

Rheological Test of Flowability and Diffusion Behavior of Carbon Fibre Reinforced Polyamide

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Received 28 November 2021, Received in revised form 27 June 2022

Accepted 30 July 2022, Available online 30 January 2023

ABSTRACT

Various materials have been produced to be used as feeder material in 3D printing application to obtain the level of mechanical properties and physical properties of a product. Before to its usage as a 3D printing feed material, polyamide-reinforced carbon fibre composites were investigated for flowability and diffusion behaviour. Using a heated nozzle to transform polymer filament into a semi-liquid that is extruded to create a structure layer-by-layer, the primary issue to prevent is delamination. For the success of this study, there are 2 main methods, namely to study the physical properties of carbon fibre reinforced polyamide composites against the composition of 20 wt.% carbon fibre and to study the temperature and rheological load on the rheological properties. Rheological test analysis found that the material flowability of 20 wt.% CF/PA at temperature parameters 210 °C, 230 °C and 250 °C against rheological loads (40, 60, 80) N recorded a range of viscosity values between 48.80 Pa.s to 97.88 Pa.s and shear rate value range between 19700 s⁻¹ to 20270 s⁻¹. Parameter optimization analysis using Taguchi method found that the largest factor contributing to the viscosity of CF/PA composite feed material was the addition of load applied. Moreover, the microstructural results of CF/PA composites show that smoother surfaces and good polymer structural bonding occur at an extrusion temperature of 250 °C. As a result, the rheology-derived flow rate may be used to tackle the problem of delamination and layer separation in 3D printing.

Keywords: Carbon fiber; polyamide; viscosity; shear rate; temperature; load

INTRODUCTION

3D printing is the technology of additive manufacturing (AM), which continues to grow allowing printing and complex lightweight structures, which can hardly be achieved by other manufacturing methods (Wickramasinghe et al. 2020). With the discovery of a variety of technologies AM, this method has been developed to the medical, aerospace, automotive, food and engineering because of many advantages such as the production design complexity, cost effectiveness, saving time and scored (Calignano et al. 2017). Fused deposition modeling (FDM) uses a heated nozzle to convert a polymer filament into a semi-liquid form that is extruded to form a structure through layer-by-layer deposition (S. Ding et al. 2019). FDM is one of the most widely used and rapidly growing 3D printing methods compared to other additive manufacturing processes (Turner et al. 2014).

The AM 3D printing methodology is essentially an extrusion process on a much smaller scale, and the rheological features are useful in understanding the behavior of materials during AM processing (Hemmati et al. 2008). According to previous study by using a polyamide as a material exhibit a strong shear thinning behavior when melts with increasing molar mass demonstrate a strong viscosity

(Seo & Seo 2018). In addition to melting rheology, several other factors such as filament bending, local friction during filament feeding and nozzle clogging can be considered for a complete assessment of the print ability of the material (Nabipour & Akhouni 2021). Previous research has found that the most common failure in 3D printing is delamination, which is caused by inconsistencies in interfacial bonding between the adjacent layers during printing (Polyzos et al. 2021). The compatibility of the matrix phase with the reinforcing phase, as well as the surface conditions of the reinforcing phase, are the most important factors in interfacial bonding in 3D printing (Latif et al. 2019). Thus, this paper aim to measure the printability performance of the mixtures on the rheology to prevent delamination occurs in 3D printing.

Filament curvature and local friction can limit the maximum force that can be applied to the filament, thus indirectly affecting the printing ability of the material (Go et al. 2017). Nozzle blockage can occur due to filament burning at the nozzle or blockage at the nozzle outlet. In the case of filled filaments, the presence of filler can cause nozzle blockage, and this is a physical limitation that depends on the size and fraction of the filler volume relative to the nozzle exit diameter (Go et al. 2017). It has been found that

printing speed has a direct effect on the dimensions of the extruded filaments (Geng et al. 2019). This can affect the quality of the printed part and thus impose printing speed as a limitation when evaluating the material to be printed. Although it is acknowledged that various factors can cause 3D printing failure.

