THERMAL DEGRADATION BEHAVIOUR OF CARBON FIBRE WASTE AS A FILLER IN POLYMER HYBRID COMPOSITES

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CERTIFICATION OF APPROVAL

Thermal Degradation Behaviour of Carbon Fibre Waste as A Filler in Polymer Hybrid Composites

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Syazwan Fikri Bin Mohammad

ABSTRACT

Carbon Fiber and Glass Fiber Reinforced Polypropylene (CFGFRP) is a hybrid polymer composite has been one of crucial material in many modern products. Nevertheless, there are several downsides of CF which degrading its astonishing mechanical properties, hardening the material, and reduce material flexibility. Therefore, recycled Carbon Fiber and recycled Glass Fiber Reinforced Polypropylene (rCFrGFRP) is an initiative for future cost saving during production. In this paper (rCF/rGF/PP) hybrid polymer composites were prepared under injection moulding process. The thermal degradation behaviour of this composite was investigated considering the combined effect of rCF and rGF on PP matrix via thermogravimetric analysis (TGA) and oven heating process. Hardness tests were taken to investigate mechanical properties effect of the composite. Finally, these can be showing the combination effect of rCF and rGF on thermal degradation of the composite.

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CHAPTER 1: INTRODUCTION

1.1 Background Study

Carbon fiber (CF) is a strong composite material which was found by Thomas Edison while he was inventing light bulb (American Chemical Society, 2003). However, it was not established in the bulb technology from the time when the tungsten filament was created. After decades, the used of CF continue to fulfil in modern industry especially in aerospace and automotive. Astonishing of stiffness, tensile strength, chemical resistance, temperature and low weight are the important elements that made the material a strong competitor in modern technology (Huang, 2009). Additionally, CF is also widely used as a filler in hybrid polymer for example carbon fiber reinforced polymer (CFRP).

Hybrid polymer composite is made up at least 2 filler elements for instance carbon fiber (CF), glass fiber (GF), polypropylene (PP) to form carbon fiber reinforced polymer (CFRP).

1.2 Problem Statement

There are various downsides regarding to Carbon Fiber Reinforced Polymer (CFRP) such as properties degradation after expiring date and high manufacturing cost. The first encountered issue is mainly because when after this hybrid polymer past it expiring date, it will cause the material to harden, inflexible and unreliable for its astonishing mechanical properties. However, many studies support by recycling CF, it will reproduce new material with similar mechanical properties (Baker & Rials, 2013). Thus, this invention can be beneficial to local and global material advancement.

As the used of carbon fiber (CF) is growing, it will produce CF waste intensively. Based on a paper research from General Motors Research & Development Center to optimize the carbon fiber properties by studying various advanced fabrication of the material. The estimation of CF demand in aerospace, industrial and sport equipment are expending by 1.1 estimation ratio (Huang, 2009). Even though, the demand of CF is expending, the manufacturing cost is still beyond our expectation. It is mainly because of the difficulties in producing the CF lignin. Researchers from The University of Tennessee explain that the complexity of producing CF lignin to meet price and strength condition is the reason to overpriced of CF material (Baker & Rials, 2013).

To reduce the cost of production, recycling of CF is an alternative method that is being used in recent years. Besides, Pimenta and Pinho (2011) on their research of recycling Carbon Fiber Reinforced Polymers (CFRP) in major application found that countless initiatives have been taken from European, Japanese, USA and UK material companies in using CF waste for CFRP production for cost reduction of hybrid polymer composite. Thus, it is important to study the behaviour of CF in hybrid polymer composite.

1.3 Objective

The objectives of this research are:

i. To conduct thermal test on the designed composites and study on material thermal degradation.
ii. To conduct Rockwell hardness test on the designed composites and study on the effect of thermal degradation towards hardness.

1.4 Scope of Study

The scope study of this research can be divided into these components:

i. To observe mass loss due heating process through heating test and Thermogravimetric Analysis (TGA) of the composites.
ii. To conduct Rockwell hardness test.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction of Carbon Fiber (CF)

Carbon Fiber (CF) is a strong material with lighter in weight than common metals. It is generally used in aerospace, automotive sports, sport equipment's and wind turbine due to its unique mechanical properties which are high strength and very light. Hence, the demand of this material composite has been increased throughout years to suite human needs. McConnell (2008) in his journal agrees that the increase of carbon fiber is factual and expanding every year. However, he does believe the price of CF would not be affordable in future as the CF supply will not meet demand for at least five years from his finding. The downside of this marvellous material may be high cost of production.

2.2 Recycled Carbon Fiber (rCF)

As the CF advancement rapidly, it will also lead to excessive CF waste. Likewise, Lin and Schlarb (2019) believe that CF waste is proportional to CF demand in 2020 which could reach 160000 tons. This can be an excellent alternative for substituting existence of CF with new material, recycled Carbon Fiber (rCF). rCF is claimed to be similar as original CF's mechanical properties hence the same properties would be conserved in the hybrid composite.

2.3 Recycled Carbon Fiber Reinforced Polymer (rCFRP)

Carbon Fiber Reinforced polymer (CFRP) are common material used in industry. Recently there are two types of composite Reinforced Polymer and Advanced Composite. CFRP can be categorised under Reinforced Polymer because it majorly uses resin such as epoxy, thermoset, and thermoplastic and more as a composite binder. The composition of CFRP can be divided into two types. First is fiber which carry contain special mechanical properties such as high tensile strength. While, another element is matrix which act as the composite binder (refer Figure 2.1).

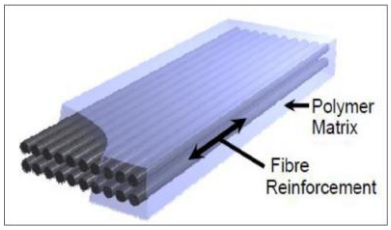

Figure 2. 1: Fiber and matrix illustration of CFRP (V. L. & Akshay, 2017)

CFRP is designed to meet abundant criteria such as low weight, resistance against fatigue, high tensile strength and chemical resistance. Due to these properties, involvement of CFRP is expending outside from early tradition which are aeronautics, motorsports and sport equipment. Now, it enters most of manufacturing which then before was unfamiliar like construction and industrial technology.

Although, popularity of CFRP is expending the major downside of its production is the high cost of production. Producing CFRP requires advanced and expensive technology. Based on demand and supply, CFRP manufacturers are not able to meet consumers' expectation in recent. However, the problem can be solved by recycling CF and use it in CFRP production. Based on previous study mechanical properties such as rCF is claimed to be similar with pure CF. Therefore, this could solve reduce cost production of CFRP.

Degradation of rCFRP is still uncertain. Less researches had been done in several years. Hence, suggestion on looking degradation record from pure CFRP are necessary to obtain numerous knowledge henceforward predicting thermal degradation behaviour in rCFRP after literature reviews.

2.4 Thermal degradation of Carbon Fiber Polymer composite

Due to its massive application in aerospace, aviation, and motorsport, it is important for manufacturer to study thermal behaviour of carbon fiber polymer composite. This is because these area fields cannot afford to lose material performance during application. Such degradation will cause product damage, thus lead to danger for consumers. For example, the use of carbon fiber is intensive in motorsport and aeronautic. Accident is not permitted therefore for those field areas, it is vital to use suitable material to avoid hazard in future (Baker & Rials, 2013; Lin & Schlarb, 2019; McConnell, 2008; Pimenta & Pinho, 2011).

