Suitability of recycled HDPE for 3D printing filament

Haruna Hamod

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ABSTRACT	
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Supervisor (Arcada):	Valeria Poliakova
Examiner:	Mirja Andersson

Abstract:

3D printing technology has been popular lately and the most used filament is ABS and PLA. There is a concern about how the filament should be made of recycled material, therefore this research was aimed to find recycled HDPE specification and extrusion parameters for 3D filament. Comparisons were made between ABS, PLA and recycled HDPE in order to obtain these parameters. The mechanical properties of recycled HDPE such as melt flow, tensile strength, young modulus and yield strain was observed to go along the PLA parameters used as 3D filament. The result shows that recycled HDPE has the potential properties as 3D filament with essential puling speed and specific cooling method employed.

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Abbreviation

ABS: Acrylonitrile butadiene styrene

HDPE: High density poly ethylene

rHDPE: Recycled high density polyethylene

MFI: Melt flow index

PLA: Polylactide or poly (lactic acid)

SLA: Stereolithography

SLS. Selective laser sintering

FDM: Fused deposition modelling

FFF: Fused filament fabrication

RepRap: Self replicating rapid prototyper

Foreword

All praises and adoration is due to almighty Allah for making it possible for me to finish this thesis throughout the ups and down, I say Alhamdulillah!

Firstly, I would like to thank Arcada University of Applied Science for given the opportunity to study in an international atmosphere and most especially to the lecturers at the department of energy and materials technology for sharing their academic knowledge and life experience during my studies.

My profound gratitude and appreciation goes to my supervisor Valeria Poliakova and examiner Mirja Andersson for their enormous support and guidance for the success of this thesis.

Secondly, to my parent who has been given me words of encouragement and prayers all the time, I pray may you eat the fruit of your labour.

Last but not the least, to friends and fellow students who has in one way or the order contribute to my knowledge, I say thank you!

1 Introduction

1.1 Background

Plastic usage has become an integral part of society with growth in population and technological development, making plastics production increasing more than expected for its ability to replace metal, wood, paper and glass in variety of engineering application. The tremendous uses range from domestic and industrial application, which can be found and seen in products like credit cards, computers, calculators, milk jugs, shampoo bottles, detergent plastic bottles, cosmetics, toys etc. However, the usage of this plastic has caused some substantial environmental burden on both land and water pollution as plastics tends to decompose very slowly and taking up landfill and seashores.

A significant concern has been raised and seen over the years with the rate at which plastics are used and disposed within communities of different countries occupying landfills and the advocacy of more ecological environment has been highlighted in many ways and recycling is part of them. If recycling is done more often, then plastics waste that ends up in the landfills will be less and will reduced the need for new raw material while serving as a resources to manufacture new product.

1.2 Research question

3D printing technology nowadays has emerged to be known for different application and used in many areas but concern is raised on how the filament should be more eco-friendly and reproduced from recycled material. The necessities for 3D filament and printed products to be more sustainable influence this research. The engineering properties of ABS and PLA that makes it suitable for filament would be examined and analysed to obtain relevant information and be compared to recycled HDPE. These comparisons will enable proper parameter optimization of the recycled HDPE that will be suitable to produce the filament.

1.3 Aims and Objectives

The aims and objectives of this thesis are:

1. Recycling HDPE to produce filaments for 3D printer
2. Finding the specifications and parameters used for 3D printers filaments.
3. Comparing and evaluating the engineering properties of ABS, PLA and recycled HDPE.
4. Determining the properties suitable for 3D printer filament from recycled HDPE.
5. Using the filament to print a product.

2 Literature review

This chapter of literature review is categorized in two parts; the first part gives some important explanation of those plastics involved with 3D printer filament (types, processes, applications and uses). The second part review extrusion, recycling, machining processes, 3D printing and earlier research done concerning HDPE as a filament.

2.1 3D printing

3D printing inception could be traced back to the late 70s, when the inkjet printer was invented. In 1984, the co-founder of 3D systems Charles Hull created the first 3D printer, the process of rapid prototyping that enables 3D object to be created from digital data. Further down the lane, Hull obtained the patent for stereo-lithography (SLA) that was used to established 3D systems and developed the STL file format which would complete the electronics transmit file for 3D printing objects form computer aided design (CAD) software. The stero-lithography apparatus continued to be developed until the first commercial 3D printer was made available to the general public, SLA-250. This technological research has been engaged and applied in limited areas of industries such as the medical and engineering, the medical applications are production of implants and prosthetics. Although it shows a tremendous potential in others application as the future unfolds and series of research and experiment is done. [1, 2]

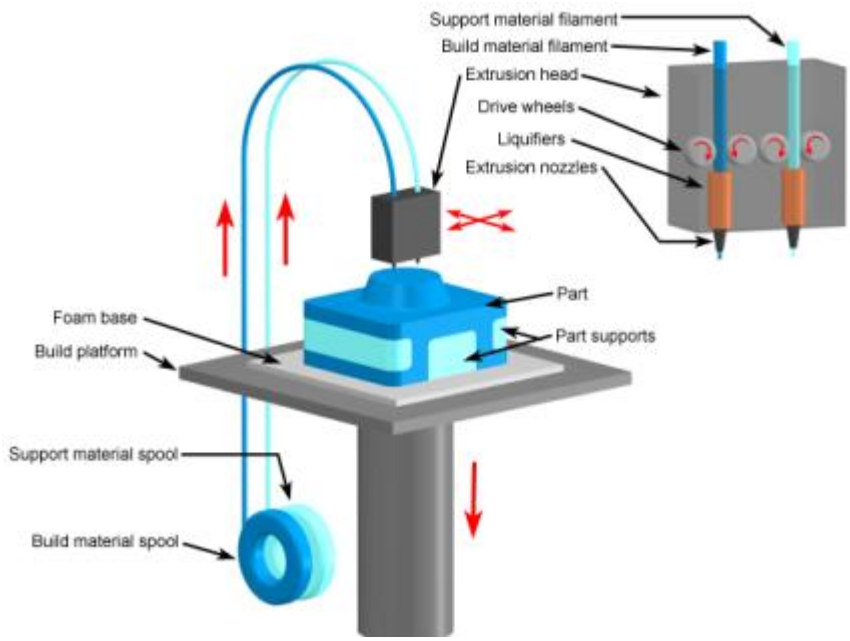

Figure 1 Schematic view of 3D printing [2]

2.2 Technology Overview

3D printing also referred to as additive manufacturing (AM) or rapid prototyping (RP) is defined as the process of joining making three dimensional solid objects from a digital model, i.e. it is a prototyping process whereby a real object is created from a 3D design. The objects are achieved during the process by creating a lay down successive layers of material. It is considered distinct from the conventional machining technique that typically depends on the removal of material by milling, drilling, boring, cutting etc. Various technologies and manufacturing methods are used in 3D printing few common ones are briefly explained below.

