DEVELOPMENT OF NYLON-6/WASTE TIRE POWDER (NYLON-6/WTP) THERMOPLASTIC ELASTOMER FOR HIGH PERFORMANCE APPLICATIONS

LIM CHIN AIK

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Universiti Tunku Abdul Rahman

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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

Signature : ______________________

Name : Lim Chin Aik

ID No. : 10AGB05045

Date : ______________________

APPROVAL FOR SUBMISSION

I certify that this project report entitled **"DEVELOPMENT OF NYLON-6/WASTE TIRE POWDER (NYLON-6/WTP) THERMOPLASTIC ELASTOMER FOR HIGH PERFORMACE APPLICATIONS"** was prepared by **LIM CHIN AIK** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons.) Petrochemical Engineering at Universiti Tunku Abdul Rahman.

Approved by,

Signature : _________________________

Supervisor : Dr.Mathialagan Muniyadi

Date : _________________________

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ABSTRACT

This project was mainly illustrated about the application of elastomer as filler in thermoplastic composite. Thus, the primary objective of this project was to investigate the effect of waste tire powder (WTP) loading on properties of nylon matrix composites. Various WTP loading (10-40 wt%) were blended together with nylon by using rheometer Brabender® Plastograph® EC 815652. The introduction of WTP into nylon-6 matrix decrease the tensile properties of the composites and chemical resistance. On the other hand, good compatibility between WTP and nylon-6 had enhanced the impact properties. Moreover, nylon-6/WTP composites retain the processing conditions of neat nylon-6 which there was not significant different between T_m and T_c of nylon/WTP composites as compare with neat nylon-6 as can be observed from the Differential Scanning Calorimetry (DSC) analysis. The reduction in tensile properties can be proven and explained from the evaluation of Scanning Electron Microscopy (SEM) micrograph of the tensile fractured surface.

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LIST OF SYMBOLS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
DSC	Differential Scanning Calorimetry
EPDM	Ethylene Propylene Diene Terpolymer rubbers
E_{Mod}	E-modulus (MPa)
E_b	Elongation at break (%)
FTIR	Fourier Transform Infrared Spectroscopy
ΔH_m	melting enthalpy (J/g)
ΔH_c	heat of crystallization(J/g)
ΔH_{100}	Melting heat for 100% crystalline nylon-6, 240J/g
M_0	Initial mass (g)
M_1	Mass of the specimens after immersion (g)
PP	Polypropylene
SEM	Scanning Electron Microscopy
T_m	Crystal melting point (℃)
T_c	crystallization temperature
Tg	Glass-transition temperature (℃)
TGA	Thermogravimetric calorimetry
TPE	Thermoplastic Elastomer
UTS	Ultimate tensile strength (MPa)
W_p	Weight fraction of polymer in sample
Wt%	Weight percentage
WT	Waste Tire
WTP	Waste Tire Powder
X_c	Degree of crystallinity (%)

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Waste rubber is a well-known non-biodegradable disposal that will cause a lot of environmental impact. The amount of waste rubber keep increasing and mostly are sourced from automobile industry such as waste tire and others such as used gloves and rubber mats. According to Rubber Manufacturer Association (2009), about 1.4 billion waste tire are produced globally each year at which 40 % of it used as feedstock for generating energy, 26% are reduced into granulated rubber, 13 % are disposed in landfills, 5% are used in civil engineering applications and the remaining 16% are used in miscellaneous.

In the nearest decade, a lot of research and studied were carried out in containing and coping up with the environmental impact caused by tires disposal. However, those technologies and recycling management introduced have their own limitations which reduce the utilization of waste tire. Generally, waste tire are disposed either into landfilling, stockpiling and legal dumping. However, landfilling of waste tire causes a lot of environmental problem such as, providing a breed spot for mosquito and others animals. In addition, pyrolysis of waste tires will produce a new product such as artificial reefs, fillers in polymer composites, bio-oil, flue gas and bio-char after heating the waste tire without the presence of oxygen. On the other hand, pyrolysis process is not a zero discharge process and toxic gases will be emitted to the atmosphere during the heating process (Martinez et al, 2012).

With the increasing popularity of introducing filler into polymer matrix to produce composites, research and development have been carried out in producing thermoplastic elastomers by incorporating waste rubber such as waste glove, waste tire powder and scrap rubber into thermoplastic (Sienkiewicz, 2012). Such new invention results in a new materials which exhibits combined properties of both thermoplastic and elastomer as well as processing of thermoplastic materials which is economic and reversible.

In this research, waste tire powder was introduced into nylon-6 to produce thermoplastic elastomer composites. Nylon-6 is a high performance polymer such as high strength, high heat resistance and can be recycled by using different processing method while retaining its excellent performance. (Wang et al, 2014). However, due to high cost and brittle properties, nylon-6 is hard to be processed. Upon this new research, incorporating waste tire powder would help to reduce the cost and improve the process ability while retaining its thermoplastic properties.

1.2 Problem Statement

Increased amount of waste tire disposal.

Since the introduction of automobile industries, tires have been the main part of most vehicles which the rate of consumption and disposal had increased annually. Based on the survey conducted by Sienkiewicz and his fellow colleagues (2012), about 1.4 billion of unit’s production in the worldwide which corresponds to an estimated 17 million tonnes of used tire each year. According to an estimation, 3.4 and 4.6 million tonnes of waste tires discarded annually in Europe and USA and almost 88% of discarded tires are produce from China, India, European countries, Japan and USA. For European countries, the annual cost of their disposal in EU countries has been estimated at nearly 600 million euros (Sienkiewicz, 2012).

Table 1.1: The used tires (in kilo tones) recovery in Europe Union (ERTMA, 2010).

Country	**(A)**	**Reuse of part-worn tires**			**(E)= A-B+C+D**	**Waste Recovery**		**Tire**	**(J)**	**(K)= B+C+D+F+G+I**	**(L)=K/A (%)**
		(B)	**(C)**	**(D)**		**(F)**	**(G)**	**(I)**			
France	381	36	0	43	302	38	128	147	0	392	103
Germany	614	10	84	45	475	0	215	260	0	614	100
Netherland	65	0	13	2	50	1	39	10	0	65	100
Portugal	92	1	2	18	71	0	50	26	0	97	105
Spain	292	31	0	27	234	8	114	112	0	292	100

#**A**: Used tires Arising; **B**: Reuse; **C**: Export ; **D**: Re-treading; **E**: Waste tires arising; **F**: Civil Engineering;

G: Recycling; **H**: Energy; **I**: Landfill / Unknown; **J**: Total Recovery; **K**; Used tire treated

Limitation on recycling and re-processing of waste tire due to the presence of cross-linking.

Tires are made of rubber which mainly contains fillers such as carbon black and other compounding ingredients such as vulcanizing agent, activators and processing aid (Bekhiti.,Trouzine. & Asroun., 2014). Upon vulcanization process crosslinks are formed and rubber chains are transform into three dimensional network which are stiff and not recyclable or re-processed. The cross-linked elastomer are designed to be high strength and elasticity for withstanding harsh mechanical and weather condition. At the same time, tire life time in a landfill is considered to be 80 to 100 years. There are no harm from the landfilling of waste tire but the amount of waste tire keep increasing annually and there will be insufficient land for disposal of waste tire in future. Besides, the presence of crosslink limit the recycling or reprocessing of waste tire into other new products (Martinez et al, 2012).

Nylon-6 is a high performance polymer but it is expensive and hard to process due to high processing temperature.

Nylon-6 is a semi-crystalline and are very tough materials with excellent thermal and chemical resistance. In common, the impact resistance and flexibility of nylon-6 will increase with moisture content, while the strength and stiffness below the glass transition temperature. Nylons tend to sustain its performance under a high temperature environment. Due to its high heat resistance, large energy is required in processing nylon-6 and the cost of processing will be increased. Hence, addition of waste tire powder may act as processing to improve the process ability of nylon-6. Besides, nylon-6 is also very viscous and brittle upon processing which restricts its applications on high deformable and high impact appliances. Hence, by introducing tougher and elastic waste tire powder, the brittleness of nylon-6 can be reduced and the impact properties can be improved (Pang, 2013).

1.3 Objectives

1. To develop thermoplastic elastomer through melt blending of nylon-6 and waste tire powder using brabender internal mixer.
2. To study the effect of nylon-6/WTP ratio on the processing torque, tensile properties (ultimate tensile strength, E-modulus and elongation at break), swelling resistance, impact strength and SEM morphological properties of nylon-6/WTP composite.
3. To compare the properties of Nylon/WTP blend with neat Nylon.

CHAPTER 2

LITERATURE REVIEW

2.1 Thermoplastic Elastomer

2.1.1 Introductions

Thermoplastic elastomers (TPEs) are a new class of polymeric material produced from the blending of thermoplastic and elastomers. TPEs are rubbery in nature with similar elasticity to that of the commercial thermosetting rubbers. Moreover, TPEs can be reprocessed as well as recycled on the similar was as thermoplastic material (Deniz, Karakaya & Ersoy, 2009). In recent years, TPEs are mainly produced by blending virgin, uncured rubber and thermoplastic materials. This produced TPEs material contain rubbery features while retaining the thermoplastic characteristic of the matirx.

Thermoplastic elastomers are one of the most competent material in the plastic market nowadays. TPE was introduced to the market since 1960 and they have grown rapidly (Salman & Muhammad, 2011). The earlies discovered TPE were made using Ethylene Propylene Diene Terpolymerrubbers (EPDM) and Polypropylene (PP). However, no crosslinking present in the blending which causes the hard thermoplastic formation and it has a decreased tensile strength as well as low stability in organic solvents. The solution to resolve the low thermal stability and chemical resistance of TPEs was by replacing EPDM by butyl rubber and natural rubber which undergoes dynamic vulcanization with improved properties.

2.1.2 Properties of Thermoplastic Elastomer

A thermoplastic elastomer has similar features as that of commercial elastomer except the replacement of chemical cross-linking by physical cross-links. The ability to form physical cross-links is the opposite of the chemical and depends on the structural requirements of the elastomer. To resolve the problem, thermoplastic elastomers must be of two-phase materials, and individual molecule must be made of two different types of structure. The constituent part are elastomeric part and the stiffer physical cross-linking part (Shanks & Kong, 2012).

Among the inherent properties of thermoplastic elastomer are good tensile strength and high tear resistance as well as high chemical, ink or paint resistance. Besides, it has a good properties of flexibility, resistance to compression and high dimensional stability. With the thermoplastic properties, TPEs can be reprocessed and recycled by mean of heat melting and solidification in similar manner to thermoplastic. TPEs can be processed through extrusion, blow moulding and injection moulding and can be reshaped if the temperature is increased until the softening point of TPE. Lastly, uncoloured TPEs can be tailored from optically transparency to opaque, depending the material. Generally, TPEs are colourless, transparent or opaque material that can be easily changed into different coloured products (Rolf Klein, 2011).