Thermal degradation for carbon fiber polymer composite can be observed in reduction weight percentage (wt%) of the material. The thermal degradation process may reduce the material mass as several components oxidise. The oxidation and chemical reaction with surrounding change the chemical composition after it reaches melting point. For polymer, it is expected to show thermal degradation once the temperature exceeds 160 °C ("Lotte Chemical Titan Titanpro® PP Homopolymer Titanpro® 6331," 2016).

2.4.1 Reduction in Weight Percentage (wt%)

Thermal degradation of carbon fiber are mostly will affect in reduction of weight percentage. It can be obtained by conducting Thermogravimetric analysis (TGA). In this analysis, its study wt% behaviour as temperature differ over time. This quantitative experiment is generally to study thermal degradation of material. There are several studies regarding to TGA on CF, GF and PP.

According to Rasana, Jayanarayanan, Deeraj, and Joseph (2019) on their finding regarding to thermal degradation of nano scale multiwalled carbon nanotubes (mwCNTs)/ micro scale Glass fibers (msGF)/ polypropylene (PP) hybrid composite found presence of MWCNT and msGF shifting up thermal degradation effect (Rasana et al., 2019). Several tests were conducted to eight different types of polymer hybrid composite which consisted three MWCNT, one msGF and four hybrid composites (refer Table 2.1). Among eight material, they were subbed into three different thermal comparison which were pure PP versus mwCNT, pure PP versus msGF, and pure PP versus mwCNT/msGF/PP hybrid composite. Well dispersion of mwCNT from (1 wt% to 3 wt%/ N1 to N3) (refer Table 2.2) resulted shifting thermal degradation curve at 11% from the original value. However, at maximum value (5 wt% of mwCNT/ N5) it was suggested the presence of flux might reduce thermal stability as heat dispersion in micro structural was important to ensure degradation at normal condition (refer Table 2.2). Introduction of msGF (20 wt% of msGF/ G) in the hybrid composite showed notable shift compared to pure PP and mwCNT/PP hybrid composite (refer Table 2.2). As two elements were added together, significant improvement of thermal stability had been recorded from (H1 to H2). Nevertheless, beyond 2 wt% of mwCNT in mwCNT/msGF/PP (H3 and H5) hybrid composite didn't boost thermal stability as performed by H2. The highest peak temperature for this experiment was H2 at 466.6 °C with the weight loss rate of 33.7%/min (refer Table 2.2 and Figure 2.2).

Table 2. 1: Sample codes and compounding weight percentage of fillers in polypropylene and composites (Rasana et al., 2019).

Material	Polypropylene (P) (wt%)	Glass fiber (G) (wt%)	MWCNT (N) (wt%)
P	100	0	0
N1	99	0	1
N3	97	0	3
N5	95	0	5
G	80	20	0
H0.5	79.5	20	0.5
H1	79	20	1
H2	78	20	2
H3	77	20	3
H5	75	20	5

Table 2. 2: Non isothermal decomposition parameters of neat P, nano, micro and hybrid CNT composites (Rasana et al., 2019).

Material	T_{onset}(°C)	T_{10}(%)	T_{50}(%)	Dt_{max}(°C)	dw/dt (%/min)
P	400	410	460	444	20.9
N1	425	432.5	455	452.5	28.2
N3	444	450	460	462	34.4
N5	431	437.5	463	460.5	29.9
G	444	449	465	461	26.6
H1	450	452	465	462	27.6
H2	452.5	459	464	466.6	33.7
H3	445	448	469	464.3	40.7
H5	435	442.5	467	462	25.6

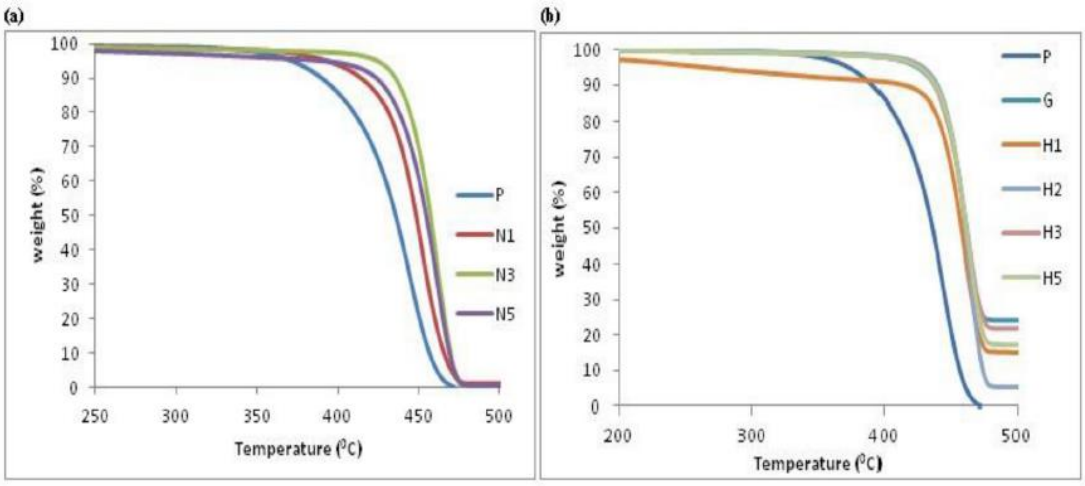

Figure 2. 2: The thermal decomposition profiles of a) neat P and nanocomposites (b) neat P, micro and hybrid composites (Rasana et al., 2019).

Based on Rezaei and his colleagues finding regarding thermal effect of different carbon fiber length in short carbon fiber (SCF) reinforced polypropylene (PP) composites. SCF were chopped into five different lengths (0.5, 1, 3, 5, and 10 mm) to form 10 wt% of SCF in the SCF/PP polymer hybrid composite. It is found that as the carbon length in the hybrid composite increased, the composite would thermally less affected (refer Figure 2.2 and Table 2.1) (Rezaei, Yunus, & Ibrahim, 2009). Additional observation on degradation temperature and wt% loss of the composite sorted by different length showed from 10 wt% to 90 wt% of SCF/PP polymer composite shifted the range from originally from 362 °C – 441 °C to 400 °C – 463 °C (for 0.5 mm length of SCF), 403 °C – 464 °C (for 1.0 mm length of SCF), 406 °C – 468 °C (for 2.0 mm length of SCF), 410 °C – 472 °C (for 5.0 mm length of SCF) and 415 °C – 473 °C (for 10 mm length of SCF) (refer Table 2.1). This experiment may suggest that introduction of rCF could improve PP matrix properties.

