Recycling of HDPE from MSW waste to 3D printing filaments

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Abstract:

This thesis investigates the suitability of Fortum CIRCO® HDPE for fused deposition modelling (FDM). Fortum CIRCO® HDPE is recycled high-density polyethylene sourced from municipal solid waste, provided by Fortum Waste Solutions Oy. The literature review covers rHDPE's future in additive manufacturing. HDPE's general ubiquity does not carry on into 3D printing. Challenges of warping and poor first layer adhesion in FDM are exaggerated with HDPE. The experiments are targeted towards exploring the feasibility of producing a high quality rHDPE filament suitable for FDM. Filament characterization techniques are used to compare rHDPE filament to a reference HDPE material. Also, printed parts are tensile tested to analyse mechanical properties of rHDPE and HDPE. This thesis illustrates that it is possible to produce rHDPE filament, although extrusion parameters have not been fully optimized. During the extrusion process a heated water bath was found to be crucial in producing the most round and the most regular filament. rHDPE filament has a more elliptical shape and greater diameter variation than pristine HDPE when produced with the available setup. Both recycled and pristine filament samples produced comparable print quality and similar Young's Modulus values during tensile testing. In addition, the recycled material exhibited higher yield strength and greater ductility. rHDPE has potential as a feedstock material for FDM. Further research is required to improve the extrusion process.

Keywords:	rHDPE, FDM, 3D printing, filament, CIRCO® HDPE, MSW
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ABBREVIATION

3D	Three dimensional
ABS	Acrylonitrile butadiene styrene
AM	Additive manufacturing
CAD	Computer-aided drafting
FDM	Fused deposition modelling
HDPE	High density polyethylene
MFR	Melt flow rate
MSW	Municipal solid waste
PLA	Polylactic acid
rHDPE	Recycled high density polyethylene
STL	Stereolithography

FOREWORD

I would like to express my gratitude to my supervisor Maiju Holm for her professional advices, supervision and support throughout this thesis.

I would also like to thank my professor Mirja Andersson who introduced me to this interesting project and company Fortum Waste Solutions Oy (previously Ekokem) who provided this work with recycled HDPE.

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Helsinki, 2018

Kateryna Angatkina

1 INTRODUCTION

1.1 Background

Global production of plastic is increasing year by year. Plastic waste is not only generating huge numbers of landfills but also polluting oceans [1]. Most plastic materials are not decomposable and release harmful toxins when burnt. A variety of solid waste management techniques emerged, and plastic recycling became the most effective solution to protect the environment and reduce use of natural resources [2].

HDPE is the most produced plastic in the world with a wide range of applications. Domestic trash contains a large amount of plastic waste because of its widespread use in plastic packaging. Generally, plastic municipal solid waste is difficult to recycle due to other solid waste mixed with plastics. Production of high quality recycled HDPE requires complex and costly separation steps [2].

3D printing is one of the most promising technologies of the 21st century. A wide range of 3D printers are used for professional and consumer applications. They can fabricate complex three-dimensional objects from children's toys to fossil models [3].

HDPE has good chemical resistance and low water absorption. It is a non-toxic material with excellent mechanicals properties [4]. HDPE is not popular in 3D printing since it is a challenging material to work with, unlike PLA and ABS which are very common thermoplastics in FDM printing [5].

Only recently has rHDPE been considered as a feedstock for 3D printing [6]. The introduction of rHDPE into the 3D printing community will be another step in solid waste minimization as HDPE is the majority of municipal solid waste [5].

1.2 Aim and objectives

The aim of this research is to determine the suitability of rHDPE sourced from MSW for 3D printing.

Objectives:

- Uncover 3D printing challenges of HDPE in FDM printing
- Compare the material properties CIRCO® rHDPE to pristine HDPE
- Produce by extrusion 3D printing filament from CIRCO® rHDPE
- Evaluate produced CIRCO® rHDPE filament

2 LITERATURE REVIEW

2.1 3D printing

2.1.1 A brief history of 3D printing

3D printing or additive manufacturing (AM) technology has been around since the beginning of the new millennium. In 1984, Charles Hull developed an apparatus for creating three-dimensional objects from layers of cured liquid photopolymer by exposer to UV light. Two years later this technique was patented and called "stereolithography" [3]. *3D Systems* which was started by Charles Hull developed the first 3D printer for commercial use, SLA-250 [3].

By the end of 1988, 3D printing technology had gained more recognition. At the University of Texas, Dr. Carl Deckard and Dr. Joe Beaman developed selective laser sintering (SLS) which was a fundamentally different way of three-dimensional printing by using powder instead of liquid material [6].

Scott Crump, the founder of *Stratasys*, invented Fused Deposition Modelling (FDM) and the first commercial device based on FDM technology appeared in 1991 [6]. *Solidscape* started serial production of 3D inkjet printers at an affordable price, in 1993. The first colour 3D printer, Spectrum Z510, appeared in 2005 and it was developed by *ZCorporation* [6].

2.1.2 3D printing technology

There are two main branches of 3D printing technology which are selective deposition printers and selective binding printers, see Figure 1. The basic principle of selective deposition printers is the deposition of raw material into layers by squeezing out soft material through a print nozzle. Selective binding printers binds material with help of heat or light by using powder or a light sensitive photopolymer as a feedstock material [3].

The basic printing process consists of three central phases: creation of a 3D model, use of printer software to slice the model and finally model fabrication. A model of the part is created by computer-aided design software. The CAD model of the design is converted into an STL file. The printer software calculates the mechanical paths of the print nozzle by slicing the STL file into 2D sectional layers [6].

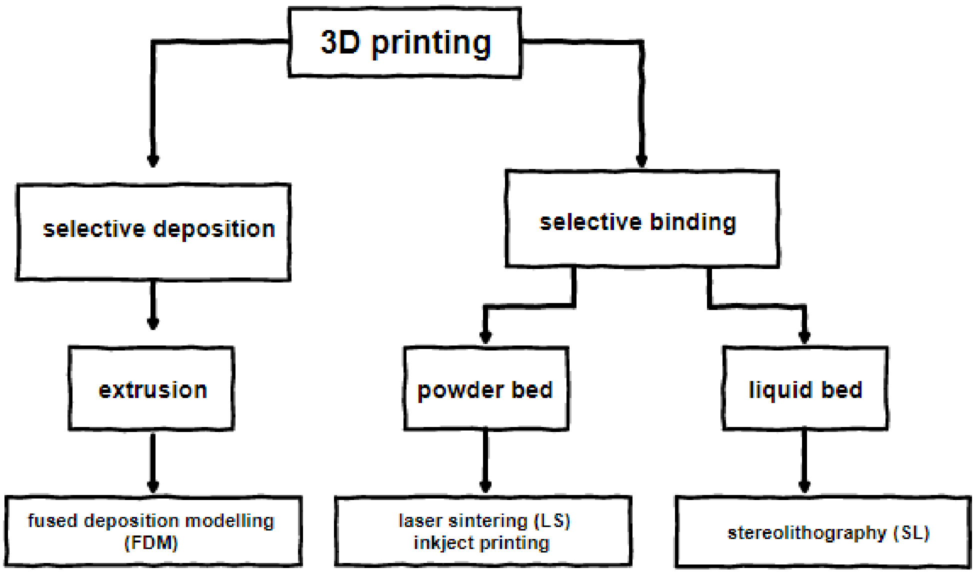

Figure 1. Two main branches of 3D printing technology [7]

2.1.3 FDM

In an FDM printer, feedstock as filament is fed from a spool through an extrusion nozzle. The nozzle has a heater block which melts a thermoplastic polymer. The melted material is extruded onto the printer build platform as shown in Figure 2. The flow and temperature of a material are set in printer software [8].

Most FDM printers deposit material by moving the print head or the platform along a set of horizontal and vertical axis while tracing the calculated path of the 2D layer. The first layer of the printed material usually outlines the base of the model. After the first layer's contours are filled, the print nozzle is raised a set layer height and continues to lay down the second layer. The printer lays down one cross-section of the model after another until the model is finished [3].

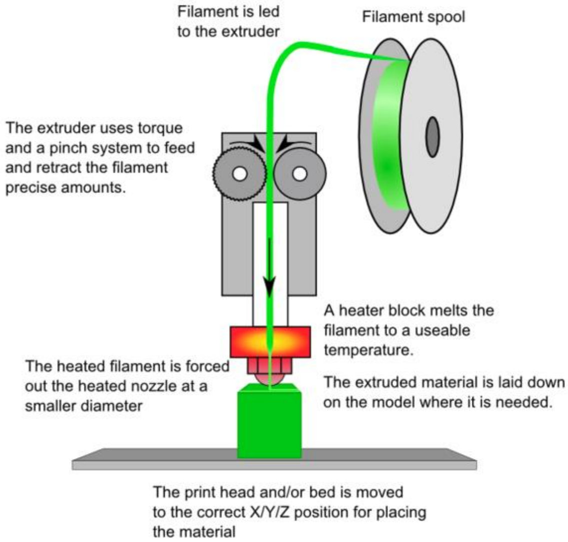

Figure 2. FDM printer process [9]

2.1.4 Filament requirements

A high-quality filament has a constant diameter across the entire length of the spool. During manufacturing filament diameter variation is inevitable. A diameter tolerance of ± 0.05 mm is considered as a standard for 1.75 mm nominal filament diameter. Inconsistent performance or a failed print is usually caused by irregular diameter of the filament [10]. Undersized filament can cause backflow inside the heater block of the extruder as shown in Figure 3.

Filament diameter dimensions affect the functionality of the extruder’s gripping mechanism. The drive gears of the extruder grip the filament and push it forward into the heated nozzle. Grip failure can occur with underside or oversized filament. Too thin a filament cannot be griped properly by the extruder drive gears and pushed forward inside the print nozzle. Too wide and the filament simply will not fit into the nozzle opening or it can strip some plastic off the surface by the drive gears leaving nothing to grip on to. In all cases no material is extruded out of the extruder’s nozzle and the printing process halts [10, 11].

The roundness of the filament also plays a significant role in good printing performance. Elliptical filament can cause similar failures of the gripping mechanism as filament diameter irregularity [10].

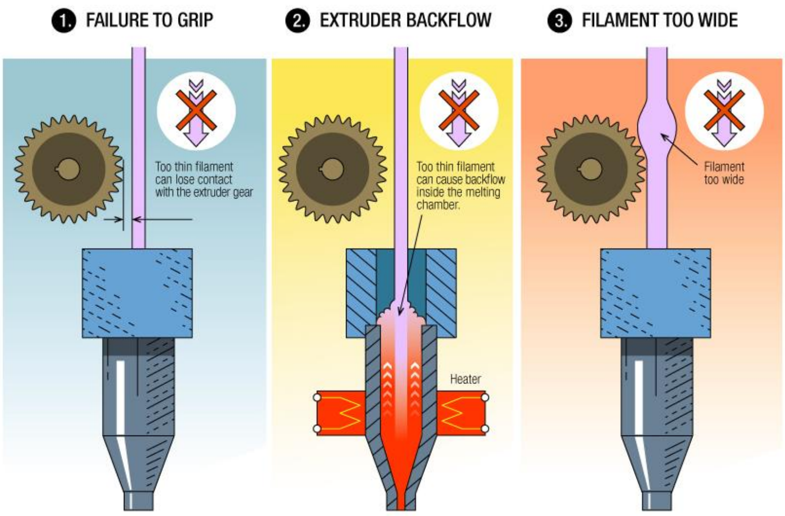

Figure 3. Failures of the gripping mechanism of the printer [10]

2.1.5 3D printing bed surface

The print bed is a surface where material is deposited onto by the extruder nozzle. There are both retaining and releasing properties required from a bed material. To create successfully printed parts, the first layer of the filament must stick to the print bed material. Good adhesion of the first layer prevents the printed part from moving around and allows for an aligned second layer. Warping is also a concern with poor adhesion of the first layer. Excessive warping causes dimensional errors as well as possible complete delamination from the bed. Finally, the printed part and the bed material must separate without damaging each other [10].

