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Relationship between screw structure and properties of recycled glass 1 fiber reinforced flame retardant nylon46 2 Shuidong Zhang, Peng Wang, Lingcao Tan, Hanxiong Huang, Guo Jiang^{*} 3 Lab for Micro Molding and Polymer Rheology, College of Mechanical and Automotive 4 Engineering, South China University of Technology, Guangzhou 510640, China 5 6 Abstract 7 8 Considering environmental protection and economic requirements, the reuse of recycled 9

glass fiber reinforced flame retardant nylon 46 (RGFFRPA46) is of significant importance. In this paper, the mechanical, thermal, rheological and flame retardant 10 properties of the recycled RGFFRPA46 were evaluated by varying shear screws. To 11 establish the relationship between screw structure and the properties of RGFFRPA46, the 12 carboxyl content (CC) and viscosity-average molecular weight ($\overline{M_v}$) of PA46, the 13 distribution and average length of glass fiber (\overline{L}) and microscopic appearance of 14 RGFFRPA46 were investigated. The results showed that the shear force resistance time 15 of RGFFRPA46 increased with the increase of the shear strain and the ratio of length to 16 diameter of the used extruder. As a result, $\overline{M_{\nu}}$ of PA46, \overline{L} and percentage of glass 17 fiber length (V_i) , which was higher than critical length decreased, whereas, the CC of 18 PA46 increased significantly. The mechanical and rheological properties were sensitive 19 to these variations, especially the decrease in impact, tensile, flexural strength and shear 20 viscosity of RGFFRPA46. Furthermore, the influence of degradation on $\overline{M_v}$ of PA46 21

^{*} Corresponding author: Jiang Guo. Fax: +86 20 87110029, E-mail address: starch@scut.edu.cn or jiangguo@scut.edu.cn (G. Jiang)

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and \overline{L} were against to the thermal properties of RGFFRPA46 after extrusion. However, the flame retardant properties of RGFFRPA46 were independent on the degradation. All of them achieved a UL-94 V-0 classification, and pass Glow Wire Ignition Temperature (775 °C) test along with LOI of 36.7 %. When RGFFRPA46 was extruded by a single screw extruder with the lowest shear force, only 5.8% decrement in mechanical properties with negligible reductions in thermal and flame retardant properties. Keywords Recycled; nylon 46; screw structure; molecular weight; glass fiber; properties Introduction Nylon 46 (PA46) is an AA-BB type polyamide prepared from tetramethylene diamine and adipic acid, which was first synthesized by Carothers,¹ and then commercialized by DSM under the name Stanyl.² Compared with the most widely used PAs (e.g., PA 6 and PA 66), PA46 has higher amide content and symmetrical chain structure, endowing it with higher mechanical and thermal properties and faster process cycle than other engineering plastics.³ Because of its good thermal stability, higher stiffness and fatigue resistance, PA46 were successfully applied in various fields such as construction area,⁴, ⁵ fiber industries,⁶ modern electronic and automotive applications.⁷ PA46 based blends and composites also drawn a lot of research interests.⁸⁻¹¹ Glass fiber reinforced flame retardant nylon 46 (GFFRPA46) have higher performance

as engineering material than the unfilled ones. They were applied as insulating parts in
various fields, especially in electrical engineering and electronics by micro injection

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molded. For example, GFFRPA46 is an importance material to fabricate the electronic 1 relay. In general, more than 50 wt% of RGFFRPA46 were produced from scraps and 2 unqualified injection products during the micro injection mold. Because of environmental 3 legislation like the EU-directives (2008/98/EC) causing an increase in demand for 4 recycling the electrical waste and electronic equipment (WEEE),¹² it is important to reuse 5 6 the RGFFRPA46. Scraps of RGFFRPA46 from 'runner and gate' molding systems and 7 defective products can be processed by micro-injection molding to generate more than 50 wt% RGFFRPA46. The RGFFRPA46 pieces with varying shapes are often granulated 8 9 the particles with uniform size to meet the demands of reusing in micro-injection molding. As the reproduced RGFFRPA46 have good mechanical, thermal and flame 10 retardant properties, it can be used solely or added at high proportion in the 11 12 micro-injection molding.

It was reported that the variation in the fiber length and distribution significantly 13 affected the mechanical and thermal properties.^{13, 14} Practically, once the V_i decreases, the 14 part of the GF with length shorter than L_c could not reinforce the polymer, resulting in a 15 decrease in mechanical and thermal properties of composite. When a fiber reinforced 16 composite was prepared by different process machine, the fiber would suffer because of 17 the varied breakage by those machines. Franzen et al.¹⁵ investigated the effect of the 18 process machine on the microstructures and properties of the prepared fiber-reinforced 19 composites. They found that the composite mixed with a Buss Kneader had shorter 20 average fiber length than that one mixed with a twin screw extruder. Rozman et al.¹⁶ 21

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revealed that the composites prepared by internal mixer had higher flexural properties 1 than those prepared by high speed mixer and twin screw extruder. Ville et al.¹⁷ studied 2 3 fiber breakage during compounding with a polyamide matrix in a Buss kneader. They confirmed that the length of GF was significantly decreased to 350 µm from 3500 µm 4 during compounding. These previous work showed that the degree of breakage from 5 extruder was lower than other machines. Ramani et al.¹⁸ surveyed the mechanism of fiber 6 fracture in fiber reinforced composites. They found that the residence time, fill-up, and 7 the intensity of mixing during extrusion compounding had predominant influence on 8 fiber fracture. The methods to reduce fiber fracture included process optimization and 9 machine design. Zhuang et al.¹⁹ investigated the influence of shear force with different 10 screw speeds and variable channel depth on fiber degradation. The main impetus of fiber 11 degradation was presented fiber-equipment and fiber-fiber interaction. According to the 12 characteristic parameters of residence time distribution (RTD) model, these two 13 interactions would be improved by the enhancement of mixing capacity. Due to the high 14 shear forces acting in the molten polymer, the shear forces will be transferred to the fibers 15 that lead to decrease in the reinforcement effect of fibers. Additionally, polymeric 16 material after thermomechanical degradation during processing harm its mechanical 17 properties.²⁰ 18

In this study, RGFFRPA46 was recycled with one co-rotating twin screw and three different single screws. During extrusion, the glass fibers in matrix were damaged and affected the properties of the resulting products. To establish the relationship between the

1 screw structure and the properties of extruded RGFFRPA46, effect of the the parameters 2 including the CC and $\overline{M_V}$ of PA46, \overline{L} and its distribution, and the morphology on the 3 mechanical, thermal, rheological, flame retardant properties and the content of bromine 4 of the extruded RGFFRPA46 were systemically evaluated.