The implementation of composite materials in engineering structures has led to the manufacture of lightweight components such as polymer-reinforced carbon fibers (Friedrich & Almajid 2013). However, many disadvantages are shown by composite materials related to low electrical conductivity (Gaztelumendi et al. 2017). The electrical function of composites such as bipolar plates can be improved by incorporating conductive polymer composite (CPC) materials, such as carbon fibers (CF) and carbon nanotubes (CNTs) into ordinary composite manufacturing processes (Radzuan et al. 2019). Adding this conductive filler into the polymer matrix produces a conductive polymer composite with an increased electrical conductivity of 90 % (Mohd Radzuan et al. 2017). The electrical conductivity of CF is considered to be 625 S/cm. Also the CF length, diameter and distance of the electron tunnel were taken to be 5000 μm , 7.5 μm and 0.01 μm , respectively. While the volume fraction for CNT is 0.2 % and the electrical conductivity of CNT is 100 S/cm. The length and diameter of the CNTs were 5 μm and 20 nm, respectively (Haghgoo et al. 2019).

With high demand for practical applications, polymer matrix composites have become sophisticated in the design and development of materials for 3D printing, which can improve polymer properties by combining matrices and reinforcement to achieve composite systems with more useful structural or functional properties that cannot be achieved by stand-alone polymers (Blok et al. 2018). In recent years, the development of polymer composite materials compatible with existing 3D printers has certainly created more opportunities for PA in FDM applications, especially in the development of PA-based composites. According to (Zhang et al. 2020) there are many achievements in developing new 3D printed FDM polyamide-based composites with improved performance (X. Wang et al. 2017).

Carbon fiber (CF) is a commonly used reinforcement to improve the mechanical properties and electrical conductivity of polymer matrix materials. FDM is one of the widespread 3D printing technologies for producing fiber-reinforced polymer composites. Meanwhile in FDM process, making filament of polymer composite is accomplished by mixing material (matrix and reinforcement) before extrusion process (Calignano et al. 2020). According to (Zhang et al. 2020) research in extrusion processing should be carried out to ensure homogeneous fiber dispersion in the polymer matrix. In fiber-reinforced polymer composites, the fiber orientation and the vacancy fraction of the composite play an important role in determining the final properties

of the composite. However, in practical FDM technology, each layer is formed through filament deposition and the direction or arrangement of filament deposition (and therefore reinforcement, if any) changes during contour formation and filling. Thus, the fibers can be oriented differently inside the printed part depending on the position of the component on the building platform, which can provide isotropic mechanical behavior for FDM polymer/fiber made composite products.

METHODOLOGY

Firstly, column chromatography grade polyamide (PA) thermoplastic polymer matrix from Sigma-Aldrich company while carbon fiber (CF) powder grade CFP-7-50 from Shenzhen Yataida High-Tech company was used in this study. PA matrix is given in the form of a powder of size 50 to 160 μm . Polyamide matrix and carbon fiber raw materials are mixed with a filler load composition of 20 wt% carbon fiber. The mixture materials between PA and carbon fiber was then mixed using a mechanical mixer. This mixing process will be started with 80 wt.% polyamide powder first followed by a composition of 20 wt.% carbon fiber. The mixture composition is then placed into the mixing chamber of a mechanical mixer machine with a rotational speed of 1500 rpm for 30 minutes at room temperature. CF/PA feed materials must first be tested for their rheological behavior. Rheology test was important before going through the fused deposition modeling (FDM) process to study material deformation and material flow due to external forces. After rheology test was done, result of shear stress τ (Pa), flow rate Q (cm^3/s), shear rate $\dot{\gamma}$ (s^{-1}), viscosity η (Pa.s), and melting flow rate (MFR) can be read.

Rheological tests were performed using a Shimadzu CFT-500D model capillary rheometer machine with a cylinder length of 10.0 mm and an orifice with a diameter of 1.0 mm. Previous studies stated CF/PA for Fused Deposited Modelling (FDM) with a 260°C nozzle temperature found that it has a significant impact on mechanical characteristics (de Toro et al. 2020). Thus, three level of rheological test temperature was selected lower than the printing temperature within the range of temperature 210, 230, 250 °C. with a rheological load at (40, 60, 80) N. Besides that, the process parameters were optimized using Taguchi experimental method using two different parameter levels at temperature (°C) and rheological load (N) as in Table 1. Optimization parameter using Taguchi method was used through Minitab 19 software. The orthogonal arrangement of L₉ Taguchi was used to determine the shear rate and viscosity of CF/PA composites in each composition through rheological tests at three different rheological temperatures and loads. Table 2 is a conoth of the orthogonal arrangement of L₉ to be applied in the Minitab software.