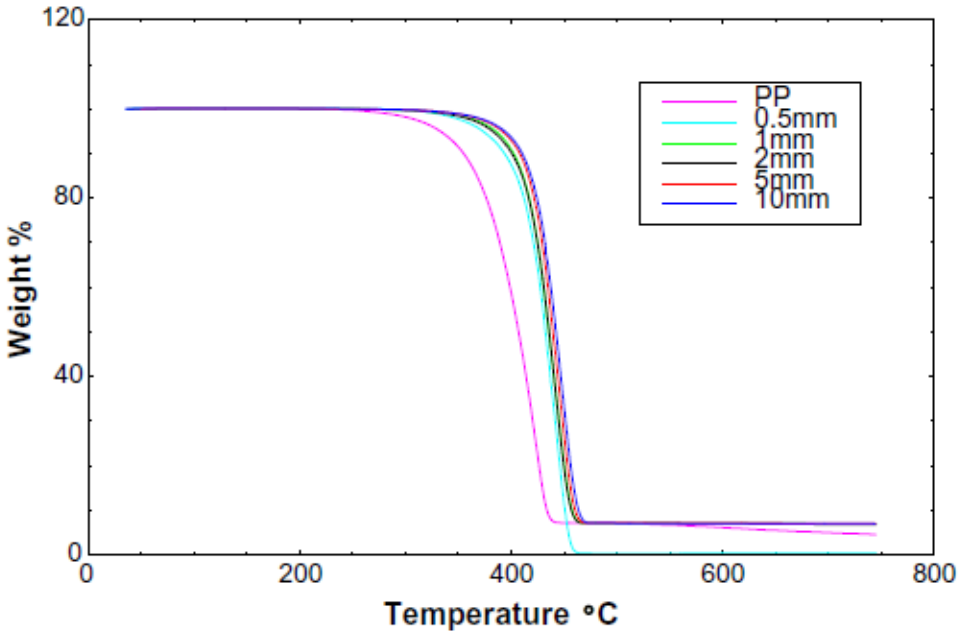

Figure 2. 3: TGA curves of PP Matrix and SCF/PP composites with 10% fiber content (Rezaei et al., 2009).

Table 2. 3: Percentage weight loss in SCF/PP composites at different temperatures (Rezaei et al., 2009).

Weight loss(%)	PP	Degradation temperature				
		SCF/PP composites				
		0.5 mm	1 mm	2 mm	5 mm	10 mm
10	362	400	403	406	410	415
20	378	409	414	418	420	422
30	399	421	425	426	428	432
40	404	424	427	428	430	440
50	413	429	430	431	432	445
60	425	432	433	435	436	450
70	428	437	441	442	445	456
80	434	441	448	451	454	461
90	445	450	453	457	460	465
93	441	463	464	468	472	473

2.4.2 Hardness Performance of the Polymer Hybrid Composite

Adding new element in polymer composite may enhance its mechanical properties from previous. Various study supports similar effect after the polymer had been modified. To understand the effect of it, numerous studies suggest that carbon fiber loading content in the polymer hybrid composite is corresponding to the mechanical properties.

Researchers form Kocaeli University had conducted a study on the effects of maleic anhydride grafted polypropylene (P-g-MAH) as a PP compatibilizer in short carbon fiber (SCF) reinforced polypropylene. The study emphasizes how SCF and P-g-MAH as are essential catalysts to improve the mechanical properties of polymer hybrid composite. Table 2.4 show the composition of CF in seventeen specimens. Generally, CF has higher strength compare to PP matrix. Due to that, tensile strength was also shown great improvement in their finding. Based on Figure 2.6 as the CF content in wt% rose, the tensile strength for the polymer hybrid composite respectively increased (Karsli & Aytac, 2011).

Table 2. 4: Composition of materials in PP/P-g-MAH/CF and DSC Result (Karsli & Aytac, 2011).

No.	Composite	T_m (°C)	ΔH_f (J/g)	X_c (%)
1	PP	167.0	61.6	29.5
2	PP-g-MAH	172.0	67.2	32.1
3	98 PP/2CF	167.1	77.1	37.7
4	96 PP/4CF	165.7	67.8	33.8
5	94 PP/6CF	166.7	62.8	31.9
6	92 PP/8CF	166.0	58.0	30.2
7	80 PP/20CF	166.5	56.2	33.6
8	97.5PP–2.5PP-g-MAH/2CF	166.4	66.5	33.3
9	97.5PP–2.5PP-g-MAH/4CF	165.5	66.1	33.7
10	97.5PP–2.5PP-g-MAH/6CF	166.7	63.2	33.0
11	97.5PP–2.5PP-g-MAH/8CF	168.3	62.5	33.3
12	97.5PP–2.5PP-g-MAH/20CF	167.0	42.2	25.9
13	95 PP–5.0PP-g-MAH/2CF	168.3	68.6	35.2
14	95 PP–5.0PP-g-MAH/4CF	165.2	64.0	33.5
15	95 PP–5.0PP-g-MAH/6CF	165.7	60.0	32.0
16	95 PP–5.0PP-g-MAH/8CF	166.7	51.0	27.9
17	95 PP–5.0PP-g-MAH/20CF	167.1	50.0	31.4

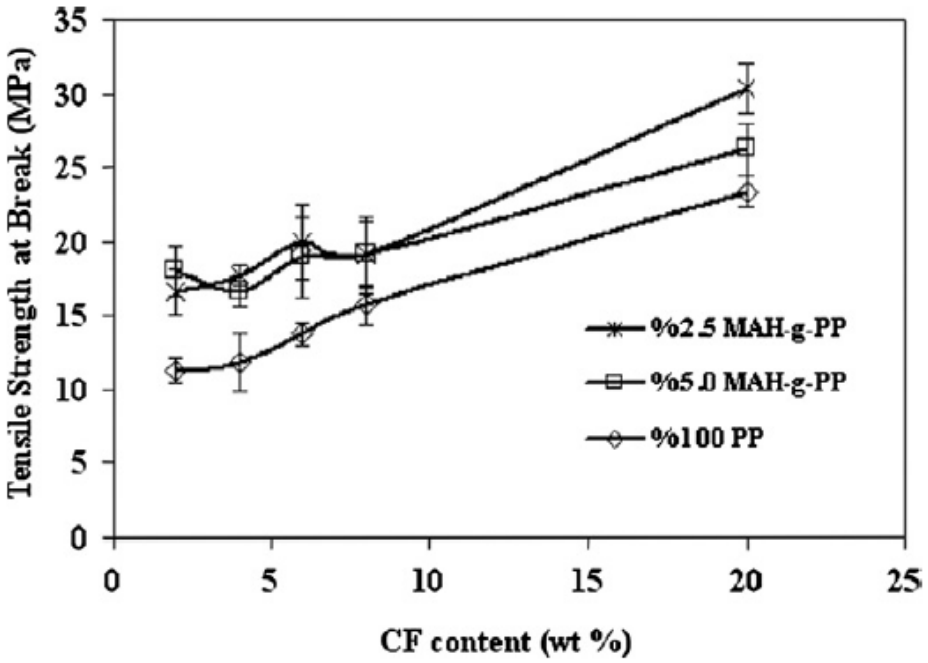

Figure 2. 4: Effect of CF contents on tensile strength short carbon fiber reinforced PP and modified PP composites (Karsli & Aytac, 2011).

