

**Polyamide 6 fibre recycling by twin-screw melt extrusion of
mixed thermoplastic polymers**

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Chapter 4

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Abstract

Textile wastes consist of multi-component materials are hardly recycled due to challenge to sort and separate the waste into a single component. Textile products dominantly produced from a non-renewable source that can be recycled several times before the end of life. Mixed-waste can be recycled together without sorting by thermo-mechanical process to produce hybrid fibres. The aim of this study was to investigate the potential on upcycling polyamide 6 (PA6) polymer mixed with secondary polymers via one-step twin-screw melt extrusion. Three secondary polymers were chosen in this study; thermoplastic polyurethane (TPU) which has interaction with PA6, and two polymers which do not have interaction with PA6; polyethylene terephthalate (PET) and polypropylene (PP). Different blending composition was prepared between PA6 and secondary polymers before being extruded into hybrid fibres through melt extrusion. The secondary polymers were then removed from the hybrid fibres to investigate the properties of the leftover of PA6 component. The fibres were characterised using attenuate total reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR) and microscopy techniques, the mechanical and thermal properties were investigated via tensile strength and differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The results showed that the blending of PA6 with interacting polymer TPU creates novel fibres morphology with multi-connected porous fibres. The mean diameter of PA6:TPU hybrid fibres when the PA6 content at 50% and 80% are 136 and 126 μm , respectively. Thermal and chemical results demonstrated strong interaction happen between PA6 and TPU. Meanwhile, the co-extrusion of PA6 with non-interacting polymer PP and PET formed PA6 micro and nanofibres in the blend, respectively. In PA6:PP blend, the SEM images show the PA6 microfibres with mean diameters of 0.76 μm and 1.13 μm developed in the hybrid fibres with PA6 content 50% and 60%, respectively. The phase inversion between PA6 and PP happened at the composition of 65% of PA6 showing the development of PA6 microfibres in a unique fibre morphology. In PA6:PET blend, PA6 nanofibres with mean diameter of 532 nm to 1026 nm were obtained. The diameter of PA6 nanofibres increase when PA6 content increase in the blend. Later, single jersey knitted fabric was produced from PA6:PP 60:40 blend composition and was treated later to remove the PP component. The treatment process exposed the development of PA6 microfibres fabric which has excellent behaviour in wicking and improved in ball burst strength compared to the untreated fabric. The success of upcycling PA6 fibres with value added properties through single-step melt extrusion can be applied to other mixed polymer waste.

Table of Contents

Acknowledgements	ii
Abstract	iii
Table of Contents	iv
List of Tables	viii
List of Figures	ix
Chapter 1 Introduction	1
1.1 Overview	1
1.2 Research aim and objectives.....	3
Chapter 2 Literature Review	5
2.1 Textile waste.....	5
2.2 Textile recycling	6
2.2.1 Mechanical recycling.....	7
2.2.2 Chemical recycling.....	7
2.3 Polyamide	8
2.3.1 Polyamide 6 waste.....	9
2.3.2 Recycled polyamide 6 fibres	10
2.3.2.1 Mechanical Properties.....	10
2.3.2.2 Chemical Properties.....	11
2.3.2.3 Thermal Properties.....	12
2.4 Textile fibres	13
2.4.1 Single component fibres	13
2.4.2 Bicomponent fibres	15
2.5 Polymer blend in textile fibre.....	21
2.5.1 Factors affect the properties of blend fibre	22
2.5.2 Interacting blend of PA6:TPU	24
2.5.3 Non-Interacting blend of PA6:PP and PA6:PET	26
2.5.3.1 Morphology of the blend.....	27
2.5.3.2 Thermal properties	30
2.5.3.3 Mechanical properties	30
2.6 Melt spinning.....	31
2.7 Conclusions	33
Chapter 3 Methodology	34
3.1 Materials	34
3.2 Fibre extrusion	35

3.2.1 Drying of polymer pellets	36
3.2.2 Melt extrusion.....	36
3.3 Fibre treatment	37
3.4 Microscopy.....	38
3.4.1 Light microscopy	39
3.4.2 Scanning electron microscopy (SEM).....	41
3.5 Capillary rheometry.....	42
3.6 Thermal properties.....	43
3.6.1 Differential scanning calorimetry	44
3.6.2 Termogravimetric analysis	46
3.7 Fourier Transform Infrared Spectroscopy	48
3.8 Tensile properties	48
3.9 Roller Drawing	49
3.10 Winding.....	51
3.11 Knitted fabric production	52
3.12 Fabric testing	53
3.12.1 Sample preparation.....	53
3.12.2 Fabric specification	53
3.12.3 Fabric structure	55
3.12.4 Bending length	55
3.12.5 Vertical wicking test.....	55
3.12.6 Ball burst strength	57
Chapter 4 Production of partially miscible hybrid fibres of polyamide 6 and thermoplastic polyurethane polymer	58
4.1 Introduction	58
4.2 Part 1: PA6:TPU-D	59
4.2.1 Viscosity and fibre production	59
4.2.2 Morphology of the as-spun fibres.....	60
4.2.3 Polymer extraction	64
4.2.4 Fibre and pore diameters.....	69
4.2.5 Thermal properties	73
4.2.6 X-ray diffraction	79
4.2.7 Chemical properties	81
4.2.8 Tensile properties	84
4.3 Part 2: PA6:TPU-A.....	87
4.3.1 Morphology of the fibre	88