TABLE 1. Parameters for DOE

Parameter	Levels		
	1	2	3
A Temperature (°C)	210	230	250
B Load (N)	40	60	80

TABLE 2. Orthogonal arrangement L₉

No. of exp. (L ₉)	Parameter	
	Temperature (°C)	Load (N)
1	210	40
2	210	60
3	210	80
4	230	40
5	230	60
6	230	80
7	250	40
8	250	60
9	250	80

In addition, the scanning electron microscope or SEM analysis of the Hitachi S-3400N model, is capable of providing two-dimensional images at high resolution that are useful for evaluating a wide range of materials for surface cracking, defects, contamination or corrosion. In addition, SEM was performed to evaluate the morphological variation of the micro surface of the collected powders in different degrees of reduction. Using 20x and 3000x double magnification with an acceleration voltage of 10.0 kV is able to show the surface of almost all samples clearly. Thermogravimetric analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. This experiment was performed at Quantum Skynet Solutions. Differential calorimeter scanning (DSC) is a thermoanalytic technique in which the difference in the amount of heat required to increase the temperature of a sample and a reference is measured as a function of temperature. This experiment was performed at Quantum Skynet Solutions.

RESULT AND DISCUSSION

RESULTS OF FLOWABILITY PROPERTIES OF FEED MATERIALS

Table 3 records the results on the effect of different temperature and load influences on the flow behavior of CF/PA composite feed material with a load of 20 wt.% filler. Previous studies has reported that the flow behaviour index's lowest value exhibits a sudden decrease in viscosity (Aslam et al. 2016). Based on the observations that have been conducted, the flow behavior of the CF/PA feed material recorded with increasing rheological load will increase the viscosity value and the shear rate value. With additional of the test temperature will reduce the viscosity of the CF/

PA composite feed material. Most research has found that the addition factor of the rheological extrusion temperature range will reduce the viscosity value of polymer composites (Penumakala et al. 2020).

As a result of the dissolution of the polymer molecular chains during heating, the viscosity of the polymer composite feed material will decrease at high temperatures. This dissolution process increases the amount of empty space in the polymer molecular chain, which will increase the smoothness of the polymer composite feed material (Magalhães Da Silva et al. 2016). According to (Q. Ding et al. 2020), although an increase in high temperature can drain the feed material better, there will be a more serious decrease in thermal oxidation decomposition. The oxidation surface will damage the fusion of the molten polymer causing the tensile strength to decrease.

Figure 1 shows a graph of viscosity against shear rate to determine the flow characteristics of CF/PA composite feedstock. This graph was derived from rheological result data obtained from the addition of temperature and load. The results of this graph show the viscosity was inversely proportional to the shear rate for each of the temperatures of 210 °C, 230 °C and 250 °C. The viscosity value of CF/PA composite feed material increases with increasing shear rate value, this indicates that the resulting value is rare for polymer matrix material. This condition tends to produce dilatant flow characteristics or shear thickening, where the viscosity values are found to result in the range of 48.80 Pa.s to 97.88 Pa.s with shear rate values between the ranges of 19700 s⁻¹ to 20270 s⁻¹.

According to (Negi & Osuji 2009) under stable flow, shear thickening always occurs when the critical shear rate is reached. However, the measured shear rate can be influenced by the geometry-dependent wall slip phenomenon. Moreover, during the oscillation dynamics, the critical strain for shear thickening decreases with increasing frequency, and it reaches the minimum critical strain for the onset of shear thickening in the high frequency regime. Accordingly, the dynamic frequency behavior of low oscillations can be reasonably interpreted in terms of stable shear behavior. The critical dynamic shear rate remains stable in the low frequency regime, in line with the critical shear rate for stable shear thickening (Chang et al. 2011).

