THERMAL AND MELT FLOW BEHAVIOUR OF KENAF FIBRE REINFORCED ACRYLONITRILE BUTADIENE STYRENE COMPOSITES FOR FUSED FILAMENT FABRICATION

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ABSTRACT

The thermal and melt flow behaviour of polymer and fibre materials are vital in producing filaments for fused filament fabrication (FFF), especially for custom-made composites materials. The degradation temperature and melting temperature of commercialised acrylonitrile butadiene styrene (ABS) filament, neat ABS polymer and different loadings of kenaf fibre (KF) reinforced ABS composites were investigated using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) respectively. Melt flow index (MFI) was then investigated in view of the viscosity of the materials using a melt flow indexer at 230 °C and 5 kg weight loading. The kenaf fibre reinforced ABS composites were prepared using an internal mixer at 180 °C and crushed to the form of granules. It was found that the addition of kenaf fibre lowered the decomposition temperature and increased the melting temperature of composites as compared to the neat ABS polymer (0% KF – ABS). Meanwhile, the neat ABS polymer had higher value of MFI as compared to the commercialised ABS filament. The value of MFI increased as the kenaf fibre loading increases. Thus, the neat ABS polymer and kenaf fibre reinforced polymer composites are suitable as feedstock filament material for FFF since the low viscosity of commercialised ABS filament is able to be printed from open-source 3D printers.

Keywords: *Fused filament fabrication (FFF); natural fibre composites; melt flow behaviour; thermal properties; kenaf fibre.*

1. INTRODUCTION

Fused filament fabrication (FFF) is the one of the most significant techniques for additive manufacturing (AM) and recently, has been widely used in aerospace (Kumar & Nair, 2017), automotive (Page, 2018), medical (Wang *et al*., 2017) and defence (Rathee *et al*., 2017) industries. The applications of AM in defence support services are to provide platforms with the ability to sustain their systems, recover their ability after damage and reduce costs in the supply chain (Busachi *et al*., 2015). In addition, AM manufactures military personnel equipment, such as body armours kit and special tools for mission requirements (Busachi *et al*., 2016). AM has also contributed to defence support services for the Royal Navy's platforms (Busachi *et al*., 2017). FFF is commonly used in producing conceptual models, prototypes and engineering components (Mohan *et al*., 2017). A three-dimensional object can be produced by successive layer upon layer of materials, where a filament is melted inside a liquefier at a temperature above its melting point and pushed at the nozzle die by solid upstream filament (Carneiro *et al*., 2015). The schematic set up of FFF is shown in Figure 1. It is used for its simple fabrication process (Maasod & Song, 2004), ability to fabricate geometrically complex shapes (MacDonald & Wicker, 2016), less expensive machining (Wong & Hernandez, 2012) and cost-effectiveness (Mohan *et al*., 2017), especially for defence industries in producing weapons (Busachi *et al*., 2016). Besides that, FFF produces less waste material (Grujovic *et al*., 2017).

Thermoplastics are commercial material filaments, such as acrylonitrile butadiene styrene (ABS), polylactic acid (PLA) and polycarbonate (PC), used in FFF for producing three-dimensional printed parts. McMains (2005) stated that thermoplastic filament is used for FFF as it is safer to maintain and less expensive. Wang *et al*. (2017) indicated that custom-made composite material filaments are possible to be used as feedstock material for FFF. However, custom-made composite material is limited to the suitability of melt viscosity since the molten viscosity should be high enough to provide structural support and low enough to enable extrusion (Wang *et al*., 2017).