2.3 Different 3D printing technologies

2.3.1 Stereolithography

SLA is the original and the oldest 3D printing process technology developed by Hull in 1984. It works by converting liquid resin into solid materials with high-intensity light, which is often ultraviolet (UV) laser. This UV laser beam traces the slices of an object on the surface of this liquid causing the thin layer to be hardened. Once the UV laser beam has drawn a 2D path along the surface, the freshly polymerized model layer is lowered into the surrounding resin bath to be followed by another fresh surface curing and joining until complete object is printed. [3]

2.3.2 Selective Laser Sintering

Selective laser sintering (SLS) is a rapid prototyping technique with powder based manufacturing similar to SLA except for the UV beam replaced by lasers and the resin replaced by powdered bed. The powder has the attractive feature of being self-supporting for the generated product. This process involves a layer of plastic powder spreads over the machine base area uniformly and the object is built by using a laser to selectively fuse together successive fine plastic powder into layers. Upon completion, the remains of the unsintered powder will eventually be removed for reuse purpose. This technology is one of the most economical methods and its tolerant in terms of deigns guidelines. [3]

2.3.3 Fused Deposition Modelling (FDM) / Fused Filament Fabrication (FFF)

It is an additive manufacturing technology where by thermoplastic material that has a melting point below 300^0C are used. This melted thermoplastic is extruded from a temperature-controlled nozzle to produce the layers of object to be created at a certain high degree of accuracy. FDM is most commonly used process that works with thermoplastics material such as ABS, PLA, and HDPE etc. In this process, the 3D object designed with CAD software is imported as an STL file to the 3D printing software that would enable temperature controlled and the thermoplastic material layer by layer. The material changes from solid to semi-liquid state during the extrusion process to form layers upon layers. Each new layer will stack on top and fused with the previous layer when the material is harden almost immediately. [4]

Above all, the major working principle of all 3D printing are similar since almost all of the technology need 3D design (e.g. CAD or CAM) at first, which is a digital representation of an object before the client printer control software send the instruction to the printer and provides a real-time interface functions and settings.

2.4 Materials for 3D printing

2.4.1 Plastics

Plastics today continue to be an exciting material to use because of their diverse characteristics and usefulness in all works of life. There are numerous areas being developed yearly to utilise the properties of plastic and new processing technologies are emerging to exploit and take advantage of their easy manufacture process into all types of end product. They are produced from polymers; the polymers are formed from a repetitive long chain of methane or ethane molecules. This joining process of these molecules is best known as polymerization.

Polymerization is a chemical reaction process by which monomers are joined together in order to form long molecular chains called polymers. An example of such polymerization is polypropylene; Polypropylene is made by joining several structural molecule unit of propylene into large monomers by polymerization to form polypropylene. [5]

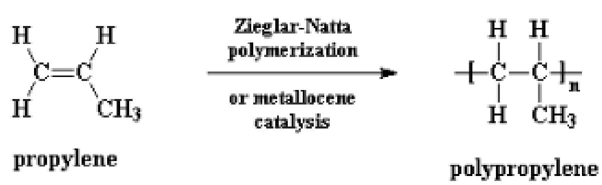

Figure 2 Ziegler-Natta polymerization of PP [6]

2.4.2 Plastics classification

Plastics are often classified as follows:

2.4.2.1 Thermosets

Thermosetting polymers are usually made from low molecular weight such that when they are heated they become very high cross-linked structure, thereby forming an infusible and insoluble product. The polymeric process is irreversible when it’s cured through heat or catalyst.

Thermoset has two-phase formation; phase one is the formation of the long-chain molecules while the second phase is the cross-linking of long chain molecules that usually takes place when heat and pressure are applied. In the thermoset material, the highly cross-linked molecules produces by strong chemical bonds enhance the high mechanical and physical strength of the materials when compared to that of thermoplastic. [5] Figure 3 shows the structure view of thermoset and their crosslinking.

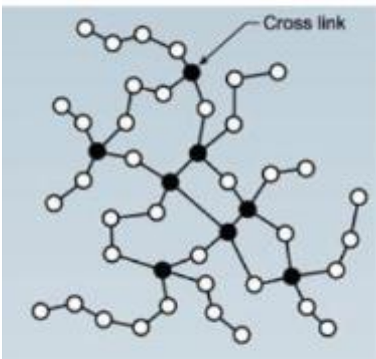

Figure 3 Structural crosslinking of thermoset [6]

2.4.2.2 Elastomers

Elastomers materials are conventionally member of rubber family that consists of a long chain-like molecule similar to thermoplastic. The molecules are joined together by chemical bonds to assume a bit of cross-linked structure. It is a group of material with different properties that has excellent processing behaviour. Their characteristic enables it to be shaped within its design temperature range, possessing elastomeric behaviour without cross-linking during production. Its glass transition is below the room temperature that makes is soft and rubbery. Its young modulus is relatively low while the yield strength is high when compared to other polymers [7].

2.4.2.3 Thermoplastics

Thermoplastic material consists of long chain-like molecules that are linked together by intermolecular interactions force. As the material is heated up to a high temperature passing the glass transition point the intermolecular forces are weakened that makes it becomes soft and flexible and the material becomes solid as it cools down. [5]

Thermoplastics can take two different types subdivision structures within thermoplastic group depending on the degree of intermolecular interactions that occurs between the polymer chains.

- Amorphous structure: This type of structure is responsible for the elastic properties of the thermoplastic materials, as the polymer chain adopt a bundled structure disorderly or a random structure.
- Crystal structure: The polymer chains acquire an orderly compacted structure directly responsible for mechanical properties of resistance to stress or loads and the temperature of the thermoplastics materials. [8]

Table 1 shows the essential characteristic and common features properties of amorphous and crystalline structures.

Table 1 Common characteristics of amorphous crystalline (pp. 4-5) [5]

Amorphous	Crystalline
• Mostly transparent	• Usually opaque
• It has low shrinkage because of the random arrangement of molecules produces little volume change	• High shrinkage: this is because as the material solidifies the molecules in the polymer are tightly packed to a high aligned structure
• Poor fatigue and wear resistance because of random structure of molecules	• Good fatigue and wear resistance because of the uniform structure
• Low chemical resistance; this is a result of the same random arrangement of molecules which are more open that enables chemicals to penetrate deep into the material and destroy many of the secondary bonds	• High chemical resistance; this is because the tightly packed structure that prevent chemical attack deep within the material

An example of standard thermoplastic which are widely referred to simply as ‘‘plastics’’ include Polypropylene (PP), Polyvinyl Chloride (PVC) Polycarbonate (PC), Polystyrene (PS), Polyamide

(PA), poly-methyl methacrylate (PMMA), Polyethylene (PE), Polylactide Acid (PLA), Acrylonitrile-butadiene-styrene (ABS) etc. These materials have different properties and are used in various applications but for the relevancy of this research ABS, PLA and HDPE are explained as follows.

2.4.3 ABS

Acrylonitrile-butadiene-styrene is a thermoplastic family of ‘‘terpolymers.’’ It is the combination of three different monomers Acrylonitrile, Butadiene, and Styrene to form a single polymer. The involvement of these materials contributes to the outstanding impact strength and high mechanical strength that makes it an opaque and stiff thermoplastic polymer suitable for tough consumer products. It has broad processing properties strong and durable at low temperatures with good heat and chemical resistance.

The following figure is the structures of the forming monomers and the representation of the property mix and arrangement of SAN and ABS [9].

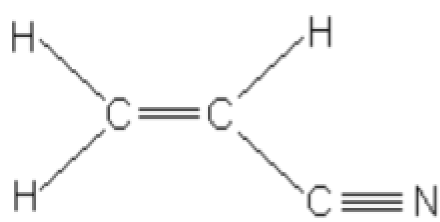

Figure 4 Chemical structure of acrylonitrile [10]

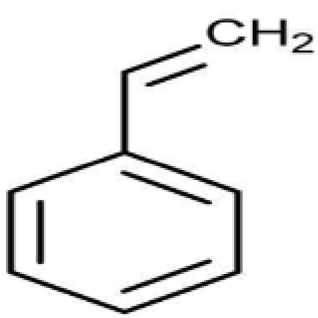

Figure 5 Structure view of styrene monomers [10]