According to past research, it was found that flocculation of micro-sized particles can significantly affect the rheology of the material. This phenomenon depends on the size of the gap between the plates. Using small gaps the material exhibits flocculation-like, gel-like behavior, and shear thinning effects are observed under stable flow at lower shear rates (> 3 s⁻¹), as well as under oscillating dynamic motion with low strain amplitude. However, by using larger gap measurements, the micro-sized flocculation effect is less significant at low shear rate regimes, and therefore the fluid viscosity is relatively low and stable.

OPTIMIZATION OF EXTRUSION PARAMETERS

Minitab 19 software was used to analyze the viscosity test data obtained. The S/N values calculated to identify the influence of each control factor (temperature and load) on the viscosity variation are summarized in Table 4. The Taguchi technique creates a standard orthogonal arrangement to

consider the effect of several factors on target values and determine the experimental plan. Then, the experimental results were analyzed using mean and variance analysis to influence factors with SN ratio (Najim & Mohammed 2019). This allows for the collection of data needed to determine which factors are most effective in the quality produced with the minimum number of experiments.

TABLE 3. The flowability of the composite feed material at different temperature and load ranges with a load of 20 %bt filler

Feed material	Load (N)	Viscosity, η (Pa.s) with temperature			Shear rate, $\dot{\gamma}$ (s ⁻¹) with temperature		
		210 °C	230 °C	250 °C	210 °C	230 °C	250 °C
20 %bt CF/PA	40	51.10	49.50	48.80	19700	19800	20100
	60	73.73	72.94	72.78	19950	20170	20210
	80	97.88	96.90	96.76	20040	20240	20270

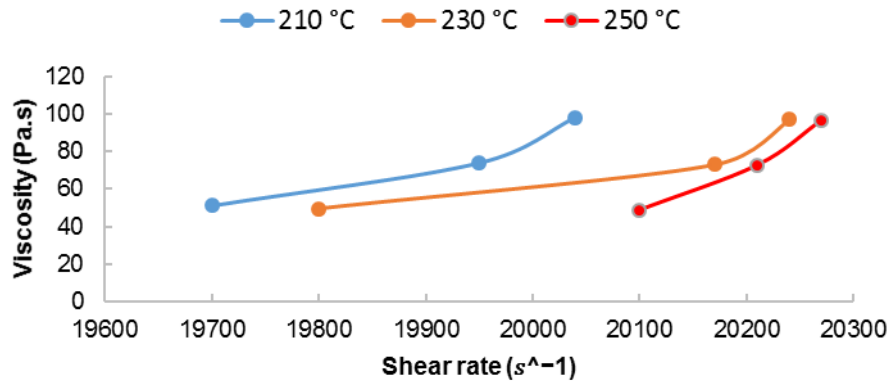


FIGURE 1. Graph of viscosity against shear rate of CF/PA composites

Next, ANOVA Analysis was conducted to identify the most significant control factors on the modeled responses and interactions between the control actors considered in the design (Fantous & Yahia 2020). The results obtained using ANOVA analysis are summarized in Table 5. This analysis was performed by considering the 95 % confidence level. The importance of control factors in ANOVA was determined by comparing the F-values for each control factor. On the other hand, the contribution of each control factor to the modeled response can be determined. ANOVA was used to analyze the effect of maximum temperature (Factor A) and load (Factor B) on the modeled reaction i.e. viscosity variation. The results of the analysis of variance show that load is the parameter that contributes the most to the viscosity which is 99.88 %. In addition, the P value obtained for viscosity is 0.000 which is less than 0.05. This indicates that load is an important parameter in influencing the viscosity of composite materials.

The main effect graph for the SN ratio is shown in Figure 2, with parameter optimization based on the largest peaks for each temperature and load parameter. The main effect graph was generated using the SN ratio from Table 5. Load is the most important parameter in optimizing the extrusion process, as shown in Figure 1. The lowest viscosity value is an important criterion for flowability in FDM as a function

of temperature and load. Therefore, the smaller statistical performance is better used for the calculation of the S/N ratio in viscosity properties (Pa.s). The molten viscosity should be in an optimal state i.e. low to allow extrusion and not too low to provide structural support (X. Wang et al. 2017).

