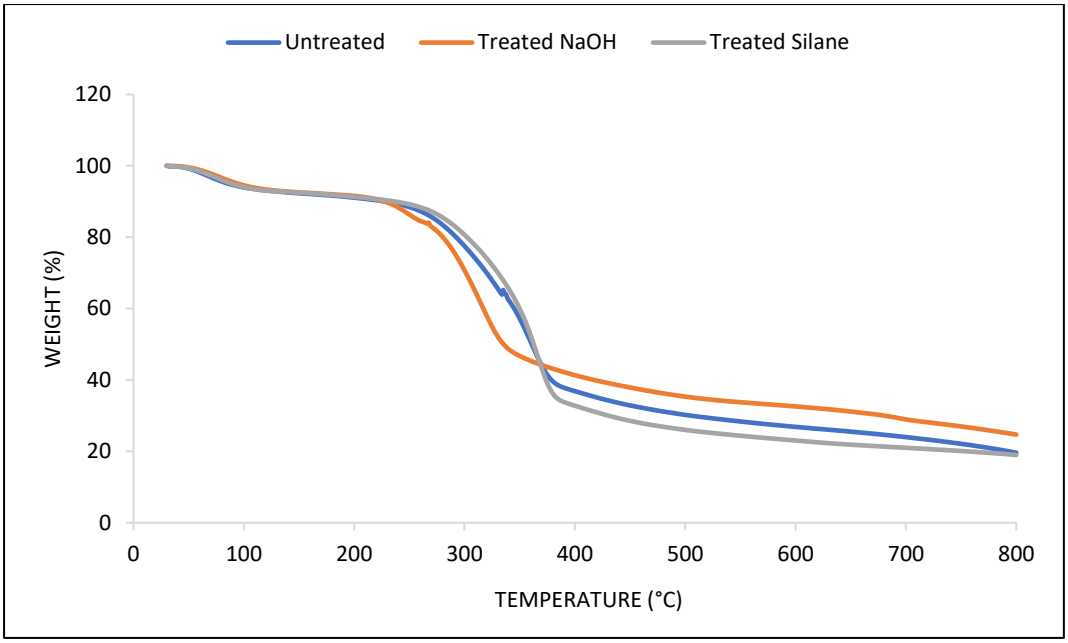
The DSC analysis will be carried out to obtain information about the heat transfer that takes place in the sample. Transitions include residual reactivity, solvent evaporation, melting, crystallization, crystal transition temperature, and glass transition temperature. Differential Scanning Calorimetry (DSC) analysis was performed with a heating rate of 20°C /min, from 30 to 800°C. Differential Scanning Calorimetry (DSC) was obtained using DSC Q20 V24.11 Build 124. The sample will be evaluated using four different samples, such as pure recycled PP, treated NaOH, treated silane, and untreated wood dust.

3. Results

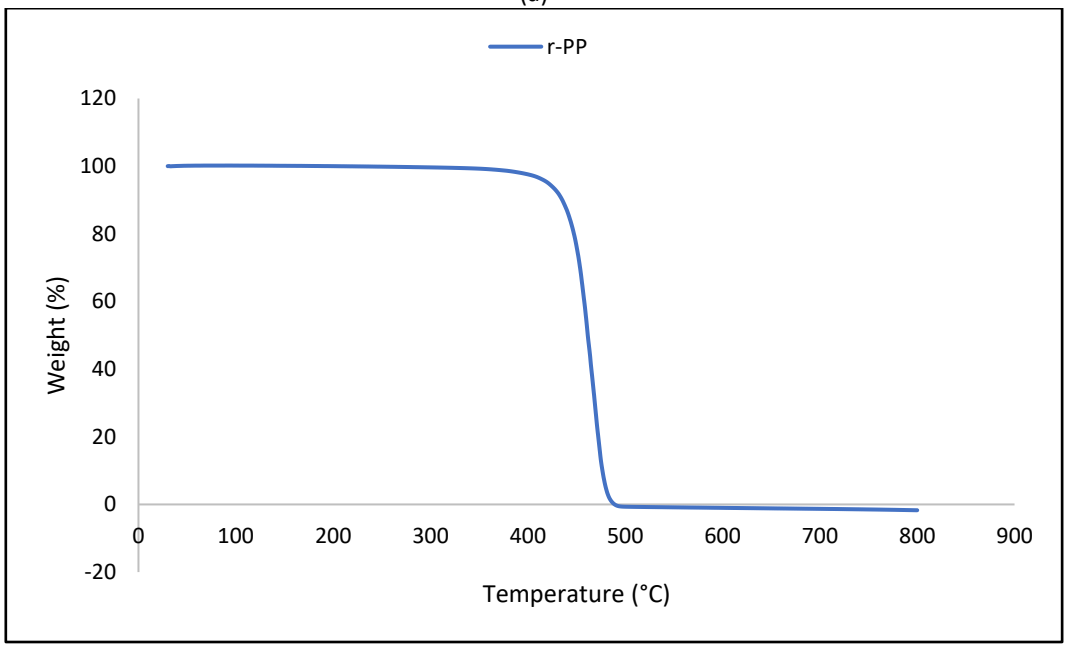
3.1 Thermogravimetric analysis (TGA)