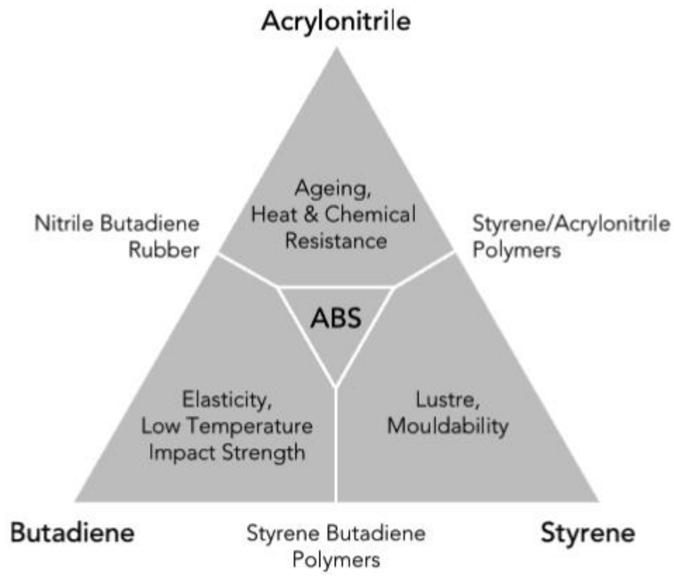

Figure 6 Basic Structure/property relationships of styrene polymers (pp.11) [11]

The good balance of properties between the monomers has two phases of polymer blend. The first copolymer phase is styrene-acrylonitrile copolymer (SAN) that gives the material its rigidity, hardness and heat resistance while the toughness is as a result of polybutadiene rubber particles that is evenly distributed in SAN matrix that gives the second phase blend. [11]

The combination of polyacrylonitrile, polybutadiene and polystyrene to form a single polymer gives it high impact strength, rigidity and hardness within the temperature range of -40°C to 110°C when compared to other engineering plastics. It has a mechanical behaviour of yielding plastically rather than tearing and the failures are ductile. ABS has a good long-term load carrying ability with stresses above their tensile strength, but modulus of elasticity and hardness are higher. Its relatively low creep nature and high heat resistance makes it a good dimensionally stability and electrical insulation material properties. ABS plastics are used in wide range of application in industry nowadays. Its uses among many others manufacturing methods are injection moulding, blow moulding, extrusion and as a filament for 3D printing products. [11, 12]

2.4.4 PLA

PLA is a term referred to as poly (lactic acid) or simply polylactide biodegradable thermoplastic made from lactic acid. It is categories among the biopolymers because of its renewability and degradability to nature, and has made it more interesting for wide range commodity applications. The lactic acid is versatile due to its application in the food, textile, pharmaceutical and chemical industries. They are naturally organic acid that can be produce in various ways of chemical synthesis or fermentation. [13, 14]

This fermentation could be derived from carbohydrate sources such as corn, wheat or sugarcane. It is a low cost process that combines significant environmental economics benefits of synthesizing lactide and PLA in melt instead of solutions that enable the production of workable biodegradable commodity polymer made from renewable resources. Below is an illustration overview of PLA production by fermentation process developed and patented by Cargill Dow LLC.

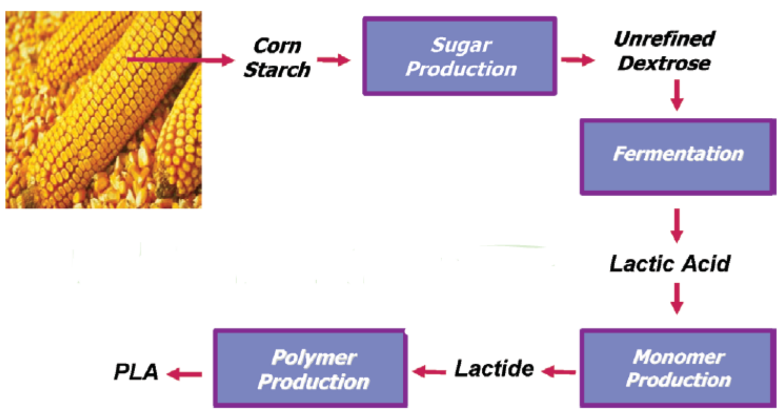

Figure 7 An overview of PLA production fermentation process (Cargill Dow LLC patented) [13]

Poly (lactic acid) material properties are good in comparison with many other bio-based materials. These properties depend on the processing temperature, component isomers, molecular weight, crystallinity, annealing time and glass transition (Tg). PLA is high strength and high modulus thermoplastic, which can be processed by most conventional plastic processes like injection moulding, extrusion, blow moulding and even used as 3D printing filament.

Table 2 below list some PLA material properties related to this research following the ISO standard testing conditions [14].

Table 2 Material properties of PLA

Physical Properties	Nature Works PLA
Melt flow rate (g/10 min)	2.4 – 4.3
Density (g/cm^3)	1.25
Mechanical properties	
Tensile strength at yield (MPa)	53
Elongation at yield (%)	10-100
Flexural modulus (MPa)	350–450
Thermal properties	
HDT (°C)	40–45, 135
Melting point (°C)	120–170
GTT (°C)	55–56

2.4.5 HDPE

Polyethylene is a thermoplastic material available in different forms and grades for various applications. Being the most important and dominant polymer that covers the largest part of plastic family, the polymer chain and molecular structure is critically important for the polymer synthesis preparation. Polyethylene is vinyl polymer that makes up the largest family of polymer. They are made from the monomer ethylene and are identified to how the chains are formed. The structure repeating unit can be seen in figure 8.

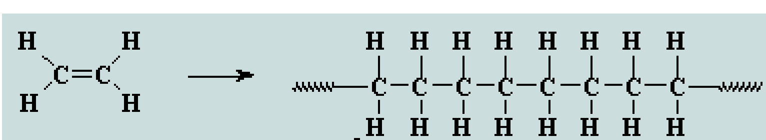

Figure 8 Repeating unit chains of polyethylene [6]

The degree of how highly dense these materials are depends on crystallinity which in turns depends on the molecular weight and what polymer branching structure it has. HDPE has as excellent mechanical properties like high compressive tensile strength, high stiffness and melting point, and greater crystallinity that make it outperform other polyethylene. However, there are other polyethylene that differs in densities, hyper-branched, crystallinities and categories according to their molecular weight and chain structure, listed in the table below [15]:

Table 3 Characteristics of different PE grades (pp. 19) [15]

PE	Density (g/cm³)	Degree of Crystallinity (%)	Number of branches (per 1000 carbon atoms)
LDPE	0.910 – 0.925	20– 30 (methyl); 3– 5 (n-butyl)	40 -50
LLDPE	0.910– 0.925	-	-
MDPE	0.926– 0.940	4– 6	
HDPE	0.942– 0.965	4 (Phillips); 5– 7 (Ziegler)	70 – 90

Generally, HDPE is considered exceptional because of its impact strength relative to other thermoplastics (one of the best impact resistance thermoplastic) and has an excellent machinability. It is somewhat hard and more opaque with range flexibility depending on production process and it can withstand rather higher temperatures. The processing temperature window for HDPE for example is, melt temperature (160-250°C) and an expected shrinkage of 1,5 - 3 %. It is used for products like rods, trays, detergent bottles, cosmetic containers, domestic water pipes, food boxes, used in automobile parts, industrial applications and the list goes on. The most common engineering properties of HDPE can be seen in table 4. [15]

Table 4 Common engineering properties of HDPE (pp. 31-34) [15]

Property	Unit Value
Density	0.942– 0.965 (g/cm³)
Melting point	120 - 135°C
Tensile Strength	20 - 40MPa
Strain at break	100 – 1000 (150%)

Tensile modulus	413- 1241MPa
Elastic modulus	0.2 – 1.2 (GPa)
Glass transition temperature	110°C
Coefficient of Thermal expansion	100 - 120×10^{-6} m/m ºC
Thermal conductivity	0.38- 0.51W/mK
Notched Impact strength (charpy)	2 -12 kJ/m²
Resistance	Above 100°C
Crystallinity	Greater than >90% (high crystalline)
Flexibility	More rigid

2.4.6 Filament Processing and testing

The processes involved in filament manufacturing and testing are described as follows.