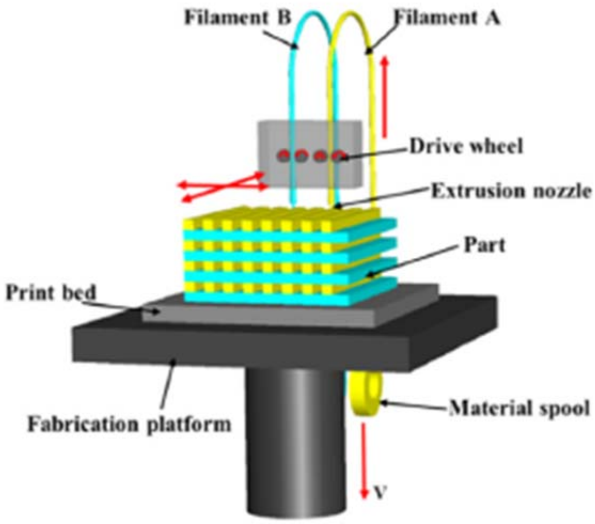

Figure 1: Schematic of FFF setup (Wang *et al*., 2017).

Nowadays, natural fibre reinforced composite (NFRC) materials have been well developed in industries. NFRC is a composite material consisting of polymer matrix with natural fibres. NFRC is widely used in numerous applications due to its lightweight properties, low cost, less damage to processing equipment, biodegradability and good relative mechanical properties (Mohammed *et al*., 2015). Currently, natural fibres have been widely used to replace synthetic or man-made fibres in engineering applications. The advantages of natural fibres, such as low cost, abundantly available, environmentally friendly, low density and higher strength performance, are the reasons that they have been chosen over synthetic fibres (Ahmad *et al*., 2015). There are numerous types of natural fibres used as reinforcement for thermoplastics, such as flax, hemp, jute, coir, kenaf and wood. However, the drawback of natural fibres, such as short lifetime, albeit with minimum environmental damage upon degradation (Mohammed *et al*., 2015), and restricted processing temperature, limit their performance and usage (Gallo *et al*., 2013). Recently, experimentation and investigation of NFRC filaments for FFF technology have been growing along with concerns and awareness on environmental friendly materials. However, the melt flow behaviour of NFRC materials is one of the main issues for FFF. Ramanath *et al*. (2008) stated that melt flow behaviour apparently affects the quality of printed specimens, which depends on pressure, temperature and physical properties, including melting temperature and rheology behaviour.

Up to now, several studies have been conducted on the melt flow behaviour of polymers and NFRC. Singh *et al*. (2016) studied the melt flow behaviour of ABS extrusion grade (ABS-EG) and ABS P400. ABS-EG was selected as an alternative material for FFF filaments, as it is commercially available. The ASTM-D-1238-95 standard was applied for 10 min at 230 °C barrel temperature and 3.8 kg load. The results obtained indicate that the melt flow index (MFI) of ABS-EG and ABS P400 were almost similar, which were 2.381 and 2.398 gm/10 min respectively. Then, both the ABS-EG and ABS P400 filaments were tested using a scanning electron microscope (SEM). The results from the SEM showed that fabricated filaments of ABS-EG contain a lot of air pockets as compared to ABS P400, where there is no presence of air pockets. The study concluded that the fabricated filaments are affected by process parameters of extrusion, including screw speed and barrel temperature. Islam *et al.* (2013a, b) investigated the MFI of kenaf fibre reinforced recycled polypropylene (RPP) using a MFI tester (Dynisco Instrument) at 230 °C with standard weight of 2.16 kg. Three experiments were conducted, which were RPP; 10%, 20%, 30%, 40% and 50% of raw kenaf fibre RPP (RKPC); and raw kenaf fibre with maleic anhydride grafted polypropylene (MAPP) reinforced RPP (MRKPC). The results of the MFI obtained for RPP; 10%, 20%, 30%, 40, 50% of RKPCP; and MRKPC were 5.58, 2.54, 1.78, 0.46, 0.40, 0.22 and 0.33 g/10 min respectively. The reduction of flow properties for RKPCP was due to the presence of kenaf fibre, whereas MRKPC had the lowest flow due to the presence of MAPP. They also found that the molecular weight of composites and viscosity had an indirect relationship. The results indicated that the higher the percentage of fibre in composites, the more viscous was their melts. Similarly, Mohammad & Arsad (2013) reported that as the loading of kenaf fibre in composites increased, the viscosity increased because the molecular weight of composites increased.