Common 3D printing bed materials are shown in Table 1.

Table 1. Common 3D printing bed materials [12]

Material	**Print bed surface**
PLA	Blue painter's tape, heated glass
ABS	Kapton tape, hair spray ABS slurry (mixture of ABS and acetone)
Nylon	Garolite (fiberglass/epoxy laminate)

2.2 Plastics

Plastic is made up of synthetic or natural polymers (resins). Polymers are long chains of monomers. A monomer is a main base molecule that repeats throughout the polymer structure, see Figure 4. Polymers can be synthesized by polymerization or polycondensation of monomers in the presence of catalysts under strictly defined temperature conditions and pressures [13].

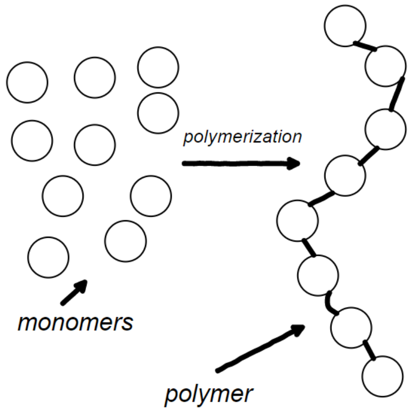

Figure 4. Monomer vs. Polymer [13]

Plastics can be divided into two groups: thermoplastics and thermosets. Thermoplastics are capable of reprocessing by remelting when heated and solidifying upon cooling. Unlike thermosets which have an irreversible curing process hence thermosets cannot be remelted and reused [2]. Thermoplastics consist of long independent polymers chains that are linked together by intermolecular interactions force. Thermosets are composed of chemically crosslinked polymer chains, and therefore they cannot be reshaped upon cooling as thermoplastics, see Figure 5 [13].

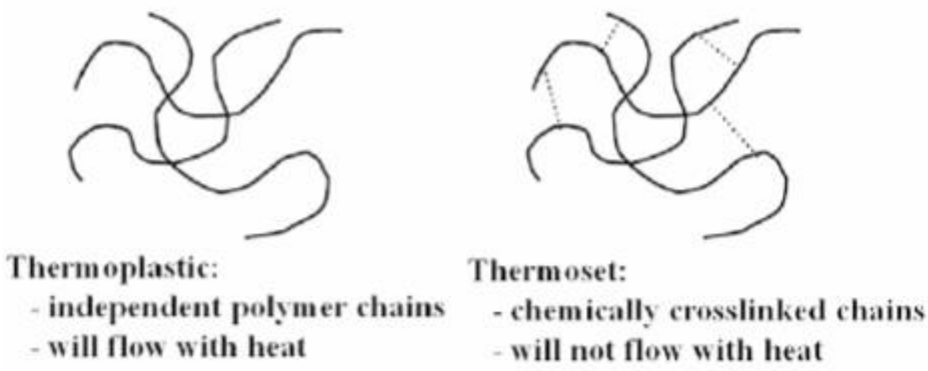

Figure 5. Thermoplastic vs. Thermoset [13]

Thermoplastics have two types of structure in the solid state: amorphous and semi- crystalline. In the solid state, amorphous polymers consist of random and disordered polymer chains and semi-crystalline polymers have both random and ordered molecular structures, see Figure 6 [14]. The polymer morphology affects both the physical properties and have a significant effect on the behaviour of the material during processing [14].

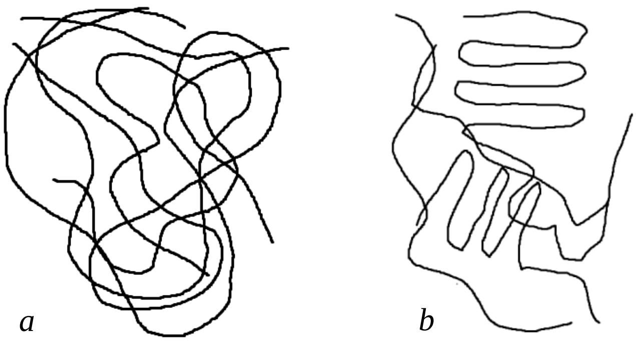

Figure 6. Amorphous (a) and semi-crystalline (b) structural representation [14]

Semi crystalline thermoplastics have a quite narrow temperature band during melting and solidification. Unlike amorphous materials which are melted and cooled over a wider temperature span [15].

2.2.1 Commonly used materials for FDM printing

In FDM, the most used materials are thermoplastics such as polylactic acid (PLA), acrylonitrile butadiene styrene (ABS), polycarbonate (PC), polyamides (nylon), polyvinyl alcohol (PVA), polystyrene (PS), high-impact polystyrene (HIPS) and polyethylene (PE) [5].

The most common materials are PLA and ABS. PLA is a sugar based biodegradable polymer, and therefore an environmentally friendly printing material. PLA can be made in a variety of colours and printed parts have smooth finish and appearance [5]. ABS is a carbon chain copolymer which consists of polymerised butadiene, acrylonitrile and styrene monomers [6]. ABS prints can be dissolved in acetone. Acetone is used in vapor polishing of ABS printed parts. Acetone vapour fuses the individual print layers of the part together and results in a glossy finish [16].

Polycarbonate (PC) is synthesized by a condensation polymerization of bisphenol A and phosgene. PC has a high gloss and clarity. Nylon is made by the reaction of the monomers diamine and a dicarboxylic acid. Nylon is strong, durable and flexible material. Polystyrene is made from the monomer styrene by free radical vinyl polymerization. PS is inexpensive, rigid and the second most used plastic in the world. PVA and HIPS are used mainly as soluble support materials. PVA is polyvinyl acetate and water soluble. HIPS is a limonene soluble polymer [5].

Advantages and disadvantages of various thermoplastic used in FDM are summarized in Table 2.

Table 2. Advantages and disadvantages of various thermoplastic used in FDM [6].

Thermoplastic	Advantages	Disadvantages
PLA	Environmental friendly High print speed and resolution	Brittle and low heat resistance Less flexible
ABS	High strength High heat resistance and melting point Flexible	Production of air born microparticles
PC	Resistance to scratches and impact Hight strength, durability and heat application	Not UV stable
Nylon	Excellent layer adhesion Self-bonding and lubrication	Sensitive to moisture
PVA	Conductive Biodegradable Water soluble	Water soluble
PS	Low thermal conductivity	Poor strength
HIPS	High strength and durability Non-toxic and recyclable	Limonene soluble
PE	High resistance to impact Recyclable	Difficult to bond Poor strength

2.2.2 High-Density Polyethylene

Polyethylene is a thermoplastic polymer which consists of a long chain of repeating methylene -(CH2)- groups [17]:

$$-CH_2 - CH_2 - CH_2 - CH_2 - CH_2 -$$

Polyethylene is formed by the process of additional polymerization. A monomer, ethylene, with a double bond is linked together with other similar monomers without producing a side product such as water. The repeating units of addition polymers have the same chemical composition as the monomers [17]:

$$nCH_2 = CH_2 \quad \rightarrow \quad \sim(CH_2 - CH_2)_n\sim$$

Ethylene Polyethylene

High density polyethylene (HDPE) is a linear polymer and formed by the Ziegler-Natta process. HDPE has a density range between 0.94-0.96 g/cm ^{3} and low-density polyethylene (LDPE) has the density 0.915 to 0.930 g/cm ^{3}. The high density of HDPE is due to molecules with little or no branching unlike LDPE which has some long branches and many short branches, see Figure 7 [18].

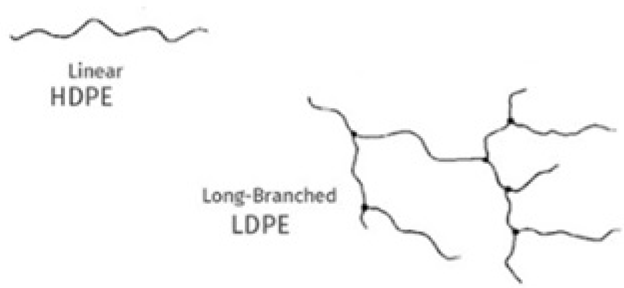

Figure 7. Schematics of molecules of HDPE and LDPE [19]

HDPE is semi-crystalline polymer with small amorphous regions [15]. HDPE has high crystallinity between 60 and 80% in a solid state and therefore shows high tensile strength [18, 20]. HDPE, as a semi-crystalline thermoplastic, has higher shrinkage values than amorphous plastics between solidifying and transition temperatures [15].

HDPE is used in injection moulding, extrusion, rotational moulding and thermoforming manufacturing processes [4].

Typical characteristics of HDPE [4, 15]:

- Toughness and stiffness
- Cold resistant
- Practically indestructible, but brittle after prolonged UV exposure

- No water absorption
- Very low dielectric losses
- High chemical resistance (insoluble in all solvents)
- Good barrier properties against gases and vapours
- High shrinkage

2.2.3 Challenges of HDPE in FDM

Warping and shrinkage of HDPE in FDM printing

HDPE is a semi-crystalline polymer and contains both amorphous and crystalline regions within the same polymer matrix. Semi-crystalline materials are prone to higher shrinkage during melt to solid transition than amorphous materials. The shrinkage is higher in semi crystalline materials due to its crystalline regions, the ordered and aligned molecular chains take up less space than the disordered polymer chains in amorphous region. Upon cooling, semi crystalline plastic's volume continues to decrease as the molecular chains are very closely aligning, packing themselves in their crystalline formation [14, 15].

HDPE shows a high shrinkage due to its higher crystallinity which makes it very sensitive to differential cooling rates. HDPE shrinkage rate is 4-6% therefore the rate of cooling is crucial for FDM process. To prevent 3D printed part of HDPE from warping, the layers of filament need be slowly cooled down during printing. The shrinkage between the melt and solid state is important [21].

The shrinkage resultant warping of the part being printed increases at faster cooling rates. The cooling time of 3D printed part is dependent on the wall thickness, melt temperature and rate of crystallization. The thin sections of mid-print part are cooled more rapidly than thick sections and consequently shrinkage in the former will be greater, causing the whole part to warp from differential cooling and shrinkage [21].

In HDPE deposition, a heated enclosure system for the 3D printer can be beneficial in controlling the shrinkage and warpage of the printed parts. The use of infrared (IR) lamps to heat and control the temperature of a printed part during printing can bring very positive results [22].

Adhesion and bonding of HDPE

In FDM, adhesion and bonding of the filament is a key element to good printability of the material [23]. HDPE poor adhesion results from low surface free energy, low polarity, and it lack functional groups on the surface [24].

Wetting is important in the adhesion of two materials and it can be used to determine the surface energy of a material. Wettability is a measurement of a contact angle (θ) between a solid and liquid. Contact angle (θ) is the tangent angle formed by a drop of liquid which is placed on a substrate surface, see Figure 8 [23].

When the angle is $\theta > 90°$, the attraction between liquid and solid is poor. A good wetting has a strong attraction between the liquid and the solid, when the angle is $\theta < 90°$ [23].

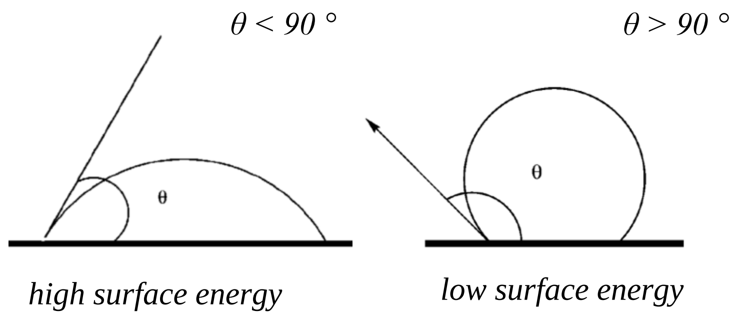

Figure 8. Contact angle measurement (θ) of wettability of material [23]

In general, HDPE has the water contact angle around 104 ° which means that the material has no polarity within its molecules, hence low surface energy. HDPE has adhesion problems due to weak boundary layers, therefore wettability is a crucial importance to the solution [23].