2.4.6.1 Recycling

Plastics can be found in almost all domestic and industrial products which given more concern and raise questions on how to recycled and manage the waste. Recycling today has been beneficiary to us human and the environment at least to some extent yet a lot still need to be done to improve on. Plastic recycling or reprocessing plastic is the process by which waste or used plastic are reprocessed and developed into useful new products or processed to the same products or processed to completely different products.

The first step towards making recycling possible is by identification of the plastics, which was developed by the society of plastic industry (SPI). The identification methods consist of three arrow triangular shape with a number indicating the type of resin/polymer used in the plastic. Often the plastic is abbreviated at the bottom triangular shape sign shown in figure 9. There are other practical ways of recognizing a plastic such as burning tests, observation test and floatation test. An example of floatation test is separation; it involves separation of different plastic from the other by soaking them in water, the ones with less density like polypropylene will flow up while others sinks down. It is the basic one that is simple and easier to use. Some certain plastics are also recognized by the burning test according to their flame colour but experienced or expert engineers often make this observation. Nevertheless, in the recycling stream today, plastics are more recognized and separated according their resin codes with technologies such as optical cameras and different sensors [16].

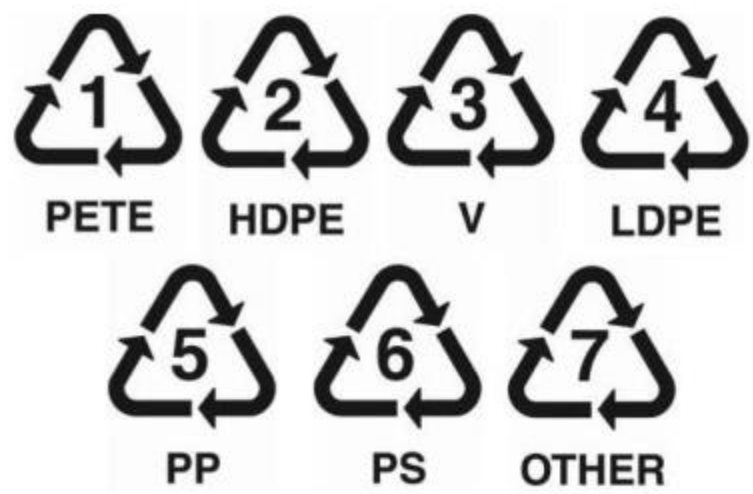

Figure 9 SPI identification codes [16]

Mechanical recycling

Mechanical recycling process involves reprocessing of plastic residues (post-consumer products) into new, the same or different products entirely. The practical main goal of mechanical recycling is to make new products by limiting the use of new raw material through a mechanical process.

It is regarded as the simplest and straightforward method of recycling. The steps generally consist of the following: collection, separation, grinding or shredding, washing &cleaning, drying, pelletizing, manufacturing and reprocessing by suitable method of the desired product usually injection moulding or extrusion. The process are shortly explained below which varies from material to material and sorted according to their resin code before further processing.

- **Collection**

  Generally, collection stage is often done to gather all kind of plastics into single place for further treatment. The collection is of the plastic waste in a large-scale industry are managed efficiently by categorization. They are classified into two groups, post-consumer plastic and post-industrial plastic waste depending upon the sources of plastic waste. Post-consumer plastics are recycled to another consumable product of the same type or different product such as PET bottles to food grade materials while the post-industrial plastic waste are recycled for industrial application like chairs, tables used in household appliances and automotive.

- **Separation and sorting**

After the materials are collected to a certain place, the separation phase is done to sort and separate the plastic according to resin identification codes. As the quality of the resin partly relies on the sorting process, it is important the sorting of the plastic is done efficiently to avoid different plastic mixtures along the way. Improper sorting could also affect the quality of the resin produced, so attention should be paid when sorting. In the large-scale industry several methods and technology are used for sorting.

- **Shredding**

Shredding process involves grinding the collected, sorted plastic material into smaller pieces called flakes. The flakes are then put forward for further processing in an extruder to produce pellets.

- **Washing**

Washing is usually done to remove contaminant from the plastic like dust, grease, labels, oil etc. They are washed with surfactants (detergents) or sodium hydroxide (Na0H) solution. The washing could either be done before the plastics are shredded into flakes or washed afterwards, depending on post-processing requirements and level of contamination.

- **Drying**

The drying phase simply requires enough heat so that all moisture is left out of the plastic and it is done using a drying machine at recommended drying temperature of the material.

- **Pelletizing or granulation**

Once the plastic flakes gotten from shredding are properly dried, they are process in an extruder to obtain pellets. The processing is done according to the material data sheet of the specific material. If for examples polyethylene (PE) is recycled and to be process to obtain pellets the temperature zones of the extruder would range between 190 - 200°C.

2.4.6.2 MFI

Melt flow index often shorten as (MFI), is a common measurement that is used to characterize thermoplastic polymers. It measures how easily or poorly a thermoplastic polymer is able to flow, by defining the mass of the polymer in grams per time period i.e. the output rate (flow) of the polymers in grams that occurs during a certain period of time. The result of this output flow rate gives an indication of how the thermoplastics will be easy to injection mould, hard to injection and easy or hard to extrude [17].

Melt flow index is an assessment of the molecular weight of plastic and is an inverse measure of the melt viscosity. I.e. the higher the melt index value, the lower is its viscosity and therefore, the lower the average molecular weight of the polymer. In terms of the mechanical strength, the lower the melt flow rate results the higher the mechanical strength and the higher melt flow results the lower the mechanical strength. This strength values could be observed in tensile stress, young's modulus of the material among others. During testing, it is important to note the weight load and temperature of the polymer because different weight load would produce different MFI results. [18].

The testing of MFI is according to ISO 1133 standard or ASTM D1238 standard depending on the choice to follow. It is practically done by applying certain kilogram of load via a piston at a melting temperature of the polymer and the polymer flow out through an orifice die while been measured at time interval. It is typically expressed in terms of grams of polymer that flows out per minute period (g/min). An example of this test for polyethylene (PE); a weight load of 2,16kg is applied onto a piston at temperature of 190°C when pushes down, the plastic melt flow out through a die and cut to be measured and calculated in g/10min. However, some polymers types often report melt index at different conditions, some polymers are measure at higher weight and temperatures and even different orifice die diameter. For example, polypropylene and ABS with weight load of 2,16kg melting at 230°C

2.4.6.3 Extrusion

Extrusion process is considered as one of the most used processing techniques in the plastic industry. It is a method by which raw material is forced through a die in a continuous process to form the desired shape die called extrudate (the product). The manufacturing products are mainly used to produces different cross-sectional components such as profiles, pipes, films, window frames, wire insulations, plastic sheets, filaments etc.

The following are the major components of an extruder:

- Hopper
- Screw
- Die
- Barrel
- Heater bands
- Cooling
- Pulling device
- Cutting device

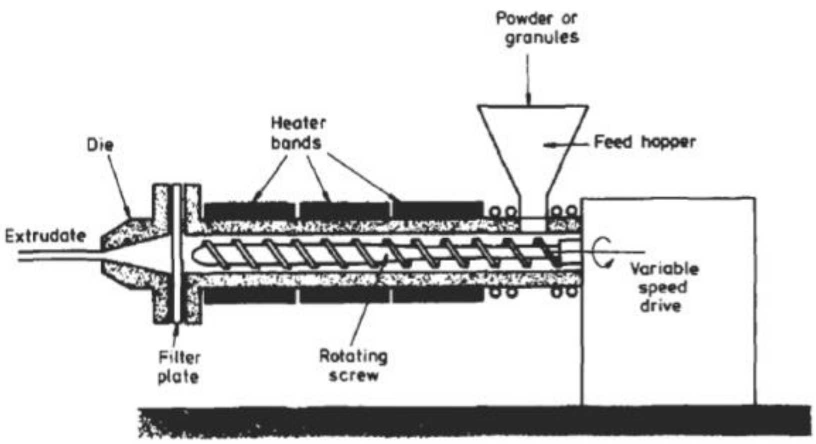

Figure 10 Schematic view of an extruder (pp. 246) [5]

Hopper

The hopper of an extruder is where the thermoplastic material is been feed through, either by gravity or regulatory device. They can be of different shape and size but the most commonly use shape is of the cone type.