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4.3.2 Thermal properties	88
4.3.3 Chemical properties	91
4.3.4 Tensile properties	94
4.4 Conclusions	95
Chapter 5 Manufacturing of hybrid fibre from polyamide 6 and polyethylene terephthalate	96
5.1 Introduction	96
5.2 Viscosity and fibre production	97
5.3 Polymer extraction	98
5.4 Fibre morphology	99
5.5 Fibre diameters	103
5.6 Thermal properties	105
5.7 Tensile properties	106
5.8 Conclusions	111
Chapter 6 The effect of non-interacting blend between polyamide 6 and polypropylene polymer	112
6.1 Introduction	112
6.2 Viscosity of the polymers	112
6.3 Fibre morphology	113
6.4 Fibres diameters	117
6.5 Thermal properties	121
6.6 Chemical properties	130
6.7 Tensile properties	132
6.8 Conclusions	140
Chapter 7 Properties of upcycled polyamide 6 fabrics	141
7.1 Introduction	141
7.2 Production of PP-40 hybrid fibre	141
7.3 Production of PP-40 knitted fabric	142
7.4 Fabric	143
7.5 Physical properties	144
7.6 Fabric Strength	146
7.7 Wicking properties	148
7.8 Bending length	149
7.9 Conclusions	150
Chapter 8 Conclusions and Future work	151
8.1 Conclusions	151



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PEPUSATAKAMAN TUNKU TUN AMINAH

8.2 Contributions.....	153
8.3 Suggestions for future work	154
List of References	156
Appendix A	168
Appendix B	169



List of Tables

Table 2.1 Porous fibres and fibre diameter.	18
Table 3.1 Types of polymer.	34
Table 3.2 Blend composition of the fibre.	35
Table 3.3 The temperature setting, screw speed and take-up speed of melt extruder (refer to Table 3.2 for blend composition).	37
Table 3.4 Solvents and extraction method of secondary polymer.	38
Table 3.5 Draw ratio of the feed roller and draw roller	50
Table 4.1 Shear rate and viscosity of the PA6:TPU-D hybrid fibre.	60
Table 4.2 Processing parameter of TPU-D 20.	63
Table 4.3 Mass loss of PA6:TPU-D hybrid fibres after being treated in DMSO and formic acid for 3 and 24h.	65
Table 4.4: Thermal properties of as-spun fibres sample.	75
Table 4.5 TGA results for PA6:TPU-D hybrid fibres before and after DMSO treatment.	76
Table 4.6 ATR-FTIR wavenumber for PA6, TPU-D and hybrid fibres.	82
Table 4.7: Carbonyl phase absorbance intensity of the TPU-D and hybrid fibres	83
Table 5.1 Shear rate and viscosity of PA6 and PET polymer.	97
Table 5.2 Fibres mass loss in formic acid and NaOH treatment.	99
Table 5.3 The morphology of fibres based on Jordhamo model.	103
Table 5.4 DSC results on PA6:PET hybrid fibres.	108
Table 6.1 Mass loss of PP component after treated with toluene.	115
Table 6.2 Viscosity ratio of PA6 and PP and expected morphology for the hybrid fibres.	117
Table 6.3 Percentage of fibres diameter reduction after drawing	120
Table 6.4: Results of DSC test on as-spun PA6:PP hybrid fibres (untreated).	126
Table 6.5: Results of DSC test on as-spun PA6:PP hybrid fibres (treated).	126
Table 6.6 Thermal degradation of as-spun PA6:PP hybrid fibres (untreated and treated).	128
Table 6.7 Tex of the as-spun and drawn PA6:PP fibres.	140



List of Figures

Figure 2.1 Ring opening polymerisation of PA6 (Richards, 2005).....	9
Figure 2.2 Interaction of water molecules with PA6 polymer (Reimschuessel, 1998).	9
Figure 2.3 Stress strain diagram of PA6 filaments at different draw ratio (Reimschuessel, 1998).	11
Figure 2.4 Principle of synthetic fibres production (Imura et al., 2014).	14
Figure 2.5 Shapes of spinneret holes and as-spun fibre cross-sections (NPTEL, 2013).	15
Figure 2.6 The cross sectional view of 4DG™ fibre (Fiber Innovation Technology, 2015).	15
Figure 2.7 Different mechanism used to produce various types of bicomponent fibre a) core/sheath, b) side by side, c) pie wedge and d)island in the sea (NPTEL, 2013).....	17
Figure 2.8 Types of bicomponent fibres a) side-by-side, b) core-sheath, c) pie-wedge, d) island-in-the-sea (Ayad et al., 2016).	17
Figure 2.9 Cross sectional view of the high surface area filament (Pourdeyhimi and Chappas, 2012).	20
Figure 2.10 Schematic diagram of two immiscible polymer blend fibre with different composition of polymer A and polymer B: <i>left</i> - polymer A as a major component, <i>middle</i> - co-continuous phase when the composition of two polymers almost in the same amount, <i>right</i> - polymer A as a minor component.	22
Figure 2.11 Size of disperse phase based on polymer viscosity.....	23
Figure 2.12 Size of disperse phase based on dispersed phase content.	23
Figure 2.13 Size of disperse phase based on shear rate.	24
Figure 2.14 Basic chemistry of thermoplastic polyurethane (Huntsman, 2010).	25
Figure 2.15 Schematic diagram of polymer melt extrusion.	32
Figure 2.16 Different types of rotating screw (top view); a) single screw, b) twin screw.	32
Figure 2.17 The movement of high viscosity polymer in an extruder.	33
Figure 3.1: Preparation of fibres for cross-section view under LM: 1- prepare the perforated plate, 2- insert needle threader through one of the holes, 3-insert a bundle of fibres into the wire loop of the needle threader, 4- pull the wire loop and the fibres through the hole, 5- cut the fibres at both sides of the plate, 6- the cut fibres (show by arrow) ready to be viewed under LM.	39
Figure 3.2 Examples of fibre cross-section images captured by LM: a) PA6 fibres and b) TPU-D 50 untreated fibres.....	39
Figure 3.3 Illustration of bulk and true cross-section area.....	40