HDPE's poor wetting properties require to be bonded quickly before the molten material solidifies. The surfaces of the HDPE need to be warmed up prior to adhesive application to avoid the formation of poorly wetting surfaces which will lead to weaker bonding [25].

HDPE filaments needs to be applied the bed at a temperature significantly higher than the basic melting temperature of the HDPE filament to produce a lower viscosity fluid that successfully wets bed surface [25]. HDPE wetting properties can be improved by introducing polar groups into the material or increasing surface energy. It will lead to improved adhesion by providing fewer weak boundary layers through better wetting [23].

HDPE filament exhibits rather good self-bonding since the diffusion between two surfaces of HDPE filament at a high temperature occurs without complications [23].

Printed layers of HDPE bind easily to each other during the printing process. Since HDPE has difficulties binding to other materials an appropriate build surface must be used [5].

2.2.4 rHDPE from MSW in FDM

HDPE is a reusable thermoplastic polymer with the recycling code «2» as shown in Figure 9. In general, thermoplastics can be reprocessed many times by melting and solidifying into new products. Unlike thermosetting polymers waste which are transformed into fuels or chemicals and cannot be directly reused as a recycled material [2].

Figure 9. HDPE recycling code [26]

rHDPE from MSW was introduced into the consumption cycle as a material for secondary applications due to its inferiority to virgin HDPE. But even as a recycled material, HDPE still possesses unique characteristics such as water and chemical resistance, moderate impact and tensile strength, and excellent abrasion-resistant [21].

In MSW, HDPE is mixed with solids and other plastics. A recycling schema to obtain a homogeneous composition stream of HDPE from plastic waste is complex and costly [2]. In this research, rHDPE from MSW was reprocessed by using a mechanical recycling approach which involves several steps such as collecting, separating, grinding, washing, drying and re-granulating [2].

rHDPE from MSW has three major problems see Table 3.

Table 3. Problems of rHDPE from MSW [2]

Contaminants	small amounts of a different types of plastics may change the properties of the bulk material (HDPE)
Additives	plasticizers, substances, stabilizers and colouring. The recycled plastic usually has a grey colour because of the colour variety of the waste products.
Degradation	recycled plastics show worse mechanicals properties than the pristine material due to prolonged exposure to temperature, ultraviolet radiation, oxygen and ozone

Research related to rHDPE for additive manufacturing is scarce. Nevertheless, the accessibility of HDPE waste generates a great deal of interest among 3D printing enthusiasts and encourages efforts to apply recycled materials for good use. Especially milk jugs as a rHDPE material were adapted to 3D printing in numerous recycling research projects [5].

2.2.5 CIRCO® recycled HDPE

rHDPE was provided by Fortum Waste Solutions Oy formerly known as Ekokem. Fortum is a clean-energy company which is located in Finland. *Fortum's mission is to engage customers and society to drive the change towards a cleaner world [27]*. Fortum Recycling and Waste Solutions unit offers material efficiency improvement in the Nordic countries [27].

The batched of rHDPE used in this paper was recycled from MSW which was refined in the Plastic Refinery at the Riihimäki Circular Economy Village, Finland. rHDPE was returned manly to be used as an industrial material [28].

The physical and mechanical properties of Fortum CIRCO® recycled HDPE are shown in Table 4 below.

Table 4. The physical and mechanical properties of rHDPE (by Fortum CIRCO®) [29]

Property	**Value**
Color	Grey
Density at 23 °C	947 kg/m^3
MFI 2,16 kg/190 °C	0,35 g/10min
Tensile strength at break MD	10,7 MPa
Elongation at yield MD	39%
Tensile strength at yield MD	24,8 MPa
Young's Modulus	376 MPa
Impact strength	115 kJ/m^2

2.3 Filament manufacturing

2.3.1 Extrusion

Extrusion is a manufacturing process of homogenization of a raw material into a uniform shape product by squeezing it through a die. It is a continuous operation where the raw material in the form of pellets are fed through a hopper into the heated barrel of the extruder. In the barrel, the pellets are melted, blended and compressed through a die, see Figure 10 [4].

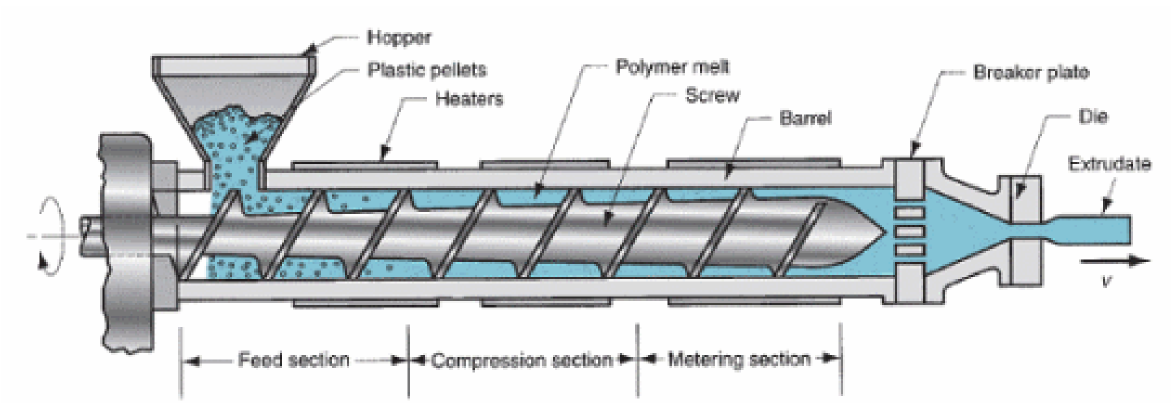

Figure 10. Schematics of a single screw extruder [30]

In the extruder, the most important component is the screw-barrel system since the material is melted and processed from the barrel temperature and mechanical energy which is imparted from the screw rotation [31].

The screw barrel system is divided into three sections [31] as shown in Figure 10 above:

- Feed section, where the raw material in the pellet form is compacted together
- Compression section, where the melted material is sheared by the screw flights and as the screw channel depth gradually gets shallower, see Figure 11.
- Metering section, where all the polymer is molten, and the screw channel depth is constant

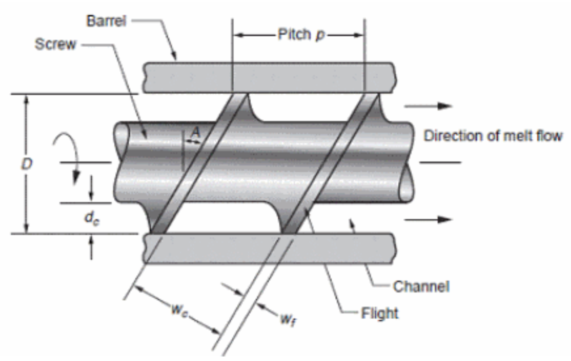

Figure 11. Geometry of the extruder screw [30]

Screw

In manufacturing industry, the screw differs with respect to materials since the efficiency of the extrusion process is mostly determined by the screw design. In the barrel the polymer is stretched and sheared with the heated walls by the screw flights with each screw turn. Frictional heating from the screw rotation and conduction heat from the barrel walls are both contributed to polymer melting. The processing material is constantly pushed from the feed section to the metering section by the screw. But the melt cannot be conveyed to a die if the molten polymer is stuck to the screw and not to the barrel walls [4, 31].

Die

The extruder die's function is to shape the melted material into a desired cross-sectional profile. The molecular orientation and surface aesthetics of a product can be also controlled by die design. High molecular orientation can be beneficial by increasing tensile performance or it can cause the polymer to shrink, warp and even easily split in the machine direction [14].

Die swell

At the die exit, polymer materials tend to swell due to expansion of viscoelastic fluids. The elastic recovery occurs when elastic stresses contract and relax in the fluid during extrusion process. For example, PE increases in size by 15-20% relative to die cross-sectional area [14]. Die swell is dependent on shear stress, polymer molecular weight distribution and geometric dimensions of the screw and die such as the length to diameter ratio of the die [4]. To achieve a desired shape of the product extrudate is pulled away from the die and drawn down to its final dimensions by a puller. The draw speed depends on the product size exiting the die versus the size required in the final product application [14].

Cooling

Controlled cooling is important in production of final products for satisfactory dimensions and performance. The extrudate is commonly cooled with water or air. At first the cooling extrudate forms surface skin while the centre stays molten. To prevent warpage of the part it is necessary to cool uniformly on all sides preferably at a slow speed. Water cooling systems allow the extrusion line to operate at higher rates. For semi-crystalline polymers the extrudate needs to be cooled slowly and evenly for precise product dimensions [14].

Puller

Final dimensions of the product are controlled by the extruder screw speed, cooling strategy and the puller speed. The draw and tension of the extrudate are regulated by the puller. Changes in the product dimensions are usually caused by periodic variations in the puller speed or extruder output. The extruder output rate and the puller speed must match each other. The puller can cause marks on the final product's surface by applying too much pressure with the puller rolls. For good performance the puller must be properly aligned with extruder at an appropriate distance [14].

2.3.2 Filament characterization techniques

There are limited standards which relate to AM. Filament based standards for AM are very general leaving no guidelines. Most of material descriptions and processes involved in their fabrication are unavailable due to copyrights or formula secrets in AM business [32].

Generally tested properties of FDM filaments are [32]:

- Filament diameter consistency
- Porosity
- Moisture content
- Thermal properties
- Mechanical properties of filament
- Melt Flow Rate

Filament Diameter Consistency

A 3D printed part's layers thickness and the extrusion diameter are determent by the nozzle diameter which is generally fixed in a FDM printer. A common commercial filament has an average diameter of 1.75 mm or 3 mm. A nonuniformity in diameter leads to improper feeding or jamming of feedstock and even nozzle clogging. Deviation in the diameter is inevitable in the actual manufacturing process [32].

Porosity

Porosity is determined by void content percentage in the filament. High void percentage of the filament can cause a great deviation in mechanical properties. The void content of filament can also be used to identify quality of the filament [32].

Moisture content

Moisture content influences the melt viscosity. High moisture content of the filament negatively affects print quality. 3D printing materials with low moisture content usually produce better quality prints. Inside the hot print nozzle, moisture in the filament vaporizes and leaves gaps in the strand of filament. Gaps can have a negative result on inter layer adhesion. For example, Nylon, PC and ABS are hydroscopic materials and a standard test method for determination of moisture in plastics by loss in weight, ASTM D6980, can be used to find the moisture content these thermoplastics [6, 32].

Thermal Properties

Melting points and glass-transition temperatures of polymers are studied to determine the thermal stability of the thermoplastics. In FDM the printing temperature of the material above the melting point of polymer and at high temperatures thermoplastics degrade. Differential scanning calorimetry (DSC) is used to study thermal degradation [32].

Mechanical properties of material

The most relevant mechanical properties of material processability in FDM are the tensile strength and strain. The mechanical properties of recycled polymers can considerably vary with impurities in their structure. The material properties of strength, ductility and toughness can be affected [32].

Tensile properties predict how the material will react to forces being applied in tension. Tensile tests show the ability of a material to withstand maximum load before rupture [33]. A tensile test of plastics consists of loading a specimen in tension. The test provides data of the tensile stress-strain relationship [34]. A typical tensile specimen is illustrated in Figure 12.