5 **Experimental**

6 Materials

Commercial grade glass fiber reinforced flame retardant PA46 with 30% glass fiber were
purchased from (Stanyl TE-250F6 as PA46-F6) DSM Co. Ltd. The flame retardants,
decabromodiphenyl ethane (DBDPE) and Sb₂O₃, the analytical grade formic acid and
alcohol were purchased from the Chemical Reagent Factory of Longxi (Shantou, China).

11 Sample preparation

12 The raw material (PA46-F6) was dried at 80 $^{\circ}$ C, for 8 h before injection mold. The relay cover (the size was $15.7 \times 12.5 \times 14 \text{ mm}^3$, the wall thickness was 0.4 mm) was injection 13 molded (Germany, ARBUR 420M, ø=25 mm, L/D=28) with 2-plate mold, the 14 temperature from feed inlet to nozzle was 290 °C, 295 °C, 295 °C, 300 °C and 295 °C, 15 respectively. The mold temperature was kept at 60 °C. The nozzle diameter of the 16 machine was 2.5 mm and the sprue diameter of 2-plate mold was 2.8 mm. The rotational 17 speed of screw was 25 m/min, three-stage injection rate was 70, 100 and 90 mm/s, 18 respectively. The injection volume was 12.5 ± 0.5 cm³, the injection pressure, reserve 19 pressure and screw back pressure was 1650±100 bar, 1800±100 bar and 5 bar, 20 respectively. The cycle time of process was 14 s, with the plasticizing time, injection time, 21

the reserve pressure time and the cool time was 1.0 s, 1.2 s, 0.5 s and 8 s, respectively. 1 The recycled PA46-F6 (RPA46-F6) (about 140 kg) including the scraps and unqualified 2 injection products was collected and smashed to 5 \times 3 \times 2 mm³ by low speed crusher 3 (Sweden, Rapid Co Ltd, PMD-2L). 4 5 L1=4.5D 6 1.2=4D L3=1.5D I < SJ-50 : D=50.0mm , L/D=10.0 , Compression rate =3.0 , l=D ,H1=0.15D , H2=0.07D ,H3=0.05D 7 -L1=13.8D L3=4.7D 12=11 50 8 II < SJ-30 : D=30.0mm , L/D=30.0 , Compression rate =3.0 , 1=D ,H1=0.15D , H2=0.07D ,H3=0.05D 9 -L1=18.0D-L2=15.0D L3=7.0D 10 Ⅲ 、 SJ-40 : D=40.0mm , L/D=40.0 , Compression rate =3.0 , l=D ,H1=0.15D , H2=0.07D ,H3=0.05D 11 L1=18. 0D L2=15. 0D L3=7. 0D 12 IV 、 T-30 : D=30.0mm , L/D=40.0 , Compression rate =3.0 , 1=D ,H1=0.15D , H2=0.07D ,H3=0.05D Figure 1 Screw structure of different extruder 13 RPA46-F6 was dried at 80 °C for 8 h before reprocessed by four different laboratory 14 extruders produced by Nanjing GIANT Machinery Co., Ltd, China. Figure 1 presents the 15 screw structure of four kinds of extruder. The sequence of screw shear force of them was 16 IV>III>II>I. The first extruder (Figure 1, (I)) was a special short single extruder (S50). Its 17 screw diameter (D), ratio of length to diameter (L/D) and die diameter (D_0) was 50 mm, 18

kg/h. Temperature profile along S50 was set at 305 °C, 310 °C, 315 °C and 310 °C,
respectively.

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10 and 3 mm, respectively. The screw speed and feeding rate was set at 75 rpm and 10

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The second extruder (Figure 1,

The second extruder (Figure 1, (II)) used was a single screw extruder (S30). The D,
L/D, and D_0 was 30 mm, 30 and 3 mm, respectively. The screw speed and feeding rate
was set at 75 rpm and 10 kg/h. The third extruder (Figure 1, (III)) was a single screw
extruder (S40) and the D, L/D, and D_0 was 40 mm, 40, and 3 mm, respectively. The
screw speed and feeding rate was set at 75 rpm and 15 kg/h. Temperature profile along
the two single extruders was 305 °C, 305 °C, 310 °C, 315 °C and 310 °C, respectively. The
RPA46-F6 extruded product by S50, S30, S40, and T30 were dubbed as RPA46-F6-S50,
RPA46-F6-S30, RPA46-F6-S40, and RPA46-F6-T30, respectively.
The fourth extruder (Figure 1, (IV)) was a co-rotating twin-screw extruder (T30) and
its D, L/D, D_0 was 30 mm , 40, and 3 mm, respectively. The screw speed and feeding rate
was set at 75 rpm and 20 kg/h. Temperature profile along the extruder was
295 °C,300 °C, 305 °C, 305 °C and 300 °C, respectively.
The specimens (127×13×4 mm ³ , 63.5×12.7×3.2 mm ³ , 165×13×3.2 mm ³ , 127×12.7×3.2
mm ³) for mechanical and thermal properties test were injection molded (Germany,
ARBUR 420M, ø=25 mm, L/D=28) with 2-plate mold, then dried at 80 °C for 8 h. The
screw rotational speed was 20 m/min and three-stage injection rate was 110, 120 and 90
mm/s, respectively. The injection volume was 58.5 ± 1 cm ³ . The injection pressure, reserve
pressure and screw back pressure was 1350±100 bar, 1500±100 bar and 20 bar,
respectively. The cycle time of process was 3.5 s and the plasticizing time, injection time,
the reserve pressure time and the cool time was 2.8 s, 1.3 s, 0.5 s and 30 s, respectively.