As expected from the researchers, there was also relation between hardness and CF content in the hybrid composite. Based on the Rockwell hardness test, the amount of CF content determined the fiber loading in the composite structural. When the number increased, hardness value would also correspondingly increased (Karsli & Aytac, 2011). Based on Figure 2.5, PP/P-g-MAH/CF recorded higher hardness value compared to pure PP. Insignificant differences of hardness value was spotted between higher amount of P-g-MAH (5 wt% of P-g-MAH) and lower amount of P-g-MAH (2.5 wt% of P-g-MAH) in the hybrid composite. Therefore, it can conclude as the CF content is increased we can expect increases in hardness value (Karsli & Aytac, 2011).

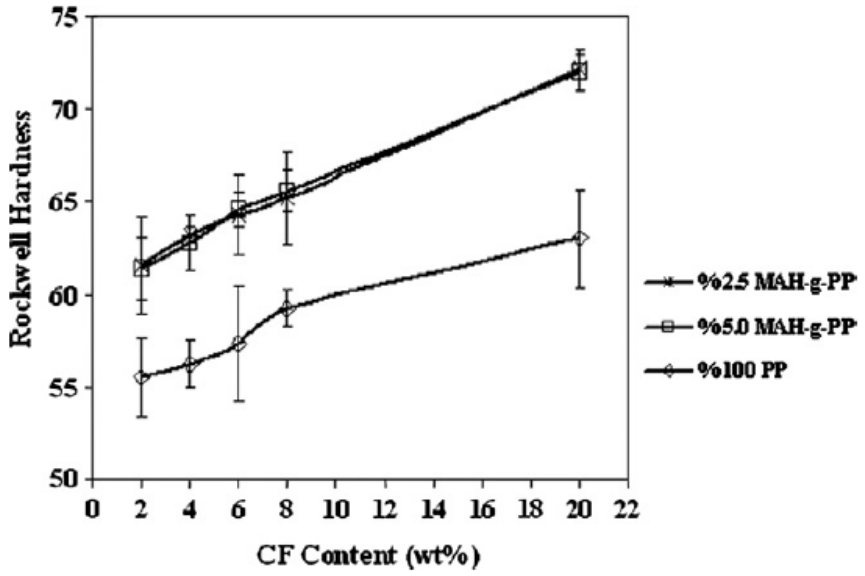

Figure 2. 5: Hardness values versus fiber weight fraction for short carbon fiber (Karsli & Aytac, 2011).

2.5 Literature Review Table

Year	Material	Parameters	Findings	Reference
2019	Glass fiber (GF) /Multi-walled carbon nanotubes (MWCNT)/ Polypropylene (PP)	Injection molding to ASTM-D638. Composition ratio (PP/GF/MWCNT) (wt%): 1. 100/0/0 (P) 2. 99/0/1 (N1) 3. 97/0/3 (N3) 4. 95/0/5 (N5) 5. 80/20/0 (G) 6. 79.5/20/0.5 (H0.5) 7. 79/20/1 (H1) 8. 78/20/2 (H2) 9. 77/20/3 (H3) 10. 75/20/5 (H5) TGA Temperature Range: 25 °C – 500 °C.	Pure PP has the highest degradation rate compare to all specimens. Introduction of msGF (20 wt% of msGF/ G) in the hybrid composite showed notable shift compared to pure PP and mwCNT/PP hybrid composite (refer Table 2.2). As two elements were added together, significant improvement of thermal stability had been recorded from (H1 to H2). The highest peak temperature for this experiment was H2 at 466.6 °C with the weight loss rate of 33.7%/min (refer Table 2.2 and Figure 2.2). the presence of flux might reduce thermal stability as heat dispersion in micro structural was important to ensure degradation at normal condition.	(Rasana et al., 2019)

2009	Short carbon fiber (SCF)/ Polypropylene (PP)	Pressing technique of fabrication (15 cm × 15 cm) with thickness of 1 mm and 3 mm. the composites were contained 10 wt% of SCF. SCF were cut into 5 different lengths 1. 0.5 mm of SCF 2. 1.0 mm of SCF 3. 2.0 mm of SCF 4. 5.0 mm of SCF 5. 10.0 mm of SCF The TGA was conducted under ambient temperature to 500 °C at a heating rate of 10 °C/min.	It is found that as the carbon length in the hybrid composite increased, the composite would thermally less affected (refer Figure 2.2 and Table 2.1). Additional observation on degradation temperature and wt% loss of the composite sorted by different length showed from 10 wt% to 90 wt% of SCF/PP polymer composite shifted the range from originally from 362 °C – 441 °C to 400 °C – 463 °C (for 0.5 mm length of SCF), 403 °C – 464 °C (for 1.0 mm length of SCF), 406 °C – 468 °C (for 2.0 mm length of SCF), 410 °C – 472 °C (for 5.0 mm length of SCF) and 415 °C – 473 °C (for 10 mm length of SCF) (refer Table 2.1).	(Rezaei et al., 2009)

2011	Short Carbon Fiber (SCF)/ Polypropylene (PP)/ Maleic Anhydride grafted Polypropylene (PP-g-MAH)	SCF were cut 1 cm × 1 cm Extrusion parameter: • 230 °C • 100 rpm Composition ratio (PP/PP-g-MAH/SCF) (wt%): refer Table 2.4 All specimens were tested under Rockwell hardness to obtain hardness value	The amount of CF content determined the fiber loading in the composite structural. When the number increased, hardness value would also correspondingly increased. Based on Figure 2.5, PP/P-g-MAH/CF recorded higher hardness value compared to pure PP. Insignificant differences of hardness value was spotted between higher amount of P-g-MAH (5 wt% of P-g-MAH) and lower amount of P-g-MAH (2.5 wt% of P-g-MAH) in the hybrid composite.	(Karsli & Aytac, 2011)

2015	Carbon Fiber (CF)/ Polyetherimide (PEI)	Composite dimension (Length*Width*Thickness): 450 mm × 450 mm × 2 mm Composite ratio % (Fiber/Resin): 58/42 %volume of PEI: 50% TGA Temperature Range: 25 °C – 1000 °C Heating Rate: 2/4/6/8 & 10 °C/min	Thermally stable up until 450 °C and the composite decomposition was happened rapidly in between range of 450 °C - 550 °C.	(Natassia L Batista & Botelho, 2015)
2010	Carbon Fiber prepreg/ Glass Fiber prepreg/ Epoxy	B-Stage of impregnation was used to produce the specimen Heating Elements: 5 & 10 A current supply on specimen. ASTM D 3039 (Tensile test)	Only 1% of tensile strength was abridged from initial properties after continuously been heated under 80 degree Celsius from 50 hours. Carbonization interrupt the long matrix of CF; thus, it decreases the ability of thermal resistance in CFRP.	(Kim, An, Yoon, Jo, & Moon, 2010)