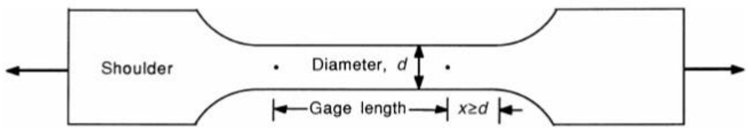

Figure 12. Typical tensile specimen [34]

The tensile stress-strain relationship is the ratio of the applied force (F) over cross sectional area of the gage section (A) at a constant deformation rate [32]:

$$\delta = \frac{F}{A}$$

Where δ is the stress [MPa], F is force [N] and A is cross sectional area [mm^2]

The definition of strain is:

$$\varepsilon = \frac{\Delta L}{L_0}$$

Where ε is the strain, ΔL is the extension of the length [mm] and L_0 is the original length [mm].

Young's Modulus, the elastic modulus, is a measure of the stiffness of the material. It is defined as the relationship between the stress and strain in an elastic material:

$$E = \frac{\delta}{\varepsilon}$$

Where E is the modulus [MPa], δ is the stress [MPa] and ε is the strain [32].

A typical stress-strain relationship for a polymer material with a yield stress greater than the failure stress is shown in Figure 13. Characteristic values associated with the material under test can be determined [33]:

- Stress at Yield (MPa)
  The lowest stress at which strain increases without a corresponding increase in stress.
- Stress at Break (MPa)
  The tensile strength of the material at failure.
- Strain at Yield (%)
  The corresponding tensile strain of the material at the yield stress.
- Strain at Break (%)
  The corresponding tensile strain of the material at the failure stress.

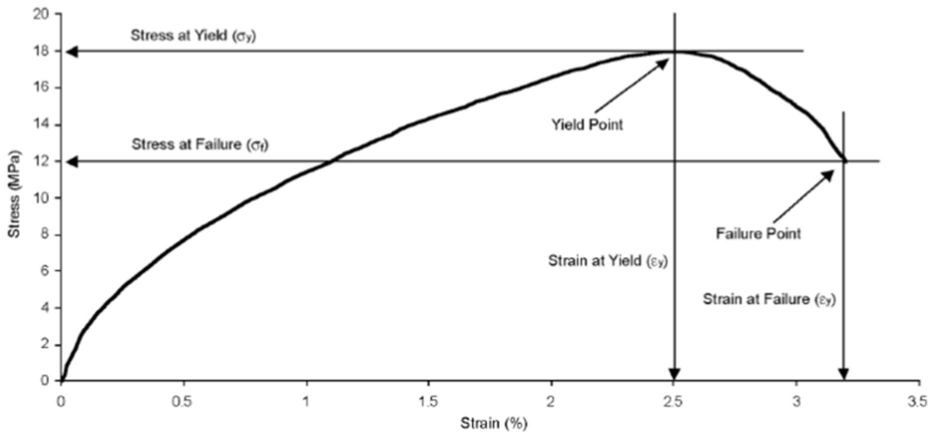

Figure 13. Stress - strain curve for polymers [33]

Melt Flow Rate

In FDM, the molten polymer is extruded through the print nozzle. Polymers that are not viscous enough or less viscous can be difficult to deposit. Melt flow rate (MFR) tests are done to determine the flow characteristics of material [32]. MFR is the number of grams of material extruded from a die in a certain time under a certain applied load [35]. A schematic of MFR basic test is illustrated in Figure 14 according to ISO 1133:2005 [35].

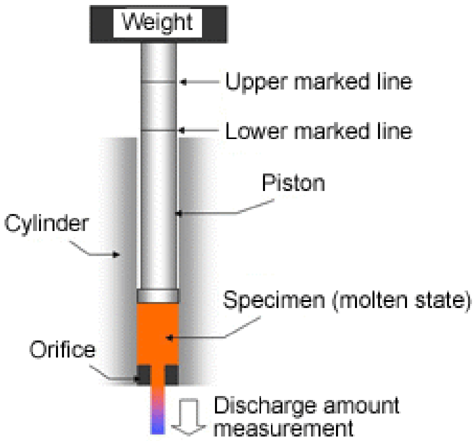

Figure 14. Schematic of MFR basic test, ISO 1133 [36]

MFR values are not directly comparable since the weight used to drive the MFR grader piston the temperature can be different. The most common commercial filaments have a high melt flow rate indicating low viscosity [35], see Table 5.

Table 5. MFR of the most common commercial filaments

Material	**MFR [g/10 min]**	
ABS (220°C/10.0 kg)	0.49 to 36	[37]
PLA (190°C/2.16 kg)	1.5 to 36	[38]
PC (260°C/5.0 kg)	11 to 29	[39]
HIPS (200°C/5.0 kg)	3,0 to 12	[40]

3 METHODS

3.1 Materials and equipment

3.1.1 Materials

The following materials were used in this research: recycled high-density polyethylene (rHDPE), CIRCO®, supplied by Fortum Waste Solution Oy, MFR 0,35 g/10min. (190 °C/2.16 kg); high-density polyethylene (HDPE), supplied by Borouge, MFR 0.4 g/10 min. (190 °C/2.16kg).
HDPE from Borouge was used as a reference material to analyse mechanical and flow properties of rHDPE in relation to pristine HDPE.

3.1.2 Equipment

Equipment available at the Arcada's production laboratory were used in this research:

- A single screw Eco Ex model extruder made by KFM
- Extrusion plastometer, Mitaten MEP 2/PC
- Injection moulding machine, ES 200/25HL CC88, made by ENGEL
- Material testing machine, M 350-5CT, made by Testometric
- FDM printer made by miniFactory

3.2 Filament manufacturing

rHDPE and reference HDPE were provided in pellet form. The material granules were fed into the extruder hopper where they were heated to molten state and forced through the die producing filament. Both materials were not pre-dried before extrusion process.

Filament extrusion line consisted of a single screw Eco Ex model extruder, cold air gun set up to cool and support extrudate in the air, a water bath and a pulling unit with a coiling device, see Figure 15 and 16. A 3 mm circular die was used to achieve 1.75 mm diameter of the filament by drawing to final diameter. The water cooling section had a length of 75 cm and was position at 10 cm from the extruder die. The filament puller was position at 175 cm from the extruder die.

Figure 15. KFM extruder, Arcada 2018

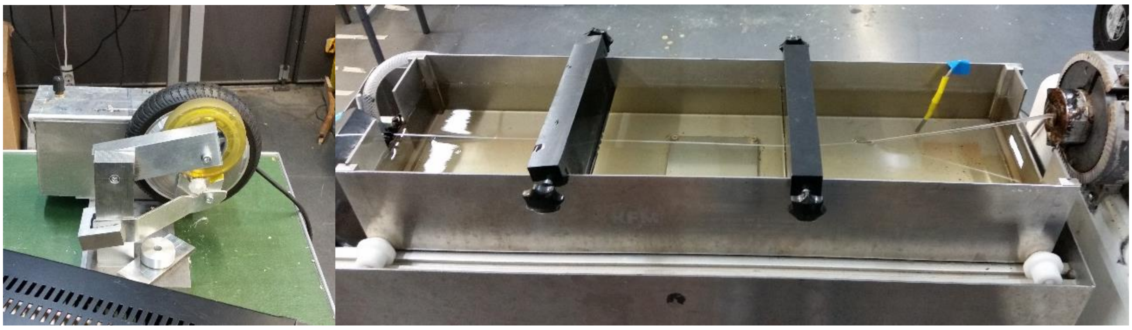

Figure 16. Puller and cooling water bath, Arcada 2018

The extrusion process parameters for rHDPE were selected based off the results of several trial runs. The same parameters were used for pristine HDPE extrusion see Table 6 and Table 7. The water bath temperature was set to 50 °C to have a slow cooling process and to control diameters of filaments. The cooling water temperature was crucial to the roundness and diameter tolerance of the filament. To cool the extrudate uniformly on all sided, it was positioned at 90 ° at the water bath dam entrance see Figure 17.

Figure 17. Water bath dam entrance, Arcada 2018

Table 6. Temperature of various extruders' zones of rHDPE and pristine HDPE

Die	Zone 4	Zone 3	Zone 2	Zone 1
200 °C	195 °C	190 °C	180 °C	180 °C

Table 7. Extrusion process parameters for rHDPE and pristine HDPE

Parameters	Value
Extruder screw speed [rpm]	20
Pulling unit speed [m/min]	3.4
Water bath temperature [°C]	50
Water bath length [m]	0.75
Total cooling section length [m]	1.75

3.3 Testing

3.3.1 Filament quality control

Filament roundness

The measurements of diameter of rHDPE and pristine HDPE filaments were obtained by using a Vernier calliper. The diameter of the filament was measured twice per point of measure, vertically and horizontally as shown in Figure 18. Dimensions of length "α" and "β" were recorded at interval of 1 m, see Figure 19.

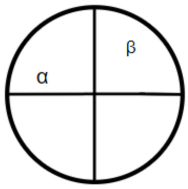

Figure 18. Schematic of filament cross section

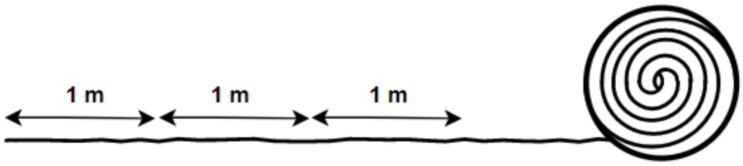

Figure 19. Measurement of the diameter of the filament at interval of 1 m

Porosity and impurities

Surfaces and cross sections of the rHDPE and pristine HDPE filaments were inspected under an optical microscope to find the void fraction and imperfection of the filaments. Cross section samples were prepared by cutting filaments at 90 ° angle by scalpel.

Thermal Properties

TA Instruments DSC Q2000 was used to find the melting point and glass transition temperature of unidentified foreign material extracted from rHDPE filament. A single sample of 2.06 mg of material was tested with a temperature range of -90.12 to 397.04 °C at 20.00 °C/min (heat only). Ramp 10.00 °C/min to 200.00 °C and 10.00 °C/min to 0.00 °C with isothermal time of 3.00 min.

3.3.2 Melt Flow Rate

MFR test was conducted according to ISO 1133:2005. rHDPE filament and pristine HDPE filament were tested. The extrusion plastometer was heated to 190 °C, see Figure 20. 4 g of each tested material was placed inside a preheated barrel. The piston was put into place and load of 2.16 kg was applied on top of the piston. Extrudate was cut off after the piston reached the indexer. The cut off time interval time was 120 sec. Five samples of each tested materials were taken and weighted to the nearest 1 mg. MFR was calculated according to the following equation:

$$MFR(T/m_{nom}) = \frac{600m}{t}$$

Where *m* is the average mass of the samples [g] and *t* is the time interval [s]

Figure 20. Material testing machine, M 350-5CT Testometric, Arcada 2018

3.3.3 Tensile testing of raw materials

Injection moulding machine, ENGEL ES 200/25HL CC88, was utilized to produce the test samples. Samples were made according to the ASTM D638-67T. The dumbbell sample type IV was chosen as a primary specimen as shown in Figure 21.

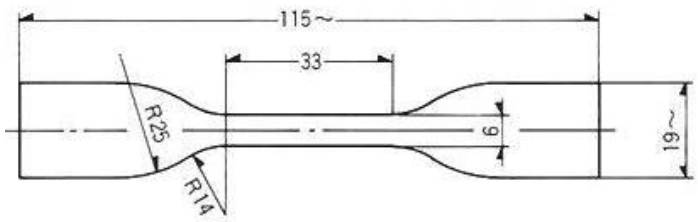

Figure 21. Dumbbell sample type IV according to ASTM D638 Standard [41]

Parameters for injection moulding were optimized for rHDPE, see Table 8. Samples for a reference material, pristine HDPE, were produced with the same parameters to compare them with recycling samples.