Ten specimens were collected to measure the properties.

RPA46-F6 and its extruded products were grinded by a high-speed knapper. The
powder sample (about 5.0 g) was dissolved in formic acid solution (1000 mL) at 40 °C
for 24 h using ultrasonic. Then the solution was cooled to room temperature and filtered
to remove the GF and other insoluble substance. The resin was purified by precipitation
using ethanol, and then dried at 80 °C for 12 h.

6 Characterization.

Intrinsic viscosity ($[\eta]$) and carboxyl contents (CC) test. The purified PA46 (0.15 g) was dissolved in 90% formic acid (25 mL) and the solution was measured at 25 °C by using an Ubbelohde viscometer. $[\eta]$ and the viscosity-average molecular weight ($\overline{M_V}$) of the PA46 samples were calculated from Eq.1 and 2. The latter is the Mark-Houwink equation for PA46 and the corresponding values of K and α is 0.0464 cm³/g and 0.76, respectively.²¹

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$$[\eta] = \frac{\sqrt{2(\eta_{sp} - \ln \eta_r)}}{c} \qquad (1)$$

14 $[\eta] = KM^{\alpha}$ (2)

15 CC was determined using the Waltz-Taylor method.²² The purified PA46 (1.0 g) was 16 dissolved in 50 mL benzyl alcohol and the solution was heated to 100 °C. Titration of this 17 solution was performed using a Tacussel electronic PHN 850 titrator, with a 0.1 N 18 KOH/ethanol solution. A control titration was performed using 80 ml pure benzyl 19 alcohol.

20 Glass fiber length measurement. A bum-out test was used to estimate the average GF

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electron microscope.

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length according to a standard procedure. The sample with 10.0 g was kept in a muffle 1 furnace at 650 $\,^{\circ}$ C for 3 h to remove polymer matrix. The lengths of the residual GF were 2 3 measured using SEM (S-3700, Hitachi), with sampling of at least 1000 fibers. Average glass fiber length (\overline{L}) was calculated according to the following equation, 4 where l_N and N was denoted as the specific GF length and the sum of GF, respectively. 5 $\overline{L} = \frac{l_1 + l_2 + l_3 + \dots + l_N}{N}$ (3) 6 7 Percentage of GF length was calculated according to the following equation. N_i was 8 denoted as the number of GF length between specific scope, for example, the length 9 ranged from 0 to 50 um, 50 to 100 um and so on. Percentage $(\%) = \frac{N_j}{N} \times 100\%$ (4) 10 11 Scanning Electron Microscopy. The microstructures of the fractured surface after tensile failure at room temperaturewere gold-sputtered and studied with a scanning 12

Tensile testing, flexural testing and impact strength studies. Tensile and flexural properties of the samples were measured on a Universal test machine (Reger, RGT-20A) according to ASTM D638-03 and ASTM D790-07, respectively. The notched impact strength was performed by ASTM D256 using an impact tester (Sans Tested, ZBC1400-2). In all cases, five specimens were tested to determine an average value.

19 Rheological measurement. The shear viscosity of samples was measured using a

capillary rheometer (Rheograph 25, GÖTTFERT). The capillary die used had a
length-to-diameter ratio of 30:1. Before testing, samples were dried at 80 °C for 8 h. The
test temperature was 290 °C, 295 °C, and 300 °C, respectively. The shear stress ranged
from 4.4 to 29.5 bar and shearing rate was from 9 to 1040 S⁻¹.

Thermal properties measurement. RPA46-F6 and its extruded products were crashed
by high-speed knapper and dried at 80 °C for 8 h. TGA analysis was carried out using a
Perkin-Elmer Pyris thermal analyzer under air atmosphere at a heating rate of 10 °C/min
and the scanning temperature range was from 50 to 600 °C. HDT under loading (1.82
MPa) was performed according to ASTM D-648 (Sans Tested, ZKW1302-1). More than
three specimens were tested determine an average value.

Flame retardant measurement. The LOI value was measured according to ISO 11 4589-1984 and the dimension of all the samples were $130 \times 6.5 \times 3.2 \text{ mm}^3$, 25 specimens of 12 each sample have been used to LOI test. The vertical burning tests were performed 13 according to UL-94 and the dimension of all the samples were $127 \times 10 \times 0.75$ mm³. GWIT 14 is defined as the temperature, which is 25°C higher than the maximum temperature of the 15 glow wire, and does not cause ignition of the material during three subsequent tests. 16 Ignition is defined as the appearance of flames on the surface of the specimen for at least 17 5s. GWIT tests (775 °C) were performed in accordance with IEC 606955-2-13:2000, 18 respectively, the dimension of all the samples were $80 \times 80 \times 0.75$ mm³, and the values 19 reported are average from five tests. Weight loss (wt) % of each sample was calculated by 20

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1 Equation (5), where the M_0 was the weight of untested specimen and M_1 was the weight

2 of tested specimen. All measurements were performed with five specimens and averaged.

3 Weight loss
$$(wt)\% = \frac{(M_0 - M_1)}{M_0} \times 100\%$$
 (5)

Bromine content determination. XRF (Shimadzu EDX-720) was applied to measure
the content of bromine of RPA46-F6 and its extruded samples, 1.0 g powder sample was
tested, the values reported reflected an average from ten tests and the voltage and current
was 5 kV, 5 uA, respectively.