Screw

The screw is one of the most important component of the extruder and its design crucially for proper mixing and processability of the polymers. It consists of three main geometrical zone-sections feed zone, compression zone and metering zone.

- **Feed zone/section:** This section is the deepest part of the screw where the pellets are introduced. The function of this zone is to preheat and convey the material forward to the subsequent zones. This continues at least as long as the material is solid and below the melting point.

- **Compression zone/section:** Compression zone or transition zone as it may be called is where the melting of the pellets takes place. The depth of the screw gradually becomes shallower so as to compact the plastic towards the barrel and transcends from the feed depth to the metering depth.

- **Metering zone/section:** The metering zone of the screw is where the melting of plastic is completed and ready pumping through the die. The depth here is constant and the supply material is uniform temperature and pressure. This zone is also referred to as pumping zone. (pp. 246 – 247) [5]

Die

The die is design and manufacture according to specific size of an extruder and the desired shape of the final product to be produced. Many die designs are available, depending on the extrusion process and the specification of the die is essential such that the final product would be produced accurately. There are several types of die that work corresponding to the job at hand:

- Lace die: they are used for producing pellets.

- Sheet die: to produce sheet shape products.
- Tube die: to produce tubes, profiles, hollow shapes etc.

The practical concept of extrusion process goes by feeding thermoplastic raw material (pellets or granules) into the hopper by gravity or regulatory device; this granules is fed through the feed throat on the screw feed zone, the rotation screw convey the plastic granules forward to the melting zone where the granules is melt through friction with the rotating screw and heating from the heater bands, afterwards the granules is pushed to the metering zone of the screw, this is where the homogenized material is brought to the desired process temperature then the molten plastic will pass through the die to take the desired shape. After forming the orifice shape of the die the profile will be channelled for cooling, during this cooling the plastic solidifies and take its final shape. The cooling could be done either by water-cooling or air-cooling among several other types.

2.4.6.4 Tensile testing

Tensile testing is the mechanical properties testing commonly used to obtain various data such as tensile strength, stress, yield, elasticity, toughness, strain, elongation and so on, for different material (metals, plastics, composites etc.). The test is used to determine the amount of force required to break a material. It measures the displacement and extent at which the material is been stretch before reaching to that breaking point. The core aspect of this test is to determine the strength and deformation of a material, as the force applied increase so does the elongation at the gage length until it fails. The strength of a material is often the main concern before its selection for any engineering applications, which are measured on how much it can deform before it fractures. The obtained data is used to specify the material, to design parts to withstand application force and as a quality control check of materials. [19]

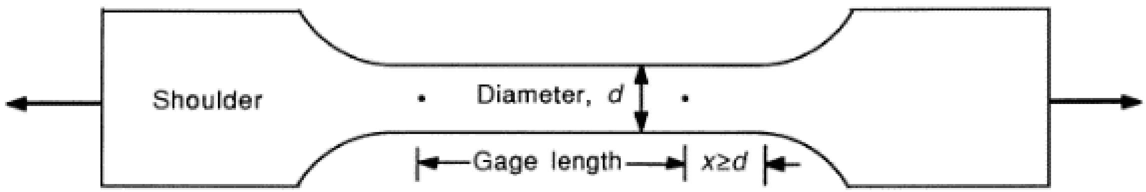

Figure 11 Example of tensile test specimen [19]

Tensile test piece also referred to as dog-bone are used for the test shown in figure11. It has wide shoulder for the gripping of the machine. The gage section plays an important role of the specimen for which its cross-sectional area are subjected to stretching until it fails or breaks when forces are applied to the shoulder. However, the gage length should be large enough and greater than its diameter because the stress state will be more complex than simple tension if reduced gage length is present. The test provides a stress-strain curve diagram that gives information about the mechanical properties of the material tested and is done according to ISO standards.

2.4.7 Materials for 3D filament

2.4.7.1 ABS as 3D filament

The thermoplastic properties of ABS is explored and found suitable as a filament because of its durability, mild flexibility, high strength, high glass transition temperature, impact resistance and very good heat resistance. Printing with ABS normally operates with hot end nozzle and bed at a recommended temperature. It is approximately printed with temperature between 230 – 256^{0}C and bed temperature of 80 to 110^{0}C depending of the printer. For ABS adhesion on print bed, different recommendations are made to use polyimide tape (Kapton tape or PET tape) and ABS juice. The ABS juice is basically mixture of ABS and acetone. The polyimide tape or ABS juice is applied on the print bed to get the first layer of prints to stick. The 3D printer nozzle diameter, layer height and printing speed settings are taken to consideration to obtain a substantial printed part.

2.4.7.2 PLA as 3D filament

PLA is considered to be the easiest material to work with during printing. It is a tough biodegradable thermoplastic with a little brittleness as it cools down. They are normally extruded between 160 – 220^{0}C and print at high speeds. A hot printing bed is not necessarily mandatory but warming the bed up with a temperature of about 60^{0}C could beneficial to the printed object. On the other hand, cooling fan is recommended to be installed and pointed at the extruder in order to speed up the cooling process because PLA is quite slow to cool down. They are used in medical application and food packages. Its application in the medical world is surgical implants, as it possesses the ability to degrade into lactic acid in the body system.

2.4.7.3 Earlier research concerning HDPE as a Filament

Specific information about recycled HDPE for 3D printer filament is difficult to find because the processing and quality varies, but some of the studies about HDPE as 3D printing filament has been done and few of them will be reviewed.

Christian Baechler analysed the testing procedures of diametrical consistency needed to ensure HDPE filament could feed consistently into the 3D printer. During the extrusion the following observation were made known.

- It was observe that variance of the extrusion rate should be avoided such that the extruder itself is automated with constant material feed rate that will enhance successful filament consistency. In other words, there should be constant rate of extrusion.
- Filament consistency should be measured along the way at an interval rate such that optimization would be improved.
- The filament was successfully extruded at an average rate of 90 mm/min and used to print parts.
- During printing, the addition of a heated print bed is recommended to limit the effects of thermal warping vulnerability on printed parts. [20]

Andreas Bastian described HDPE filament printing process and changes in his research. In an attempt to print with HDPE filament, several changes were made to the temperatures and printing speed in other for obtain suitable outcome. The hot bed of the printer was optimized such that HDPE would adhere and the extrusion temperature. After several temperature and printing speed changes, the data used were; a print speed of 5mm/s, a bed temperature of 70^{0}C and an extrusion temperature of 230^{0}C [21].

The RepRap community also gave some recommendation according to an experiment run that involve HDPE as filament. It shows that the filament size and extruding temperature corresponds by indicating viscosity changes, as the extruding temperature get higher so does the filament size diameter decreases. In regards to the printing, a foam-core board was discovered to be compatible with HDPE as a print bed material. [22]

2.4.7.4 Diameter tolerance

In normal circumstances, filament should maintain constant diameter across the entire spools, but due to manufacturing processes, there is always tolerances allowed depending on the size of filament been produced. For examples a 1.75mm filaments diameter allow tolerances of ±0.3mm. Then again, careful attention should also be paid during extrusion because inconsistence extrusion also leads to inconsistence diameter. (Inconsistence extrusion = inconsistence diameter).

2.4.7.5 Filament roundness

The regularity of the filament during production is of important across the entire length of the spool. This is because the filaments undergo some sort of compression upon making contact with wheels due to gripping and rotation winding.