For every polymer, it has its own glass-transition temperature (Tg) and crystal melting point (Tm). At very low temperatures, both thermoplastic and elastomer are in stiff and brittle. At Tg or Tm TPEs start to melt while at service temperature TPEs are commonly in rubbery state. Increasing the temperature above T_m results in molten formation where both thermoplastic and elastomer phase are in a viscous liquid and mouldable state. The TPEs have the properties which are intermediate between its two constituent phases. Hence, a TPE will have two service temperatures (Amin et al, 2011). There are some other factors which govern the mechanical properties of thermoplastic elastomers, which include effect of molecular weight of soft elastomers and rigid thermoplastic used and the ratio of hard to soft phases. Thermoplastic elastomers have high T_m strength as compared to homo polymers of the same molecular weight. In addition, the tensile strength of a thermoplastic elastomer are dependent ratio of stiff thermoplastic. When the ratio of hard thermoplastic to soft elastomers increased, the product shows strong rubbery features at initial stage which later become leather-like and transform into hard and flexible plastic. The composition of thermoplastic also affects the T_m and thermal stability of resulted thermoplastic elastomer (Amin et al, 2011).

2.1.3 Advantages and Disadvantages of Thermoplastic Elastomers (TPEs)

Table 2.1 will show the advantages and disadvantages of TPEs

Table 2.1: The advantages and disadvantages of TPEs. (Amin et al, 2011)

Advantages	Disadvantages
• Providing wide design options due to excellent dimensional stability.	• Decreased in chemical and environmental resistance
• Cost effectiveness.	• Decreased in thermal resistance.
• Economical, efficient and fast processing.	• Cost of materials are higher than thermoset materials.
• Can be insert moulded with other olefin based material without adhesives usages.	• Cannot be used with filler while making large amount of product with low cost.
• Low density.	• Not stable at outdoor environment.
• Elastic recovery properties within a temperature range.	
• Resistance to solvent or oil.	
• High adhesion property.	
• High resistance of surface tear.	
• Low permeability and colourable.	
• Wide range of hardness	

2.1.4 Applications of Thermoplastic Elastomers (TPEs)

Thermoplastic elastomers serve a wide range of markets due to its low cost and equipped with rubbery and thermoplastic properties. Major applications of TPEs are in replacements of synthetic and natural rubber, in foam cushioning insulation materials and sheets. Construction application such as, asphalt modification (Wang et al, 2015). Besides, TPEs also applied in electrical and electronic appliance such as insulation for indoor/outdoor electrical cable, optical fibre, welding cable insulation, insulation of high voltage automobile cables, coil forms and parts in transformers, general cable insulation with high flame retard, housing for composite and in microelectronic chips (Li et al, 2014).

In addition, TPEs are used in transportation and automotive parts, such as car dash boards and knobs, bumpers, joint boots, suspension bushings, car window and door trim, floor mats, gear knobs, flexible grip, mirror case, automotive gaskets, coloured interior components and lead acid batteries casings. Besides, TPEs are used in medical industry such as medical devices like blood bags seal, and urine bags seal, artificial heart valves and joints, syringes or tubing and packs of medicines. (Technical contribution for Medplast Supplement, 2007).

According to Amin (2011), For the household and consumer products, TPEs are applied in door and window handles for household, plastic furniture, coat hangers, lacquer, varnishes, paints, weather stripping applications, shoe soles, belts, sports equipment, electrical accessories and casing, expansion joints, ring gaskets, handles for knives and scissors, and other non-slip grip equipment, syringes, nebulizers, toys, plastic eye wear, razors and shavers, safety equipment, soft contact lenses for eyesight, lenses for cameras and monitors.

Moreover, TPEs also used in industrial and office, workshop helmets, and parts for motors, projectors and copiers. For food and beverage, the usage of TPEs on food packaging, document lamination films, packing foam. Lastly, TPEs also used in militaries, latest in substitution of metals in applications such as aircraft, space ships which require strength as similar to metals but with less weight (Amin et al, 2011).

2.2 Waste Tire

2.2.1 Introductions

Tire is a composite engineering structure made up of a polymeric matrix and reinforced with steels wire. The old age tire had improved and innovated to a pneumatic tire so that it has a wide range of service environment. For example, automobile tire made up of about twelve components and truck tires about twenty (Ramarad, 2015). The tire contain the crosslinking molecular structure, this three dimensional chemical network prohibited tire to reshape and resize. Table below shows the composition of main materials used in passenger and truck tire manufacturing.

Table 2.2. Composition of materials used in tire manufacturing. (Ramarad, 2015)

Type of tire	Car/ Passenger	Truck
Rubber/ Elastomer	41-48	41-45
Carbon black	22-28	20-28
Metal/ Steel	13-16	20-27
Textile	4-6	0-10
Additives	10-12	7-10

These components shown are vulcanized to achieve specific properties to a tire composite. Vulcanization is the irreversible process of rubber crosslinking with sulphur which discovered by Charles Goodyear in 1839 (Kumar & Nijasure, 1997). In the crosslinking process, the formation of cross-linked structure of rubber is done due to the transverse bond connect to rubber chains and this causes the tire become an elastic, insoluble and infusible thermoset composite which cannot be reprocessed (Ramarad et al, 2015).

2.2.2 Source and Impact of Waste Tire to the Environment

With the rapid expansion of automobile, the production of tires must be enough to avoid shortage of tires to the market. Since the production increase, the number of disposed tires will increase too. Those used tires were disposed after used by car, truck, passenger vehicles, small vehicles, passenger aeroplane and jet. Due to the friction between the tires and the land or road, the rubber on the surface of the tires will lost and cause the tires have no friction ability anymore. So, the tires will be substitute or replace with a newly build tires and the old tires will be discarded as waste. The waste tires will be then sent to spot for landfilling. There are numerous environmental problem because of landfilling. Firstly, the tires ponding the water and the water will become the place for mosquito larva breeding's which are lead to fatal diseases such as dengue, chikungunya and malaria. Discarded tires could also present breeding ground to other animals such as rodents and snakes. (Naik & Singh, 1991; Li et al, 2006)

In addition, waste tires pose a fire risk and they are difficult to be extinguished once they are ignited. This is because the present of water increase the production of pyrolytic oil which providing a mode of transportation to carry the oils flow around and aggravated contamination of soil and water. Once the combustion triggered, the burned tires will emit pollutants and toxic airs that will threaten the public health (Martinez, 2013).

Moreover, according to Martinez and co-workers, the additive which added into the tires such as sulphur will affect the eco-toxicity of landfills by leaching of metal resulting these materials would retard or kill the beneficial bacterial in the soil. Lastly, landfilling will cause a country to lack of land as the waste tires occupied a lot of space due to the tire life is very long (80 to 100 years). If keep landfilling the tires, the land will insufficient for development of a country or further usage.

2.2.3 Properties of Waste Tire Powder (WTP)

Waste tire is an elastic materials which made up of natural rubber, synthetic rubber, carbon black and supported by steel cord and chemicals. Before doing the research on the waste tire, the steel wire have to be removed by using magnetic separation and textile fibre was removed by density too. Then, the waste tire can be grinded into powder. The properties of the waste tire powder are dependent of its size. Research conducted by Horace Moo-Young (2003) shows that, as the size of granulated increase, physical and chemical properties such as the thermal stability increase up to (200 ℃) as well as the compressibility. WTP can be produced through two principal processes which are ambient and cryogenic. Ambient is a method in which waste tire rubber is processed at room temperature and cryogenic method uses liquid nitrogen to freeze and produce brittle scrap tire which are then hammered in hammer mill and shattered into small and smooth particles (Bekhiti et al, 2015). According to Melik, rubber powder which crushed mechanically in ambient temperature has a very low density nearly about 0.83, cohesion varied from 6.5 to 50 kpa and with different of the rubber particle size, it give different friction angle. The elements present in waste tires are Carbon: 85.16%; Hydrogen: 7.27%; Nitrogen: 0.38%; Sulphur: 2.30%; Oxygen: 0.54% and Ashes: 4.36% (Pang, 2013).

2.2.4 Method of Managing Waste Tires

Recovered waste tires are considered to be a valuable material as their composition and properties are a source of high value raw materials. The development of recovery method led an effective conversion of energy which can be used in manufacture new goods. Figure 2.1 illustrates the flow of waste tire utilization.