The disadvantages of natural fibre, such as low thermal stability, limit the usage of NFRC for FFF. Torrado *et al.* (2015) investigated the effect of jute fibre reinforced ABS composites on FFF and found that the high temperature of the extrusion process can cause decomposition of jute fibre. Montalvo *et al.* (2018) studied on wood plastic composites (WPC) and found that the decomposition of WPC is between 300 and 500 °C. Therefore, it is vital to study the thermal stability of NFRC before the NFRC filament is produced and extruded on FFF.

The aim of this study is to investigate the thermal degradation temperature, melting temperature, glass transition temperature and melt flow behaviour of commercialised ABS filaments, neat ABS, as well as different loadings of kenaf fibre reinforced ABS composites using thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and melt flow indexing.

2. MATERIALS AND METHODOLOGY

2.1 Materials

Kenaf (*Hibiscus Cannabinus*) flour core fibres were supplied by ZHF Industries Sdn. Bhd, while ABS pellets (100% pure) were supplied by Macrocom (M) Sdn. Bhd. Commercialised ABS filaments was obtained from a 3D printer supplier.

2.2 Preparation of the Blends

A sieve shaker was used to obtain an average length of 150 µm for the kenaf fibre flour. The ABS pellets and kenaf fibre were dried in an oven at 80 °C for 24 h before being mixed in an internal mixture. The blends were made by mixing ABS with different percentages of kenaf fibre (KF), which were 0, 2.5 and 5 wt.%, which identified as 0% KF – ABS (neat ABS), 2.5% KF – ABS and 5% KF – ABS respectively. A HAAKE Rheomix OS internal mixer was used to mix 2.5% KF – ABS and 5% KF – ABS. The mixture was mixed at 180 °C and 50 rpm for 12 min for each mixing, with a maximum of 50 g allowed for each mixing. First, the ABS pellets were fed into the internal mixer for 5 min and then, the kenaf powder was added into the mixer. Then, a crusher machine was used to crush the composite materials into the form of granules, while the ABS commercialised filaments were cut into 3 mm length.

2.3 Thermal Gravimetric Analysis (TGA)

The instrument used for the TGA test was a Mettler Toledo TGA. The temperature used for the samples is 25 to 550 °C with heating rate of 10 °C/min and the atmosphere used was nitrogen gas. The granules with 2.5% KF – ABS and 5% KF – ABS were weighed to be between 5 – 15 mg and placed in the chamber. The TGA test was conducted to measure the change in mass of the sample as a function of increasing temperature and the final residue yield on set from degradation of temperature was recorded.

2.4 Differential Scanning Calorimetry (DCS)

The instrument used for the DSC test was a DSC 4000 system. The samples were weighed to be around 5 to 10 mg with 10 °C/ min heating rate. Measurements of melting temperature (T_m) and glass transition temperature (T_g) were recorded as a function of temperature in the range of 30 – 250 °C, and calculated by analysing the different peaks obtained from the graphs.

2.5 Melt Flow Index (MFI)

The MFI was determined using a melt flow indexer (Ray Ran- 6MPCA advanced melt flow system). The melt flow properties of the polymer and composites were measured at temperature of 230 °C with weight loading weight of 5 kg. All the samples were measured for weight of 12 g. Then, 300 s of pre-heating was conducted when samples were fed into instrument's chamber.

3. RESULTS AND DISCUSSION

3.1 Thermogravimetric Analysis (TGA)

The TGA analysis of the commercialised ABS filament, 0% KF – ABS, 2.5% KF – ABS and 5% KF – ABS are presented in Figure 2. Table 1 summarises the TGA results of decomposition temperature and final weight after decomposition. The commercialised ABS filament decomposition temperature was slightly higher as compared to 0% KF – ABS by 1.26%. On the other hand, 0% KF – ABS had higher decomposition temperature as compared to 2.5% KF – ABS and 5% KF – ABS. Figure 5 shows that the increase of fibres decreased the thermal stability as compared to the 0% KF – ABS.