2009	Carbon Fiber (CF)/ Epoxy 8552/IM7 and M18-1/G939	Oven convection 180/190 & 200 °C for 500 days	Temperature range of 180 °C to 200 °C mechanical properties of CFRP faced significant reduction.	(Wolfrum, Eibl, & Lietch, 2009)
2016	Carbon Fiber (CF)/ Epoxy (JA-02 epoxy resin)	Composite dimension (Length*Width*Thickness): 12.2 mm × 12.2 mm × 12.2 mm Curing: 90 °C (2 hours), 110 °C (1 hour), & 130 °C (4 hours) Composite ratio % (Fiber/Resin): 38/62 Dynamic Mechanical Analysis (DMA) temperature setting: 90/110/120/130 & °C)	Oxidation layers appears on surface layer of the composite. At 180 °C, cracking occurred.	(Zhang, Sun, & Gu, 2016)
2019	Carbon Fiber (CF)/ Epoxy (EPIKOTE RESIN	Composite ratio % (Fiber/Resin): 50/50	CF matrix diminished indicating oxidation happen during the analysis (refer Figure 4 to	(Zöllner, Lieberwirth,

	05475, EPIKURE Curing Agent 05443, & HELOXY Additive 112)	TGA Setting: 15 K/min at 1000 - 1200 °C	observe oxidation spots). This shows that the fiber matrix is damaged.	Kempkes, & Fendel, 2019)
2019	Recycled Carbon Fiber (rCF)/ (PEEK)	Accelerated thermal ageing of epoxy resin and 3D carbon fiber epoxy braided composites	PEEK-rCF are slightly lower than those of PEEK-vCF.	(Lin & Schlarb, 2019)
2019	(CaCO3, TiO2 & Al2O3)/ Epoxy (LY-551, HY-951)	Composite ratio % (Fiber/Resin): 10/90, 15/85, 20/80, & 25/75 Flexural strength test Tensile test SEM	Filler proves to improvise mechanical properties of polymer hybrid up until fiber content is 20%wt. However, after the critical %wt, mechanical properties are degrading.	(Venkateshwar Reddy, Rajendra Prasad, Mohana Krishnudu, & Hussain, 2019)

CHAPTER 3: METHODOLOGY

3.1 Process Flow

There were seven main processes in this paper, Figure 3.1 explain in detail in process flow chart.

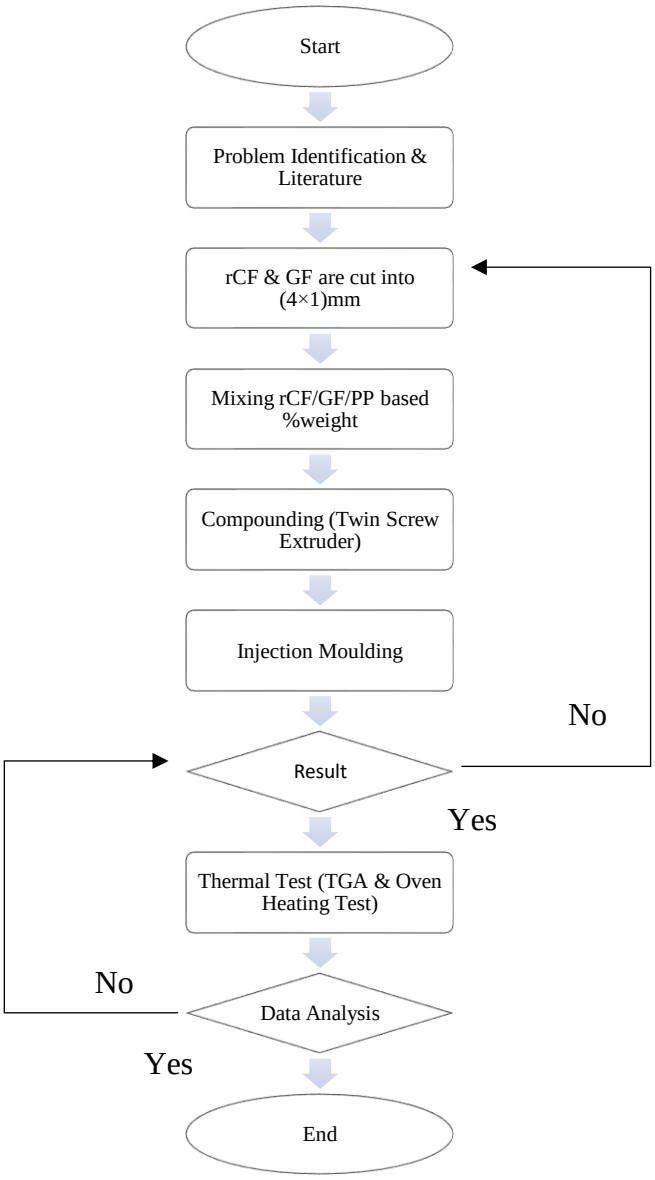

Figure 3. 1: Process flow

3.2 Material

The materials used for this experiment were carbon fiber Prepreg 3K, 2x2 Twill Weave Carbon 6-Month waste and glass fiber Prepreg 7781 E-Glass 6-Month waste from Fibre Glast Developments Corporation (USA). Both materials were cut into small fiber size (4 mm × 1 mm). For the rCF were blended to produce fine fiber size. Titanpro® Polypropylene Homopolymer with Titanpro® 6331 grade was used for epoxy resin.

3.3 Compounding/ Extrusion

Compounding is the process of mixing at least two elements to form long compounded element. The material was palletized as preparation to proceed injection moulding process. ZSE 27 MAXX - twin screw extruder was used in this process (refer Figure 3.2) under parameters as stated in Table 3.1.

Figure 3. 2: ZSE 27 MAXX - twin screw extruder

Table 3. 1: Compounding parameters

Parameters	Unit	Value
Melting Temperature	°C	200
Screw Speed	rpm	50

The final product of compounding will result into four composition ratios of different wt% of rCF, rGF and PP (refer Table 3.2). These products were initially palletized before injection process could be taken subsequently. The shape of the hybrid polymer composite can be referred in Figure 3.3.

Table 3. 2: Fabricated polymer hybrid composite composition

Weight% of PP	Weight% of rCF	Weight% of GF
100	0	0
90	5	5
80	10	10
70	15	15

Figure 3. 3: CF/GF/PP pallets

Finally, dog-bone specimens were fabricated using injection moulding process. Four main compositions of 100, 90, 80 and 70 wt% of PP were produced (refer Figure 3.4).

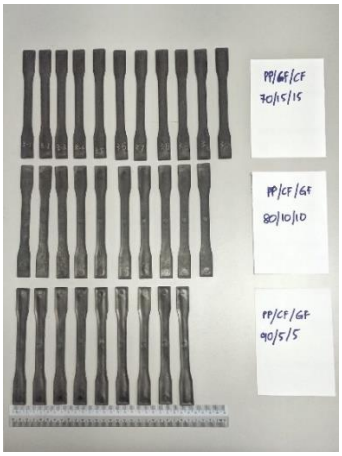

Figure 3. 4: Dog-bone specimens

3.3 Experiment Setup

3.3.1 Thermogravimetric Test

Thermogravimetric test was conducted by Central Analysis Laboratory (CAL) from University Teknologi Petronas. Four samples (4 mm diameter size) were sent for the testing. Testing temperature were set between 29.1 °C and 594.5 °C.

3.3.2 Heating Test

Heating test was conducted to observe change in mass and dimension of the specimens. The experiment was conducted at B17-02-08 (under UTP Mechanical department bloc) using an oven as shown in Figure 3.5.