The TGA analysis of the wood dust with the treatment of NaOH, silane and untreated is presented in Figure 2(a) and the analysis of recycled PP is presented in Figure 2(b). Table 1 summarizes the TGA results of starting degradation, decomposition temperature, residue temperature and final weight after decomposition of all the studied materials. A nitrogen atmosphere was employed in this TGA study to analyse the thermal stability, thermal degradation, and mass of wood dust fibre and recycled PP. It is essential to analyse the degradation behaviour of recycled PP and wood dust at a different temperature to ensure that both materials are suitable to be used for FDM filament [17].

Referring to Figure 2(a), it can be observed that the degradation begins at 71.74°C for untreated wood dust, 75.66°C for treated silane and 79.11°C for wood dust treated with NaOH. The degradation happened because of the evaporation of moisture content in the wood fibre. The wood dust has a mass loss of not more than 10% during this degradation phase. This finding is similar to Krishna and Kanny [10]. Krishna and Kanny [10] have identified at an earlier stage of degradation fibres begin to lose moisture as their moisture evaporates during this phase. The weight loss of fibre at this stage is presently less than 9% of the total weight. Degradation of wood dust treated with NaOH has a higher temperature compared to untreated and silane treated because NaOH treatment has removed chemical content such as hemicellulose and lignin during alkaline treatment.



(a)



(b)

Fig. 2. TGA graph of (a) NaOH treated, silane treated, and untreated wood dust and (b) Recycle PP

Table 1
Result of the TGA test

Materials	Degradation temperature (°C)	Decomposition temperature (°C)	Residue Temperature(°C)	Final weight after decomposition (%)
Treated NaOH	79.11	320.22	694.67	24.231
Treated Silane	75.66	373.15	564.98	18.981
Untreated	71.74	364.06	799.77	19.605
Recycle PP	355.16	475.93	499.52	0.038

Meanwhile, the decomposition of wood dust begins at higher temperatures 250-450°C, as hemicellulose, cellulose, pectin, and lignin begin to decompose which is comparable to Krishna and Kanny [10] also reported the chemical composition of the fibre, such as cellulose, hemicellulose, pectin, and lignin, is progressively destroyed at temperatures ranging from 300°C to 500°C. Wood dust treated with NaOH decomposed at a lower temperature of 320.22°C, followed by untreated wood dust at 364.06°C. This finding is like Kumar *et al.,* [13], in which fibre treated with NaOH was lower than untreated fibre. This is because the lignin and hemicelluloses were removed during the alkali treatment. Wood dust treated with silane decomposed at the highest temperature of 373.15°C, which is comparable to Jamadi *et al.,* (2021) [17], which reported that fibre treated with silane decomposed at temperatures ranging from 300°C to 400 °C. This may be the cause since raw wood dust fibre is formed of smooth fibre surrounded by hemicellulose and lignin, although in minor quantities. Amorphous structures of hemicellulose and lignin, which are softer than wood dust fibre, serve as the glue between the fibres. As a result, these impurities initiate more active sites and increase thermal decomposition early [14]. Wood dust treated with silane lost the most weight that is 35% during the decomposition phase at 250-450°C if compared to wood dust treated with NaOH and untreated which only lost 30%.