Over all, the importance of high quality filament in 3D printing is to ensure uniform filament diameter (consistency) during production process and ability to optimize this diameter at a bending angle to rap around the spool that will be used to suit the 3D printer available. The most commons standard filaments are 1.75mm and 3mm.

The data in the following table are some of the recommended processing parameters specification during filament production that could also be optimized to suit available extruder and 3D printer. The comparisons are made between ABS and PLA that would be used in processing HDPE and rHDPE to produce the filament. These numbers ranges between different grade and standard testing results. [10, 13, 14]

Table 5 PLA, ABS and HDPE comparison

Properties	PLA	ABS	HDPE
Melt flow rate (g/10 min)	2.4 – 4.3	22- 48	4 – 8
Tensile strength (MP)	50 - 55	30 - 52	20 - 40
Strain at yield (%)	10 - 100	3 – 75	>100
Young's modulus (MPa)	3500	1700 – 2800	200 – 1200
Melting temperature (^{0}C.)	120 - 170	200 - 230	120 - 190
Glass transition Temperature (GTT) (^{0}C.)	50- 60	100	80 - 110
Extruding Temperature (^{o}C)	160 – 220	210- 230	130 - 190
Glass transition Temperature GTT (°C)	55–56	105	110
Crystallinity	~37	N/A	
Cooling time	Long	Medium	Medium

3 Method

In this chapter, the research methods used are explained. The methods can be divided into three different part; processing the material, testing the material, filament production and 3D printing. The material processing part is categorised as recycling where the plastic shredder, granulation process and injection moulding are used. The testing part was carried out using melt flow index plastometer and testometric tensile testing machine. The third part involves using extruder for the filament production while 3D printer was used to print the filament gotten from the production.

3.1 Recycling

Collection of material

Generally all sort of plastic are gathered into a single place for further treatment but in this study only HDPE is gathered, since it's the only material involved in recycling process. Different HDPE containers were collected in a plastic bag shown in figure 12.Most these collected material are from domestic products such as; used shampoo bottles, detergent containers, cleaning agent bottles, milk jars to mention few.

Figure 12 Collection of HDPE plastics

Sorting

The collected plastics were sorted and separated into two categories: (1) similar plastic of the same colour and (2) different coloured plastic together. This categorisation was performed to ensure proper process and avoid contaminant along the way for further post-processing.

Washing and separation

The plastics was soaked in 60°C of water for approximately 5 hours in order to remove the labels and the glue while washing at the same time. The labels were removed manually with hand by peeling. Afterwards, they are separated into similar plastic of the same colour and the different colour plastics are put together. The labels could have been removed alternatively by chemical solution but this choice is made since it peeled off easily and easy process to avoid chemical contamination or extensive washing post-processing.

Figure 13 Soaked HDPE plastic

Figure 14 Manual peeling of the labels

Figure 15 Similar washed plastic

Figure 16 Mixed coloured washed plastic

Shredding and Drying

The plastic shredder was used to shred the HDPE material into recyclable material. Similar plastic was shredded together and the mixed plastic together to obtain the flakes that will be used produce pellets in extrusion. Some of the big plastic was unable to fit in the shredded and was cut with hacksaw machine to reduce its size to enable the fitting.

The shredder works by throwing the plastic through a hopper into the shredder that cuts the plastic into recyclable material (flakes). The flakes are collected through a 5mm filter located at the bottom of the shredder to get an approximate uniform flake size.

After getting the plastic flakes from the shredder, the flakes are dried in flexible modular drying unit. This is done in order to remove extra moisture from within the plastic flakes. If not done, extra moisture may affect the material when being processed. In this case the moisture could be noticed as runny plastic melt or air bubbles within the plastic melt.

Figure 17 Plastic shredder (Arcada plastic laboratory, June 2014)

Granulation process

The granulation process was when the recycled HDPE flakes are produced to pellets (granules) using the extruder. This was done in order to get a uniform size pellets that would enhance the output flow of the molten plastic coming out of the extruder and used for the filament production. Figure 17 shows the mixed coloured flakes that were shredded together and figure 18 shows the produced pellet gotten from extrusion.

Figure 18 Mixed coloured flakes

Figure 19 Recycled HDPE pellets

3.2 Melt flow index

The phase of this experiment is to perform MFI test for the materials involved. This is done with melt flow index plastometer. As mentioned in the literature study, melt flow index (MFI) is used to determine how easily or poorly material is able to flow and it's measured in grams per minute's period (g/min). The test is done according ISO 1133 standard using Mitaten MFI extrusion plastometer. The materials involved are HDPE and rHDPE and were tested according to ISO 1133 standard.

Since the part of this research objective is to find filament specification and compared to optimized HDPE to rHDPE material properties. ExxonMobil™ HDPE HMA 014 grade was used as virgin HDPE in order to compare the values to the recycled one. The choice of this material is due to two factors. The first reason was the material fairly good mechanical properties and possibility to process with extrusion and this grade is specifically recommended for extrusion. The second factor is the availability of these materials in Arcada plastic laboratory. The materials are always preheated for 5min at 190^0C temperature before applying load of 2.16kg onto the piston that will pushes down the molten plastic through a die. After performing recycling process on the HDPE materials collected to get the rHDPE pellets, MFI test is also performed at 190°C melting temperature with the same load as virgin HDPE. Each material test was run 4times consecutively to obtain average figures. Figure 20 shows the extrusion plastometer used to perform the melt flow test.

The data was obtained by setting a cutting interval of 30 seconds on the plastometer. In other words, for every 30 second the plastometer is extruding a cutter is set to cut the molten plastic and weight afterwards. The following calculation method is used to obtain the data.

Recycled HDPE weighs 0,18g in 30 seconds, In 1 min equals 0,36g and in 10 min will equals 3,6g/10min.

Figure 20 Melt flow index plastomer (Arcada plastic laboratory, June 2014)

3.3 Injection moulding

Injection moulding took place in other to produce the dog-bones to be tested with tensile testing machine. Input values such as nozzles, cylinders, hopper temperatures, clamping force, cooling time, injection speed, injection times was used according to the moulding parameters of ExxonMobil™ HDPE HMA 014 grades. Although some of these values were optimized in other to produce dog-bone piece that suit the purpose of this research. This is due to the fact that the values on material data sheet are used for many different types of process and it is not said to be specific a certain product. After several optimization runs of the dog-bones the parameters shown in table 6 were the input values for the virgin HDPE and the same value were used to produce the recycled HDPE. Engel 90 CC was the injection moulding machine model used for the production.

Table 6 Optimized set up parameters for the injection moulding

Parameters	Value	Unit
Temperatures		
Hopper	50	^{0}C
Cylinder 2	190	^{0}C
Cylinder 3	190	^{0}C
Cylinder 4	180	^{0}C
Nozzle	200	^{0}C
Mould temperature	43	^{0}C
Clamping force	300	kN
Cooling time	15	s
Injection		
Injection speed	50	m/s
Injection time	4	s
Holding pressure		
Holding pressure	140	Bar
Holding time	5	s
Plasticizing stroke	25	mm

The Virgin HDPE and recycled HDPE dog-bone produced from injection moulding are shown in figure 21 and 22 respectively.