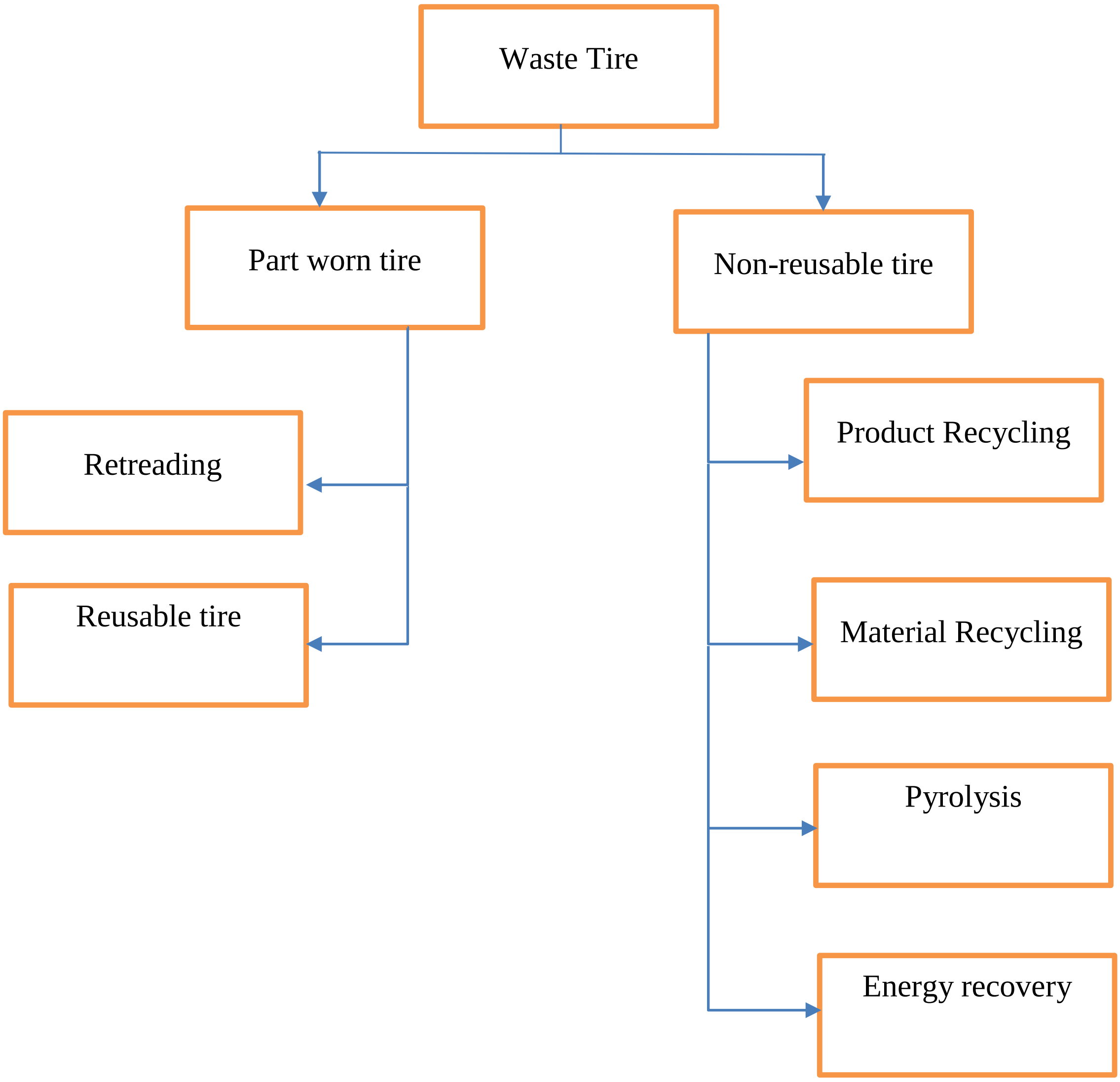

Figure 2.1: Flow of waste tire utilization

Figure 2.1 shows that, there are two type of waste tire which will undergo different treatment respectively. The first type of waste tire named part worn tires which the tire can be used on the road. This type of waste tire will be reused and re-treaded after collected. Re-treading is defined as the process of extending the lifetime of tire as long as the casing of the tire is in a good conditions. Besides, only tires which passed certain inspection and have been verified that the tire carcass have no damage due to wear and tear can go through re-treading process. Re-treading process can be done using hot or cold process (Sienkiewicz et al, 2015). Throughout low temperature method, the prepared carcass is coated with a layer of rubber compound which will act as binder and form the vulcanized thread with certain mixture pattern are then pressed into the body of rubber envelope and cured using an autoclave. The high temperature process happen at elevated pressure where the new rubber compound of the respective composition is laid on the carcass. The mixture are then vulcanized in moulds producing the patterns of tire tread (Sienkiewicz et al, 2012). The re-treading process is more favourable for truck and aircraft type's tires because the process consume about 30% of the energy and 25% of raw materials to get a new tire. According to the largest tire manufacturers, truck tire are generated through re-treading due to cost saving.

The second type of waste tire named end-of-life tire which the tire are non-reusable or unable to reprocessing. According to Sienkiewicz (2012), the processing method of these tire can be divided into four types. The first type is product recycling which the whole tire and shredded tires without any further treatment. For example, the tire are used as construction engineering material due to their size, shape, high elasticity, excellent damping resistance, noise proving and shocks absorption. The second type is material recycling which different from product recycling but is a most common method to manage the waste tire. The waste tire will undergo de-vulcanized procedure and then is grinded into powder form and will be used as additive or filler in a new products.

Next, pyrolysis mainly is used to produce charcoal and syngas from the burned waste tire for others industries usage. The pyrolysis is the process of decomposing the rubber component in the presence of heat but absence of oxygen to avoid oxidation. During pyrolysis process, tire will be burned under at a temperature of 400 ℃ in oxygen free environment containing inert or a self-generated atmosphere, leading to the volatilisation and the decomposition of the different structure those compound the feedstock (Martinez, 2012). Combustion of feedstock at this conditions or environment will trigger dehydration, cracking, isomerisation, dehydrogenation, and aromatisation as well as condensation reactions. Pyrolysis mainly is used to produce charcoal and syngas from the burned scrap tire.

Lastly, another method to manage the waste tire is energy recovery. A used waste tire contain more than 90% organic material and has a calorific value of about 32,000,00J/kg which increased the competition to other fuels (Sienkiewicz et al, 2012). Mostly, the cement industry will highly demand the tire as fuel. This is because the tire will be burned in a cement kilns at temperature above 1200 ℃ and under this temperature the tire will fully combusted. The ash and wire mesh are bound to the clinker and it is does not pollute the environmental as the lower emission compare as coal combustion. Beside the cement industry, waste tire also used as a feedstock for the production of stream, electrical energy, paper manufacturing industries, lime and steel processing (Uruburu et al, 2012; Sienkiewicz et al, 2012).

2.3 Nylon

2.3.1 Introductions

Nylon is one of the most widely used polymer produced from condensation polymerization which was discovered by Wallace Carothers at DuPont and commercialized in 1938 as synthetic fibre. The nylon chain are aligned in parallel manner with strong hydrogen bonds and other secondary bonding between the individual chains which holds the chain together tightly. This is the reason for characteristic of nylon that exhibit excellent textural properties, mechanical strength, and toughness as well as high chemical resistance and thermal stability (Ting et al, 2014). Nylon is a polyamide that formed through polycondensation of hexamethylene diamine and a dicarboxylic acid at industrial scale (Swift, 2005). Different nylon can be produced base on the length of the di-acid and different length of nylon will gives different properties, which gives a broad range of applications. In this research, nylon-6 was used and Figure 2.2 show the chemical structure of nylon-6.

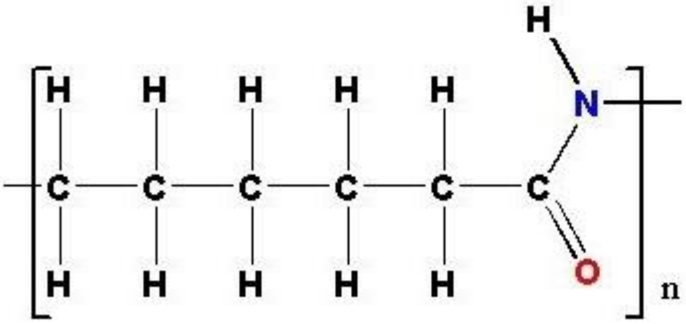

Figure 2.2: Chemical Structure of Nylon (Sherman, 2015)

2.3.2 Properties of Nylon

Nylon basically come from the family of polyamide so its properties mainly followed the polyamide properties. The important properties of nylon are the high oil and solvent resistance, toughness, fatigue and abrasion resistance, low friction and creep, stability at elevated temperature, fire resistance, good appearance and good processing ability.

The nylon properties mainly is determined by the extend of hydrogen bond in between the adjacent chains. The T_m of nylon increases with increasing the amide link (CONH group) to alkyl (CH_2) groups in the chain. The increment of crystallinity will lead to higher stiffness, density, tensile and yield strength, chemical and abrasion resistance and better dimensional. However, the crystallinity decreases the elongation, impact resistance, thermal expansion and permeability of the nylon. Moreover, water absorption is one of the properties of nylons too. By increase the crystallinity of nylon leads to higher ratio of amide group which is responsible for high water adsorption (Laurati et al, 2014).

Table 2.3: Properties of Nylon (Gruszka et al, 2005)

Density	1.14g/cm^3
Abrasion resistance	Very high
Elasticity	Very high
Sensitiveness toward exposure to sunlight	Very high
Softening point	175 ℃
Melting point	218 ℃
Susceptibility to electrification	Very high
Susceptibility to pilling	Very high
Shrinkage in boiled water	8-12%
Solvents	Phenol, Formic acid, Hydrochloric acid

2.3.3 Application of Nylon

Nylon has a wide range of usage which are involving in appliances, business equipment, consumer products, electrical and electronic devices, furniture, hardware, machinery, packaging and transportation. Firstly, the transportation consume a lot of nylon and this provide the largest market for nylon. Commonly, unmodified nylon are applied in electrical connector, wire jackets, windshield wiper and speedometer gears, and emission canisters. Besides, nylon that are softer can be used in fuel lines, hoses of air brake and coating of spline shaft. In addition, mineral filled nylon can be found in wheel caps, radiator grilles and mirror housing. Moreover, nylons that contained both minerals and glass fibre will be applied in exterior parts of the vehicle. Meanwhile, electrical and electronic industries in Western Europe and United States contribute a major nylon market because of its flame retardant properties.

Nylons uses including color-coded components, plugs, connectors, coil forms, wiring devices, terminal block and harness ties. For the industrial applications, the nylons are popular due to its well fatigue resistance and excellent impact strength particulars. The use of polyamide with superheated water in industrial applications could involve foam extrusion applications, spray coating applications, and recycling (Vinken, 2008). However, the spinning of fibres from a water solution at elevated temperatures and pressures is unable to be achieved. The nylon can be applied to sport industries because of its toughness for producing sports equipment, such as ski boots, racquet, and ice skate. The glass reinforced nylons which have the high impact characteristic which can be applied in electrical appliances tool, engine fans, radiator, brake and power steering wheels and clutch pedals. Lastly, nylon will be made into film for food packing or container by extrusion or moulding

CHAPTER 3

METHODOLOGY

3.1 Introductions

This chapter illustrates on the materials used for preparing nylon-6/WTP blend, the name of suppliers and the physical properties of materials. Besides, the composite preparation method and the conditions were discussed. Finally, the characterization of materials and evaluation method of the composites such as Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Differential Scanning Calorimetry (DSC), Tensile Test, Impact Test and Swelling Resistance.

3.2 Raw Materials

3.2.1 Nylon-6

The nylon-6 used is from Sigma-Aldrich Company. The properties of the nylon-6 pellets is tabulated in table 3.1.

Table 3.1 Properties of the Nylon-6 pallet

Product name	Nylon-6 pellets
Product number	181110
CAS Number	25038-54-4
MDL	MFCD00133998
Formula	$(C_6H_{11}NO)_n$
Appearance colour	Colourless to white to pale yellow
Appearance form	Pellets
Auto-ignitions Temperature	815 °F
Transition Temperature	T_m : 228.5 °C; T_g :62.5 °C
Density	1.084 g/ml at 25 °C (lit.)

3.2.2 Waste Tire Powder (WTP)

WTP, a recycled product which was granulated mechanically from scrap tires which was bought from Mega Makmur Saintifik Sdn. Bhd. The average particle size of WTP was 70 µm.