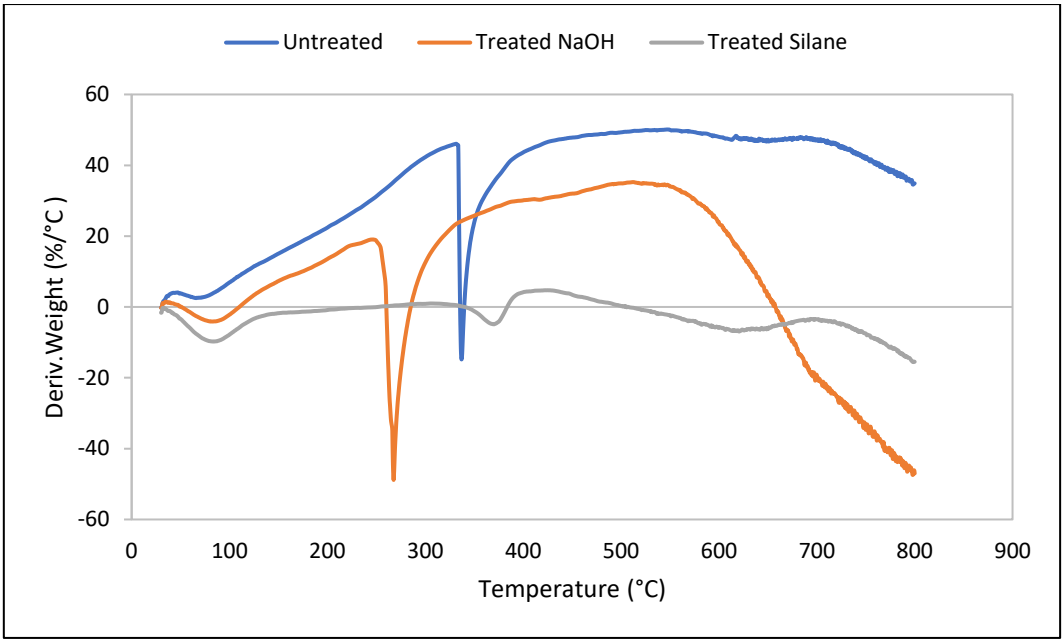
Consequently, the residual phase of wood dust decomposed further at temperatures ranging from 450°C to 800°C, which is the remaining weight that might be assigned to char or other products of thermal decomposition [18], with untreated wood dust decomposing at a maximum temperature of 799.77°C. The treated NaOH decomposed at 694.67°C, while the treated silane decomposed at 564.98°C. When treated silane wood dust decomposed at the maximum temperature, it produced a significant amount of residue; less than 19% to treated NaOH and untreated wood dust. This finding is comparable to that of Nadlene *et al.,* [19], which reported that char residue is produced when cellulose is decomposed at a high temperature and that the lowest residual char residue weight indicates better thermal stability.

In terms of TGA analysis for recycled PP, its degradation phase happened at 10–300°C. In this phase, recycled PP starts to lose moisture as it is evaporated and the weight loss in this stage is currently below 10% [20]. At temperatures of 300°C- 500°C recycled PP begins decomposing at 475.93°C meanwhile at the residue temperature between 450-600°C recycled PP decomposed at 500°C which produced 0.038%.

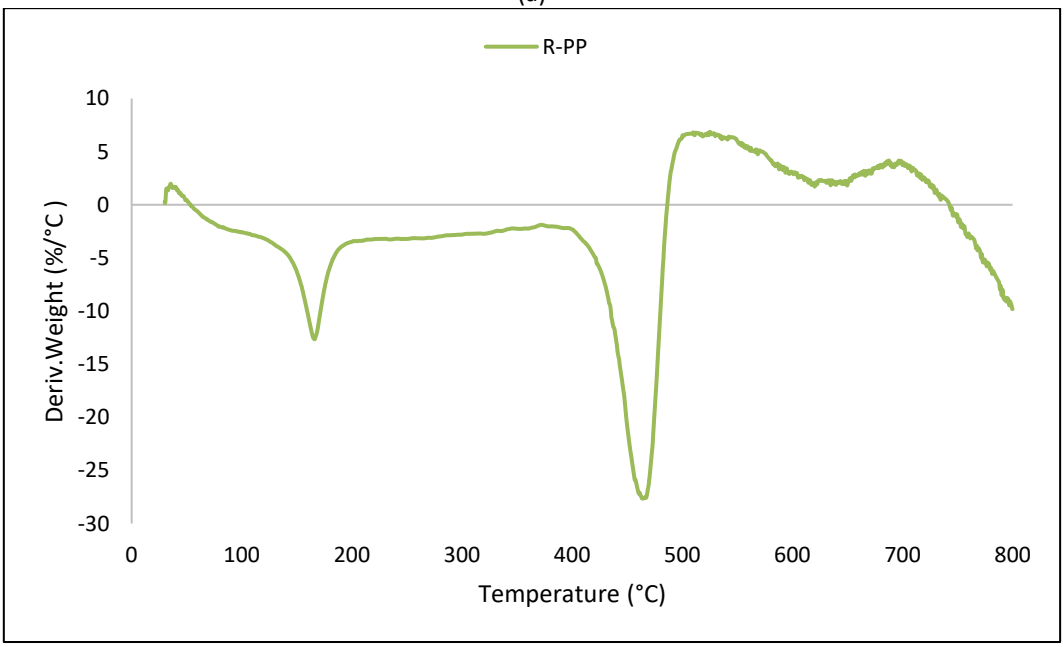
3.2 Differential Scanning Calorimetry (DSC)