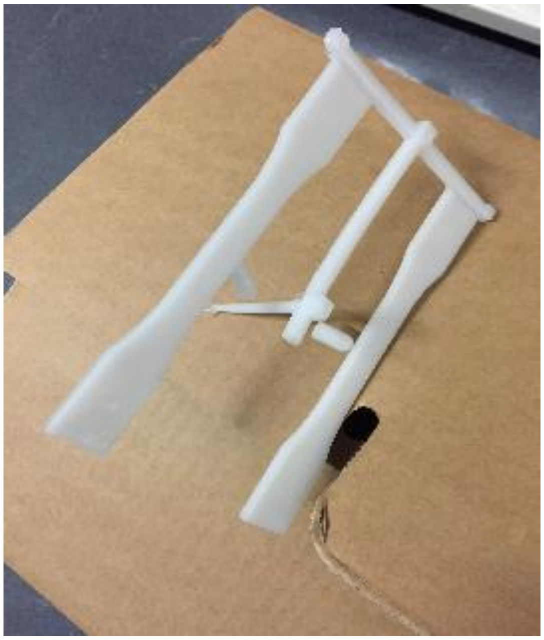

Figure 21 Virgin HDPE dog-bone piece

Figure 22 Recycled HDPE dog-bone piece

3.4 Tensile testing

In order to be able to test and compare results, the dog-bone produced from injection moulding machine were tested. This was done according to the explanation in chapter 2.4.6.4. It was done to obtain mechanical data of the dog-bone such as tensile strength, engineering stress and strain, young modulus, deformation etc. The virgin HDPE and recycled HDPE were tested with testometric tensile testing machine. The tests were conducted at a load speed of 51mm/min following ISO 1133 standard. The amount of dog-bones tested was set to be seven in order to be as accurate as possible by obtaining an average value and test specimen dimension are 120 x 12.8 x 3.1 mm.

The following figure shows the material been tested and the result obtained are listed in chapter 4.

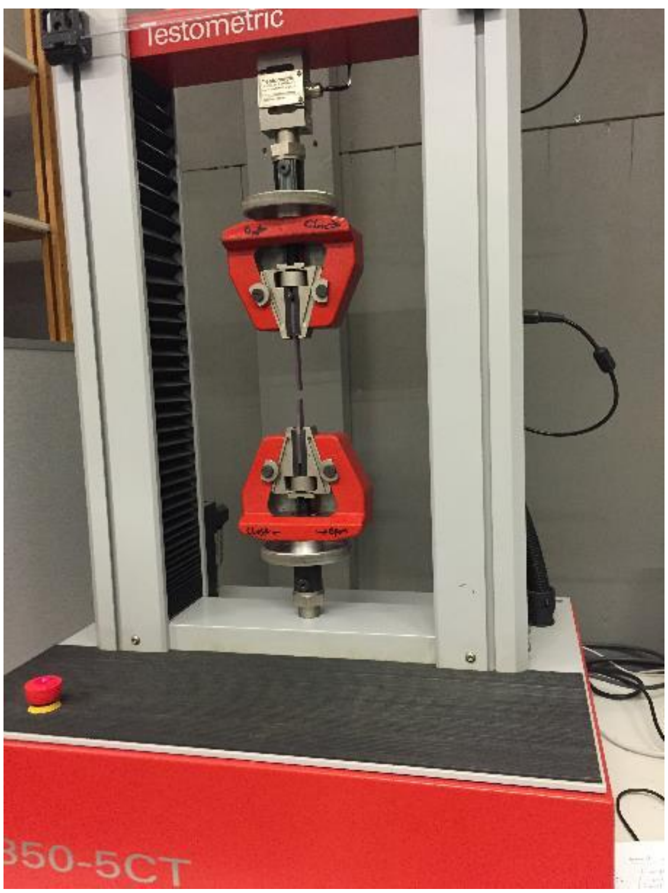

Figure 23 Dog-bone being tested with testometric tensile testing machine (Arcada plastic laboratory, June 2014)

3.5 Filament extrusion

The filament was produced using KFM Eco Ex model extruder that as six temperature zones. The first two zones function as feed zone heating temperature, the second two zones is the compression zone heating while the last two zone heats the pumping zone and the die. During the extrusion, several cooling methods were performed to obtain the filament size such as cold-air gun, water-bath, and table blower. An electric spooling device serve a puller for the filament during production while the filament is rap around a plastic profile tube.

Prior getting the desire size of the filament several optimizations was done. The optimization production is listed in the figure below and the parameters obtained are detailed in the result chapter. Figure 23 shows the manual pulling of the filament to the pulling device before been automated. After several pulling distance between the extruder and the puller, it was observed that the appropriate pulling and distance between the extruder the puller is one meter.

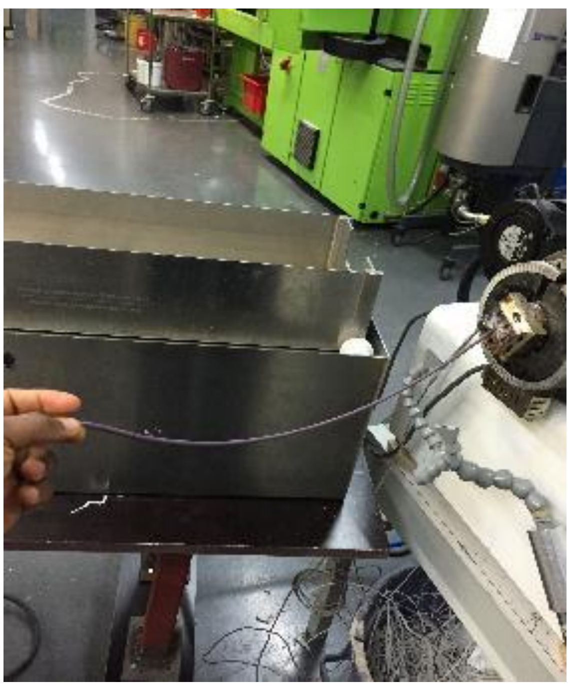

Figure 24 Manual pulling of filament

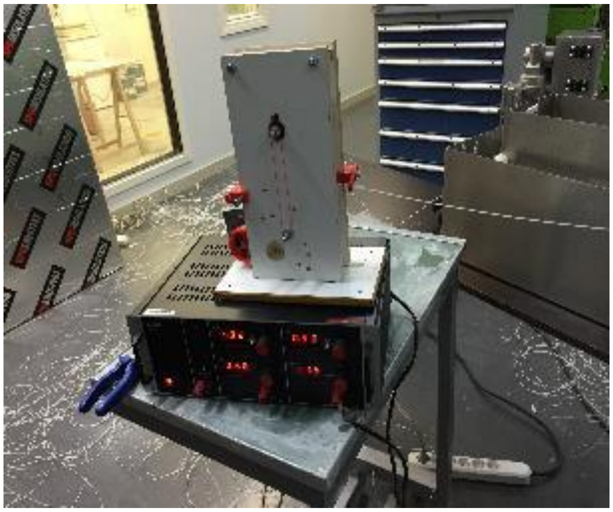

Figure 25 An electric pulling device

Figure 26 Filament extrusion (Arcada plastic laboratory, Oct 2014)

4 Results

4.1 Tensile testing & MFI

Table 7 Comparison table for 3D filament

Tests	PLA	ABS	Tested HDPE (Mean Value)	Tested rHDPE (Mean Value)
Melt index (g/10min)	2.4 – 4.3	22- 48	3,37	2,85
Tensile Strength (MPa)	50 - 55	30 - 52	25,45	25,59
Young's modulus (MPa)	3500	1700 – 2800	463,35	428,38
Strain at Yield (%)	10 - 100	3 – 75	16,12	16,12
Melting temperature (^{0}C)	120 - 190	200 - 230	190	190
Extruding Temperature (^{o}C)	160 – 220	210- 230	190	160 - 190

4.2 Filament extrusion optimization

Table 8 Optimization table of recycled HDPE (Filament Die)

Test runs	**Extrusion temperatures zones (^{0}C)**	**Extrusion speed (rpm)**	**Cooling method**	**Pulling device voltage (V)**	**Filament size (mm)**
1	175	20	Cold air gun	9,5	1,86±0,01
2	175	15	Cold air gun/ table blower	8,4	1,72±0,01
3	175	15	Room temperature	8,6	1,6±0,01
4	175	15	Room temperature	8	1,78±0,02
5	175	15	Room temperature	8,1	1,76±0,02
6	175	15	Room temperature	8,4	1,75+0,02

Figure 27 Recycled HDPE filament

5 Discussion

To determine the mechanical properties of recycled HDPE to suit 3D filament and the variability in these properties, this investigation looked at the relationship between melt flow rates, tensile strength of recycled HDPE compared to ABS and PLA and produced the filament. Table 7 and 8 shows the obtained data and optimization respectively

5.1 Changes in mechanical properties

5.1.1 Changes in melt index

The melt flow rates of recycled HDPE and virgin HDPE were studied to compare to ABS or PLA. The tested value of virgin HDPE gives 3, 37 g/10min and recycled HDPE gives 2,85 g/10min. Their difference of 0,52 g/10min indicates a decrease of 15%. This decrease in value of the recycled HDPE can be evaluated as result of degradation of the molecular chain that hinder the melt flow to become lesser. Comparing 2,85g/10min melt flow of recycled HDPE to PLA, it can observed that the flow rate only falls between the range of 2,4 – 4,3 g/10min and this signifies the possibility of recycled HDPE to be used as 3D filament.