It is found that the thermal stability of kenaf fibre was the lowest with 314.53 °C for decomposition temperature. The overall weight loss of kenaf fibre could be divided into three different phases. For the first phase, weight loss of approximately 6% from initial weight occurred at below 100 °C. The second weight loss of 12.5% occurred at around 260 °C, where this was the initial stage of thermal degradation. The final phase is referred to as major thermal degradation, which occurred with maximum weight loss at 325 °C. The weight loss occurred due to the vaporisation from fibre and decomposition of cellulose (Fauzi *et al*., 2016). As the content of kenaf fibre was increased, the decomposition temperature of the composites decreased since kenaf fibre has low thermal stability as compare to ABS polymer. Both formulations of kenaf fibre reinforced ABS composites also degraded in three phases. For 2.5% KF – ABS, in the first degradation phase, the moisture evaporation started at 250 °C and ended at 348 °C. The second decomposition phase occurred in the range between 370 and 460 °C, while the third phase was between 465 and 490 °C. For 5% KF – ABS, the first decomposition phase occurred between 240 and 340 °C, the second phase was between 350 and 450°C, while the third phase was between 450 and 480 °C. The decomposition steps for kenaf fibre reinforced ABS composites was a very rapid and complicated process.

These results are supported by Azwa & Yousif (2013), where the addition of natural fibre in composites cause the thermal stability to reduce due to less stable fibres. Similarly, Tawakkal *et al*. (2014) studied the effect of kenaf fibre loading reinforced PLA composites on thermal properties and concluded that decreasing trend of degradation temperature for TGA curves of kenaf / PLA composites were found with increasing kenaf content. El-Shekeil *et al*. (2014) investigated the influence of fibre content on thermal properties of kenaf fibre reinforced poly (vinyl chloride) / thermoplastic polyurethane poly-blend composites and found that composites with lower fibre content had a higher thermal stability as compared to higher fibre contents. The increasing percentage of fibre lowers the thermal stability due weakening of hydrogen bonding and decrease of mobility of cellulose chains in cellulose (Aji *et al*., 2011).

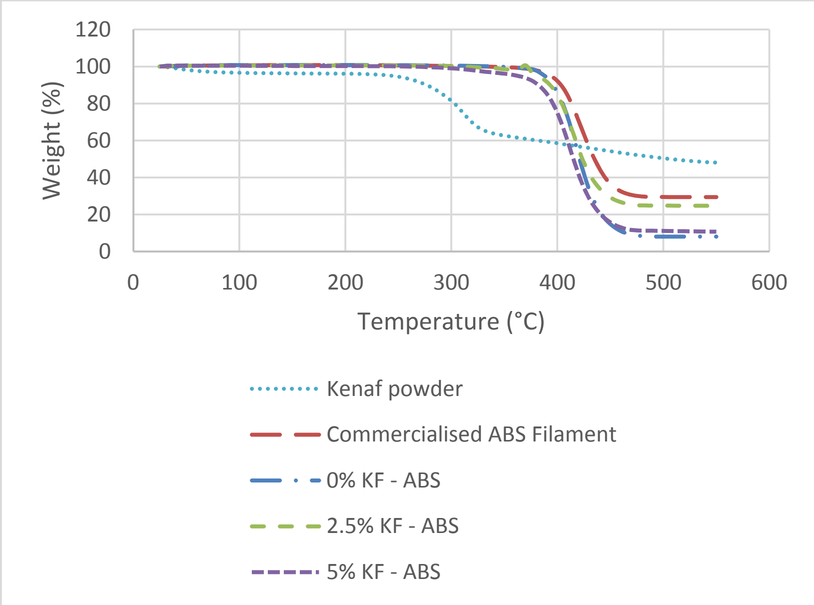

Figure 2: TGA curves.