3.3 Preparation of Nylon-6/Waste Tire Powder Composites

The composites were prepared by melt blending method using rheometer Brabender® Plastograph® EC 815652. The composite mixing was carried out based on the compounding formulation as in Table 3.2**.** The mixing was carried out at melting temperature of nylon-6, (230 ℃) for 7 min at 60 rpm. The graph of processing torque of the composites was obtained from the Brabender. The nylon-6/WTP composites were pressed into 1mm film using hydraulic hot and cold press machine GT-7014-A30C at 260 ℃. The composite were first preheated for 8 minutes and pressed for 4 minutes, followed by 3 minutes of cooling.

Table 3.2: Ratio of nylon-6/WTP.

Composition (wt%)	
Nylon-6	WTP
100	0
90	10
80	20
70	30
60	40

3.4 Characterization

3.4.1 Fourier Transform Infrared Spectroscopy (FT-IR)

FTIR was carried out using Perkin Elmer Spectrum ex1 to identify the types of chemical bonds and functional groups in neat nylon-6 and nylon-6/WTP composites. The analysis was carried out to determine the absorption band with a wavelength from 4000 cm^{-1} to 400 cm^{-1} with 2 scans at a resolution of 4 cm^{-1}.

3.4.2 Scanning Electron Microscopy (SEM)

The morphology of neat nylon-6 and nylon-6/WTP composites were observed using SEM at accelerating voltage of 2kV. Prior to scanning, the samples were placed on a disc and held in place using a double-sided carbon tape then coated with platinum particles to avoid sample charging. The model of equipment used was JOEL JSM 6701F.

3.4.3 Differential Scanning Calorimetry (DSC)

Mettler Toledo TOPEM was used for determining the melting points, crystallinity and crystallising temperatures of the nylon-6 and nylon-6/WTP composites. The samples were heated from 25 ℃ to 300 ℃ at a rate of 10 ℃/min under nitrogen flow of 10 ml/min. This was immediately followed by cooling back to 25 ℃.

The degree of crystallinity is calculated using equation 3.1:

$$X^{m}_{c} = \frac{\Delta H_m}{W_p \ x \ \Delta H_{100}} \ x \ 100\% \qquad (3.1)$$

Where

X_c= Degree of crystallinity (%)

ΔH_m = Melting enthalpy (J/g)

ΔH_{100} = Melting heat for 100% crystalline nylon-6, 240J/g

W_p = Weight fraction of polymer in sample

3.5 Performance Test

3.5.1 Tensile Test

According to ASTM D638, tensile test was performed under an ambient condition in order to measure the elastic modulus, ultimate tensile strength and elongation at break of nylon-6 and various filler loading of nylon-6/WTP composite. The test was carried out through Tinius Olsen H10KS-0748 with 500 N load cell and at a crosshead speed of 50 mm/min. A least 10 tensile tests were performed for every individual loading of WTP to obtain average values. Scanning Electron Microscopy (SEM) was conducted to evaluate the fractured samples cross-section morphology.

3.5.2 Impact Test

Izod impact test is carried out with the sample dimension 44 mm*12.7 mm* 3.2 mm and the load applied was 7.5 J and ASTM D256 standard on un-notched samples was followed. Zwick Impact tester was used to measure the total impact energy (Joule) required to fracture the standard test specimen. The impact strength of the specimens will be calculated by using equation 3.2.

$$\text{Impact strength} = \text{Impact load} / \text{cross sectional area of the specimen} \quad (3.2)$$

3.5.3 Swelling Resistance

Nylon-6 and nylon-6/WTP samples were cut into dumbbell shaped from the compression moulded sheet. Then, swelling test was carried out using toluene as a solvent in accordance with ASTM D471-79. The test pieces were weighed using an electronic balance and initial mass (M_0) and were recorded in grams. The test pieces were then soaked in toluene for 72 hours and at room temperature, 25 ℃, in dark place. After 72 hours, the test pieces were weighed again and the mass of the specimens after immersion (M_1) in toluene were recorded. Solvent resistance of composite was determined from the swelling percentage of samples in toluene. The higher swelling percentage indicates the lower solvent resistance of composite. Swelling percentage was calculated based on equation 3.3.

$$\textbf{Swelling Percentage} = [(M_1 - M_0) / M_1] \times 100 \quad (3.3)$$

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter will discuss about the experimental results of waste tire powder as filler in nylon-6 composites. The effect of WTP loading on the processing, mechanical properties, morphological properties, chemical resistance, FT-IR spectroscopic analysis and DSC of nylon-6/WTP composites was investigated.

4.2 Characterization of Nylon-6/WTP composites

4.2.1 FT-IR Spectroscopic Analysis

An infrared spectrum can detect the unknown functional group of a sample with absorption peaks which correspond to the frequencies of vibrations between the bonds of the atoms. Different material will have unique combination of atoms and thus, there are no two compounds that producing the same infrared spectrum. Figure 4.1 shows FT-IR result of neat nylon-6, the peak 3290cm^{-1} and 3082cm^{-1} indicate the presence of free stretched amide (N-H) groups of the samples (Nanjundaswamy & Dasnkoppa., 2012). Beside, according to Abdalla Abdal-hay with co-workers, the amide II band will shows a peak on approximately 1545 cm^{-1} wavelength. Hence, the absorption band at 1551 cm^{-1} is assigned to the amide II band vibrations. In addition, the others properties of amide absorption bands such as bands 1264 cm^{-1} and 1202 cm^{-1} (Amide III, C-N-H vibration), 691 cm^{-1} (Amide V), 1462 cm^{-1} (C-N group,) and 1171 cm^{-1} (Stretching C-N group) can be observed from the result (Wang et al, 2014; Thombare et al, 2013).

Wang claimed that infrared absorption band that near to 2936 cm^{-1} and 2869 cm^{-1} are belonged to symmetric and stretching vibration of $–CH_2$ while 1373 cm^{-1} belonged to methylene group. Moreover, the absorption band 2370 cm^{-1} and 2345 cm^{-1} are mainly contributed to the presence of carbon dioxide while 2186 cm^{-1} is assigned to carbon monoxide existence (Glascoe et al, 2009; E.V. Benvenutti et al, 1999). Lastly, there are few functional groups can be viewed from the FT-IR result, such as peak 1639 cm^{-1} is assigned to the hydroxyl group (OH), peak 1122 cm^{-1} is assigned to Carbonyl compound (C-CO-C) stretching, peak 973 cm^{-1} is dedicated to deformation of tertiary methyl groups and absorption band of 960 cm^{-1} is due to the stretching of alkene group (C=C) and peak 579 cm^{-1} contribute to the existence of the alkene deformation respectively (Pang, 2013; Ragavendran et al , 2011; Chipara et al, 2012; Bacher, 2002).

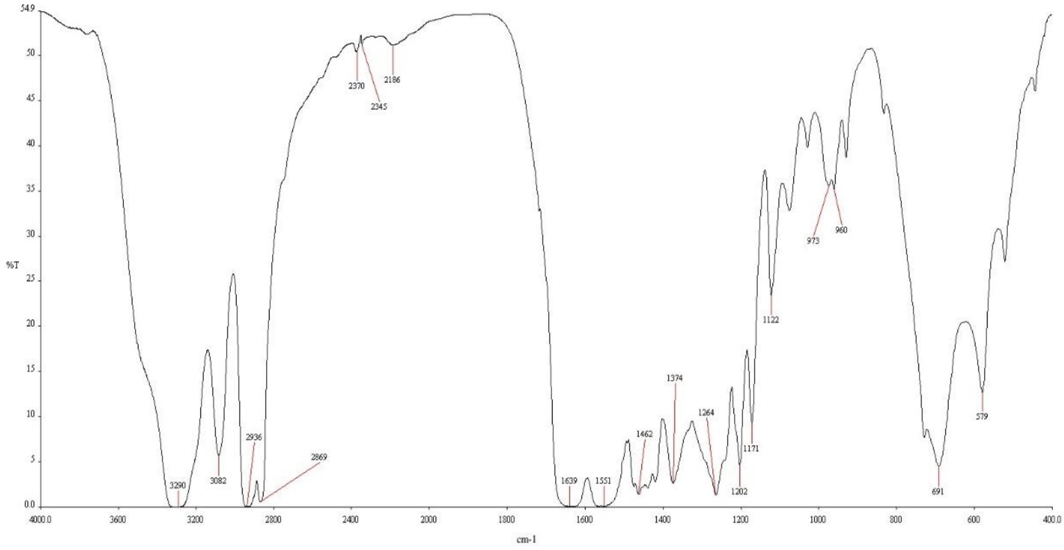

Figure 4.1: The FT-IR spectrum of Nylon-6

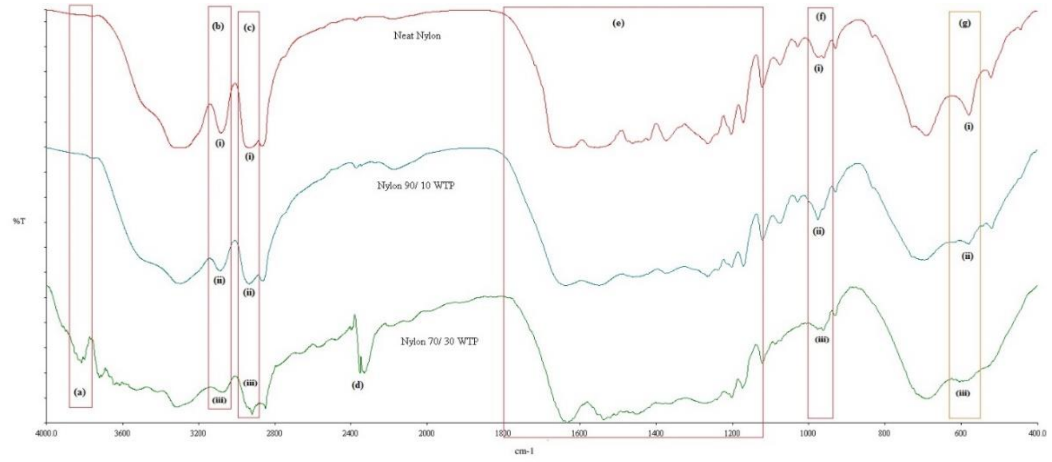

Figure 4.2: The FT-IR spectrum comparison of Nylon-6 and WTP filled Nylon-6 Composites

Figure 4.2 shows the comparison of neat nylon-6 and WTP filled nylon-6 composites. From Figure 4.2, region (a) showed a peak of 3813 cm^{-1} after the WTP loading had increased to 30 wt% and this indicates the presence of hydrocarbon compound in WTP (Unapumnuk., 2006). Besides, region (b) and (c) show a shifted absorption peak from (i) to (iii) which is from 3082 cm^{-1} to 3079 cm^{-1} and from 2936 cm^{-1} to 2916 cm^{-1} respectively. In addition, as the WTP increase to 30 wt%, there is a peak formed in region (d), absorption band formed between 2400 cm^{-1} and 2000 cm^{-1} which indicates amount of carbon dioxide increase due to partial dissociation of oxygen during heat processing (Seiferh et al, 1998). However, in region (e) the absorption peak from 1800 cm^{-1} to 1150 cm^{-1} is retained. In addition, region (f) and (g) illustrate that the absorption peak are shifting and reduced from (i) to (iii) which is from 975 cm^{-1} to 960 cm^{-1} and from 579 cm^{-1} to 560 cm^{-1} each. Hence, all the peak of nylon-6 in that region are still able to be observed either in nylon-6/WTP 10 wt% or nylon-6/30 wt% which proves that there are no chemical reaction in between nylon-6 and WTP.