Table 8. Parameters for injection moulding machine

Parameter	**Value**
Temperature	
Nozzle [°C]	220
Cylinder 2,[°C]	200
Cylinder 3 [°C]	215
Cylinder 4 [°C]	190
Mould temperature [°C]	50
Injection speed [mm/s]	55
Injection pressure [bar]	80
Holding pressure [bar]	30
Holding time [s]	7

Tensile testing was performed by using a material testing machine, Testometric M 350-5CT see Figure 22. This process was done according to the ASTM D638-67T. Each specimen was firmly held by grips from both ends and load was applied.

15 dumbbell samples of each tested material were run to find out the mean values of stress at yield, stress at break, strain at yield, strain at break and Young's Modulus. A testing speed was 2.5 mm/min.

Figure 22. Material testing machine, Testometric M 350- 5CT, Arcada 2018

3.3.4 Tensile testing of 3D printed models

Test specimens were printed using a FDM printer, see Figure 23. The printer dimensions were 150 x 150 x 150 mm with a nozzle diameter of 0.4 mm. The maximum temperature of the nozzle is 300 °C and the temperature of the print build platform is 100 °C. The recommended filament thickness is 1.75 mm.

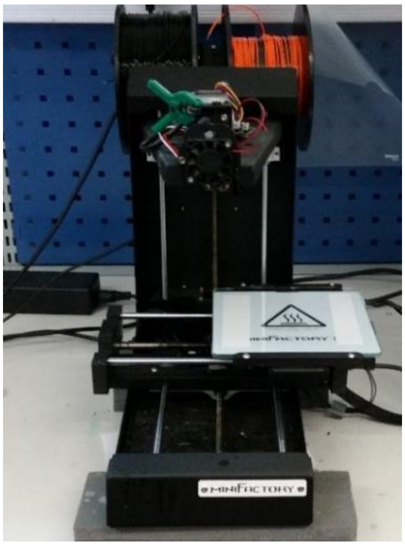

Figure 23. miniFactory printer, Arcada 2018

HDPE as a feedstock material required special preparation compare to common FDM filaments due to its semi crystalline nature. The shrinkage and warp deformation of HDPE greatly influenced deposition settings of a general FDM printer [24]. In this research a heated enclosure for the FDM printer was not introduced. Polypropylene (PP) sheet was utilized as a main print platform material. Since it was empirically established that rHDPE and pristine HDPE exhibited poor adhesion to traditional surface materials such as glass, Kapton tape and ABS slurry.

A dumbbell sample EN ISO 527-2 1BA was chosen as a primary specimen as shown in Figure 24 due to its a compact size and fast printing time. The specimen was designed in SolidWorks and converted to STL for 3D printing. A sliced model of the specimen was made by Repetier-Host software, see Figure 25.

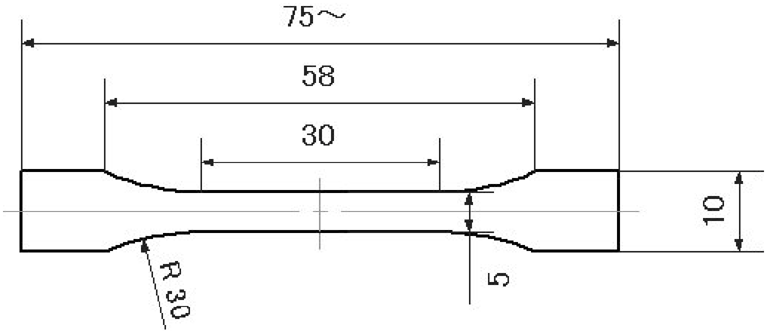

Figure 24. Tensile test specimen type 1BA, SFS EN ISO 527-2:1996[41]

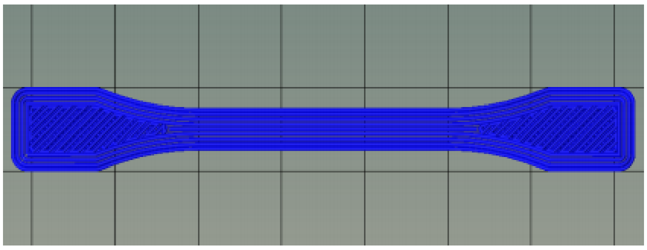

Figure 25. Sliced model of the specimen type 1BA, SFS EN ISO 527-2:1996

To test filament performance, specimens were printed horizontal to the print bed, arranging the layer orientation longitudinally along the sample's length, see Figure 26 [42]. Specimens were deposited one by one on the printer platform to get better quality prints.

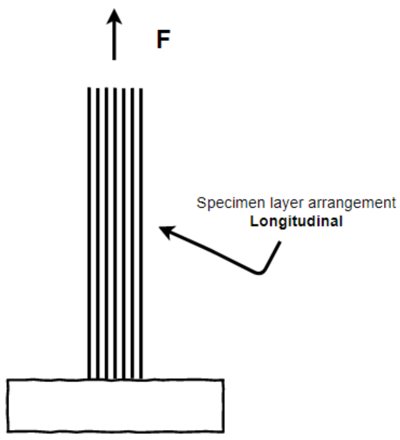

Figure 26. Specimen layer arrangement for a tensile test [42]

Five samples of each material were printed with the same printing parameters, see Table 9 below.

Table 9. The miniFactory printer setting for rHDPE and pristine HDPE tensile specimens

Parameters	**Values**
Extruder temperature [°C]	220
Platform temperature [°C]	60
Infill [%]	20
Layer height [mm]	0.2
Infill style/pattern	Rectilinear
Angle	45
Build orientation	Flat
Speed for print moves, perimeters [mm/s]	60
Speed for print moves, infill [mm/s]	80

Tensile properties of the printed rHDPE and HDPE samples were tested on the Testometric M350- 5CT machine according to the ASTM 638-67T. The selected tested speed was 2.5 mm/min.

3.3.5 The rHDPE filament prototype testing

Additional specimens were designed to observe the shrinking and warping of rHDPE and pristine HDPE materials alongside adhesion and self-bonding characteristics.

A cube and a cylinder 30x30x30 mm with thin wall features were chosen to investigate the shrinking and warping characteristics with a reference to prior research in this field [24], see Figure 27. To evaluate straight, parallel and perpendicular features of the printed specimens, a solid cube of 8 cm^3 volume was chosen as a printing object.

The extruder temperature was 220 °C and the platform temperature was 60 °C. The shell thickness for the hollow samples was 0.4 mm, one nozzle thickness one wall. For a solid cube, infill density was 20 %. The print speed was 50 mm/s for all printing samples.

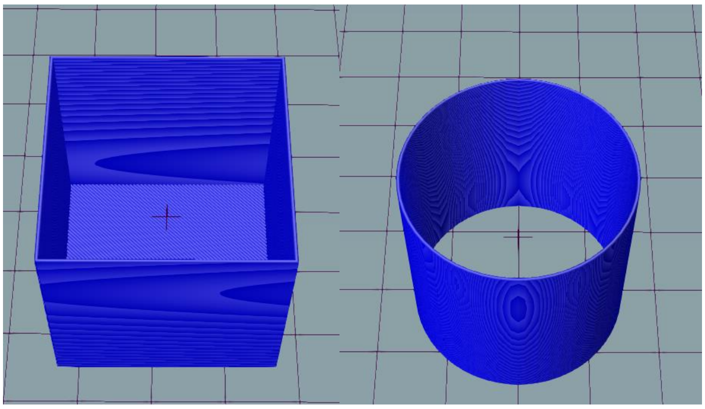

Figure 27. Sliced models of a hollow cub and cylinder for 3D printing

4 RESULTS

4.1 Filament extrusion

Several trial tests were run but optimal conditions for the rHDPE extrusion process were not found. The outcome of rHDPE filament was satisfactory. Pristine HDPE filament was better than satisfactory. The main problem during rHDPE extrusion was the material flow stoppages. In the middle of the extrusion, extrudate stopped coming out of the die that caused rHDPE filament to thin out and eventually to break. Pristine HDPE extrusion went without any complications. The pristine HDPE filament surface did not contain foreign particles while rHDPE was often peppered with foreign debris, see Figure 28.

Figure 28. Produced filament from rHDPE

4.2 Filament roundness

The measurements of the pristine HDPE and rHDPE filaments are shown in Table 10 and Figure 29. rHDPE filament is oval-shaped and its diameter is irregular across the entire length of the filament. The filament has the average value of side "α" of 1.63 mm and side "β" of 1.68 mm. The pristine HDPE filament is round shaped and uniform along the entire length of the filament with the average value of filament's diameter of 1.76 mm.

Table 10. Diameter values of pristine HDPE and rHDPE filaments

	pristine HDPE		**rHDPE**	
	α	β	α	β
Average ø [mm]	1.78	1.74	1.63	1.68
Median ø [mm]	1.78	1.74	1.65	1.67
Average Deviation ø [mm]	0.03	0.03	0.10	0.11
$\frac{average\ deviation\ ø}{average\ ø}100\%$	1.47	1.60	6.24	6.78
Max ø [mm]	1.86	1.81	1.84	1.90
Min ø [mm]	1.71	1.67	1.36	1.40
Max difference in ø [mm]	0.15	0.14	0.48	0.50
$\frac{\text{max difference in ø}}{\text{average ø}}100\%$	8.42	8.05	29.37	29.79
$\max\lvert\alpha - \beta\rvert$	0.11		0.12	
$\min\lvert\alpha - \beta\rvert$	0.01		0.01	
Total length [m]	50			

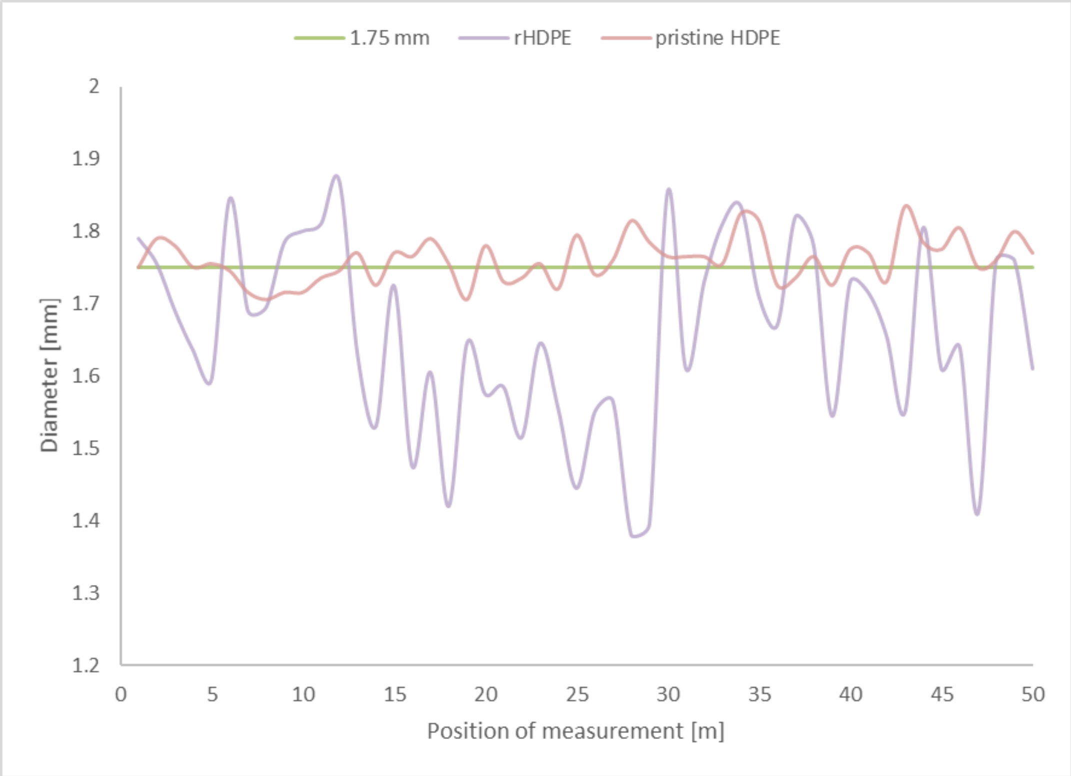

Figure 29. rHDPE and pristine HDPE averaged filament diameter over distance

4.3 Porosity and impurities

The rHDPE filament had a smooth surface with some impurities and bumps across the whole length of the filament. Foreign material contamination of rHDPE filament was overserved with a closer examination of the filament surface under a microscope, see Figure 30. Black specks and spots were found on the rHDPE filament surface. Pristine HDPE filament did not have any impurities and imperfection, see Figure 31.