Figure 3.4 Step of measuring PA6:TPU-D hybrid fibre cross-section area in ImageJ software.	41
Figure 3.5 Example of images of a) TPU-D 50 and b) PP-60 treated fibres viewed under light microscope (<i>left</i>) and scanning electron microscope (<i>right</i>).	42
Figure 3.6 Capillary Rheometer.....	42
Figure 3.7 DSC diagram.....	44
Figure 3.8 Basic DSC curve diagram.	45
Figure 3.9 DSC heating curve of PA6 and PP hybrid fibres.	46
Figure 3.10 TGA diagram.	47
Figure 3.11 Example of TGA curve.	47
Figure 3.12 ATR-FTIR diagram.	48
Figure 3.13 Tenacity against elongation curve.....	49
Figure 3.14 Filament drawframe.....	50
Figure 3.15 Filament drawing mechanism.....	51
Figure 3.16 PP-40 yarn carrier; <i>left</i> - spool and <i>right</i> -cone.....	51
Figure 3.17 Twisting machine.....	52
Figure 3.18 Double-bed flat knitting machine (10-gauge).	52
Figure 3.19 Template for sample size on top of the treated fabric.	53
Figure 3.20 Course and wale of knitted fabric.	54
Figure 3.21 Hanging pear loop illustration.....	55
Figure 3.22 Vertical wicking test using wicking solution.	56
Figure 3.23 Vertical wicking test using water.	57
Figure 3.24 Ball burst tooling clamp (left) and ball probe (right).....	57
Figure 4.1 Viscosity of PA6 and TPU-D at 230°C.	59
Figure 4.2 SEM images of PA6:TPU-D hybrid fibres and single component fibres in cross-section view (left) and longitudinal view (right).....	61
Figure 4.3 Micrograph of TPU-D fibres produced at different processing temperature (range 200°C~230°C) pores appear in the fibres produced at 220°C were shown in dotted circles.	62
Figure 4.4 SEM mages of TPU-D 20 fibres produced at different screw speed in cross-sectional view (top) and fibre longitudinal view (bottom).	64
Figure 4.5 SEM images of TPU-D 20 produced at different winder take-up speed in cross-sectional view (top) and fibre longitudinal view (bottom).....	64
Figure 4.6 Micrograph of TPU-D 20 (upper) and TPU-D 50 (lower) before (left) and after treatment with DMSO for 3h (middle) and 24 h (right).	66
Figure 4.7 SEM images of PA6:TPU-D hybrid fibres after 24 h treatment in DMSO in cross-sectional view (upper) and longitudinal view (lower).	67



Figure 4.8 SEM images of TPU-D 50 after treated with formic acid for 3 and 24 h in cross-sectional view (upper) and longitudinal view (bottom).	68
Figure 4.9: Fibre morphology of TPU-D 50 before and after treatment in formic acid.	69
Figure 4.10 Micrograph of PA6, TPU-D, TPU-D 20 and TPU-D 50 fibre cross-section.	69
Figure 4.11 Fibre cross-section and pore area of the single component and hybrid fibres (bulk and true area). ($n=30$)	70
Figure 4.12 Fibre diameter of the PA6:TPU-D hybrid fibres and single component fibres.	71
Figure 4.13 Frequencies of the pore size developed in TPU-20 and TPU-50 fibres from 30 specimen for each. ($n_{PA6-50}=1046$, $n_{PA6-80}=1170$)	72
Figure 4.14 Porous fibres using freezing spinning method. (Cui et al., 2018).....	73
Figure 4.15 DSC curve of the fibres in a) heating and b) cooling cycle.....	74
Figure 4.16 DSC curve of hybrid fibres before and after treatment with DMSO in a) heating and b) cooling cycle.	77
Figure 4.17 TG (top) and DTG (bottom) curve of hybrid fibres before and after treatment.	78
Figure 4.18 XRD results of (top)-single component fibres and hybrid fibres, (middle)-TPU-D 20 and (bottom)-TPU-D 50 hybrid fibres untreated and treated with DMSO.	80
Figure 4.19 The FTIR spectra of PA6:TPU-D hybrid fibres and single component fibres (normalised at 2866 cm^{-1}).	81
Figure 4.20 Carbonyl region of the FTIR spectra for TPU-D and hybrid fibres (normalised at 2866 cm^{-1}).	83
Figure 4.21 FTIR spectra of PA6 crystal form (α and γ -crystal) in the fibres.....	84
Figure 4.22 Mechanical properties of PA6, TPU-D and hybrid fibres. ($n=10$)	85
Figure 4.23 Tenacity vs elongation curve of PA6, TPU-D and hybrid fibres.	86
Figure 4.24 Mechanical properties of the TPU-D fibres produced at different processing temperature (200 and $230\text{ }^{\circ}\text{C}$).	87
Figure 4.25 DSC curve of the PA6 and TPU-A 20 hybrid fibres untreated and treated.	89
Figure 4.26 SEM images of TPU-A 20 hybrid fibres: (left) untreated and (right) treated in DMSO.....	90
Figure 4.27 ATR-FTIR spectra of the PA6, TPU-A and TPU-A 20 untreated and treated.	91
Figure 4.28 Carbonyl group spectra of: (top) PA6, TPU-A, TPU-A 2- untreated and TPU-A 20 treated and (bottom) larger image for the PA6, TPU-A 20 untreated and TPU-A 20 treated.	93
Figure 4.29 Tenacity and elongation of the PA6, TPU-A and TPU-A 20 untreated and treated fibres.	94