Table 1: Results of the TGA test.

Materials	Decomposition temperature (°C)	Final weight after decomposition (%)
Kenaf powder	314.53	8.36
Commercialised ABS filament	425.16	4.60
0% KF – ABS	419.83	2.65
2.5% KF – ABS	417.94	3.34
5% KF – ABS	410.39	3.46

3.2 Differential Scanning Calorimetry

Table 2 shows the melting temperature (T_m) and glass transition temperature (T_g) for the commercialised ABS filament, 0% KF – ABS, 2.5% KF – ABS and 5% KF – ABS. The results show that the addition of kenaf fibre increased the Tm of the composites as compared to the 0% KF – ABS., while the Tm of the commercialised ABS was lower than the 0% KF – ABS. The T_g of the 0% KF – ABS increased as the content of kenaf fibre increased, with the commercialised ABS filament giving the highest Tg.

Table 2: Results of the DSC test.

Materials	Melting temperature, T_m (°C)	Glass transition temperature, T_g (°C)
Commercialised ABS filament	213.67	108.46
0% KF – ABS	220.17	100.28
2.5% KF – ABS	222.50	102.43
5% KF – ABS	221.83	107.62

3.3 Melt Flow Behaviour

MFI indicates how fast a material flows in the molten state and is inversely proportional to viscosity. The MFI of commercialised ABS filament and different loadings of kenaf fibre reinforced polymer composites are shown in Figure 3. The commercialised ABS filament had the lowest MFI value, whereas the 0% KF – ABS had the highest value of MFI. The addition of 2.5% KF - ABS decreased the value of MFI, whereas the MFI for 5% KF – ABS increased. The MFI values of the composites increased as the filler loading increased. This is because the presence of kenaf fibre hindered the flowability of the ABS matrix. Lim *et al.* (2015) found that the presence of irregular shapes of natural filler provides an obstruction to the polymer melts of composites. Thus, it leads to lower MFI values for composites as compared to neat polymers. Meanwhile, another study found that the MFI values of composites are much higher than neat polymers due to the orientation of fibre along the flow lines with high aspect ratio (Tayfun *et al.*, 2016).

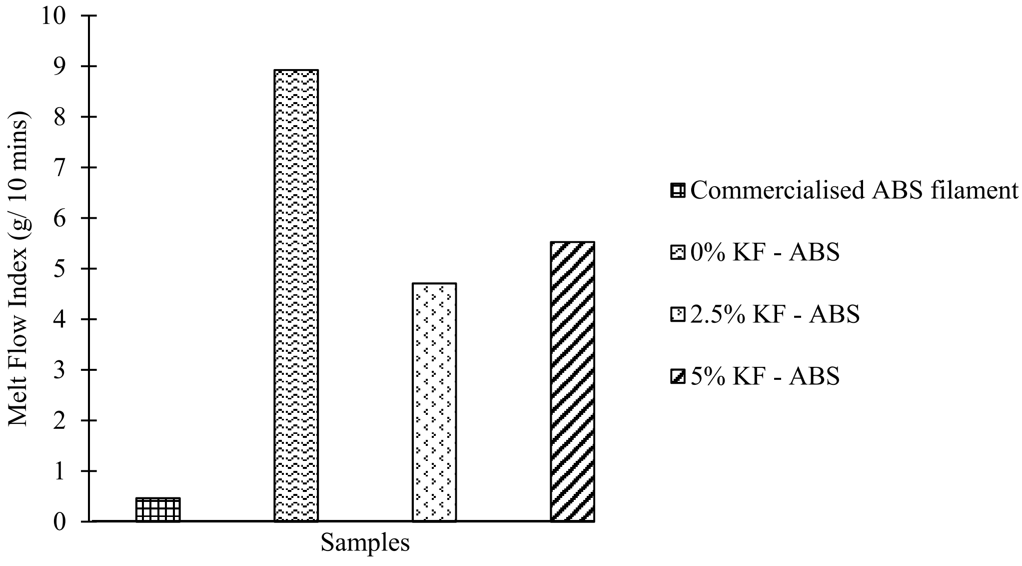

Figure 3: MFI of the ABS polymer and composites.