Figure 3(a) shows the DSC analysis of wood dust following treatment with NaOH, silane, and untreated wood dust, while Figure 3(b) shows recycled PP. Table 2 shows the glass transition (T_g) and melting temperature (T_m) data from DSC treated NaOH, treated silane, untreated wood dust, and recycled PP. The glass transition temperature and oxidative stability will be investigated to determine whether amorphous compounds can perform glass transitions (T_g).

The first peak in Figure 3(a) represents wood dust fibre, which would be recognized to have an exothermic temperature of 85.12°C at untreated more than higher compared to treated silane and treated NaOH have a lower temperature at 82.09°C because wood dust was thermally decomposed at approximately 250–600°C according to Zheng *et al.,* [21]. The melting temperature for 3 tests of wood dust fibre is 377.89°C, which is higher for untreated wood dust compared to treated silane and treated NaOH, which are 336.25°C and 264.98°C, respectively, because untreated wood dust did not provide any treatment, leaving him with a variety of substances including cellulose, hemicellulose, and others that had to be burned, raising the melting point and reducing crystallization enthalpy and crystallinity [22].



(a)



(b)

Fig. 3. DSC graph of (a) NaOH treated, silane treated, and untreated wood dust and (b) Recycle PP

In terms of DSC for recycled PP as shown in Figure 3(b), the first peak, which Glass transition temperature of 167°C, corresponds to the melting temperature of the recycled polypropylene The second peak point, which is known as exothermic, occurred at a temperature of 472°C., which corresponds to the greatest temperature that can be reached by regenerated polypropylene before it is burnt. With this Figure 3(b), you will be able to identify whether the extrusion operation was successful if the extruder temperature was set between 167°C and 472°C, which is required to use a lot of temperature-based mixtures such as recycling PP, which is used more than 90% in forming composite filaments. The DSC results referring to wood dust fibre and recycled PP are used as an indicator for the maximum peak temperature; however, they also depend on the setup and condition of the machine mechanism, in addition to other factors such as whether the mixture created has

good bonding or not and machine configuration [23]. Furthermore, if the DSC test findings are validated, the temperature range of 160°C to 210°C may be used to create an acceptable composite filament during the extrusion process.

Table 2
Result of DSC test

Materials	Glass transition temperature, T_g (°C)	Melting temperature, T_m (°C)
Treated NaOH	82.09	264.98
Treated Silane	84.77	336.25
Untreated	85.12	377.89
Recycle PP	167.77	472.29

4. Conclusions

Wood dust fibres and recycled PP have great potential for use in the development of filament for Fused Deposition Modelling (FDM). FDM contains a range of heating processes, such as extrusion and printing, which necessitates an understanding of the thermal properties of its raw materials. Developing FDM filament from by-product materials such as wood dust and recycled PP promotes the growth of its applications, which include furniture, household appliances, toys, and many more. TGA and DSC studies aid in determining the heating parameters used for the fabrication of filament from wood dust and recycled PP composite (r-WoPPC). TGA wood dust fibre exhibited that wood dust treated with silane had better thermal resistance than treated NaOH and untreated wood dust, with a higher degradation temperature of 372°C. While DSC tests indicated that the melting temperature of recycled PP is 167°C Both TGA and DSC analyses are consistent with previous research that established the heating range for FDM extrusion and printing between 160°C to 210°C.

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