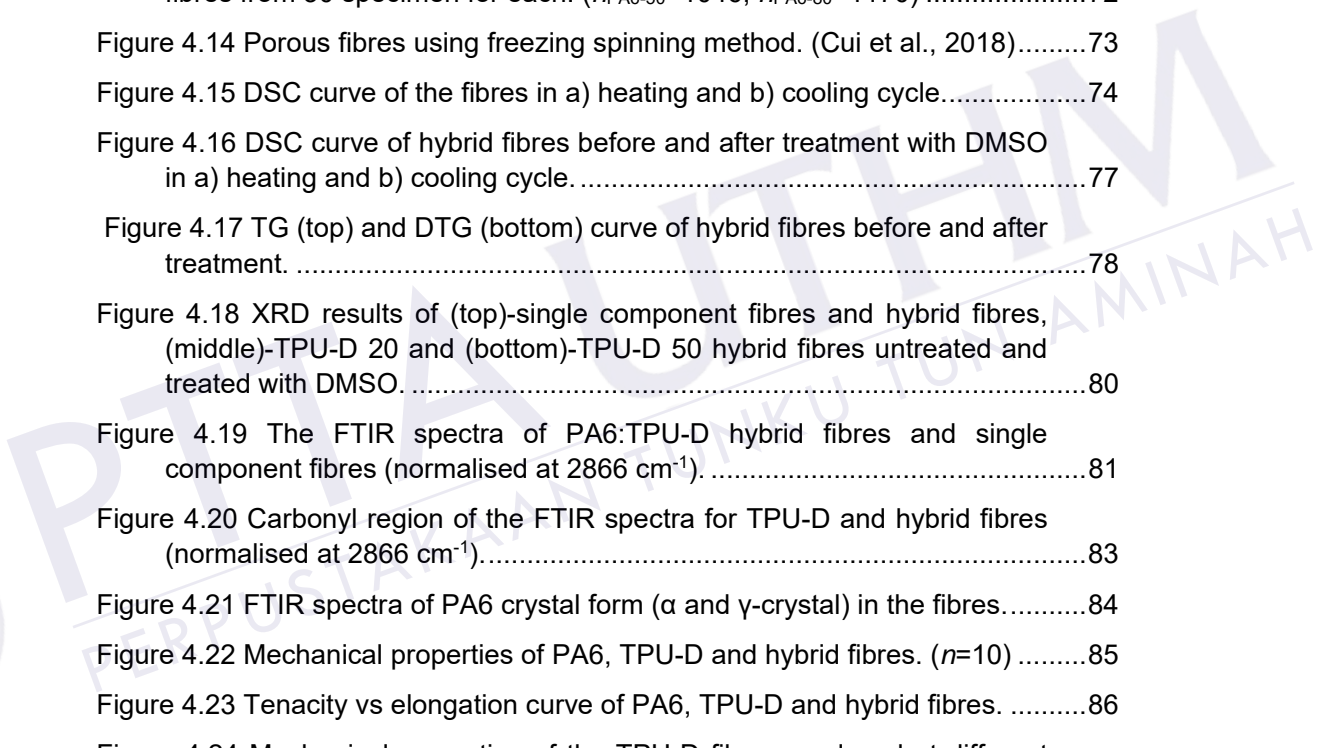


Figure 4.30 Tenacity vs elongation representative curve of PA6, TPU-A and TPU-A 20 untreated and treated fibres.....	95
Figure 5.1 Viscosity of PA6 and PET at 270 °C.	97
Figure 5.2 Cross section view of the PA6:PET hybrid fibres (taken using Leica Microscope).....	100
Figure 5.3 SEM images on PET-50 and PET-60 hybrid fibres before treatment.	101
Figure 5.4 Morphology of PA6:PET hybrid fibres.	102
Figure 5.5 SEM images of PA6:PET hybrid fibres after PET etched in NaOH.....	102
Figure 5.6 PA6:PET hybrid fibres diameter.....	104
Figure 5.7 Diameter of PA6 fibrils.....	104
Figure 5.8 DSC curve of PA6 and PET single component fibre (cooling and second heating cycle).....	105
Figure 5.9 DSC second heating curves of PA6:PET hybrid fibres (untreated and treated).	107
Figure 5.10 DSC cooling curves of PA6:PET hybrid fibres (untreated and treated).....	107
Figure 5.11 Mechanical properties of PA6:PET hybrid fibres; tenacity (top), elongation (middle) and Young modulus (bottom).....	109
Figure 5.12 Tenacity vs elongation at break of PA6:PET hybrid fibres.	110
Figure 6.1 Shear viscosity of PA6 and PP pellets at 230 °C.	113
Figure 6.2 SEM images of PA6:PP hybrid fibres before and after treated (cross-section and longitudinal view).....	114
Figure 6.3 Illustration of PA6:PP hybrid fibre structure in different weight compositions.....	115
Figure 6.4 SEM image of PP-50 showing phase separation between PA6 fibril and PP matrix.	116
Figure 6.5 Diameter of as-spun and drawn PA6:PP hybrid fibres (untreated).	118
Figure 6.6 Diameter of as-spun and drawn PA6 microfibres (treated).	118
Figure 6.7 Microscopy images of as-spun PA6:PP hybrid fibre.	119
Figure 6.8 Microfibres diameter distribution of PA6 microfibres (as-spun and drawn fibres). ($n=50$).....	121
Figure 6.9 DSC curves on second heating cycle of PA6:PP hybrid fibres (as-spun and treated fibre).	123
Figure 6.10 DSC curves on cooling cycle of PA6:PP hybrid fibres (as-spun and treated fibre).	125
Figure 6.11 Thermal degradation of as-spun PA6:PP hybrid hybrid fibres.	127
Figure 6.12 Derivative weight curve of as-spun PA6:PP hybrid fibres.	127
Figure 6.13 Illustration of PP-35 fibre morphology.	128