A higher MFI value indicates an easier flow of material through the dies, which is a low viscosity fluid, while a low MFI indicates as high viscosity fluid (Sa'ude *et al.*, 2016). The commercialised ABS filament had the lowest value of MFI as compared to other samples, indicating high viscosity of its fluid. However, the commercialised ABS filament can be printed well via a 3D printer with no clogging at the nozzle. These results contradict with Khaliq *et al.* (2017), where they obtained lower viscosity for commercialised ABS filament. They stated that different ABS filaments will give different viscosities, either low or high, due to material composition. Thus, it indicates that neat ABS polymer and kenaf fibre reinforced polymer composites are suitable as feedstock filament since their low viscosities allow them to be able to be printed using open source 3D printers. In addition, a lower viscosity material is the best for the printing process in 3D printers, but the process parameters of 3D printer can be optimised to obtain the good printing quality by adjusting the speed of extrusion. This condition can enhance the effective heating process, which corresponds to lower viscosity (Khaliq *et al.*, 2017).

Han *et al.* (2012) concluded that the strength of kenaf composites is enhanced because of the lower viscosity of polymer matrix, which allows for better wetting of fibres and enhanced strength of composites. The mechanism for enhancement of the strength of composites is closely related to fibre-polymer surface adhesion without considering the chemical treatment. Tayfun *et al.* (2016) investigated the surface modification of fibre by alkaline and silane treatments, and found that the MFI of rice straw and thermoplastic polyurethane (RS-TPU) composites were much higher as compared to neat TPU polymer. Meanwhile, Kim (2015) found that the adhesion between natural fibre and polymer matrix is hindered by lignin and volatile extractives, which leads to poor adhesion of composites between fibre and matrix. This study suggested an addition of coupling agent to increase the contact of surface area between natural fibre and polymer matrix. Based on the observation from SEM, they found that addition of maleic anhydride grafted polypropylene copolymer (MA-g-PP) into kenaf filled PP composites increased the interfacial adhesion as compared to kenaf filled PP composites without MA-g-PP. However, the addition of MA-g-PP into composites decreased the value of MFI as compared to composites without MA-g-PP, which had higher value of MFI. Similarly, Noranizan & Ahmad (2012) reported that treated kenaf fibre with alkaline treatment and compatibiliser improved the interaction of kenaf and high-density polyethylene (HDPE) composites as compared to untreated kenaf fibre. In this investigation, the viscosity of treated kenaf fibre and compatibiliser with HDPE composites was higher as compared to untreated kenaf fibre with increase of fibre loading. Wang *et al.* (2018) concluded that materials that have lower MFI have higher average molar mass. As the filler loading of fibre increases, the viscosity of the composite will increase. Thus, high average molar mass can affect the melt viscosity of the material.

4. CONCLUSION

In conclusion, the thermal and melt flow properties of kenaf fibre reinforced ABS composites were studied and compared with 0% KF – ABS polymer and commercialised ABS filament for FFF applications. The addition of kenaf fibre lowered the decomposition temperature of composites, whereas it increased the melting and glass transition temperatures of the composites. The MFI value of 0% KF – ABS was higher as compared to the commercialised ABS filament. The high viscosity of the commercialised ABS filament is due to the high molecular weight, leading to low value of MFI. The addition of kenaf fibre with ABS polymer reduced the MFI value, but the value of MFI increased with higher loading of kenaf fibre. The mechanism of rheology behaviour reflects the change in molecular weight and the interaction among components. Thus, the viscosity of the composites was affected by the amount of kenaf fibre.

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