According to (Miazio 2019) that the strength of the sample decreases with the addition of load hence there will be an increase in speed. In the range of 50 mm (s⁻¹) to 80 mm (s⁻¹), the strength of the specimen is at the same level. However, above 80 mm (s⁻¹), its strength decreases significantly from 0.62 kN to 0.35 kN. This condition occurs because defects in the extruded composite are due to the limited capacity of the extrusion nozzle and the time required to plasticize the filament is too short the possibility of blockage at the nozzle (P. Wang et al. 2019). Therefore, a load of 40 N is the optimal load value to avoid defects on the composite. The most optimal extrusion parameter is at a temperature of 250 °C and a load at 40 N where the viscosity value is at the lowest state at a value of 48.80 Pa.s. This is so that during the extrusion process using the FDM method, the flow characteristics of the feed material can flow with low viscosity values. Therefore, the mechanical properties will increase with good polymer melting.

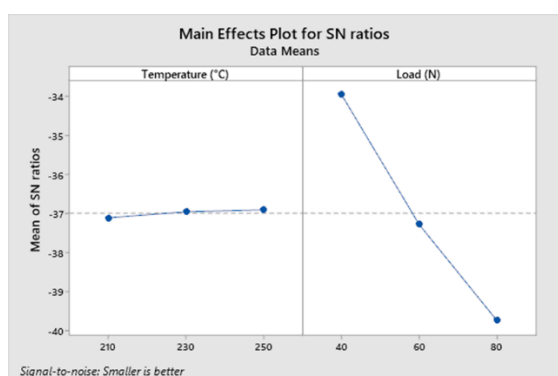


FIGURE 2. Graph of the main effect of the SN ratio for the viscosity value.

ANALYSIS OF MICROSTRUCTURE OBSERVATIONS

Figure 3 shows a cross-sectional SEM micrograph of the feed material against rheological extrusion temperature. Figure 3 shows that at extrusion temperatures of 210 °C and 230 °C inter-molecular cracking occurs. This indicates

that temperature plays a high role in the strength of the mechanical properties of the material. High viscosity and low fluidity of molten polymer at low temperatures can result in high porosity and poor bonding between lines and layers of molten polymer (Fang & Hanna 1999). With increasing temperature, the melting viscosity of the polymer decreases, the fusion between the polymer structure and the coating becomes better and there is a lower porosity so that the tensile strength and impact strength increase. Accordingly, according to (Attolico et al. 2020) the orthotropic behavior of FDM printed parts tends to decrease, while the mechanical properties improve with increasing extrusion temperature. Furthermore, according to (Zo et al. 2014) polyamide melting temperature is between 225 °C showed that an increase in extrusion temperature (<250 °C) leads to an increase in the bond quality of the polymer structure and the density and homogeneity of one part. While for the use of higher temperatures (<400 °C) there will be a more serious decrease in thermal oxidation decomposition. The oxidation surface will damage the fusion of the molten polymer causing the tensile strength to decrease (Q. Ding et al. 2020).

TABLE 4. Taguchi orthogonal arrangement L₉ showing S/N values for viscosity

No.	Temperature °C	Load (N)	Viscosity (Pa.s)	S/N (DB)
1	210	40	51.10	-34.1684
2	210	60	73.73	-37.3529
3	210	80	97.88	-39.8139
4	230	40	49.50	-33.8921
5	230	60	72.94	-37.2593
6	230	80	96.90	-39.7265
7	250	40	48.80	-33.7684
8	250	60	72.78	-37.2402
9	250	80	96.76	-39.7139

TABLE 5. ANOVA analysis for viscosity values

Sumber varian	DF	Sum of squares (SS)	Mean square (MS)	F ratio	Contribution rate%
Load	2	3367.53	1683.76	2499.28	99.88
Temperature	2	3.49	1.747	0.00	0.12
Error	6	4.04	0.67		
Total	8	3371.57			100