Figure 6.14 Thermal degradation of PA6:PP hybrid fibres (untreated and treated).....	129
Figure 6.15 Derivative weight curve of PA6:PP hybrid fibres (untreated and treated).....	129
Figure 6.16 FTIR spectra of PA6:PP as-spun hybrid fibres and single component fibres untreated and after treated in toluene. (Normalised at 1634 cm ⁻¹).....	131
Figure 6.17 Tenacity of as-spun and drawn PA6:PP hybrid fibres (untreated and treated).....	133
Figure 6.18 Tenacity-elongation curve of as-spun PA6:PP hybrid fibres(untreated and treated).....	135
Figure 6.19 Tenacity-elongation curve of drawn PA6:PP hybrid fibres (untreated and treated).....	136
Figure 6.20 Elongation at break for as-spun and drawn PA6:PP hybrid fibres (untreated and treated).....	138
Figure 6.21 Initial Young's modulus of as-spun and drawn PA6:PP hybrid fibres (treated and untreated).....	139
Figure 7.1 Paper spool (left) and cone (right).....	142
Figure 7.2 PP-40 circular plain jersey knitted fabric before treatment.....	143
Figure 7.3 PP-40 circular plain jersey knitted fabric after treated with toluene.....	143
Figure 7.4 The structure of PP-40 knitted fabric untreated (a, b & c) and treated (a', b' & c') viewed under light microscope (a,b, a' & b') and SEM (c & c').....	144
Figure 7.5 Mass and thickness of untreated and treated PP-40 fabric.....	145
Figure 7.6 Courses, wales and stitch density of untreated and treated PP-40 fabric.....	146
Figure 7.7 Ball burst strength of the PP-40 untreated and treated fabrics.....	147
Figure 7.8 Example of PP-40 treated fabrics after ball burst strength test.....	147
Figure 7.9 The representative curve of force vs extension of PP-40 untreated and treated fabrics.....	148
Figure 7.10 Vertical wicking results of PP-40 treated fabrics.....	149
Figure 7.11 Bending length of PP-40 fabrics.....	149

Chapter 1 Introduction

1.1 Overview

Textile waste has been an issue around the world with 92 million tonnes waste recorded in 2015 (Global Fashion Agenda and The Boston Consulting Group, 2017). With the increasing demand on textiles, the amount of waste generated is expected to increase up to 148 million tonnes by 2030. At the moment, only 25% of the waste is recovered via reuse and recycling route where most of it either incinerated for energy recovery or send off to landfill (Ellen MacArthur Foundation, 2017). Over two-third of textile raw materials are produced from plastic sources, hence incineration or landfill disposal has negative impacts on the environment.

Reuse and recycling can be the preferred option for textile waste. By reusing the product, the life of the product can be extended and the landfill can be avoided. However, recycling is needed for the products that are no longer useable. Four types of recycling approaches can be applied to textile waste; fabric recycling, fibre recycling, polymer recycling and monomer recycling (Ellen MacArthur Foundation, 2017; Sandin and Peters, 2018). The most widely adopted recycling approach in the industry are fabric and fibre recycling. Meanwhile for polymer and monomer recycling, the fibre production is limited to single component polymers such as polyethylene terephthalate (PET) or known as polyester fibres, Polyamide 6 (PA6), and cellulose-based fibres (Palme et al., 2017).

Even though the efforts to recycle the textile wastes into textile fibres have been in place, the conversion of textile waste into materials for clothing is extremely low (<1%) (Ellen MacArthur Foundation, 2017). The nature of textiles products that usually consists of more than one material, either by using blended yarns (e.g. polyester/cotton yarns), bicomponent fibres (core-sheath, pie-wedge, island in the sea types, etc.) or fabric coated with polymer, limits the recycling process. The mix-material products result in complex waste where component polymers cannot be practically separated by commercially relevant technologies. The sorting and separation of mix-materials waste into single component polymers is challenging and results in unsuitability of a large proportion of waste for recycling. There is a paucity of technologies and facilities to sort and separate the mixed waste materials also limits the potential of recycling multi-component products (Östlund et al., 2015).

As most of the textile products are made from plastic sources, thermo-mechanical processing can be used to recycle mixed-waste product. The mixed-waste can be directly blended, melted and re-extruded to produce blended fibres. The mixing of two different polymers is called binary blending and three different polymers is ternary blending. In polymer blends, selection of polymers, blend composition, the viscosity of the polymers and processing conditions affect the morphology and subsequently the properties of the blended fibres. In textile industry, common polymers used to produce synthetic fibres are polyethylene terephthalate (PET) (51%), polypropylene (PP) (~5%) and polyamide (PA) (5.4%) (Textile Exchange, 2018).

The possibility of mixed-waste textile product containing these polymers are high. For examples, polyamide and polyester were commercially produced as bicomponent fibres (Radici Group, n.d.). In carpet industry, polyamide was used as a face fibres and polypropylene as a bottom layer or backing fabric. Marine products such as inflatable raft and life jacket use polyamide as a base fabric and coated with polyester or thermoplastic polyurethane to make it durability against water (Erez, 2018). Complex materials exist in textiles result to the mixed-waste materials once entered the recycling centre. Even for textiles made from a single component, when collected as a waste, can also be mixed with varieties of other textile sources.

Among common synthetic polymers used for textile fibres, polyamide is considered as an expensive polymer (USD2.32-2.42/kg) compared to PET (USD1.22-1.23/kg) (YNFX, 2020). Over 4.55 million tonnes of polyamide was produced in 2014 (Wesołowski and Płachta, 2016). Two types of polyamide frequently use as textile fibres are PA6 and PA6,6 with PA6 dominates the usage in apparel industry (86%) compared with PA6,6 (14%) (Wesołowski and Płachta, 2016).