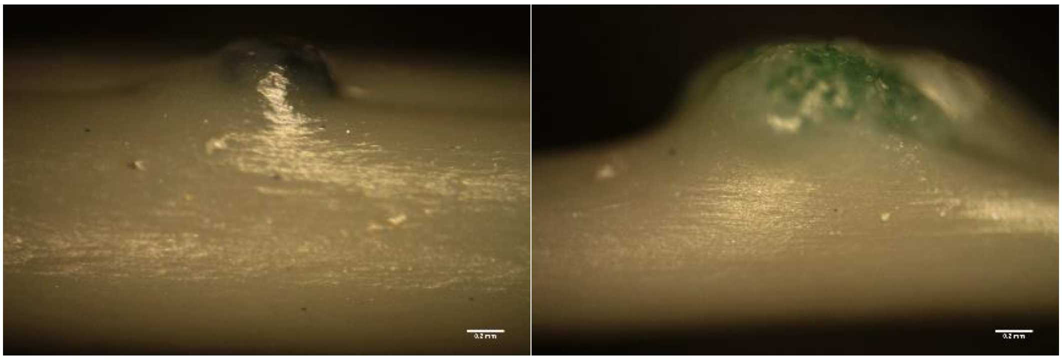

Figure 30. Impurities of rHDPE filament

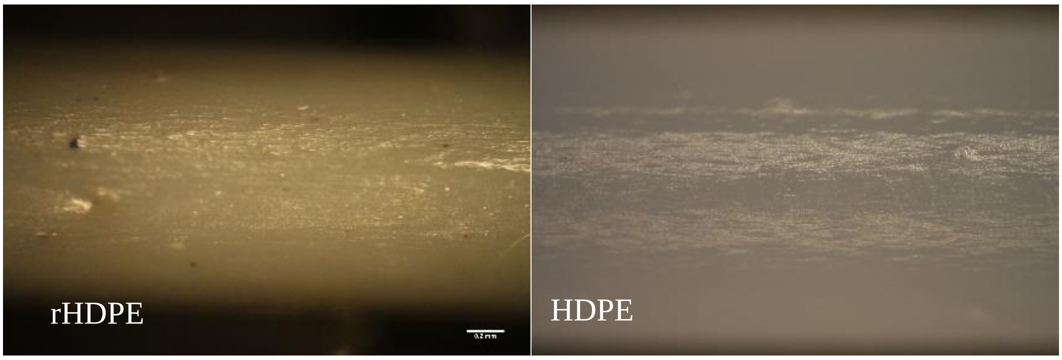

Figure 31. The surface of rHDPE and pristine HDPE filament

Several cross sections of the both filaments were taken and examined under an optical microscope to determine the void content percentage in the filaments. Some features were detected on the cross-sectional area of the rHDPE, see Figure 32, but they cannot be identified as empty spaces due to insufficient sample preparation. Therefore, the void fraction measurement was not performed.

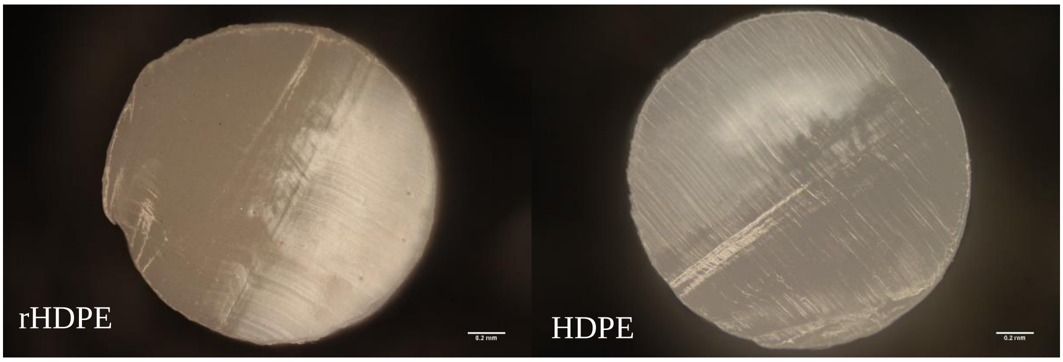

Figure 32. The cross section of rHDPE and pristine HDPE filament

Thermal properties

The melting peak of the sample is 129.22 °C during the first heating and 130.52 °C during the second heating as shown in Figure 33 below. The unidentified foreign material extracted from rHDPE filament has a high degree of crystallinity of 119.2% and 115,3%. Heat capacity of the material does not fluctuate to a large extent throughout the testing. The results differ from the normal HDPE values, though.

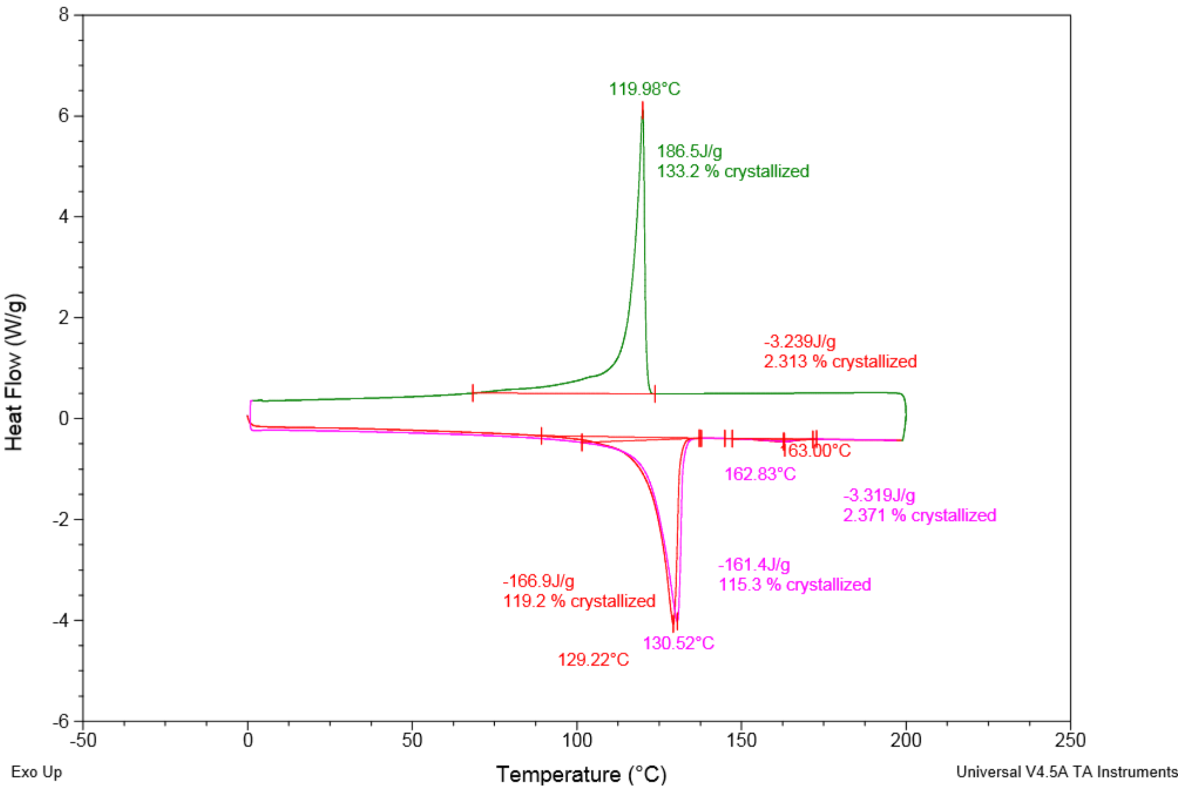

Figure 33. DSC curves for unidentified foreign material extracted from rHDPE filament

4.4 Melt Flow Rate

MFR test results of the rHDPE and pristine HDPE filaments are shown in Table 11.

Table 11. MFR results of the rHDPE and pristine HDPE filaments

Material	MFR [g/10 min]
rHDPE (190°C/2.16 kg)	0.336
Pristine HDPE (190°C/2.16 kg)	0.324

4.5 Tensile testing

Table 12 shows tensile test results of injection moulding of rHDPE and pristine HDPE samples.

Table 12. Tensile test results of injection moulding of rHDPE and pristine HDPE samples

	rHDPE	**pristine HDPE**
Stress at Yield [MPa]	12.92	9.97
Stress at Break [MPa]	10	3.48
Strain at Yield [%]	2.62	0.96
Strain at Break [%]	15.24	16.3
Youngs Modulus [MPa]	755	915.6

Table 13 shows tensile test results of 3D printed rHDPE and pristine HDPE samples

Table 13. Tensile test results of printed rHDPE and pristine HDPE samples

	printed rHDPE	**printed pristine HDPE**
Stress at Yield [MPa]	10.36	7.4
Stress at Break [MPa]	-	9
Strain at Yield [%]	5.50	3
Strain at Break [%]	-	84.6
Youngs Modulus [MPa]	336	385

Three out of five printed tensile samples of pristine HDPE broke at 21-22 mm elongation mark. All five printed tensile samples of rHDPE remained intact up to 35 mm elongation mark, see Figure 34.

Figure 34. Printed tensile specimens of rHDPE and pristine HDPE (from left to right)

4.6 3D printing

The first layers of the rHDPE and HDPE tensile samples managed to stick to the to the PP sheet. But the adhesion problem of the first layers was not completely fixed even with increased temperature of printer's build platform, see Figure 35.

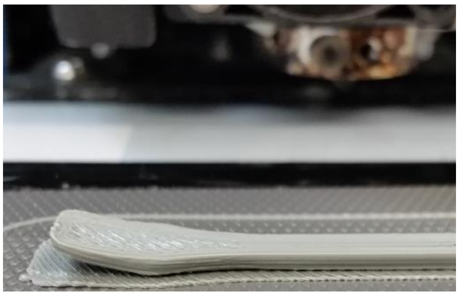

Figure 35. Warping of the rHDPE tensile sample

Printed samples of the cube shrank as they were cooled down to room temperature. The first layers of the cube models curled at the corners as shown in Figure 36.

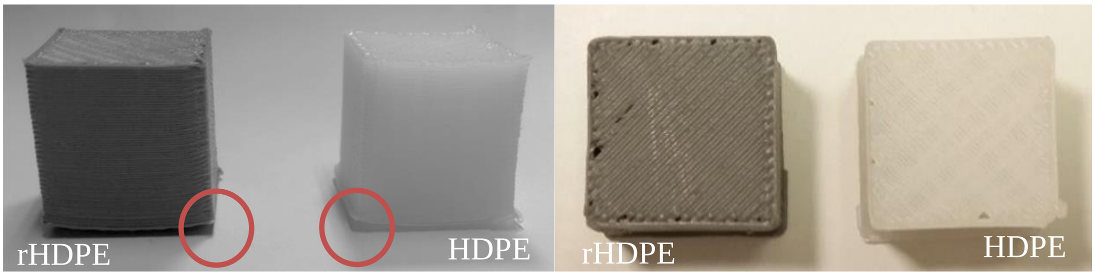

Figure 36. Warping of rHDPE and HDPE cube models

The warping of the thin walls of the hollow cube samples became apparent even during printing process. The hollow cylindrical samples did not show noticeable warp deformation due to its geometrical specification, lack of sharp features, see Figure 37.