The determination of the initial stage of decomposition of the reinforcing elements of carbon fibers and polymers is highly dependent on the properties of thermal resistance. This level of thermal decomposition must be determined to determine the appropriate mixing and injection temperatures for further processing. Temperature increases can cause more serious thermal degradation of the polymer, which results in a deterioration of the mechanical properties the melting point of the polyamide matrix starting at 178.16 °C. However, the crystallization

temperature (T_c) of the CF/PA composite is higher than that of the PA matrix around 10 °C. This can be explained by the heterogeneous nucleation effect caused by carbon fibers influencing the nucleation mechanism and growth of PA matrix crystals (An et al. 2014). Therefore, the results of TGA and DSC can be used as setting the mixing and injection temperature parameters should be above the melting point of PA (<178.16 °C) and not more than the thermal decomposition temperature of CF/PA composite (<430 °C).

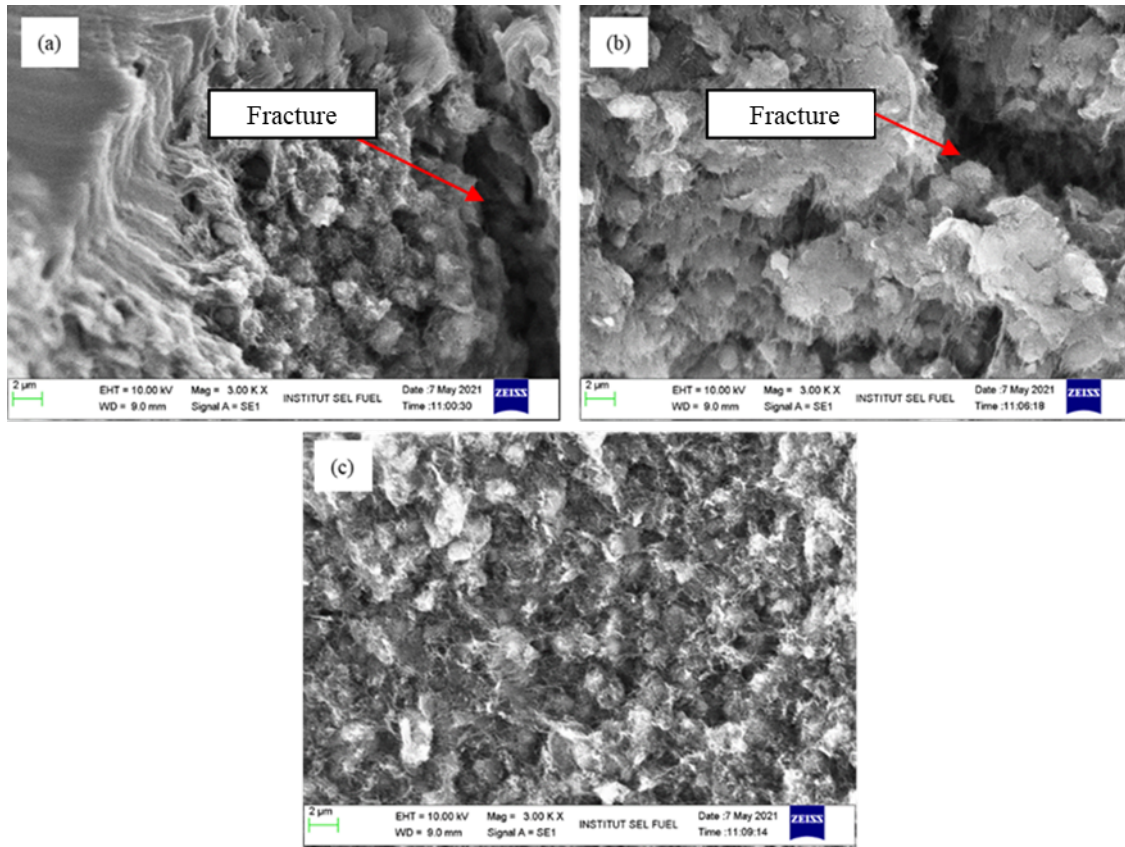


FIGURE 3. Cross -sectional microstructure of CF/PA composite feed material against rheological extrusion temperature; (a) 210 °C, (b) 230 °C, and (c) 250 °C