PA6 or also known as nylon 6 is widely used in many applications such as apparel, carpet, parachute fabrics, tire cord, ropes and tents (Wesołowski and Płachta, 2016). Increasing demand of polyamide products contribute to the abundance of polyamide waste too. For example, carpet which contains approximately 24% of PA6 fibres, is thrown to landfill every year. Fibre grade PA6 is a high value thermoplastic polymer that has a low rate of degradation in landfill. Therefore, to avoid landfill, another option for PA6 waste is by recycling the PA6 containing products. Several studies have shown that reprocessing PA6 fibres into several cycles can be done successfully. However, the repetitive process of melting PA6 fibres may slightly decrease the mechanical properties and stability of the polymer (Tuna and Benkreira, 2017).

Polyamide and other thermoplastic textiles polymers can be processed several times. Recycling the mixed-waste containing different thermoplastic polymers without separation process can be done by thermo-mechanical recycling. Two polymers can be melted and extruded into new hybrid fibres. Blending of PA6 with other materials such as acrylonitrile–butadiene–styrene (Aparna et al., 2017) (ABS) cellulose acetate butyrate (CAB) (Zhang et al., 2015) and chitosan (Dotto et al., 2017) into hybrid fibres were studied previously.

Blending of two different polymers can create interacting and non-interacting behaviour of the polymer blend. Amide group of PA6 and urethane group of TPU create interaction through hydrogen bonding. Non-interacting behaviour was found on the blending of PA6:PET and PA6:PP due to low chemical interaction (Hajiraissi et al., 2017). To improve the interaction on PA6:PET and PA:PP, compatibiliser was used (Aparna et al., 2017). However, the non-interacting behaviour of these polymers can benefit in the production of microfibres or nanofibres.

A study on the interacting and non-interacting behaviour of possible polymers found in textile waste was crucial. There is a paucity of reported work on the blending of polyamide with TPU as hybrid fibres when prior research focused on composite (Cai et al., 2019), injection moulding (Xu et al., 2019) and compressed moulding (Rashmi et al., 2017). Blending of PA6:PET and PA6:PA6 was studied before however focusing on addition of compatibiliser for improving of interfacial connection and the optimisation of the blend focus on PET and PP in the blend (Aslan et al., 1995; Liao et al., 2015; Aparna et al., 2017; Hajiraissi et al., 2017). With the increasing of textile waste collected and lack of technologies to separate the waste, added with high price of PA6, a study on the PA6 blending with other polymers into fibres is needed.

1.2 Research aim and objectives

The aim of this research is to investigate the effect of blending of PA6 polymer with interacting and non-interacting polymer commonly used in textile fibres production. Subsequently, the developed fibres will be processed into fabric form to evaluate their performance for apparel applications. The objectives of this research are to:

- i. produce interacting hybrid fibres consisting of PA6 as main polymer blending with thermoplastic polyurethane (TPU) as secondary polymer.
- ii. produce non-interacting hybrid fibres consisting of PA6 as main polymer blending with polyethylene terephthalate (PET) and polypropylene (PP) as secondary polymers.

- iii. Investigate the properties of the interacting and non-interacting hybrid fibres that blended in different weight composition by:
 - a. removal of secondary polymer.
 - b. assess the morphology of the hybrid fibres before and after removal of secondary polymer under.
 - c. characterise the properties of the fibres concerning thermal, chemical and mechanical and fibre diameter.
- iv. produce fabrics made of hybrid fibres and investigate the properties of the fabrics before and after removal of secondary polymer.



Chapter 2 Literature Review

Textile waste has been considered as an environmental issue. The scenario of textile waste management and current methods on recycling textile waste are presented in this chapter. Previous studies on recycling Polyamide 6 and its properties were reported. Different types of textile fibres; single and bicomponent fibres, were also discussed, including the method to produce fibres. Finally, the blending of two polymers in producing textile fibres, with melt spinning approach that is suitable for thermo-mechanical recycling of the polymer blend, will be discussed.

2.1 Textile waste

Textile waste has been an issue all around the world. It is reported that over 92 million tonnes of textile waste generated globally in 2015 and the numbers are expected to increase up to 148 million tonnes by 2030 due to growing global population (Global Fashion Agenda and The Boston Consulting Group, 2017). In UK alone, 921,000 tonnes of textile waste collected in 2017 which end up in landfill (WRAP, 2019).

Textile waste can be divided into two categories: pre-consumer waste and post-consumer waste. Pre-consumer waste refers to by-product materials generated by textile industries such as surplus fabrics, yarns and fibres, whereas post-consumer waste is contributed by the owners of textiles including clothing and textile household such as bed linen, curtains, towels and floor covering (Echeverria et al., 2019).

Textile waste can be either reused, recycled, incinerated or sent off to landfill. Almost 55% of textile waste collected in UK was sent to landfill and only 36% was recovered through reuse and recycling process (WRAP, 2019). Most of the waste was reused domestically through second hand or charity shop or exported to developing countries. Only small proportion of the waste was recycled, currently into down-cycle application such as wiper, insulation materials and padding mattress while less than 1% of the waste was recycled back into textile fibre (Ellen MacArthur Foundation, 2017).

The route of the textile waste can be improved, to conserve the resources. The unwanted clothes, mostly exported to third-world country where there are no proper recycling and collecting facilities, will end up wasting the valuable materials into landfill. Moreover, in 2015, East African Community announced to ban an importation of used clothing that will affect the utilisation of the collected textile waste in the future (SMART Secondary Materials and Recycled Textiles, n.d.).

Over two-third of textile raw materials derived from petroleum resources have high compostable times in landfill, compost and aquatic environments. The risk of harmful gas emission to the atmosphere such as methane (CH₄) and carbon dioxide (CO₂) is also a significant concern (UNEP, 2002). Department for Environment Food & Rural Affairs (DEFRA, 2013) estimated that 3 tonnes of CO₂ can be saved with reusing or recycling of 1 ton of general waste. Sending off the waste into landfill or incinerating bring harmfulness to the environment and contribute directly to the global climate change. The more optimum option for handling the waste is either reuse until the end of product life or recycling.