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9 **Results and Discussion**

Carboxyl content and viscosity-average molecular weight. The carboxyl content (CC), 10 intrinsic viscosity ([η]) and viscosity-average molecular weight ($\overline{M_{\nu}}$) of RPA46-F6 and 11 its extruded samples were investigated. Table 1 and 2 show the percentage (%) of 12 variation in CC and $\overline{M_{\nu}}$ of its extruded products, respectively. It was clear that the PA46 13 macromolecular was degraded by extrusion. The degradation degree of PA46 14 macromolecular was dependent on the screw shear force. The CC and $\overline{M_{\nu}}$ of PA46 was 15 8.06×10^{-5} mol/g and 3.62×10^{4} for RPA46-F6. For RPA46-F6-S50, the CC increased and 16 $\overline{M_{y}}$ decreased slightly. When the L/D of single screw increased from 10 to 40, the % of 17 increment in CC, and the % of decrement in $\overline{M_{y}}$ of RPA46-F6-S40 increased, 18 significantly. According to the results, undesired macromolecular degradation of PA46 19 during extrusion processes can be reduced by decreasing the screw length or increasing 20

the depths of the gaps, so that the maximal shear force was lowed. When RPA46-F6 was
extruded by S50, the suffering of shear force was lower than other single screws due to
its high depths of the gaps and short screw length. ¹⁹ As a result, the degree of degradation
of PA46 macromolecular was quite lower. When RPA46-F6 was extruded by S30 and
S40, the shear force increased with the increase of the screw length and the decrease of
the depths of the gaps. Therefore, the degradation degree of PA46 macromolecular
increased. The RPA46-F6-T30 showed the highest CC and the lowest $\overline{M_v}$ value,
whereas its absolute value of ΔCC and $\Delta \overline{M_{\nu}}$ were the highest. As the shear force
generated by twin screw extruder was higher than that produced by single screw extruder,
the shear by screw snipped the PA46 macromolecular chain, and degree of degradation of
PA46 macromolecular increased with increasing shear force. As a result, severe
$\overline{M_{\nu}}$ degradation of RPA46-F6-T30 occurred because of the larger strain generated by
twin-screw extruder. The degradation induced the formation of short chain fragments
with carboxylic end groups. ²³ Therefore the ΔCC of RPA46-F6-T30 was higher than that
of other extruded products. The results revealed that the degree of degradation of PA46
macromolecular during extrusion processes could be reduced by decreasing the shear
force. It can be fulfilled by decreasing screw length and residence time, and increasing
the depths of the gaps. ^{18, 19}

Average length of glass fiber (\overline{L}) and its distribution. The mechanical properties of fiber reinforced thermoplastic composites are influenced by the length distribution of the

1	fibers ¹² . Thus, the variation of GF breakage and length distribution of GF during
2	extrusion was investigated. The \overline{L} of RPA46-F6 and its extruded products are listed in
3	Table 1(list 3), and % of the decrement of \overline{L} of extruded products are listed in Table 2.
4	The \overline{L} of RPA46-F6 was 266.2 um, while the $\overline{L}_{and} \Delta \overline{L}$ of RPA46-F6-S50 was 246.1
5	um and -7.6%, respectively. When RPA46-F6 was extruded by S30 and S40, the $\Delta \overline{L}$ of
6	RPA46-F6-S30 and RPA46-F6-S40 were around -13.7% and -20.2%, respectively. This
7	indicated that the flow stresses in the melt were responsible for the \overline{L} reduction during
8	the extrusion. According to Von Turcovich and Erwin, ²⁴ the following three principle
9	mechanisms, namely fiber-fiber interactions, fiber-equipment surfaces interactions and
10	fiber interaction with the polymer, explain the phenomena of \overline{L} decrease. The main
11	factor is fiber-polymer interaction, which is related to the melt rheology. ²⁵ During the
12	processing, when the RPA46-F6 passed the solid convey zone and melting zone all the
13	interactions occurred resulting in the \overline{L} decrease. Furthermore, shear forces generated by
14	the extruder that acted the molten polymer are transferred to the fibers leading to the
15	breakage of these fibers as the shear stresses overcome the fiber tensile strength. ²⁶ It
16	could conclude that the degree of fiber breakage increased with screw length, residence
17	time and the shear intensity. The significant decrease of \overline{L} was presented in
18	RPA46-F6-T30 and its $\Delta \overline{L}$ increased to 33.2%. The larger shear force generated by
19	twin-screw extruder corresponded to the severe breakage of GF ^{16, 27} . Similar to those of
20	the PA46 degradation, the degree of GF breakage in RPA46-F6-T30 was much higher
21	than that of other extruded products extruded by single screw.

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1 Kelly and Tyson²⁸ proposed a fundamental relationship between the critical fiber length 2 (L_c) and the interfacial shear strength (τ_i) as given below: 3 $L_c = \frac{\delta_f d}{2\tau}$ (5) 4 5 Eq.5 clearly indicates that interfacial shear strength increases with the decrease of the critical fiber length thereby enhances the composite strength where d is the fiber diameter 6 (9.6 um determined by SEM and calculated from 50 fibers). According to Thomason.²⁹ 7 the value of τ_i is 32.9 MPa. When the glass fiber was processed, it loses strength in the 8 long term as a result of weathering and the fiber fracture strength (δ_f) of glass fiber is 9 1700 MPa.³⁰ From these given parameters, it could be calculated that L_c was 248.0 um 10 11 from Eq.5. 20 20 25 -C:RPA46-F6-S30 A: RPA46-F6 B:RPA46-F6-S50 V, 18 v 18 12 16 16 20 14 14 ° 12 Centage (%) 13 10 itage 6 14 4 2 50 100 150 200 250 300 350 400>450 100 150 200 250 300 350 400>450 100 150 200 250 300 350 400>450 50 50 Distribution Range of Fiber Length (um) 15 Distribution Range of Fiber Length (um) Distribution Range of Fiber Length (um) E:RPA46-F6-T30 25 -D:RPA46-F6-S40 20 20 16 Centage (%) Centage (%) 17 5 18 0 50 100 150 200 250 300 350 400 >450 Distribution Range of Fiber Length (um) 100 150 200 250 300 350 400 50 19 Distribution Range of Fiber Length (um) 20 Figure. 2. The glass fiber length distribution of RPA46-F6 and its extruded products. (A): RPA46-F6; (B): RPA46-F6-S50; (C): RPA46-F6-S30; (D): RPA46-F6-S40; (E): 21