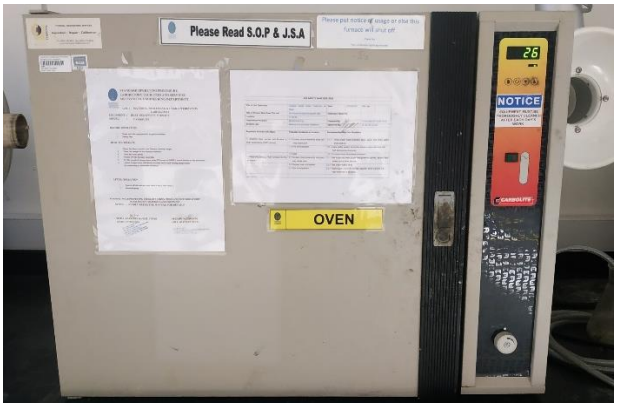

Figure 3. 5: Oven (B17-02-08)

Two sets of heating temperatures (150 °C and 175 °C) were used in this experiment. 20 minutes of startup temperature were set to allow oven temperature rose to the expected temperature. Each set of heating test consisted of four different materials composition (100 wt%, 90 wt%, 80 wt%, & 70 wt%) were heated at 1 hour. Mass and dimension of each specimen were recorded before and after they heated.

3.3.3 Hardness Test

While there are many testing methods to indicate a mechanical property out there, hardness testing is the practical method to prove that. This is because its economical and the easiest method compare to other testing standards such as tensile test and fatigue test.

First of all, the indentation hardness can be defined as a measure of material's mechanical property to withstand penetration without damaging the surface of it or form plastic deformation due to the indentation process (Lindegren, Bucan, & Boccalini, 2017). In this paper, Rockwell hardness test is applied because it is suitable for polymer materials (refer Figure 3.6). Initial tests were taken to acknowledge datum value of the hardness test. Heated tests were taken subsequently for thermal degradation study.

Figure 3. 6: Rockwell Hardness Test

3.4 Gantt Chart

Table 3.3 showed process flow of the research for 2 semesters of Final year project. Five main section were identified in order to achieve objectives.

Table 3. 3: Gantt Chart of research

Planning & Activities	Week																											
	FYP 1																FYP 2											
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
Completion of Materials preparation																												
recycled Carbon Fiber (rCF)																												
Glass Fiber (GF)																												
Grind rCF																												
Grind GF																												
Completion of Hybrid Polymer Composite																												
Extrusion - Compounding Material																												
Compounding with PP																												
Injection Moulding																												
Completion of Thermal Testing																												
Thermogravimetric Analysis (TGA)																												
Oven Heating Test																												
Hardness Test																												
Data and Result																												
Discussion																												
Completion of Final Report																												
Report																												

CHAPTER 4: RESULT & DISCUSSION

4.1 Thermogravimetric (TGA) result

TGA result can be observed in Figure 4.1. TGA method was used to determined the wt% loss curve over temperatures. Four result were tested from (a) 100 wt% of PP sample, (b) 90 wt% of PP sample, (c) 100 wt% of rGF sample and (d) 100 wt% of rCF sample.

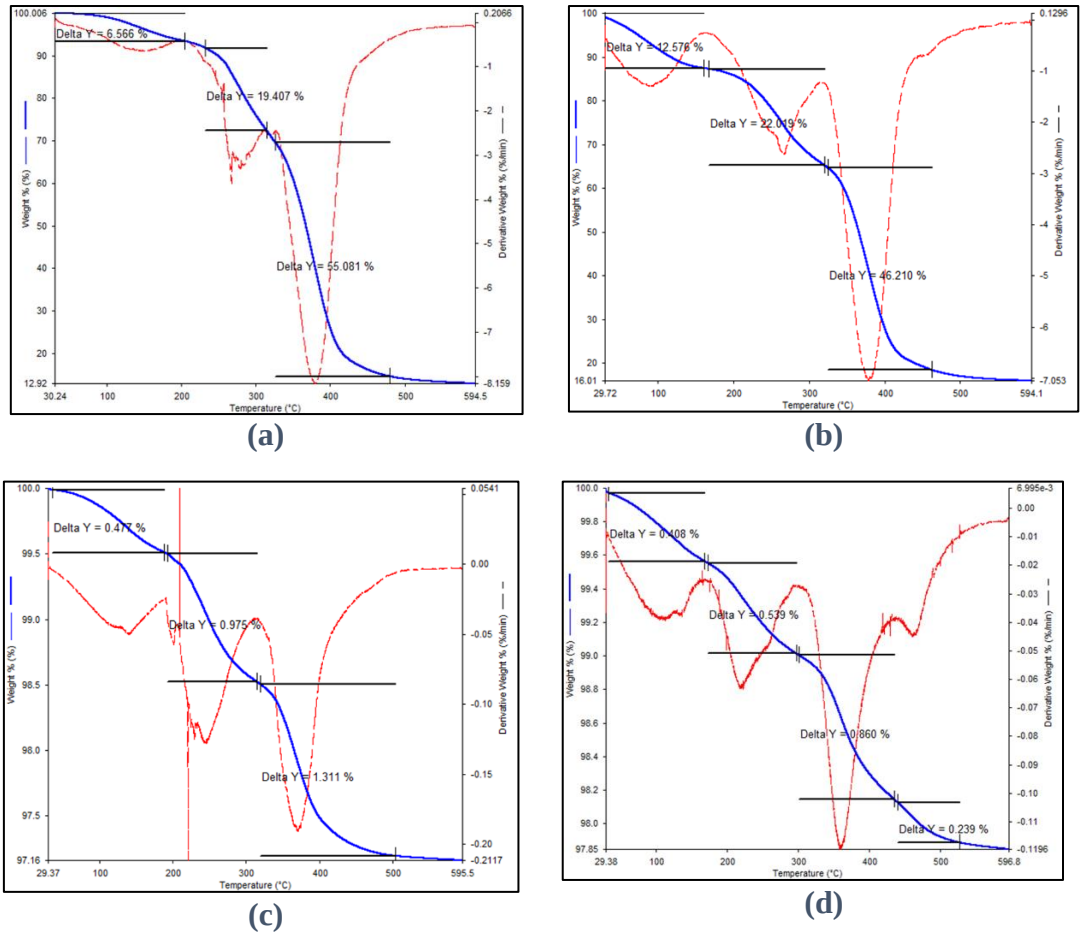

Figure 4. 1**:** (a) 100 wt% of PP. (b) 90 wt% of PP. (c) 100 wt% of rGF. (d) 100 wt% of rCF.

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For 100 wt% of PP, degradation happened in temperature range of 55 °C to 580 °C, while for 90 wt% of PP in temperature range of 31 °C to 500 °C, next, 100 wt% of rGF in the range of 250 °C to 595 °C (continuing) and 100 wt% of rCF in the range of 302 °C to 596 °C (continuing).

In DTG analysis, it indicates 3 stages of thermal degradation based on loss in wt%. 100% wt of PP was lost 6.566 wt% between temperature of 30.24 °C to 205 °C, 19.407 wt% was lost between 240 °C to 310 °C, and lastly 55.081 wt% was lost between 320 °C to 485 °C. On the other hand, in wt%. 90% wt of PP was lost 12.576 wt% between temperature of 29.72 °C to 170 °C, 22.019 wt% was lost between 180 °C to 315 °C, and lastly 46.210 wt% was lost between 320 °C to 465 °C. Comparing these two materials composition (100 wt% of PP and 90 wt% of PP), the initial was recorded higher in 90 wt% of PP. This trend also applied in second stage where 90 wt% was higher compared to 100 wt% of PP. However, the final stage was highly recorded in 100 wt% of PP.