Table 4.1: Table of chemical composition with absorption peak in nylon-6 FT-IR

Assignments	**Absorption peak / band (cm^{-1})**
Amide group	3290, 3082
Amide II	1551
Amide III	1264 , 1202
Amide V	691
C-N group	1462
Stretching C-N group	1171
Symmetric and stretching methylene, CH_2	2936, 2869
Carbon dioxide, CO_2	2370, 2345
Carbon monoxide, CO	2186
Hydroxyl group, OH	1639
Stretching Carbonyl compound, C-CO-C	1122
Tertiary Methyl groups	973
Stretching Alkene group, C=C	960
Alkene deformation	579

4.2.2 Processing Torque

The mixing torque can be used to determine the rheological property and processing parameters of the polymeric systems, such as processing temperature, rotation speed and filler loading (Cai et al, 2013). Figure 4.3 shows the torque versus time curves for nylon-6 and nylon-6/WTP composite with various loading of WTP. All the nylon-6 and nylon-6/WTP compounding were pre-mixed before put into the internal mixer. Thus, only a maximum peak can be viewed from Figure 4.3. The mixing torque usually very high from the beginning of the mixing due to the high viscosity of the composites. However, during 4th mins or 240 sec, the torque decrease slowly because of the solid mixture start to melt and flow during the shear for being applied in the mixing chamber.

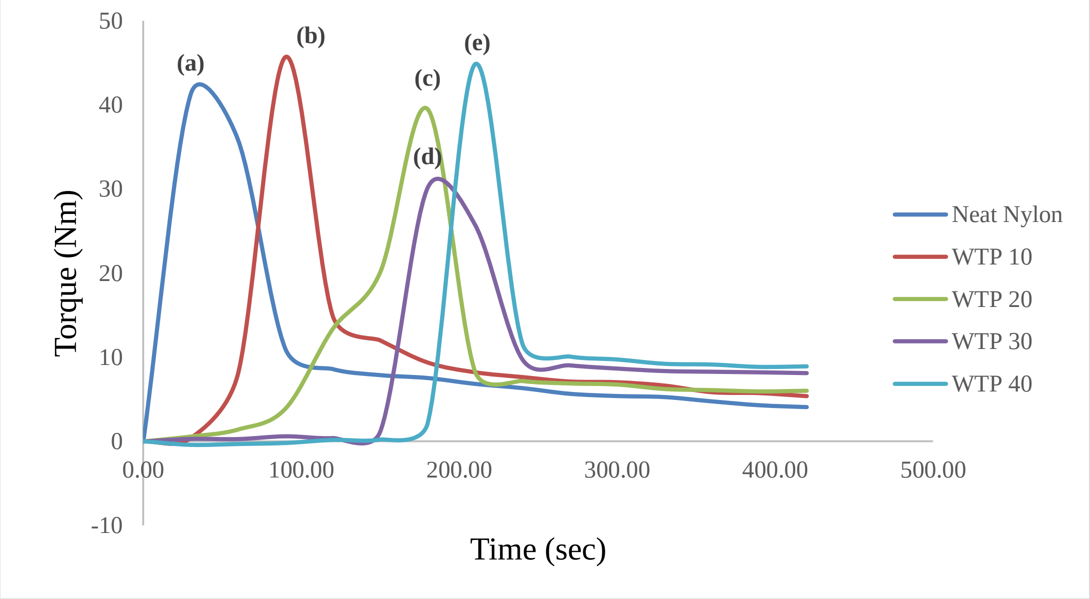

Figure 4.3: The mixing torque of nylon-6 and nylon-6/WTP composites

From Figure 4.3, the processing torque for nylon-6 is being shifted from point (a) to (e) while WTP loading increases. The melting of nylon-6 which start at point (a) was prolonged with addition of WTP because the heat supplied for melting nylon-6 was absorbed by WTP while blending nylon-6/WTP composites.

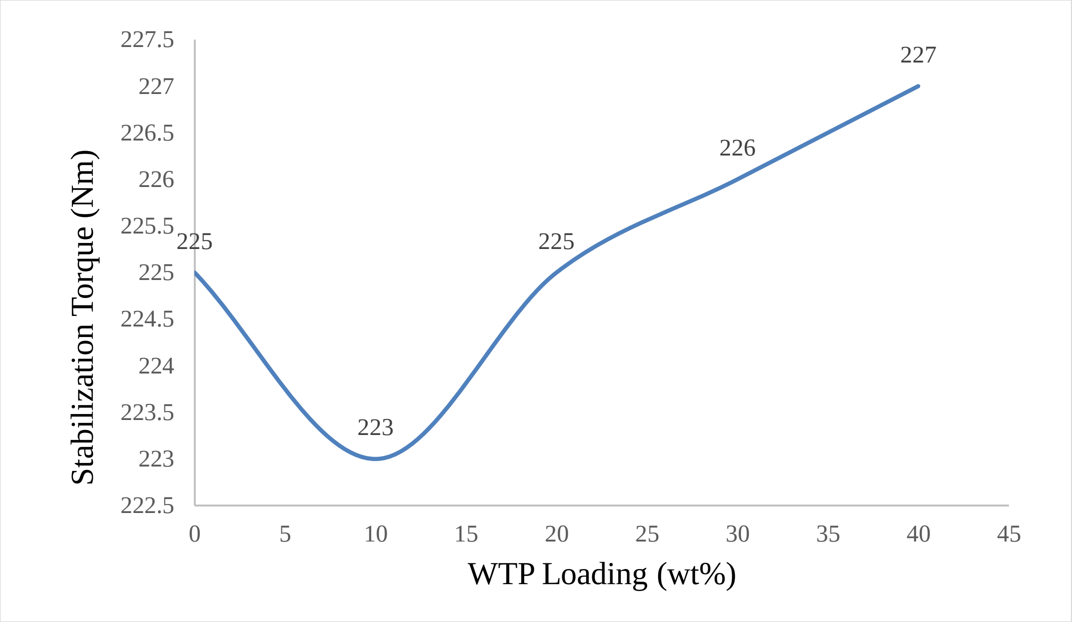

Figure 4.4: The effect of WTP loading on the stabilization torque of nylon-6/WTP composite

Figure 4.4 showed the stabilization torque of WTP filled nylon-6 composite. The stabilization torque can be defined as the completion of melting and almost constant viscosity of neat nylon-6 (Othman, 2007). When the filler loading is 10 wt%, the stabilization torque of the composite is the lowest. This indicate that the filler amount is not restricting the matrix flow and the viscosity of WTP filled nylon-6 composite. In addition, the low amount of WTP filler will have lesser friction to the internal mixer’s rotor resulted reduction of viscosity as well as the torque (Waldman & De Paoli., 2013). However, there are an opposite trend of the torque from 20 wt% to 40 wt% of WTP loading. This eventually happen is due to the flow of the nylon-6 matrix is restricted by the WTP. Furthermore, the amount of filler increase will thereby exert greater resistance to the torque created in the internal mixer’s rotors. Moreover, Othman (2007) investigated that the irregular shaped filler particles which is dispersed to the nylon-6 matrix will destroy the slip movement of the matrix and thus increase the resistance toward the flow of the composite (Balakrishna, Ismail. & Othman, 2013).

4.2.3 Differential Scanning Calorimetric (DSC)

Differential Scanning Calorimetric (DSC) is a method for analyzing first order transition of a polymeric material such as melting and crystallization. Thus, performing DSC analysis on the nylon-6/WTP to examine the possible changes in the crystalline structure and overall degree of crystallinity of the composite. The thermal parameters such as melting temperature (T_m), crystallization temperature (T_c), melting enthalpy (ΔH_m) and heat of crystallization (ΔH_c) were recorded and showed in Figure 4.3.

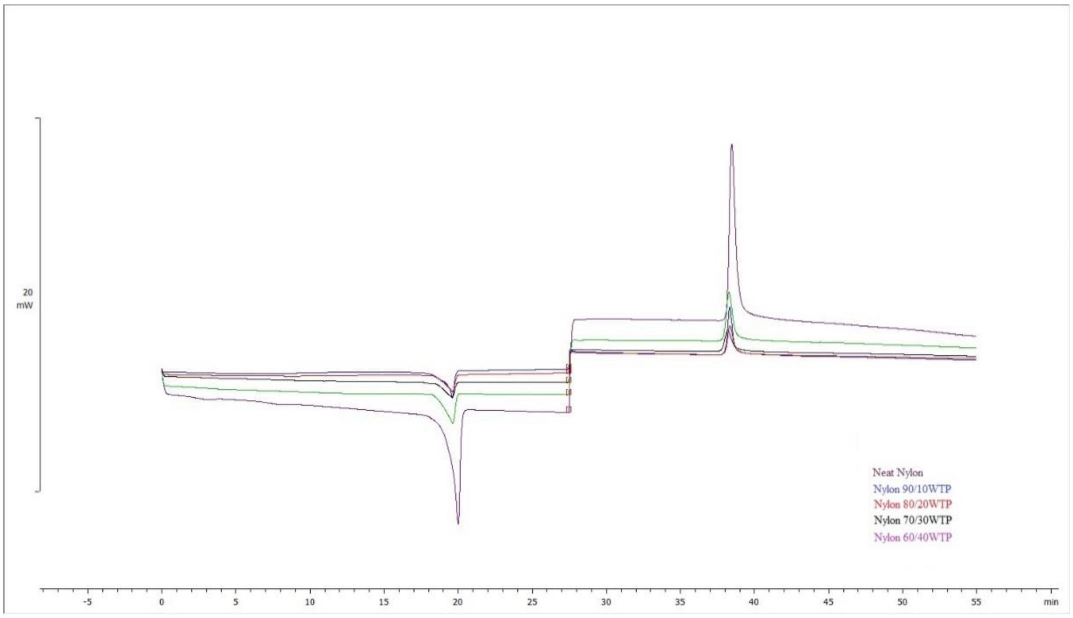

Figure 4.5: DSC of nylon-6/WTP loading composites (Heating & Cooling)

The differences between T_m and T_c of nylon-6/WTP as compare to neat nylon-6 is about 1.23% and 1.5% respectively. The result reveal that there are no significant changes in T_m and T_c of nylon-6 with increasing WTP loading which also suggest that melt blending of nylon-6/WTP can be carried out at similar processing condition as that of neat nylon-6.