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RPA46-F6-T30

1	The degradation of GF during extrusion could be characterized by fiber length
2	distribution as shown in Figure 2. The percentage of glass fibers (V_j) whose length was
3	higher than L_c and ΔV_j of extruded products are listed in the Table 3 and 4. The V_j
4	decreased with increasing screw length and shear intensity. It meant that only some part
5	of the GF work as reinforcement agent. As it can be seen from Figure 2(A), $V_{\rm j}$ of
6	RPA46-F6 was 0.535, with 8.1% of the lengths larger than 450 um. The GF length
7	distribution enabled these GF acting as reinforcement of the RPA46-F6. Since the stress
8	transfer between GF and matrix which was also related to L_c , the decrease in the GF
9	length leads to only a smaller number of the GF working as reinforcement. Compared to
10	those of RPA46-F6, the V_j of RPA46-F6-S50 decreased to 0.413 resulting in 6.1% of the
11	lengths longer than 450 um. Furthermore, the percentage of the GF with longer lengths
12	(higher than 450 um) and V_j decreased significantly as presented in Figure 2(C)~(E) and
13	Table 3. During the extrusion, the shear force (fiber-polymer interaction) in the screw
14	preplasticization would damage the GF, inducing further fiber attrition through fiber-fiber
15	interactions and fracture at the solid-melt interface. ³¹ As the length of screw
16	preplasticization in S50 was lower than the other single screw extruders, the degree of
17	fiber breakage of RPA46-F6-S50 achieved a lower value. The $V_{\rm j}$ of RPA46-F6-S50 was
18	higher than other extruded products, while RPA46-F6-T30 owned the lowest $V_{j}\!.$ The
19	results revealed that the degree of GF breakage during extrusion processes could be
20	reduced by decreasing screw length and residence time, or increasing the depths of the
21	gaps.

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Morphology. In order to study the difference in reinforcing effect of GF to PA46 matrix, 1 the fracture surface of tested specimens were analyzed by SEM. Figure 3 show that in 2 there were considerable fiber that were pulled out from RPA46-F6 and its extruded 3 samples, indicating the GF fracture occurred in these samples. The GFs were relatively 4 finely dispersed in the PA46 composites. The matrix show some crack with ductility of 5 PA46 and the long fibers with adherent protrudes from the surface leaving holes in the 6 7 matrix, from which fibers had been pulled out. Fiber breakage, which reduced the average aspect ratio of the GF, occurred in the extruded samples. It is well known that 8 shear stress concentration occurs near the fiber ends where failure initiates.²⁸ The path of 9 the crack propagation is either along the interface or through the matrix depending upon 10 the bond strength. The high-magnification images (Figure. 3 B, D, F, H and J) emphasize 11 12 GF surfaces were coated with PA46 residues, confirming good interfacial adhesion between the GF and polymer matrix. The fracture surface of RPA46-F6 and 13 14 RPA46-F6-S50 (Figure. 3 B and D) was deformed, indicating the matrix with higher 15 toughness proved. With an increase of screw shear, the ductile failure of the PA46 matrix changed to a brittle one, which was associated with the formation of crazing. More craze 16 remnants on the fracture surface were clearly visible both in PA46 matrix (Figure 3 (H 17 18 and J)), suggesting that the matrix continuity was destroyed. It was found that the fracture surfaces of RPA46-F6-S40 and RPA46-F6-T30 matrix took place while the composite 19 was cryofractured. However, the interfacial interaction between GF and PA46 was not 20

- 1 changed due to a good interaction in PA46/GF. $\overline{M_y}$ of PA46 degraded during extrusion
- 2 that reduced the PA46 chains interaction. As a result, the rigidity of RPA46-F6-S40 and
- 3 RPA46-F6-T30 matrix increased.



Figure. 3. SEM of RPA46-F6 and its extruded products. (A), (B): RPA46-F6 (×200),
(×1k); (C), (D): RPA46-F6-S50 (×200), (×1k); (E), (F): RPA46-F6-S30(×200), (×1k); (G),
(H): RPA46-F6-S40(×200), (×1k); (I), (J): RPA46-F6-T30(×200), (×1k)

The fractured surface of RPA46-F6-S50 depicted the good adhesion between GF and 14 PA46 matrix, with few crack remnants and pull-out fiber on the fracture surface. The 15 degree of GF breakage was lower level. With the further increase of single screw length, 16 the proportion of pulled-out GF and the degree of GF breakage increased significantly. 17 The pulled-out fiber portions and the degree of GF breakage of RPA46-F6-T30 were 18 higher than those of other samples. It was hard to find adherent protrude GFs in its 19 fractured surface. The variation of fractured surface was attributed to the shear force 20 generated by twin screw extruder was higher than that of single screw extruder. The fiber 21 breakage would decrease the reinforcing effect of GF in the fiber reinforced polymeric 22

1 composites ³² that was observed in the extruded samples in different degrees.

As the S50 obtained the lowest shear force due to the lowest L/D, the reduction of $\overline{M_V}$ and \overline{L} , and the pulled-out portion of GF in RPA46-F6-S50 was lower than that in other extruded products. It favored the improvement of the mechanical and thermal properties of RPA46-F6.

6 Mechanical properties. If GF is employed in an amount below some critical value, the 7 mechanical properties of the polymer composite was not improved. The degradation of 8 \overline{L} and V_j has a negative effect on mechanical and thermal properties of the polymer 9 composite.¹² It is well known that both tensile behavior²⁸ and impact strength³³ are 10 closely related to the GF length and the fiber orientation to the loaded direction. Actually, 11 once the V_j decreases, the part of the GF with length shorter than L_c could not reinforce 12 the thermoplastic, resulting a decreased mechanical properties of the composites.