2.2 Textile recycling

Textile recycling industries were developed with a target to reduce both pre-consumer and post-consumer wastes and conserve resources while minimising the amount of waste going to landfill (Echeverria et al., 2019). Moreover, most of the textile products are recyclable and have a potential to replace virgin materials. The UK government has also been promoting recycling as more and more waste has been directed towards landfills. Approximately GBP 82 million were spent on disposing the textile waste in landfill (Ellen MacArthur Foundation, 2017). Serious actions have been taken to emphasise recycling such as increasing the landfill tax. It was expected that by 2030, municipal waste including textile waste sent to landfill will reduced to 10% (Bukhari et al., 2018).

Several organisations are involved in recycling textile waste such as Waste & Resources Action program (WRAP), Department for Environment, Food & Rural Affairs (DEFRA) and Carpet Recycling United Kingdom (CRUK). WRAP has launched several plans to promote the usage of textile waste such as European Clothing Action Plan, Sustainable Clothing Action Plan (SCAP) and Love Your Clothes campaign to raise awareness of the value of clothing. CRUK focuses on carpet recycling and acts as a centre and connector for companies and organisations involved in carpet recycling. The target is to decrease the number of carpet waste that goes to landfill and develop close loop recycling of carpets.

London based company Worn Again have a collaboration with several companies to develop FIBERSORT technology with the aim to produce an automated sorting technology that can sort a variety of post-consumer textile waste (Worn Again, 2016). These show that the effort has been made by the private sector to emphasise on the recycling industry. However, these examples limit the recycling effort only onto a single component polymer. Textiles waste consists of mix-materials that encounter

difficulties to be sorted and separated were send off to other easier route either landfilled or incinerated (Robert, 2016).

Textile recycling can be divided into fabric recycling, yarn and fibre recycling and polymer/monomer recycling which can be accomplished by either through mechanical, thermal, chemical processing or combination of these processing (Ellen MacArthur Foundation, 2017; Sandin and Peters, 2018).

Fabric recycling makes use of fabrics from pre-consumer waste generated by garment industries (e.g. off-cut fabrics, rejected fabrics) or post-consumer waste contributed by the users of the textiles (e.g. unwanted clothes, old clothes) and converted into new clothes or products. Tonlè, is an example of a company that produced textile products from the pre-consumer waste and managed to save 10 tonnes of waste being send to landfill in 2014 (Explorer, 2019). For fibre and polymer recycling, the methods can be simplified as mechanical recycling and chemical recycling.

2.2.1 Mechanical recycling

Fibre recycling, also referred to mechanical recycling, mechanically shredding the fabric to reclaim fibres for subsequent conversion into yarns or nonwovens. Polyester and cotton fibres, which dominantly used in textile industry were mechanically shredded during recycling process. The reclaimed cotton fibres result to shorter and lower cotton fibre quality thus produced coarser yarn count. Therefore, cotton waste was recovered into down-cycling route such as insulation materials, blankets, wipes, etc (Sandin and Peters, 2018). Pure Waste Textiles Ltd. be able to produce new 100% recycled cotton t-shirt from shredded textile cotton waste (Pure Waste Textiles, n.d.). Apart from recycling into yarn, the recycled cotton and polyester fibres were also produced into nonwoven fabrics for household application (Sharma and Goel, 2017). Low quality fibres restrict the recycling process into fibres found suitable approaches as reinforced composites(Serra et al., 2019; Meng et al., 2019), pressed into compressed mould (Dissanayake et al., 2018), sound absorption panel (Santhanam et al., 2019) or reinforced in brick or mortar (Orasutthikul et al., 2017; Kimm et al., 2018).

2.2.2 Chemical recycling

Other method for textile waste recycling is chemical recycling, either by polymer recycling or monomer recycling. Polymer recycling can be achieved by treating the fibres chemically/mechanically while keeping the polymer or oligomer intact.

Monomer recycling is a process where the polymers/oligomers are broken down to the monomer level. Textile waste consists of synthetic fibres made from thermoplastic polymers which can be chemically recycle to produce new fibres with identical properties to the virgin raw materials. ECONYL Regeneration yarn for example, was produced by recycling textile waste and fishing nets (Moorhouse and Newcombe, 2018). Some examples of regenerated fibres produced through chemical recycling were MIPAN Regen fibres, which are made by recycling PA6 post-consumer waste, REPREVE® PA6,6 fibres made of 100% recycled polyamide fabric (UNIFI, 2008) and CYCLEAD™ fabrics which are made by recycled PA6 fibres (Toray, 2007). The separation of polyester from cotton/polyester blend for example, involved chemical process such as hydrolysis and dissolving in solvent (Ling et al., 2019).

2.3 Polyamide

Polyamides are linear macromolecules containing amide (-CONH-) linkage. Two popular variances in the textile and plastic industries are PA6 and PA6,6. Other popular polyamides available commercially are polyamide 11, 12, 46 and 69. PA6 widely used in the apparel industry (86%) compared to PA6,6 (14%) (Wesołowski and Płachta, 2016). In this study, the research will concentrate on the polymer derived from PA6 which is mainly used for clothing.

PA6 or polycaprolactam is made by ring opening polymerisation of caprolactam monomer. The ring opening of ϵ -caprolactam monomer occurs when the monomer is heated at 250-280°C at atmosphere pressure for 12-24 hrs as shown in Figure 2.1 (Richards, 2005) and the complete process of ring opening can be seen in Appendix A. The polymerisation could be initiated by water, acid or very strong base such as sodium hydride (NaH), but water is the favoured option in the industry (Richards, 2005). The amide bonds (-CONH-) developed lactam provide hydrogen bonding between chains hence contribute to the excellent properties of PA6 such as stiffness and toughness (John and Furukawa, 2012). The H-bonds also make the PA6 categorised under hygroscopic material as water molecules can form H-bonds with the amide groups as shown in Figure 2.2 (Reimschuessel, 1998).