Table 4.2: Thermal parameters of the nylon-6/WTP composites during the crystallization and melting process.

WTP loading	**T_m (℃)**	**T_c(℃)**	**ΔH_m(J/g)**	**ΔH_c (J/g)**	**Degree of crystallinity (X_c)**
0	190.91	224.54	90.72	-88.32	36.8
10	191.82	221.07	58.35	-58.6	24.42
20	191.77	220.76	66.20	-60.88	25.37
30	193.26	220.74	50.90	-45.97	19.15
40	192.53	221.17	56.14	-47.06	19.61

Table 4.2 recorded the summary of melting temperature and crystallization temperature of nylon-6/WTP composites. From the table, the crystallisation temperature (T_c) is decreasing when the loading increase to 40 wt %.The crystallization temperature (T_c) of modified nylon-6 is lower than the neat nylon-6 because of the presence of waste tire powder can restricts the surface nucleation sites, decreasing the crystallization of matrix nylon-6 as a result. However, the heat fusion (ΔH_m) and crystallization (ΔH_c) of nylon-6/WTP are lower than the neat nylon-6 and there are no significant different among all WTP filled nylon composites. The percentage of crystallinity (X_c) of nylon-6 was calculated using a heat of crystallisation of 240 J/g of 100 % crystalline nylon-6 (Fornes & Paul, 2003). The percentage of crystallinity (X_c) are relatively lower when there is the presence of waste tire powder. With addition of WTP, the mobility of nylon-6 chains are decreased which results in high viscosity of the nylon/WTP-6 blend. This result is in good agreement with the torque value obtained which also shows an increasing torque or stiffness of composites as compared to neat nylon-6. When the chain mobility is restricted, the growth of crystalline structure is retarded or hindered which may result in low crystallinity of WTP filled nylon-6 composites as compared to neat nylon-6.

4.3 Mechanical Properties

4.3.1 Tensile Properties

The tensile properties of composites is based on the interaction of the filler (Waste Tire Powder) and the polymer (nylon-6) matrix. The adhesion between the particles and the polymer matrix depends on the cross sectional area and surface area as well as the strength. Some of the mechanical properties data are presented in Table 4.3.

Table 4.3：Tensile Test Results of nylon-6/WTP.

Parameter	**WTP loading**				
	0	10	20	30	40
UTS	59.7	32.5	28.0	16.6	11.5
E-Modulus	594.7	579.6	562.4	392.0	325.2
Elongation at break	126.5	18.1	23.1	11.3	10.0

Table 4.3 shows the tensile properties test parameters of different nylon-6/WTP ratio, which are ultimate tensile strength (UTS), E-modulus (E_{Mod}) and elongation at break (E_b). Based on the table, the incorporation of WTP into nylon-6 results decrease in all the tensile properties of nylon-6.

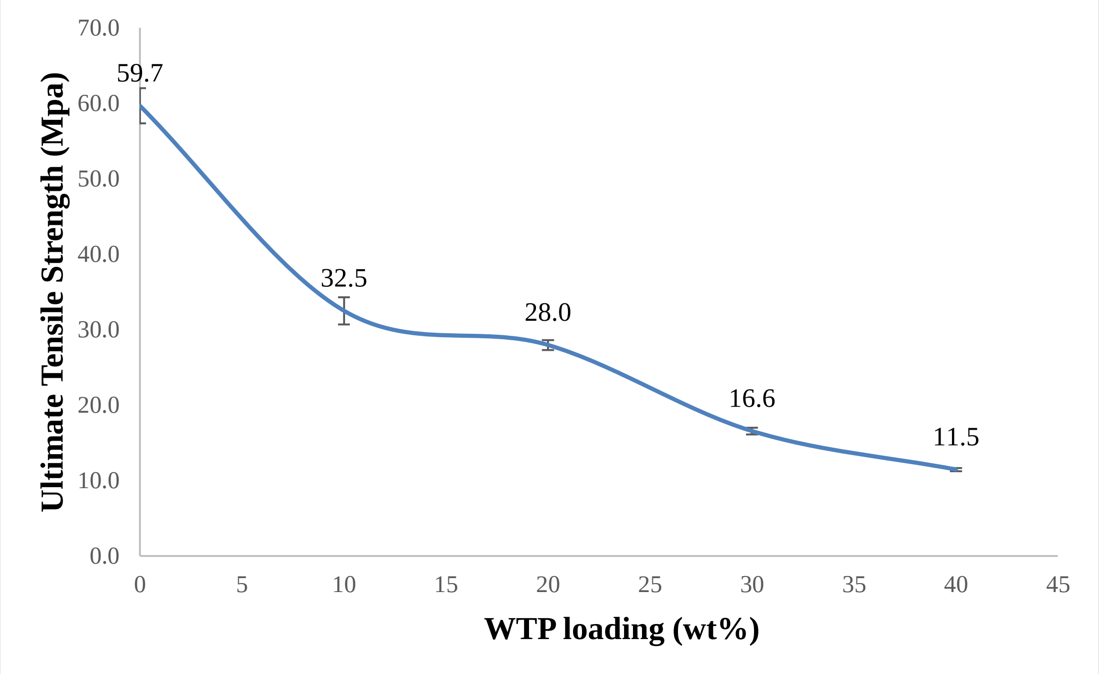

Figure 4.6: The effect of WTP loading on UTS of nylon-6/WTP composites

The ultimate tensile strength (UTS) is a parameter used to determine the energy that can be withstand by polymer while being stretched or pulled. This is depends on the adhesion between the filler and polymer matrix by accounting the size of the interface and the interaction force (Othman, 2007). From Figure 4.6, as the WTP loading increase, tensile properties of the composite decrease because of the poor interfacial adhesion between nylon-6 and WTP which was resulted from non-homogenous dispersion of WTP in nylon-6 matrix. Besides, the strength of the composite mainly is from nylon-6 matrix, addition of WTP will either increase or decrease composites tensile strength based on the interaction of WTP with nylon-6. In this research, UTS was decreased due to incompatibility between WTP and nylon-6. According to Pang (2012), both polypropylene (PP) and WTP exhibit hydrophobic properties, when WTP is added to PP, a strong interfacial adhesion is formed between PP and WTP. On the other hand, nylon-6 exhibits hydrophilic nature as the present of the amide group meanwhile WTP is hydrophobic and, hence, the incompatibility between nylon-6 and WTP will form a weak interfacial adhesion which lead to the decreasing UTS.

Furthermore, as the WTP loading increase, the WTP act as the stress concentrators in the polymer providing nucleation sites for plastic deforming and crack initiating. This can be seen determined from the SEM morphology.