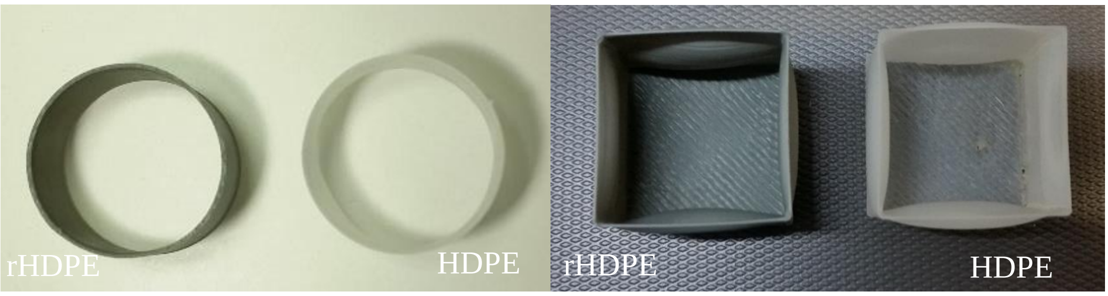

Figure 37. Warping results of the hollow cubes and cylinders samples

The rHDPE layers of the filament effectively bonded to each other as well as pristine HDPE filaments, see Figure 38. All printed samples did show any sign of layers splitting or separations.

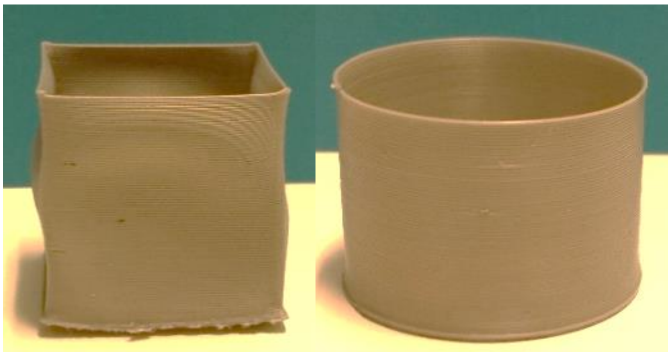

Figure 38. rHDPE printed samples of hollow cube and cylinder

5 DISCUSSION

Extrusion

The material flow stoppage was the main problem of rHDPE extrusion process. The cause of this problem was not found. The change of extrusion temperatures and screw speed during several trial runs did not have any effect on material flow performance. The material flow stoppage could be caused by blockage of die, melting instability or unsuitability of the extrusion screw since rHDPE consists of various blends of HDPE polymers with different molecular weight, molecular weight distribution and chain branching [4].

Filament roundness

rHDPE filament with a diameter of 1.75 mm and a tolerance of ± 0.05 mm was difficult to manufacture. The non-uniformity and inconsistency of diameter of the rHDPE filament might have been caused by the same unknown factors that caused the stoppage. The max diameter value of rHDPE filament was 1.90 mm and the min diameter value of rHDPE filament was 1.36 mm. On the other hand, the average diameter value of pristine HDPE filament was 1.76 mm.

Impurities

Black dots and bumps on the rHDPE filament surface could be pieces of different grades of HDPE material which did not entirely melt during reprocessing. rHDPE was recycled from MSW and the material could be contaminated with other plastics. In that case, the properties of rHDPE filament may not be consistent since small amounts of a different types of plastics may change the properties of the bulk material (HDPE).

MFR

The MFR test showed that there is no difference between raw materials values and processed materials values of rHDPE. The raw rHDPE has MFR of 0,35 g/10min and rHDPE filament MFR is 0.336 g/10min indicating a decrease of 4% in MFR value of provided raw rHDPE. Pristine bulk HDPE has MFR of 0.4g/10min and extruded HDPE filament MFR is 0.324 g/10 min indicating a decrease of 19% in MFR value of provided raw HDPE. rHDPE has significantly higher material viscosity than most common commercial filaments such as ABS and PLA.

Tensile test

The maximum value of stress at yield of pristine HDPE printed sample is 15.6 MPa and the maximum value of stress at yield of rHDPE printed sample is 16.4 MPa. The mean elongation at breaking point for 3D printed samples of pristine HDPE is 27.9 mm with minimum value of 21.6 mm. 3D printed samples of rHDPE did not fracture at 35 mm elongation mark which will be 106% from the total length of the sample. The ductility of 3D printed samples can be explained by molecular orientation of the extruded filament and alignment of the printed layers of the tested parts. Nevertheless, the unidirectional strength of the rHDPE printed samples is higher than in pristine HDPE printed samples. Injection moulded samples of rHDPE and pristine HDPE did not show any signs of stretchiness. The mean elongation at breaking point for rHDPE injection moulded samples is 10,49 mm and the mean elongation at break for pristine printed HDPE samples is 11.23 mm. Overall, rHDPE material did not show significant difference in obtained values compare to pristine HDPE. In some cases, rHDPE performed even better than pristine material.

3D printing

rHDPE and pristine HDPE filaments did not clog the print nozzle nor jam the drive gears of the extruder during printing process. The print times did not exceed 20 min, though. The rHDPE filament diameter irregularity would have caused more problems for an extruder process with a longer running time. The rHDPE and pristine HDPE printed parts show significant warping and first layer adhesion issues, but it was expected. Aside from these issues rHDPE filament does not display any irregularity in printing behaviour.

6 CONCLUSION

This work evaluates the suitability of CIRCO® rHDPE as a feedstock material for FDM printing. The empirical research of this thesis covered the entire manufacturing chain: from filament production to 3D printing. Suitable extrusion parameters for manufacturing rHDPE filament were not found since the uniformity and roundness of the filament along the whole length were not reached.

rHDPE did not show major differences compare to reference HDPE during tensile testing of printed specimens. rHDPE filament values of stress at yield, stress at break and strain at yield were even higher than values of the pristine material. In addition, rHDPE printed samples did not fracture during tensile testing.

The literature research indicates that HDPE undesirability stems from its highly crystalline polymer structure. Large HDPE prints require specific considerations to counter poor first layer adhesion and warpage from differential cooling. rHDPE did not display major disadvantages compared to pristine HDPE during printing. rHDPE filament did not clog the print nozzle nor jam the drive gears of the extruder during printing even though diameter of the rHDPE filament was not consistent and was oval-round shaped along the whole length. In conclusion, rHDPE filament from MSW is quite suitable for 3D printing.

Further research is required to improve the extrusion process. In addition, a heated enclosure system for the 3D printer can be beneficial in controlling the differential shrinkage and warpage of the printed rHDPE parts.

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APPENDIX I

برو ج
Borouge
SHAPING the FUTURE with PLASTICS

Product Data Sheet

Polyethylene

BB2581

High Density Polyethylene for Blow Moulding

DESCRIPTION

BB2581 is a multimodal high density polyethylene intended for blow moulding.

This grade has good stiffness-impact balance and excellent environmental stress cracking resistance (ESCR).

APPLICATIONS

BB2581 is recommended for use in up to 10 liters size cosmetics, personal care, household and industrial chemicals bottles.

SPECIAL FEATURES

The product has the following features:

- Superior Environmental Stress Crack resistance
- Good stiffness-impact balance

PHYSICAL PROPERTIES

Typical properties*	Conditions	Method	Value	Unit
Physical				
Density		ISO 1183	958	kg/m³
Melt Flow Rate (MFR2)	(190°C / 2.16kg)	ISO 1133	0.4	g/10min
Mechanical				
Tensile Modulus	(1mm/min)	ISO 527-2	1300	MPa
Tensile Stress at Yield	(50mm/min)	ISO 527-2	29.0	MPa
Tensile Strain at Yield	(50mm/min)	ISO 527-2	8.0	%
Flexural Modulus	(2mm/min)	ISO 178	1400	MPa
Charpy Notched strength	(23°C, Type 1, Notch A, Edgewise)	ISO 179	12	KJ/m²
Shore Hardness	(Shore D- 3 Sec.)	ISO 868	65	
		ASTM D2240	65	
Thermal				
VICAT Softening Temperature		ISO 306	128	°C
		ASTM D1525	128	°C
Heat Deflection Temperature (0.45MPa)		ISO 75-2	80	°C
Additional Properties				
ESCR	(10% Igepal, F50)	ASTM D1693-B	250	Hours
FNCT	(6.0MPa, 50°C, 2% Arkopal)	ISO 16770	45	Hours

***Data should not be used for specification work.**

PROCESSING TECHNIQUES

BB2581 is easy to extrude and can be used in all conventional blow moulding machines.

Following parameters should be used as guidelines:

Temperature

- Barrel: 170-190°C
- Die: 175-190°C
- Melt temperature: 170-200°C

STORAGE

BB2581 should be stored in dry conditions at temperatures below 50°C and protected from UV-light. Improper storage can initiate degradation, which results in odor generation and color changes and can have negative effects on the physical properties of this product.

More information on storage can be found in Safety Information Sheet (SIS) for this product.

SAFETY

The product is not classified as a dangerous preparation.

Please see our Product Safety information sheet (SIS) for details on various aspects of safety; recovery and disposal of the product, for more information contact your Borouge representative.

RECYCLING

The product is suitable for recycling using modern methods of shredding and cleaning. In-house production waste should be kept clean to facilitate direct recycling.

RELATED DOCUMENTS

The following related documents are available on request, and represent various aspects on the usability, safety, recovery and disposal of the product.

Product safety information sheet (SIS)
Statement on chemicals, regulations and standards
Statement on compliance to food contact regulations

DISCLAIMER

The product(s) mentioned herein are not intended to be used for medical, pharmaceutical or healthcare applications and we do not support their use for such applications.

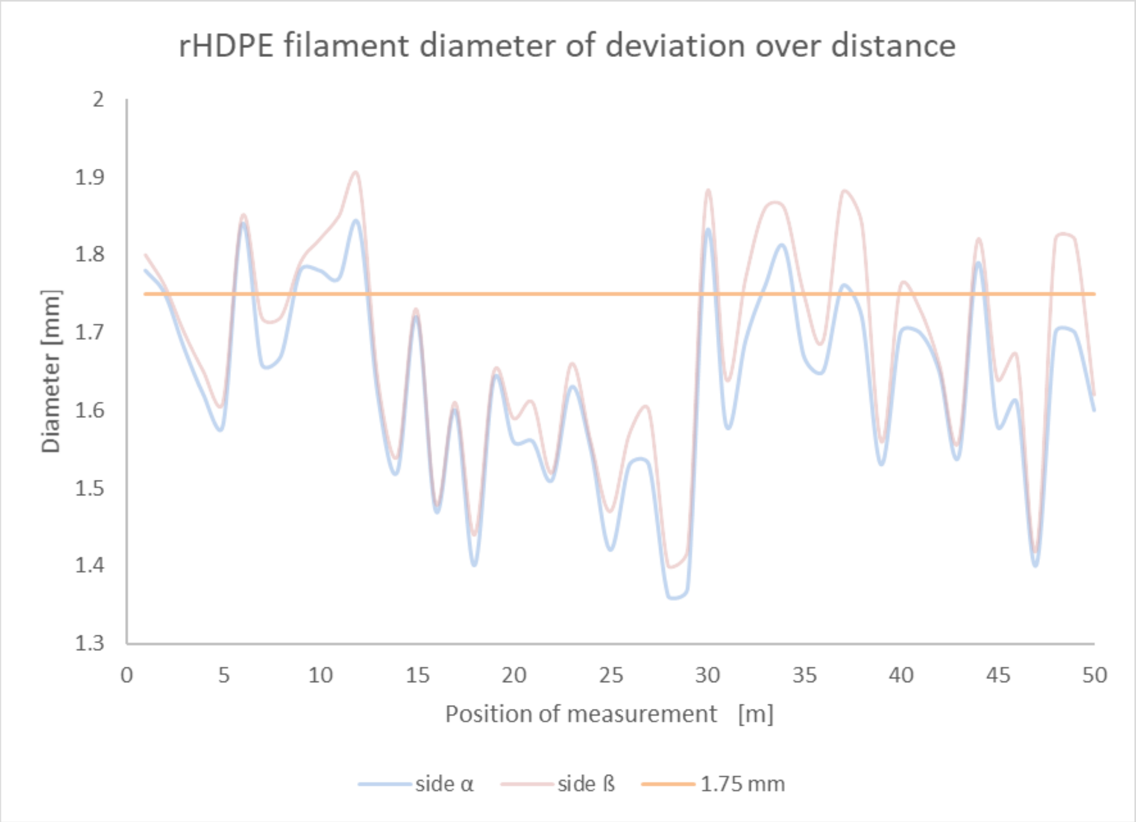
To the best of our knowledge, the information contained herein is accurate and reliable as of the date of publication, however we do not assume any liability whatsoever for the accuracy and completeness of such information.