The notched impact strength, tensile and flexural strength of RPA46-F6 and its 13 14 extruded samples are listed in the Table 3, their decrement percentage are listed in Table 4. The decrement % in mechanical properties of RPA46-F6-S50 was lower than other 15 extruded products and the % of decrement was less than 5.8%. From Table 1 and 2, 16 Figure 2B and 3C and D, it was seen that, $\overline{M_y}$, \overline{L} and V₁ of RPA46-F6-S50 was much 17 higher than RPA46-F6. RPA46-F6-S50 had the good interfacial adhesion without 18 significant pulled-out-glass fibers and craze remnants on the fracture surface. Moreover, 19 the PA46 matrix obtained the ductile fractures. As the screw length and the shear intensity 20 increased, RPA46-F6-S40 decreased significantly in mechanical properties and their 21

1	corresponding Δ Impact strength (%) of RPA46-F6-S40 increased to 15.3%. Figure 3
2	revealed the fractures of PA46 matrix changed from ductile to brittle with the increase of
3	screw shear force. It was found that the fracture surfaces showed more interface
4	debonding and less matrix deforming than those of RPA46-F6. They would cause the
5	decreased flexibility of extruded samples, which is related to the reduction of impact
6	strength. Furthermore, the increased shear force by longer screw was a result of the
7	damaged GF. The pulled-out GF and also breakage on the fracture surface accelerated at
8	that stage. The decrease of mechanical properties including Δ Impact strength(%), Δ
9	Tensile strength(%) and Δ Flexural strength(%) of RPA46-F6-T30, significantly increased
10	to -29.6%, -26.0% and -23.1% respectively. RPA46-F6-T30 matrix displayed a rigidity
11	fracture. \overline{L} and V _j of RPA46-F6-T30 was the lowest of all the samples, implying that
12	reinforced efficiency of glass fiber is in the lowest value. ^{17, 30} Moreover, the pull-out GF
13	had been observed in various GF reinforced thermoplastics as the dominant failure
14	revealing that the RPA46-F6-T30 specimen showed a brittle fracture mechanism proved
15	by Figure 3 (I and J). As the shear strain increased, $\overline{M_{\nu}}$, \overline{L} and V _j of the RPA46-F6
16	extruded products decreased, and these changes in turn led to the rather lower mechanical
17	properties of RPA46-F6-T30. ²² Due to the high shear forces acting by twin screw in the
18	molten polymer and then transferred to the fibers, the reinforcement effect of the fibers
19	decreased. It could be proved by the impact and tensile mechanical properties of the
20	composites, consequently. Additionally, polymer undergoes thermo-mechanical
21	degradation during processing which was also responsible for the changes in the 19

mechanical properties. When the RPA46-F6 was extruded by S50, its $\overline{M_{y}}$, \overline{L} and V₁ 1 could obtain rather high values, and they were contributed to the least decrement in 2 mechanical properties. 3

Rheological behaviour. For RPA46-F6, the process temperature must be carefully 4 selected. When the thermal process temperature was lower than melt temperature, the 5 viscosity of RPA46-F6 was higher which causing the difficulty during mold injection.³⁴ 6 7 Higher temperature could decrease the viscosity, however, it will induce the thermal decomposition of PA46. From Eq. 6, it could be deduced that the viscosity of polymer 8 9 was depended on the temperature. If in the low temperature range the activation energy (En) achieved an increase, whereas in the high temperature range, the activation energy 10 decreased, however. 11

12

15

1000

Where η_{α} , R and T is the viscosity, gas constant, and temperature on the absolute 13 scale, respectively. 14

B: T=295 °C

 $Ln \eta_{\alpha} = \frac{E_n}{RT} + Ln A$

1000

(Pa.S-1)

A: T=290°C

(6)

100

C: T=300 °C



Figure.4. Rheological behaviour of RPA46-F6 and its extruded products at different 18 temperature. A: 240 °C; B: 250 °C; C: 260 °C. 19

Shear viscosity versus shear rate for RPA46-F6 and its extruded samples at 290 °C, 295 20

°C, 300 °C are presented in Figure 4. All samples exhibited shear thinning behavior and 21

elucidated the characterization of Non-Newtonian Fluid. The magnitude of the shear
viscosity was strongly dependent on the temperatures. Moreover, a significant reduction
in viscosity of RPA46-F6 and its extruded products at higher shear rate was observed.
When the stress is high enough to destroy the formed network by fiber-matrix-fiber and
fiber-fiber collision, then the molten chains are free to move³⁵ and the shear viscosity of
extruded products decreases.

With increasing shear force, the shear viscosity of RPA46-F6 extruded products 7 decreased. At the same temperature and shear rate, the shear viscosity of RPA46-F6-T30 8 was lower than other samples. In general, two reasons were responsible for the reduction. 9 The one is polymer with lower $\overline{M_{y}}$ shows lower shear viscosity. The other one is that 10 when GF length reduces, opportunities for fiber orientation and fiber-matrix interaction 11 12 decreases, resulting in decreasing the shear viscosity of composites with shorter GF length³⁶. By the result of $\overline{M_y}$ and \overline{L} measurement, it revealed that $\overline{M_y}$ and \overline{L} of 13 RPA46-F6-T30 decreased significantly, compared to that of RPA46-F6. As a result, the 14 shear viscosity of RPA46-F6-T30 decreased. Further, the results demonstrated 15 granulation had the advantages of reducing shear viscosity, improving the flow properties 16 of RPA46-F6, decreasing the injection defects such as weld line, short molding, and GF 17 emergence on the surface.¹² 18