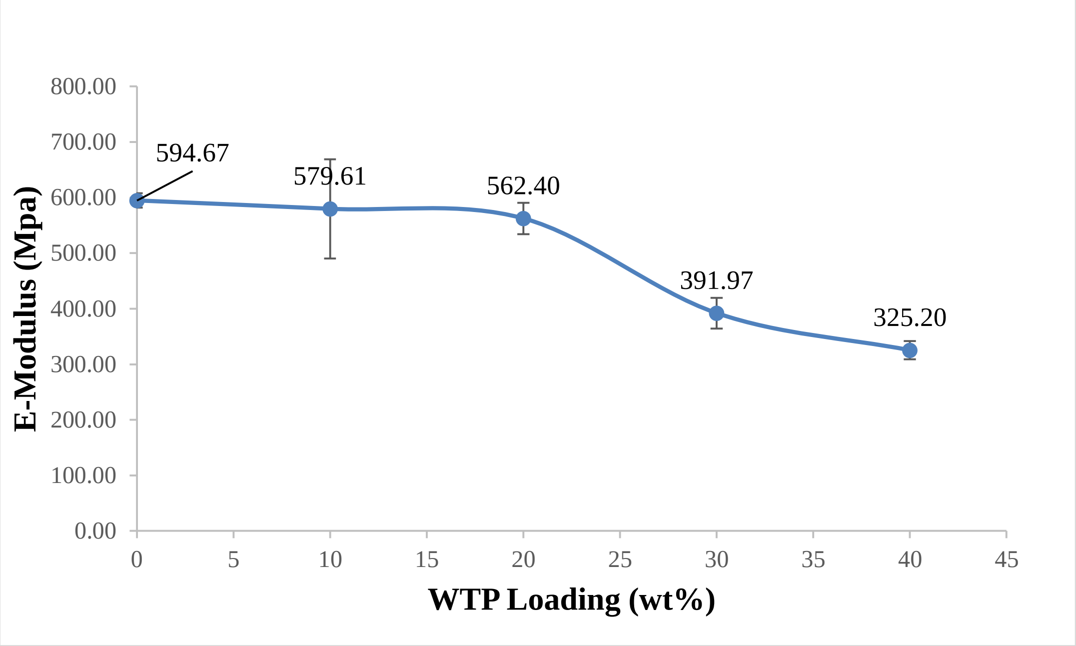

Figure 4.7: The effect of WTP loading on E-modulus of nylon-6/WTP composites

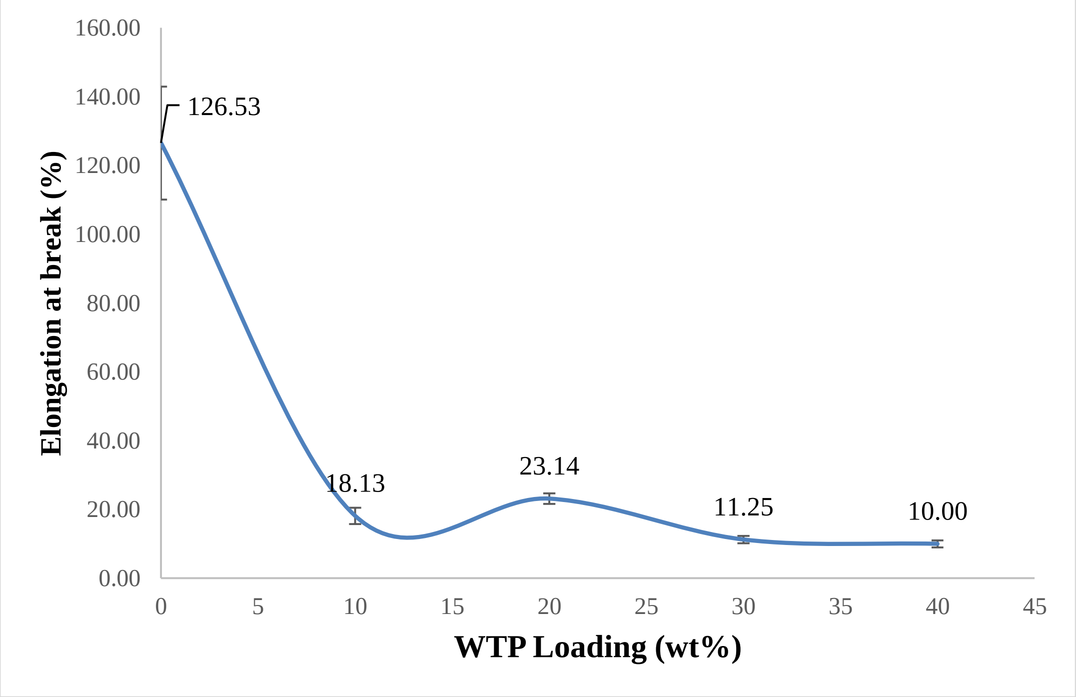

Figure 4.8: The effect of WTP loading on elongation at break nylon-6/WTP composites

E-modulus is used to determine the stiffness or brittle of filled polymer. From Figure 4.7, stiffness of the composites decrease while WTP loading is increase. This is because the WTP is an elastomer which will disperse in thermoplastic matrix and providing elasticity toward composite but not the stiffness of the composites. Nevertheless, at higher WTP loading the domination of interaction between WTP and nylon-6 can be expected to reduce and is being replaced by higher WTP interaction between WTP particles (Othman, 2007). However, as can be seen from table 4.3 and Figure 4.8, the elongation at break of the composites is decreasing when WTP loading increase. Elongation at break is defining the rupture behavior of composites materials. The addition of elastomers normally will increase the elongation because of the elastic properties of the elastomers. Although the loading of WTP increase, the elongation at break of composites was decreased. Conventionally, WTP has hydrophobic characteristics (Lin, Huang & Shern., 2008) and nylon-6 has hydrophilic properties is attributed by amide groups. The incompatibility of their phases results in low adhesion and possibility of micro-voids formation on the interface. Subsequently, unstable WTP-nylon-6 interface is less capable of transferring the stress applied from the nylon-6 matrix to WTP phase efficiently (Pang, 2013). The reduction of elongation at break might be caused by the over curing of WTP during melt blending and hot press process with high temperature (260 ℃) which is relatively higher processing temperature compare to other thermoplastic processing temperature, especially PP. Normally, the stiffness and elasticity of over cured WTP will be decrease and thus these are the reasons of the decreasing value of E-modulus and elongation at break of the composites with increased WTP loading (Pang, 2013).

4.3.2 Impact Properties

The impact test is to determine the toughness of a polymer which energy required to cause fracture at high rates of force application. The toughness is related to the amount of energy can be absorb or disperse by a material, in the form of deformation before the exerted force fractured it. (Brostow, Lobland & Khoja., 2015)

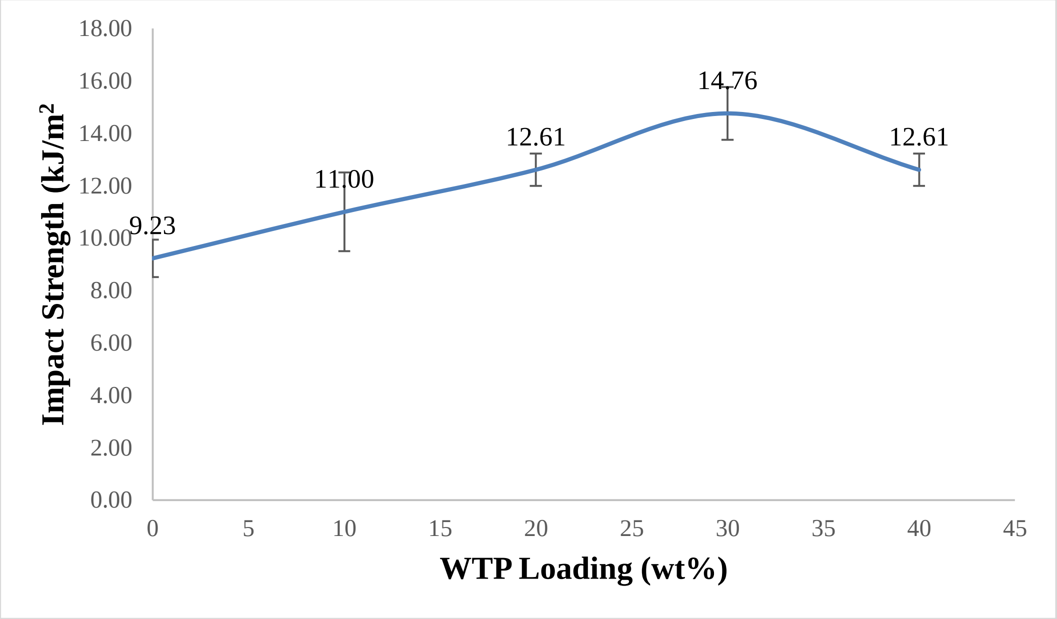

Figure 4.9: The effect of WTP loading on the impact strength of nylon-6/WTP composites

From Figure 4.6, the impact strength of the composite was increasing and reach the optimum value when WTP loading is at 30 wt% then the impact strength decrease. Normally, the impact strength of nylon-6 will be higher due to its bulk and non-oriented polymer arrangement (Meijer & Govaert., 2005). However, when comes to the filled composite, the impact strength will depend on the properties of the filler, the orientation of the filler and the interaction of filler and polymer matrix (Alcock, 2004). The elasticity of WTP in the rubberized composites capable to resist crack propagation and able to experience a higher deformation than the neat nylon-6. Thus, the samples can deform elastically results the increment of the impact strength. Nevertheless, while WTP loading is at 40 wt%, the high content of WTP will create an irregular voids with the matrix of nylon-6 which leads to the poor interfacial adhesion between the composite components. Thus, when the force exerted to the weak interfacial region, the impact strength decreased (Othman, 2003). Poor interfacial adhesion between WTP and nylon-6 can be seen from the SEM morphology of tensile fractured surface of 40wt% WTP filled nylon composites.

4.4 Swelling Test

Swelling test usually conducted to measure the crosslink density of elastomers and the chemical resistance of a composites material by measure the solvent uptake of the composites after immersed into the solvent for a particular time. (Barlkani & Hepburn., 1992) Figure 4.4 illustrate the increased weight percentage of neat nylon-6 and WTP filled nylon-6 composites.

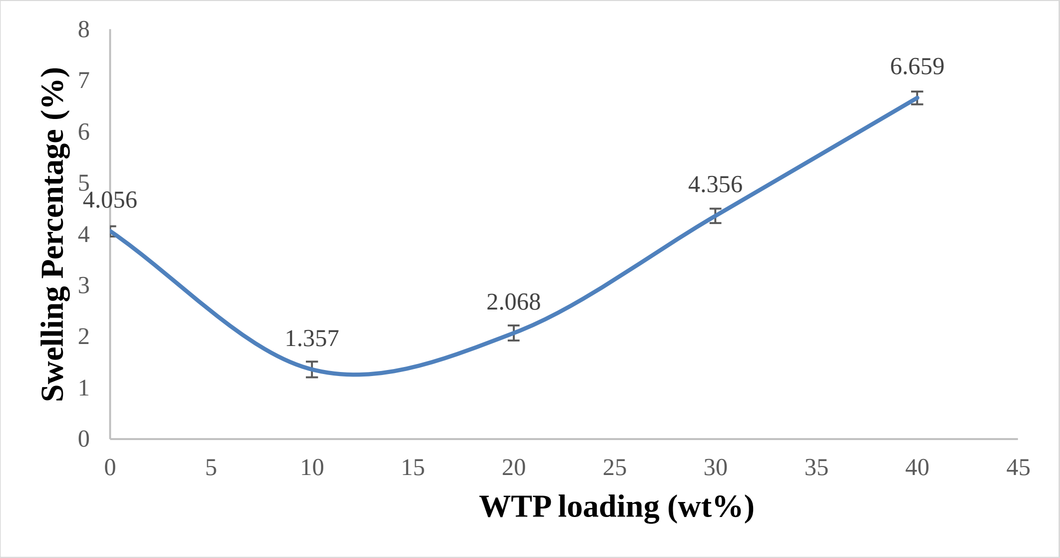

Figure 4.10: The effect of WTP loading on swelling percentage of nylon-6/WTP composite

Commonly, the weight percentage will decrease as the filler loading increase because increased amount of crosslink will prohibit the intruding of toluene into the composites (Awang, Ismail & Hazzian, 2007). Thus, when filler loading at 10 wt%, the weight percentage decrease due to the molecular interaction between WTP and nylon matrix restrict the intruding of the toluene. However, when WTP loading increase from 20 wt % to 40 wt%, the swelling percentage increase due to poor adhesion between WTP and nylon-6 which allow more solvent uptake. Besides, in-cooperating of WTP at higher loading results in micro cracks which become the path for solvent penetration into the composite. Subsequently increased the swelling percentage of nylon with increased WTP loading.

4.5 SEM Morphological Observations

From Figure 4.11(a), the surface of neat nylon-6 is smooth and indicates that the mechanical properties such as tensile strength and E-modulus is strong and excellent. When the WTP is introduced to the matrix, the tensile strength and E-modulus decrease, this can be proven by observing Figure 4.11(b) and Figure 4.12 (a). Although a smooth surface is formed, there is still agglomerations of WTP on the surface of the polymer matrix which proves the low adhesion between WTP and nylon-6. From Figure 4.11 (c), there is a rough surface form when the WTP loading increase to 20 wt% but there is a void occurred on the surface of nylon-6 matrix which can be observed from Figure 4.12(b). This proved that WTP does not form a good interaction with nylon-6 which resulted the decrement of tensile properties. As the WTP loading increase to 30wt%, there are phase separation happen between WTP and nylon-6 as shown in Figure 4.11(c) and Figure 4.12(d). From Figure 4.11 (c), the evidence of incompatibility between nylon and WTP is revealed from the detachment of WTP from nylon-6 interface. Besides, as the WTP loading increased to 40wt%, the nylon-6 matrix is completely covered by WTP which as shown in Figure 4.11 (e). The occupied matrix unable to withstand the pulling force by the tensile machine. Besides, propagation cracks and fracture can be seen on the surface and fracture of nylon-6/40wt% WTP composites which proves the over curing of WTP. Hence, the tensile properties is the lowest among the other composites.