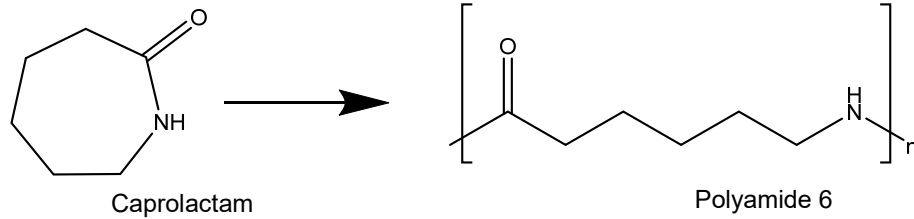


Figure 2.1 Ring opening polymerisation of PA6 (Richards, 2005).

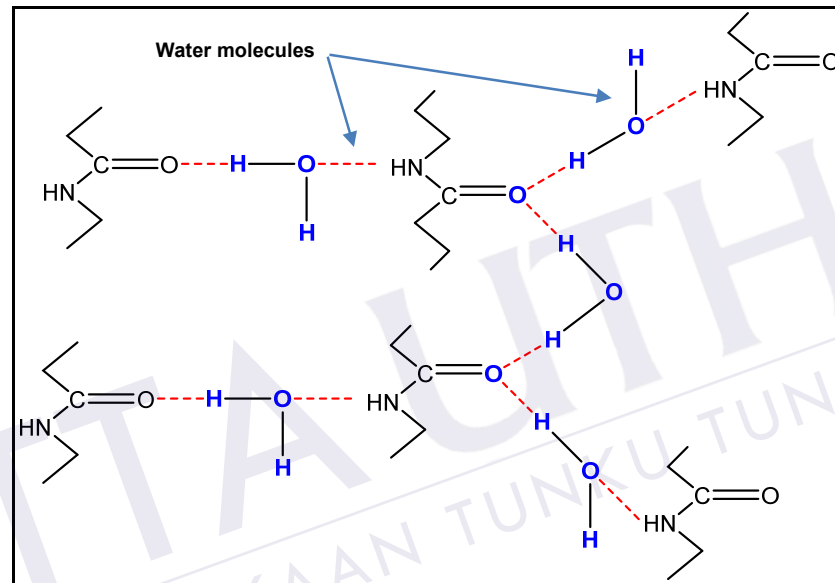


Figure 2.2 Interaction of water molecules with PA6 polymer (Reimschuessel, 1998).

The important parameter of polyamide is the molecular weight or molecular mass. Molecular weight influences the melt viscosity of the polymer namely; higher molecular weight contributed to higher melt viscosity. The melt viscosity of the polymer influences the movement of the polymer chains, namely higher melt viscosity tends to reduce the polymer chain movement (Grüner and Hopmann, 2018).

2.3.1 Polyamide 6 waste

Polyamide is a significant textile fibre with 5.7 million tonnes produced annually comprising 5.4% of the synthetic fibre market and was ranked second among all the textile fibres (Textile Exchange, 2018). The application of polyamide in textile industries is apparel, carpet and industrial filament such as tire cord and ropes (Wesołowski and Płachta, 2016).

From these applications, carpet waste can be collected easily and the amount is significant. The volume of carpet waste for disposal rose by 400,000 tonnes every year in the UK alone (Hilton et al., 2019). Carpet is classified as textiles and is extensively used in buildings and automobile as well as in aircrafts. The surface of carpet acts as a feet insulator from cold floor, sound proof and also adds to the décor of a room. 698 million m² of new carpets were produced in the United Kingdom in 2016 to replace the old carpets(Hilton et al., 2019). The huge quantity of fibres consumed to produce carpets lead to an enormous volume of waste, which dominant by nylon fibre (50-70%) (Mohammadhosseini et al., 2018).

2.3.2 Recycled polyamide 6 fibres

Early work on the extrusion of polyamide waste into filaments was conducted by Esfahani (1983) which reported that re-extrusion of PA6 and PA6,6 waste polymers up to 5 cycles is possible. A study conducted by Meyabadi et al. (2010) also showed that PA6 waste can be recycled into filaments. The blending of recycled PA6 with virgin PA6 showed an improvement in the tensile and thermal properties of recycled PA6. Other than the above, to the best of authors' knowledge, there is no published work on the recycling of polyamide waste into PA6 filaments or fibres. Other studies on this domain focused on the reprocessing of polyamide waste via injection moulding up to 16 cycles (Su et al., 2007; Crespo et al., 2013; Grümer and Hopmann, 2018). These studies proved that recycling the polyamide waste into multiple processing cycles are achievable. Besides, polyamide waste polymers were recycled into reinforced composites (Pan et al., 2016; Hasan et al., 2018), cement mortar and concrete reinforcement (Orasutthikul et al., 2017).

2.3.2.1 Mechanical Properties

The common tensile properties for filaments or fibres are tenacity, initial modulus, breaking elongation and work of rupture. Polyamide's relative mass, the extrusion speed, draw ratio, and heat treatment influence the tensile properties of the fibres.

Early study conducted by Esfahani (1983) on the recycling PA6 fibres found that the tenacity of PA6 fibre decreases slightly with the increasing number of cycles with 48.7 cNtex⁻¹ (5.5gd⁻¹) and 43.7 cNtex⁻¹ (4.94 gd⁻¹) reported for the first and fifth cycle respectively. The other tensile properties (the work of rupture, Young's modulus and breaking elongation) also show a similar trend to that of tenacity. Similarly, the study done by Meyabadi et al. (2010) also suggest that the tensile strength and modulus of recycled PA6 fibres are slightly lower than the virgin PA6 fibres. The loss of the

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