19 **Thermal stability.** For flame-retardant polymer composites, DBDPE decomposed at 20 lower temperatures than the flammable polymer.³⁶ It caused the flame-retarding effect 21 and decreasing the onset degradation temperature of composites. Previous studies 21

suggested that the onset degradation temperatures assessed at 5% of loss ratio ($T_{5\%}$) were 1 360.5 °C and 395.2 °C for DBDPE and PA46, respectively.^{37, 38} TG and DTG of 2 RPA46-F6 and its extruded products are shown in Figure 5. All samples exhibit two-step 3 degradation behaviors. This type of decomposition was caused by a depolymerization 4 mechanism was commonly observed in aliphatic polyamides.^{34, 35} The thermal 5 decomposition temperature and decrement value are listed in the Tables 5 and 6. The first 6 stage of degradation ranged from 50 °C to 150 °C and the lose weight was about 7 $1.5\pm0.3\%$. This observation was attributed to the trace amount of water in the samples. 8 9 The thermal decomposition temperature (at a weight loss of 5%, $T_{5\%}$) of RPA46-F6 and its extruded products ranged from 369 °C to 375 °C. The temperature of maximum 10 decomposition (T_{max}) was between 392 °C and 399 °C, and the total mass loss reached 11 over 40% of its final point (600 °C). The terminal residue at 600 °C was consisted of GF 12 and the char from PA46, and Sb_2O_3 . $T_{5\%}$ and T_{max} of the extruded products were lower 13 14 than those of RPA46-F6, indicating that the extrusion process leads to the reduction of 15 thermal stability of RPA46-F6. Table 6 revealed $\Delta T_{5\%}$ and ΔT_{max} of the extruded products increased with the increase of shear force. For example, $\Delta T_{5\%}$ and ΔT_{max} of 16 RPA46-F6-S50 were -0.3 ^{o}C and -1.1 ^{o}C , respectively. $\Delta T_{5\%}$ and ΔT_{max} of RPA46-F6-T30 17 were -4.8 °C and -5.8 °C, respectively. According to Bikiaris et al,⁴⁰ the higher CC may 18 contribute to lower thermal stability of polyester. So it could draw the conclusion that 19 $\Delta T_{5\%}$ and ΔT_{max} of extruded products were depended on CC. Fortunately, the lowest $T_{5\%}$ 20 was 369.3 °C, which guaranteed the RPA46-F6-S50 to obtain good thermal stability as 21 22

2

micro-injection molding.

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1 the melt processing temperature of RPA46-F6 ranged from 295 °C to 315 °C during the



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Flame retardant properties. The experiment results about the flame retardant properties 1 are summarized in Tables 7 and 8. They revealed that extruded products could maintain 2 the original flame retardant properties of RPA46-F6. The LOI of RPA46-F6 and its 3 extruded products were higher than 36.0 and meanwhile, they could maintain V-0 4 classification in the UL-94 test. From TG analysis and XRF determination of RPA46-F6 5 6 and its extruded products, they confirmed the decrease of Br content was negligible. The Br content of RPA46-F6 and its extruded products was higher than 6.35%, which ensured 7 the RPA46-F6 could achieve V-0 classification¹² and 36.0 LOI. The reason was DBDPE 8 9 could exhibit good thermal stability during the extrusion and was independent to screw shear force. As a result, extruded RPA46-F6 could maintain the original flame retardant 10 properties. 11



1	specimen for at least 5s. ⁴¹ Acquasanta et al ⁴² proposed a model (presented in the Figure 6)
2	to explain the polymer behaviour during glow wire test as follows: 3 zones can be
3	recognised in the part of the specimen which was in contact with the wire. A first zone at
4	temperature above the decomposition temperature (Pyrolysis Zone); A second zone where
5	the material is just melted, and the degradation is slower (Melting Zone); A third zone
6	where the temperature is not high enough to melt the polymer, but the material can be
7	deformed because it is above its heat distortion temperature (Softening Zone). In their
8	opinion, the processes taking place in the pyrolysis zone mainly determine the test results.
9	Furthermore, both mechanical and rheological properties can have an important influence
10	on GWIT properties. ⁴² The data of GWIT are listed in the Table 8 which confirmed all
11	extruded products pass the GWIT (775 $^\circ C$) test. By the results of the LOI and UL-94 test,
12	it demonstrated that all the extruded products could maintain the original flame retardant
13	properties of RPA46-F6. During the GWIT (775 $^{\circ}$ C) test, all of them could not be ignited
14	by the hot glow wire, as a result, the ignition time, extinguish time, ignition duration of
15	all samples was 0. Compared with that of RPA46-F6, the weight loss of extruded
16	products increased from 1.56% to 2.52%, and the RPA46-F6-T30 obtained the largest
17	value. With increasing shear force, the mechanical properties and shear viscosity of
18	extruded products decreased. According to the Acquasanta model, ⁴² Pyrolysis Zone,
19	Melting Zone and Softening Zone of extruded RPA46-F6 were longer than those of
20	RPA46-F6, respectively. The mechanical properties and shear viscosity of RPA46-F6-T30
21	were much lower than those of RPA46-F6, implying that it was easier to soften and melt 25

than other extruded products during GWIT test. The weight of melt RPA46-F6-T30
wrapped around the hot tip of the wire increased, as a result the weight loss increased.
Despite that, the extruded products could reach a GWIT higher than 775°C and pass the
test.³⁵ The results of flame retardant properties indicated that the flame retardant
properties of extruded products could match the requirement for electrical insulation
application.¹²

7 Conclusions

8 The shear force of RPA46-F6 suffered increased with the increase of shear strain and 9 L/D of screw. The increasing shear force caused the degree of GF breakage and PA46 10 degradation to increase. In the case of the samples prepared by single-screw 11 extruder, $\overline{M_v}$, \overline{L} and V_j gradually decreased with increasing screw length. However, the 12 highest % of decrement of $\overline{M_v}$, \overline{L} and V_j were observed for the RPA46-F6 extruded by 13 T30. The variance of the reductions would affect the properties of RPA46-F6 in different 14 trends.

15 (1). The reductions of V_j , $\overline{L}_{and} \overline{M_v}$, which were responsible for lowering the 16 mechanical and thermal properties and shear viscosity of RPA46-F6 extruded products. 17 Fortunately, RPA46-F6-S50 obtained high mechanical and thermal properties due to the 18 least decrement in V_j , $\overline{L}_{and} \overline{M_v}$. Compared to those of RPA46-F6, the maximal 19 decrement percent of mechanical properties were only 5.8%, with the negligible 20 reduction in $T_{5\%}$ and HDT.