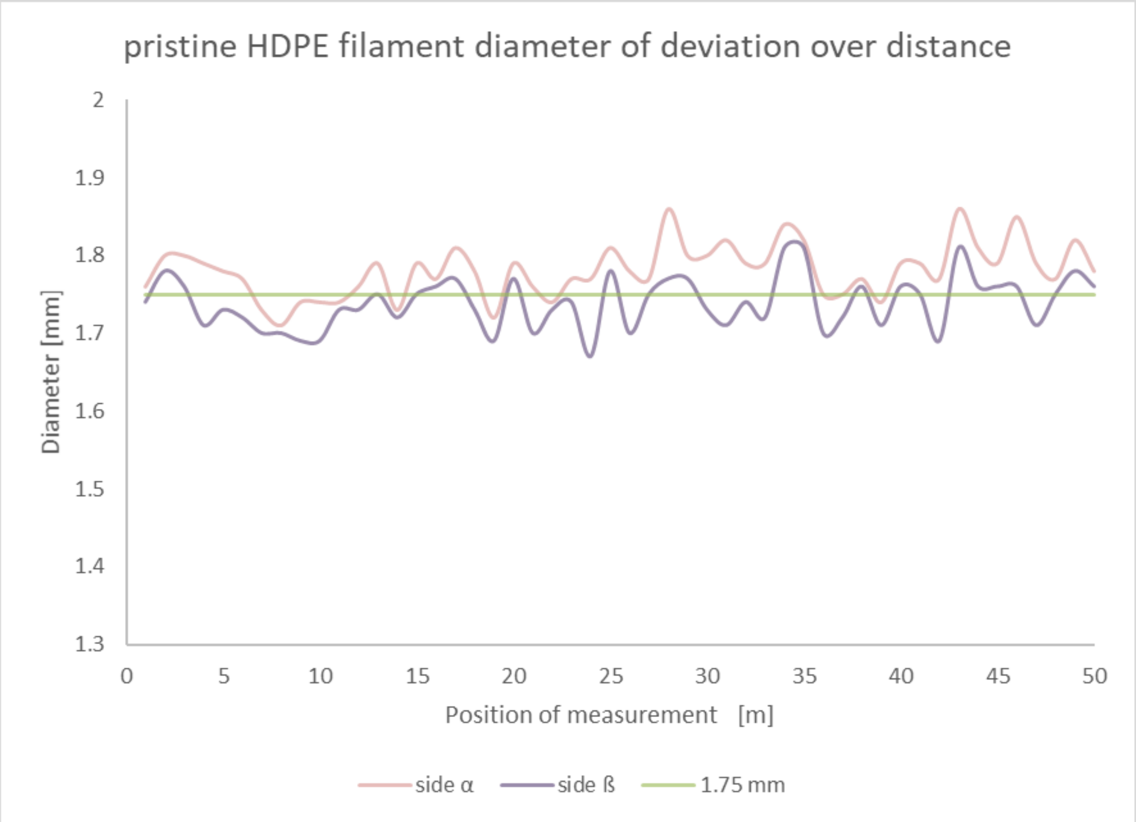
Borouge makes no warranties which extend beyond the description contained herein. Nothing herein shall constitute any warranty of merchantability or fitness for a particular purpose.

It is the customer's responsibility to inspect and test our products in order to satisfy itself as to the suitability of the products for the customer's particular purpose. The customer is responsible for the appropriate, safe and legal use, processing and handling of our products.

No liability can be accepted in respect of the use of Borouge products in conjunction with other materials. The information contained herein relates exclusively to our products when not used in conjunction with any third party materials.

APPENDIX II


rHDPE filament diameter of deviation over distance
2
1.9
1.8
1.7
1.6
1.5
1.4
1.3
Diameter [mm]
0
5
10
15
20
25
30
35
40
45
50
Position of measurement [m]
side α
side ß
1.75 mm


pristine HDPE filament diameter of deviation over distance
2
1.9
1.8
1.7
1.6
1.5
1.4
1.3
Diameter [mm]
0
5
10
15
20
25
30
35
40
45
50
Position of measurement [m]
side α
side ß
1.75 mm

APPENDIX III

Testometric materials testing machines

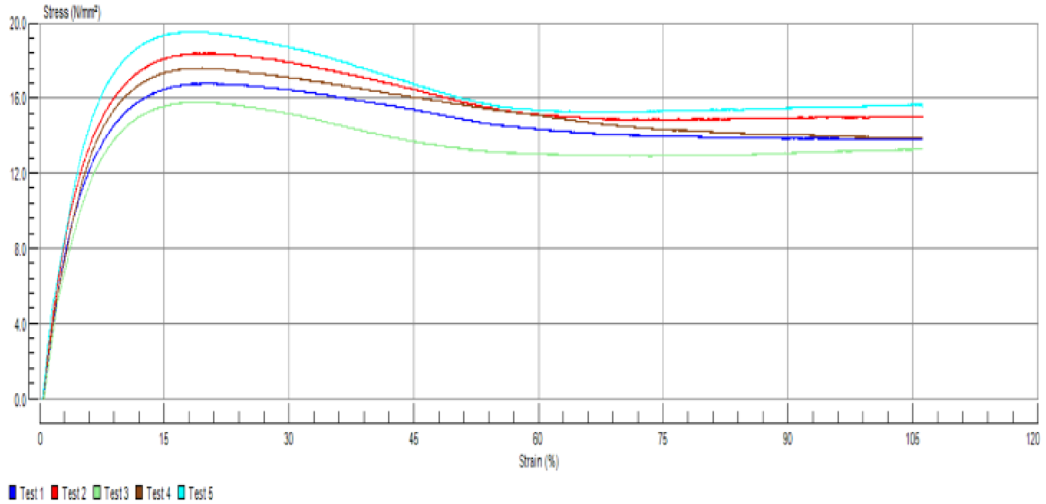
winTest™ Analysis

Group : printed samples
Material : rHDPE

Test Speed : 2.500 mm/min
Pretension : Off
Sample Length : 33.000 mm

Test No	Force @ Yield (N)	Force @ Break (N)	Force @ Peak (N)	Elong. @ Yield (mm)	Elong. @ Break (mm)	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)	Stress @ Break (N/mm²)	Stress @ Peak (N/mm²)	Strain @ Yield (%)
1	46.420	155.190	188.000	0.560	35.034	6.828	4.145	13.856	16.786	1.697
2	157.820	168.000	205.930	2.180	35.054	6.144	14.091	15.000	18.387	6.606
3	133.590	148.500	176.980	2.142	35.041	6.276	11.928	13.259	15.802	6.491
4	183.440	155.440	197.220	3.645	35.032	6.465	16.379	13.879	17.609	11.045
5	59.010	173.890	218.830	0.553	35.038	5.865	5.269	15.526	19.538	1.676
Min	46.420	148.500	176.980	0.553	35.032	5.865	4.145	13.259	15.802	1.676
Mean	116.056	160.204	197.392	1.816	35.040	6.316	10.362	14.304	17.624	5.503
Max	183.440	173.890	218.830	3.645	35.054	6.828	16.379	15.526	19.538	11.045
S.D.	60.613	10.403	16.104	1.300	0.009	0.360	5.412	0.929	1.438	3.938
C. of V.	52.227	6.494	8.158	71.569	0.025	5.703	52.227	6.494	8.158	71.569
L.C.L.	40.796	147.287	177.396	0.202	35.029	5.868	3.643	13.151	15.839	0.613
U.C.L.	191.316	173.121	217.388	3.430	35.051	6.763	17.082	15.457	19.410	10.393

Test No	Strain @ Break (%)	Strain @ Peak (%)	Youngs Modulus (N/mm²)
1	106.164	20.691	317.520
2	106.224	18.618	346.125
3	106.185	19.018	329.313
4	106.158	19.591	330.895
5	106.176	17.773	355.879
Min	106.158	17.773	317.520
Mean	106.181	19.138	335.946
Max	106.224	20.691	355.879
S.D.	0.026	1.091	15.083
C. of V.	0.025	5.703	4.490
L.C.L.	106.149	17.783	317.219
U.C.L.	106.214	20.493	354.674



Testometric
materials testing machines

winTest™ Analysis

Group: printed samples
Material: pristine HDPE

Test Speed : 2.500 mm/min
Pretension : Off
Sample Length : 33.000 mm

Test No	Force @ Yield (N)	Force @ Break (N)	Force @ Peak (N)	Elong. @ Yield (mm)	Elong. @ Break (mm)	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)	Stress @ Break (N/mm²)	Stress @ Peak (N/mm²)	Strain @ Yield (%)
1	61.730	54.900	219.710	0.463	22.831	4.342	5.512	4.902	19.617	1.403
2	59.700	176.980	222.400	0.504	35.042	5.754	5.330	15.802	19.857	1.527
3	57.350	173.260	218.420	0.540	35.062	5.556	5.121	15.470	19.502	1.636
4	58.250	72.260	217.820	0.607	25.058	4.857	5.201	6.452	19.448	1.839
5	177.220	27.020	204.530	2.920	21.605	5.825	15.823	2.413	18.262	8.848
Min	57.350	27.020	204.530	0.463	21.605	4.342	5.121	2.413	18.262	1.403
Mean	82.850	100.884	216.576	1.007	27.920	5.267	7.397	9.007	19.337	3.051
Max	177.220	176.980	222.400	2.920	35.062	5.825	15.823	15.802	19.857	8.848
S.D.	52.780	69.675	6.961	1.071	6.628	0.643	4.713	6.221	0.621	3.245
C. of V.	63.706	69.065	3.214	106.359	23.738	12.216	63.706	69.065	3.214	106.359
L.C.L.	17.316	14.372	207.934	-0.323	19.691	4.468	1.546	1.283	18.565	-0.978
U.C.L.	148.384	187.396	225.218	2.336	36.149	6.066	13.249	16.732	20.109	7.080

Test No	Strain @ Break (%)	Strain @ Peak (%)	Youngs Modulus (N/mm²)
1	69.185	13.158	415.288
2	106.188	17.436	438.422
3	106.248	16.836	406.882
4	75.933	14.718	402.570
5	65.470	17.652	266.288
Min	65.470	13.158	266.288
Mean	84.605	15.960	385.890
Max	106.248	17.652	438.422
S.D.	20.084	1.950	68.278
C. of V.	23.738	12.216	17.694
L.C.L.	59.668	13.539	301.114
U.C.L.	109.541	18.381	470.666