5.1.2 Changes in tensile strength

Recycled HDPE gives an average value of 25,59MPa and the virgin HDPE gives an average value of 25,45MPa among the seven run tested. The value of tensile strength of the recycled HDPE is observed to increase slightly. This increase is however not significant given 0,55% with respect to the virgin HDPE. Comparing the recycled value HDPE to ABS or PLA tensile strength, it is observed that there is enormous value gap between both of them. This difference could also be established with different grade of materials used for specific processing methods. As a result of this, the recycled HDPE used for this analysis may have contained different plastic grade that are produced with different methods. It is also recalled that almost 70% of the gathered HDPE plastics are bottles, therefore this could be that the plastic grades involve are meant to be produced by injection mould blowing.

5.1.3 Changes in Young's modulus

Young's modulus value of the tested virgin HDPE as average value of 446,37MPa and the recycled one gives an average value of 428,38MPa indicating the decrease of 17,99MPa, which is 4,03% from the original virgin material. It can be established that the rigidity of the recycled HDPE decreased slightly and can be linked with ABS that has mild flexibility that makes rHDPE a probable material for filament.

5.1.4 Strain

The yield strain value of both the virgin HDPE and recycled HDPE indicates an average value of 16,12% and there was no difference on the average value. This strain value can be established with PLA strain data indicating high probability of recycled HDPE for filament.

All of these tested value depends on each other in one way or the other, that is to say, melt flow results indicates a consistent decrease of 15% and young's modulus decrease of 4% while the tensile strength indicates an increase value of 0,5%. The noticeable increase and decrease during the testing goes with explanation in chapter 2.5, that low melt flow index value results in higher mechanical strength and higher melt flow index value results in lower mechanical strength. For instance, the recycled HDPE is less viscous when compared to the virgin. In terms of mechanical properties, it can be observed that the recycled HDPE shows a decrease value in melt flow rate and an increase value in tensile strength that proves the literature findings mentioned above. The slight tendency changes of young's modulus between the recycled and virgin material can be established to be the rigidity of the recyclate content, grades difference and possible degradation.

5.2 Filament extrusion

During the extrusion experiment two type of die was attempted to produce the filament. Lace die was first attempted to produce the filament with water-bath cooling but the diameter of the die (6mm) was too big to optimize to the desired filament size and the filament shrinks too fast and became tangled as soon as it touches the water. Therefore, water-bath cooling was out of the question as a cooling method.

The filament die of 2mm was first tested with virgin HDPE at the same cooling method and pulling voltage to obtain some rough data to be optimized. More than seven trials were made but the only reasonable data was recorded and it served as a preliminary for the recycled HDPE material. The preliminary set data is indicated as the first test run in table10. When the recycled HDPE was in used, the extrusion temperature remained the same all thorough while other parameters were modified.

During the second and third production, the extrusion speed and the pulling device voltage was reduced by 25% and 60% respectively of the virgin material production (20 rpm and 21 V) then the filament size got close to the wanted size and cooling method was also changed to a room temperature.

Further on the production, the room temperature cooling method was observed to be enough for cooling at one-meter distance to the pulling device. This gives the molten plastic an approximate

70% cooling before been pulled by the pulling device. Then, it finally cools down after passing through the puller. The top and bottom wheel of the puller was adjusted to ensure the filament formed the required circularity shape that would fit the 3D printer nozzle available in the lab. At this stage and up to the final production, changes were only made to the pulling device voltage by reducing it with percentages.

The filament size measurement was done with venire calliper at an interval of 5meters with a tolerance of +0,02 in order to be as accurate as possible while producing them. The tolerance of +0,02 is allowed according to previous type filament production and the available 3D printer also gives a room of 0,3 tolerance (1,75mm+0,3 tolerance).

6 Conclusion

This thesis work covers recycling process of HDPE, finding 3D filament specification and production parameters. Comparison of ABS, PLA and HDPE was done to see the probable relationship between the materials and it is observed that recycled HDPE data goes along with PLA data with respect to melt flow, yield strain melt temperature and extruding temperature. Tensile strength and young’s modulus only differs from the test performed and it is neither close to ABS nor PLA. It can be established that recycled HDPE is suitable as 3D filament because of the data relationship to PLA.

As can be seen, changes in mechanical properties were monitored through recycling by measuring flow rate and tensile properties. Increases in tensile strength of the recycled material and decrease in melt flow rate were obtained. Comparing these engineering properties, the result shows that recycled HDPE is less viscous than the virgin HDPE. This amount to the prove of lower melt flow rate results to higher the mechanical strength and vice versa while the tensile strain remains even on both virgin HDPE and recycled HDPE.

The findings in the literature review concerning 3D filament production were built upon and viable corrections were made during filament production. Inconsistency in extrusion rate was avoided by ensuring constant feed of material is available that eventually improves the constant rate of extrusion.

Based on the data obtained during filament production, the puller speed is seen to be a great influence during the optimization simply because the voltage is been reduced at several percentage before getting the desired size. The percentage reduction basically improves the filament sized up to 60% from the started 21V. The extruding temperature of recycled HDPE also contributes to the optimization process by reduction. During the optimization, the temperature was reduced by 7,9% to enhance the procedure. It can be concluded that the higher the extruding temperature of rHDPE the more viscous the molten plastic becomes therefore reducing the temperature by percentage (7,9% of 190) turns out to balance the required viscosity.

Eventually, the parameter specifications of obtained for recycled HDPE filaments are:

Table 9 Recycled HDPE filament size parameters

Extrusion temperatures zones (^{0}C)	Extrusion speed (rpm)	Cooling method	Pulling device voltage (V)	Filament size (mm)
175	15	Room temperature	8,4	1,75+0,02

The results clearly shows that the pulling speed of filament is essential and have a big impact during the optimization with drastic improvement of 60% close to the 1,6 mm filament size. Distance between the die and the pulling device and room temperature cooling method also play a significant role to obtain the filament size diameter. During the filament production, another important part is filament consistency and diameter circularity in order to obtain desire 3D filament size. It can be concluded that recycled HDPE was found suitable as filament with respect to the data obtained and in relation with PLA data.

On a final note, 3D printing with the produced filament was unable completed due unavailability of form-core board that is compatible with HDPE which will serve as a printing bed but the filament was tested with the printer available (MINIFACTORY 3D-PRINTER) in the lab to observe the diameter tolerances, circularity and ability to print. The circularity and diameter tolerance was discovered to match the printer.

7 Suggestion for further work

Investigation could be done on recycled HDPE to improve the shrinkage. One example would be adding additives and finding the relationship with cooling method.

Another investigation could be about why recycled HDPE tangles during cooling with water that makes water-bath cooling not suitable before the filament size is optimized to desire diameter.

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