21 (2). Due to non decomposition of the DBDPE during the extrusion, RPA46-F6

extruded products could achieve a UL-94 V-0 classification, obtain a higher LOI value
(>36), and also pass GWIT (775°C) test.

(3). All results illuminated that single screw with high depths of the gaps and shorter
length was the optimal extruder to recycle RPA46-F6. The mechanical, thermal and flame
retardant properties of RPA46-F6-S50 were in acceptable values. Thus, RPA46-F6-S50
could be reused solely or added at high proportion to injection molded as relay, electrical
connectors, switch and other electronics accessory.

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14 Notes and references

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		products.		
Samples	CC (×10 ⁻⁵ mol/g)	$[\eta]$ (mg/dl)	$\overline{M_v} \times 10^4$	\overline{L} (um)
RPA46-F6	8.06	1.35	3.62	266.2
RPA46-F6-S50	9.12	1.29	3.38	246.1
RPA46-F6-S30	9.85	1.24	3.20	229.6
RPA46-F6-S40	10.02	1.19	3.04	212.3
RPA46-F6-T30	10.56	1.07	2.64	177.7

Table 1. The carboxyl content (CC), intrinsic viscosity ($[\eta]$), viscosity-average molecular weight $(\overline{M_{\nu}})$ and length of glass fiber (\overline{L}) of RPA46-F6 and its extruded

Table 2. Decrement or increment percents in CC, $[\eta]$, $\overline{M_{\nu}}$ and \overline{L} of extruded

	produ	cts. (RPA46-F6 a	is the control)	
Samples	ΔCC (%)	$\Delta[\eta]$ (%)	$\Delta \overline{M_v}$ (%)	$\Delta \overline{L}$ (%)
RPA46-F6-S50	+13.2	- 7.4	-6.6	- 7.6
RPA46-F6-S30	+22.2	- 12.6	-11.6	- 13.7
RPA46-F6-S40	+24.3	- 16.3	-16.1	- 20.2
RPA46-F6-T30	+31.1	- 28.1	-27.1	- 33.2

All the Decrement or increment percents were calculated by the following Equation: $\Delta V(\%) = \frac{V_n - V_0}{V_0} \times 100\%$

 V_0 is denoted as all the parameters of RPA46-F6, V_n is that of extruded product.

products.					
Samples	V_j	Impact strength (J/m)	Tensile strength (MPa)	Flexural strength	
				(MPa)	
RPA46-F6	0.535	94.6±2.6	157.3±4.8	227.5±5.3	
RPA46-F6-S50	0.436	89.1±2.7	150.5±5.6	225.2±6.6	
RPA46-F6-S30	0.231	85.3±1.9	148.1±3.6	222.2±4.6	
RPA46-F6-S40	0.123	80.1±2.1	144.2±4.6	218.3±5.2	
RPA46-F6-T30	0.155	65.5±3.8	116.4±3.6	174.9±4.7	

Table 3. The V_j and mechanical properties of RPA46-F6 and its extruded products.

 Table 4. Decrement percents in the V_j and mechanical properties of extruded products (RPA46-F6 as the control)

		· · · · · · · · · · · · · · · · · · ·	
ΔV_{j}	Δ Impact strength	Δ Tensile strength	Δ Flexural strength
(%)	(%)	(%)	(%)
- 18.5	-5.8	-4.5	-1.0
- 56.8	- 9.8	- 5.8	- 2.3
- 77.0	- 15.3	- 8.3	- 4.0
-71.0	-29.5	-26.0	-23.1
	ΔV _j (%) - 18.5 - 56.8 - 77.0 -71.0	$\begin{array}{c c} \Delta V_{j} & \Delta Impact strength \\ \hline (\%) & (\%) \\ \hline -18.5 & -5.8 \\ -56.8 & -9.8 \\ -77.0 & -15.3 \\ -71.0 & -29.5 \end{array}$	$\begin{array}{c c c c c c c c c c c c c c c c c c c $

Samples	T _{5%} (°C)	$T_{max}(^{o}C)$	HDT (℃)
RPA46-F6	374.1	398.6	278.3
RPA46-F6-S50	373.8	397.5	278.1
RPA46-F6-S30	372.5	397.3	275.4
RPA46-F6-S40	371.7	396.5	274.8
RPA46-F6-T30	369.3	392.8	272.2

Table 5. T_{5%}, T_{max} and HDT of RPA46-F6 and its extruded products

 Table 6. Decrement value in the thermal decomposition temperature and HDT of extruded products (RPA46-F6 as the control)

extructure produces (iteration i o us the control)						
Samples	$\Delta T_{-5\%}(^{\circ}C)$	$\Delta T_{max}(^{o}C)$	ΔHDT (°C)			
RPA46-F6-S50	- 0.3	-1.1	- 0.2			
RPA46-F6-S30	- 1.6	-1.3	- 2.9			
RPA46-F6-S40	- 2.3	-2.1	- 3.5			
RPA46-F6-T30	- 4.8	-5.8	- 6.1			

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Samples	LOI	UL94	Br content (%)
RPA46-F6	36.8	V-0	6.45±0.05
RPA46-F6-S50	36.7	V-0	6.38±0.04
RPA46-F6-S30	36.7	V-0	6.39±0.05
RPA46-F6-S40	36.6	V-0	6.41±0.06

V-0

6.41±0.05

36.6

RPA46-F6-T30

Table 7. Flame retardant properties of RPA46-F6 and its extruded products.

 Table 8. GWIT of RPA46-F6 and its extruded products.

Samples	Ignition time(s)	Extinguish time(s)	Ignition duration(s)	Weight loss(wt)%
RPA46-F6	0	0	0	1.56±0.32
RPA46-F6-S50	0	0	0	1.62±0.36
RPA46-F6-S30	0	0	0	1.75±0.32
RPA46-F6-S40	0	0	0	1.81±0.35
RPA46-F6-T30	0	0	0	2.52±0.41

Content entry

An optimal extruder to recycle glass fiber reinforced flame retardant nylon46 with minimum decrement in the mechanical, thermal properties.