By observing both 100 wt% of rCF and 100 wt% of rGF, it can be concluded that rCF had higher temperature range of thermal degradation which was 29.38 °C – 530 °C (versus 29.37 °C – 503 °C). There were three stages for both degradation curve. Only for initial stage, both were similarly recorded around range of 29 °C - 197 °C for more than 0.400% lost in weight. However, for the rest stages, 100 wt% of rGF had much degradation (2^{nd} stage, 0.975% lost in weight) and (3^{rd} stage, 1.311% lost in weight).

In conclusion, heating process were affecting weight loss for all specimens (100 wt% of PP, 90 wt% of PP, 100 wt% of rGF, & 100 wt% of rCF). Pure PP was noticed with higher thermal degradation temperature range and loss in weight if it was compared to 90 wt% of PP. This can be related as the properties of rCF and rGF in the specimen 90 wt% of PP might slow down the thermal degradation as they had higher melting point. Therefore, the specimen 90% of PP can be said as the improvised version from pure PP.

4.2 Oven Heating Test

Table 4.1 describe specimens produced by injection process. The materials were classified based on their composition of PP/rCF/rGF. There were six samples for each catagory (100 wt% of PP, 90 wt% of PP, 80 wt% of PP and 70 wt% of PP) (refer Table 4.1). Initial mass for each specimens were recorded on Table 4.2.

Table 4. 1: Material composition

Material	wt% of PP (%)	wt% of rCF (%)	wt% of rGF (%)
1	100	0	0
2	100	0	0
3	100	0	0
4	100	0	0
5	100	0	0
6	100	0	0
1-6	90	5	5
1-7	90	5	5
1-4	90	5	5
1-5	90	5	5
1-2	90	5	5
1-3	90	5	5
2-6	80	10	10
2-7	80	10	10
2-4	80	10	10
2-5	80	10	10
2-2	80	10	10
2-3	80	10	10
3-6	70	15	15
3-7	70	15	15
3-4	70	15	15
3-5	70	15	15
3-2	70	15	15
3-3	70	15	15

Table 4.2 above shows Dog-bone mass by different composition at standard room temperature were measured before heating process was taken. In average pure composition of PP (100 wt% of PP) recorded 7.015 gram. Followed by 7.875 gram for 90 wt% of PP. Then, by 7.644 gram for 80 wt% of PP. Lastly, by 7.599 gram for 70 wt% of PP. In general, pure PP had the average lowest mass of all type of composition. Whereas, 90 wt% of PP had the average highest mass than other which was 7.875 gram. The other 2 types were recorded similarly to each other which was around 7.644 gram (80 wt% of PP) and 7.599 gram (70 wt% of PP). 90 wt% of PP had mass improved for 12.27 % from pure mass, while, the others (80 wt% of PP and 70 wt% of PP) increased correspondingly by more than 8.00%.

Table 4. 2: Initial mass of specimens (at 26 °C/ Standard Room Temperature)

Material	Initial Mass, m_i (g)
1	6.974
2	7.043
3	7.031
4	6.995
5	7.029
6	7.015
1-6	7.82
1-7	8.014
1-4	7.906
1-5	7.821
1-2	7.817
1-3	7.871
2-6	7.737
2-7	7.755
2-4	7.846
2-5	7.675
2-2	7.351
2-3	7.502
3-6	7.461
3-7	7.573
3-4	7.201
3-5	7.936
3-2	7.962
3-3	7.458

Mass improvement may be resulted as additional materials were added in non-pure PP composition. This caused the non-pure composition of mass to record higher than the original (refer Table 4.3 for heated mass and Table 4.4 for mass loss analysis).

Table 4. 3: Final mass of heated specimens

Material	Composition of PP (%)	Heated Mass, m_h (g)	Heating Temperature, (°C)
1	100	6.974	26
2	100	7.043	26
3	100	7.010	150
4	100	6.974	150
5	100	6.983	175
6	100	6.967	175
1-6	90	7.820	26
1-7	90	8.014	26
1-4	90	7.892	150
1-5	90	7.806	150
1-2	90	7.739	175
1-3	90	7.790	175
2-6	80	7.737	26
2-7	80	7.755	26
2-4	80	7.827	150
2-5	80	7.660	150
2-2	80	7.130	175
2-3	80	7.247	175
3-6	70	7.461	26
3-7	70	7.573	26
3-4	70	7.181	150
3-5	70	7.912	150
3-2	70	7.540	175
3-3	70	7.056	175

Table 4. 4: Percentage mass loss of specimens by different temperature

Material	Specimen mass loss, $m_i - m_h$ (g)	Specimen mass loss in percentage, $(m_i - m_h)/m_i$ (%)	Heating Temperature, (°C)	Average percentage mass loss by different temperature, (%)
1	0.000	0.00%	26	0.00%
2	0.000	0.00%	26	
3	0.021	0.30%	150	0.30%
4	0.021	0.30%	150	
5	0.046	0.65%	175	0.67%
6	0.048	0.68%	175	
1-6	0.000	0.00%	26	0.00%
1-7	0.000	0.00%	26	
1-4	0.014	0.18%	150	0.18%
1-5	0.015	0.19%	150	
1-2	0.078	1.00%	175	1.01%
1-3	0.081	1.03%	175	
2-6	0.000	0.00%	26	0.00%
2-7	0.000	0.00%	26	
2-4	0.019	0.24%	150	0.22%
2-5	0.015	0.20%	150	
2-2	0.221	3.01%	175	3.20%
2-3	0.255	3.40%	175	
3-6	0.000	0.00%	26	0.00%
3-7	0.000	0.00%	26	
3-4	0.020	0.28%	150	0.29%
3-5	0.024	0.30%	150	
3-2	0.422	5.30%	175	5.35%
3-3	0.402	5.39%	175	

The heating process taken in 3 different temperatures (26 °C, 150 °C, & 175 °C). This is to ensure the mass loss different can be studied b this method. In standard room temperature, all specimens had no mass loss. However, as the temperature rose, the mass loss followed the trend. According to LOTTE Chemical Titan, thermal degradation of PP happens when the temperature 160 °C ("Lotte Chemical Titan Titanpro® PP Homopolymer Titanpro® 6331," 2016). Hence, approaching to 175 °C the loss due thermal degradation is greater. This is because as the material absorb greater heat, it releases the bond between molecules much easily. Thus, when the temperature of heating process increased from 150 °C to 175 °C, rapid mass loss of

material can be observed. In this context, PP were primarily affected during the heating process due to its melting temperature which is 160 °C. However, it suggested that rGF and rCF were less likely to be affected due to its high melting point as shown in TGA result (refer Figure 4.1). Due that, the outcomes of heating test reflect similarly to TGA-DTG result as the degradation rapidly increased as the composition of PP reduced.