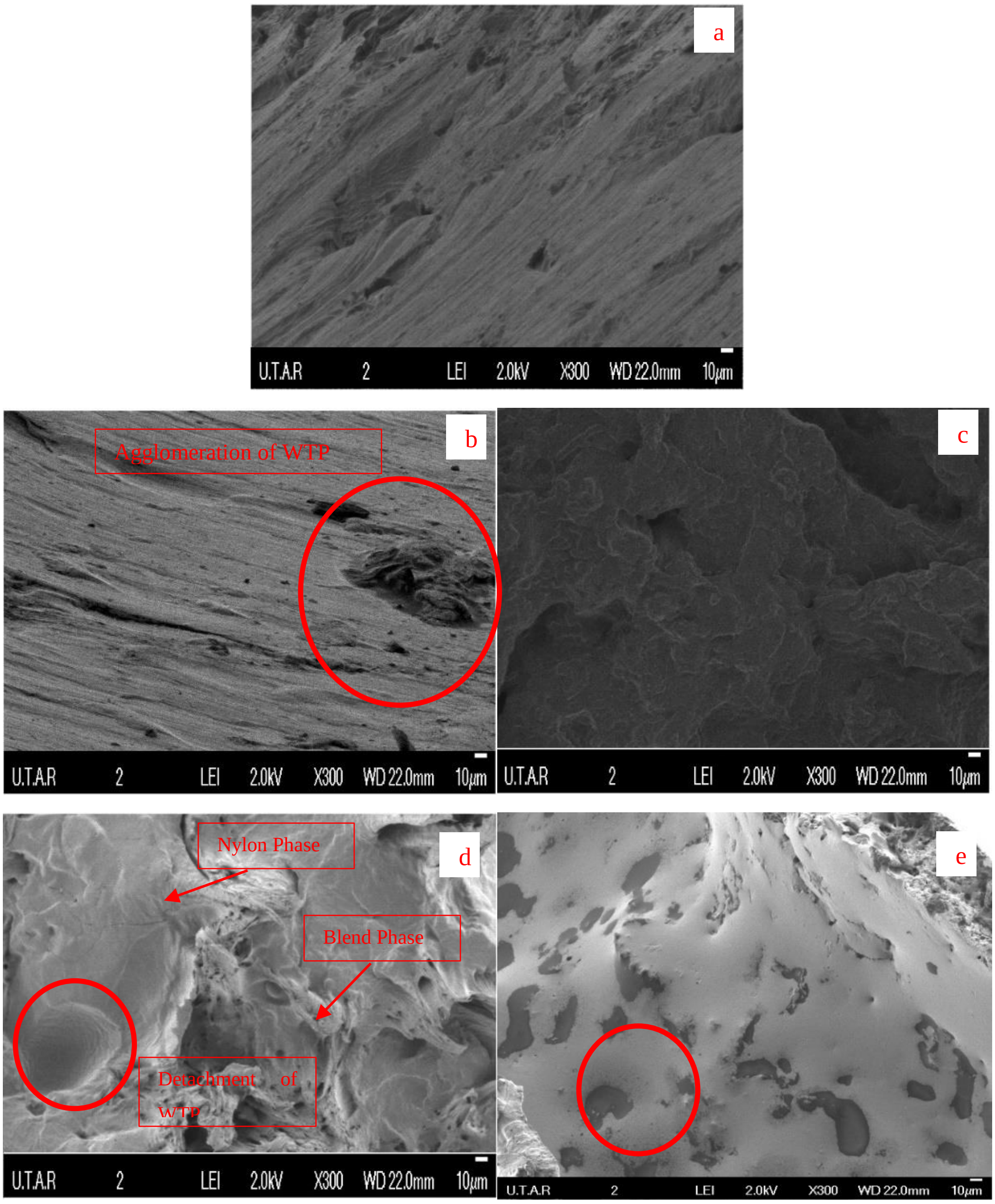

Figure 4.11: SEM micrographs of the tensile fractured surfaces of (a) neat nylon-6; (b) nylon-6/WTP composites, at 10 wt% loading; (c) nylon-6/WTP composites, at 20 wt% loading; (d) nylon-6/WTP composites, at 30 wt% loading; (e) nylon-6/WTP composites, at 40 wt% loading at 300x magnification.

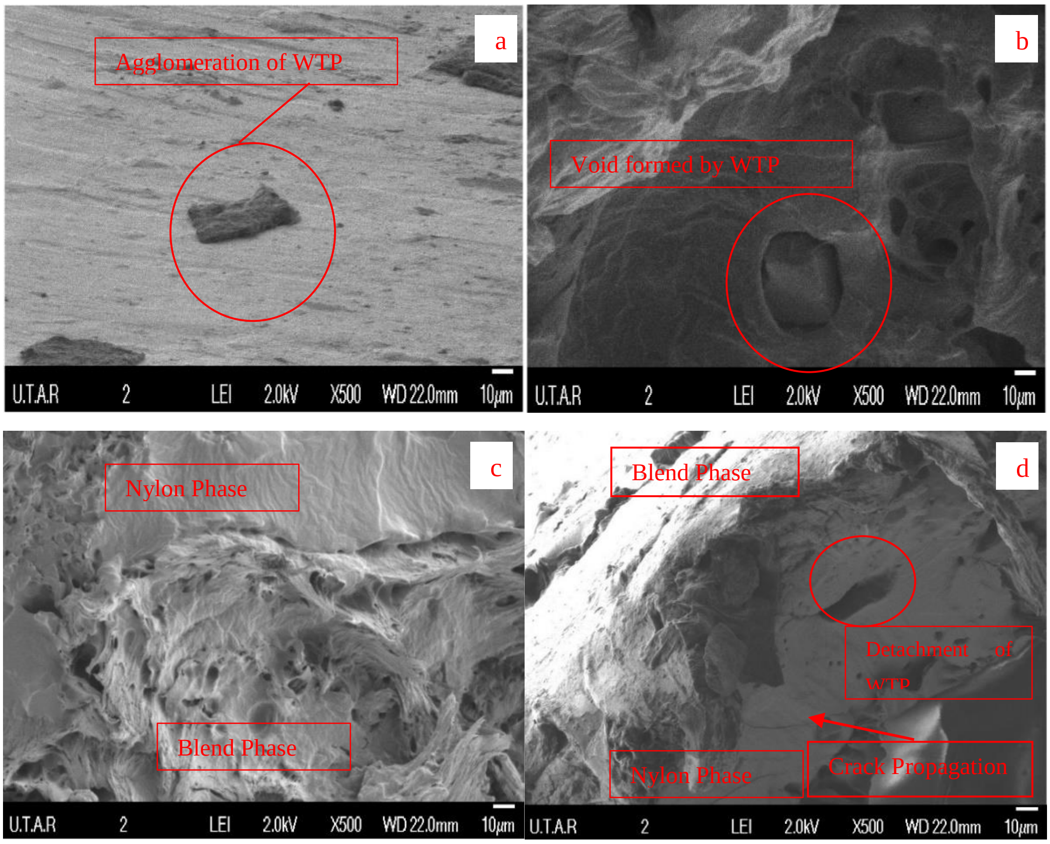

Figure 4.12: SEM micrographs of the tensile fractured surfaces of (a) nylon-6/WTP composites, at 10 wt% loading; (b) nylon-6/WTP composites, at 20 wt% loading; (c) nylon-6/WTP composites, at 30 wt% loading; (d) nylon-6/WTP composites, at 40 wt% loading at 500x magnification.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The FT-IR results implement that neat nylon-6 contain of a lot of amide group and hydrocarbon as well as carbon dioxide increase in amount when introducing WTP to produce nylon-6/WTP. Besides, the processing torque of nylon-6/WTP had been prolonged as WTP loading increase because the energy supplied to melt the composites is absorbed by WTP. For the processing conditions, nylon-6/WTP can be processed according to the conditions that processing nylon-6 because the different of T_m and T_c of nylon-6/WTP are not significant as compare to neat nylon-6.

In addition, tensile properties of nylon-6/WTP decrease dramatically as loading of WTP increase. This is mainly because of the poor adhesion between WTP and nylon-6 matrix as a result of more agglomeration of WTP while WTP is introduced at high loading. Besides, the incompatibility had caused due to WTP hydrophobic nature and nylon-6 hydrophilic properties lead to detachment of WTP and micro void formed on the surface of the nylon-6 matrix. Moreover, over curing WTP had caused crack propagation and fracture on the matrix of nylon-6. These are strongly supported by the evidence from SEM morphology of nylon-6/WTP composites. Hence, nylon-6/WTP composite had a decrement on ultimate tensile strength, E-modulus and elongation at break due to the reason stated. However, there is a increase trend on nylon-6/WTP composites as WTP loading increase. The impact resistances are primary from the elasticity of WTP elastomer properties.

For chemical resistance, swelling test had been constructed. The swelling percentage should be decreased while WTP loading increase because the cross-link density will restrict the toluene intruding to the composite. However, from this research, the swelling percentage increased as the WTP loading increased. This is because poor adhesion as well as incompatibility between WTP and nylon-6 lead to micro cracks within the composite that become the path for toluene to penetrate. Thus, the chemical resistance of nylon-6/WTP composites reduced when WTP was introduced in high loading.

In a nutshell, nylon-6/WTP composites were successfully prepared by using melt blending method. The optimum nylon-6/WTP loading is 80:20 which has a comparable process ability with improved impact and swelling resistance. However, tensile properties of the composite showed significantly low value at all WTP loading. Throughout the research, the outcome showed that nylon-6/WTP can be processed as similar to nylon-6 for impact related applications.

5.2 Recommendations

From this research, there are a few proposals for future works as listed below:

- A number of other test or evaluation can be carried out such Thermogravimetric calorimetry (TGA) depending on the application of nylon-6/WTP.
- In the future study, a coupling agent can be added to WTP for modifying WTP properties so that it can have a better compatibility with nylon-6.
- A study on size of WTP which will affects the dispersion and properties of composites should be conducted.

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