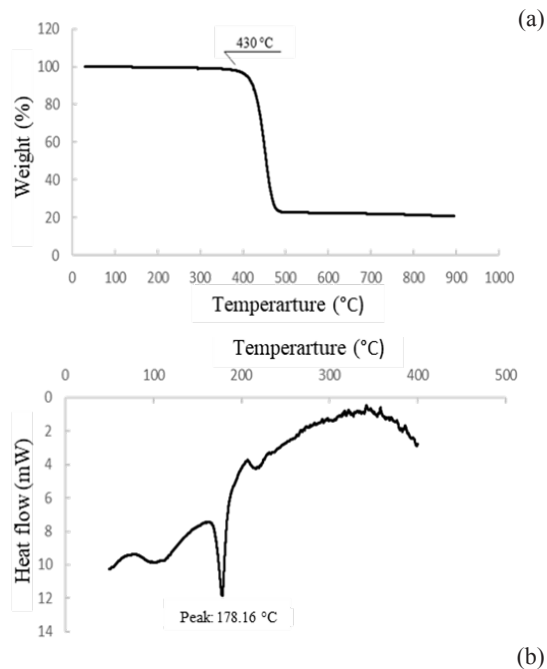


FIGURE 4. TGA (a) and (DSC) (b) analysis for CF/PA composites

Figure 4(a) shows the results of thermogravimetric analysis (TGA) analysis that has been performed on carbon fiber reinforced polyamide composites. Based on the Figure shows the CF/PA composite undergoes an early-stage

decomposition between the temperatures of 103 °C to 430 °C. This weight reduction of around 0.5 % to 2.0 % is due to moisture evaporation for the feed material in this stage (J. Wang et al. 2020). With increasing temperature, the weight of the CF/PA composite decreased to a temperature of 470 °C, with the highest rate of derivative weight value at 430 °C. The main reason for the decrease in material weight in this temperature range is the decomposition of the PA matrix. After this stage, 80 % of the sample weight is lost slowly to 900 °C. Accordingly, the weight of PA decreased to close to zero at the end of the study, indicating full decomposition of the PA matrix. For CF/PA composite materials, the carbon fiber decomposition temperature is usually much higher than 1000 °C (Peng et al. 2019). Therefore, the weight balance should be associated with the presence of carbon fibers. According to (Grund et al. 2019) while considering TGA for CFRP, observed that polymer matrices decompose completely at relatively low temperatures (usually between 200 °C and 400 °C), while carbon fibers tend to decompose at temperatures between 400 °C and 1000 °C, depending on fiber primers and heat treatment.

In the production of carbon fiber composites, apart from determining the properties of thermal resistance, finding the melting point of the matrix material is also important. The polymer matrix should be selected to have a melting point lower than the thermal decomposition temperature of

the carbon reinforcing fibers. The phase change of the PA matrix is illustrated in Figure 4(b) with the appearance of endothermic peaks during the differential scanning calorimetry (DSC) study was conducted. The resulting T_g value at the endothermic peak represents

CONCLUSION

The study on the flow properties of CF/PA composite feedstock by using a carbon fiber filler load of 20 wt.% was successfully performed with good flow rate without getting stuck on the rheological nozzle. As a result of the rheological study, the flow characteristic of the resulting CF/PA composite feed material is dilatant or shear thickening where the viscosity value increases as the value of the shear rate increases. Viscosity values were found to result in the range of 48.80 Pa.s to 97.88 Pa.s with shear rate values between the range of 19700 s⁻¹ to 20270 s⁻¹ against temperature factors of 210 °C, 230 °C, and 250 °C with loads of 40 N, 60 N, and 80 N. Based on this study, carbon fiber reinforced polyamide composites have undergone a rheological process to study the flowability of the feed material for use in the FDM method. To achieve the goal of this composite material for bipolar plate applications, a study on electrical conductivity is proposed to be done in the future.

ACKNOWLEDGEMENT

The authors wish to express their gratitude and appreciation to the Ministry of Higher Education (MOHE) Malaysia and Center for Research and Instrumentation Management (CRIM), UKM for their financial support under Grant Number, FRGS/1/2020/TK0/UKM/02/18 which enable them to complete this study.

DECLARATION OF COMPETING INTEREST

None

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