4.3 Rockwell Hardness Test

Rockwell testing method was used to determine the hardness value. Based on Table 6 there were 3 different temperatures 26 °C, 150 °C, and 175 °C. 4 types of composition were tested in 3 different temperatures (refer Table 4.5).

Table 4. 5: Rockwell hardness test result

Material	Rockwell Hardness value, (R)	Rockwell Hardness value by heating temperature, (R)	Heating Temperature, (°C)
1	85.9	82.7	26
2	79.5		
3	75.6	75.9	150
4	76.2		
5	70.1	70.6	175
6	71.1		
1-6	71.4	69.8	26
1-7	68.2		
1-4	77.9	76.7	150
1-5	75.4		
1-2	68.7	69.5	175
1-3	70.3		
2-6	89.2	86.2	26
2-7	83.1		
2-4	74.7	74.3	150
2-5	73.8		
2-2	2.5	39.8	175
2-3	77.0		
3-6	78.2	70.8	26
3-7	63.4		
3-4	77.0	76.8	150
3-5	76.6		

3-2	56.3	66.3	175
3-3	76.2		

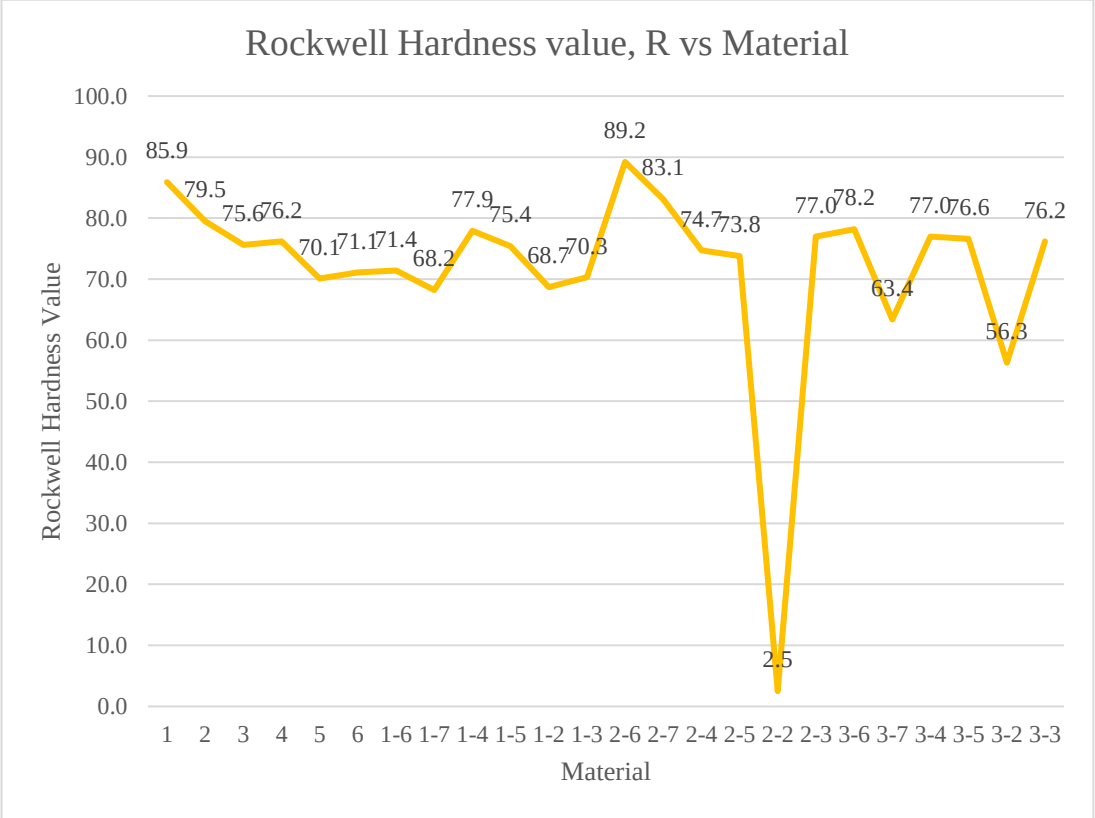

Figure 4. 2: Graph indicate hardness result based on 26 °C (every 2 specimens in each category), 150 °C (continuation specimen 3-4 of each category) & 175 °C (continuation specimen 5-6 of each category).

For 100 wt% of PP, in standard room temperature the hardness value was 82.7 (refer Table 4.5 for average value and Figure 4.2 for detailed results). As the temperature increased to 150 °C the value reduced to 75.9. As the temperature were set to 175 °C, the value further shrunk to 70.6. For 90 wt% of PP, in standard room temperature the hardness value was 69.8. As the temperature increased to 150 °C, in contrast, the value rose to 76.7. As the temperature were set to 175 °C, the value reduced to 69.5. For 80 wt% of PP, in standard room temperature the hardness value was 86.2. As the temperature increased to 150 °C the value reduced to 74.3. As the temperature were set to 175 °C, the value rapidly reduced to 39.8. For 70 wt% of PP, in standard room temperature the hardness value was 70.8. On the other hand, as the

temperature increased to 150 °C, the value amplified to 76.8. As the temperature were set to 175 °C, the value went down to 66.3.

The result summarises, as the temperature increase the hardness of material will be degraded. This is because, mechanical properties of the hybrid polymer composite are depending on resin and filler mechanical properties. Based on previous studies shows that fiber components such as GF and CF have reinforced the original polymer properties. The reinforcement had also increased the melting point of the material from the original form. As the materials were exposed to heat, it resulting to loss of molecular bond in the hybrid polymer composite. This had made the structural of strong material to be weaken due to thermal effect because as the temperature exceed 160 °C, numerous amounts of PP molecule lost. Due to that, original hardness value may be affected. This finding suggests, heating process will be weakened the material's hardness which can be referred in the previous study (refer Figure 2.5).

Besides, further observation can be done by looking into two compositions 100 wt% of PP and 80 wt% of PP. However, there still inconsistency when recording the data for the rest compositions of material which were in 90 wt% of PP and 70 wt% of PP. Firstly, the issue happened specially after the high temperature exposure of every material compositions. Degradation caused the material's surface to be uneven in shape as shown in Figure 4.3. This condition is not suitable for measurement, however several attempted resulted low values. Other that, the machine accuracy maybe downgraded as the usage had passed its prime lifetime.

Figure 4. 3: Specimen failure during Rockwell Hardness Test

CHAPTER 5: CONCLUSION AND RECOMMENDATION

To sum up, thermal degradation shown based on TGA result indicated as expected result. rCF prepreg had improve the PP-matrix like other hybrid polymer composite that used good material's condition.

Apart from that, it can also be concluded that as the temperature increases the mechanical properties shrinks. Rate of thermal degradation may increase as the composition increased.

Hardness test were not showing the expected result; however, 70 wt% of PP had shown higher record of hardness compared to another CF and GF compositions.

Due that, deep structural analysis may explain on thermal loading under microscopic factor. Thus, it could enhance the mechanical properties of the hybrid polymer composite. Numerous testing needs to be done especially on heating and hardness test. Due to lack of laboratory access, the data cannot be fulfilled and insufficient.

The results of this experiments may not be accurate. However, the objective of this experiment is achievable.

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