Applicability of recycled HDPE for Rotational Molding

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Abstract:

This thesis work is focused on studying use recycled HDPE instead of raw HDPE in rotational molding process in order to create significant environmental and economic benefits. The study examines the difference between properties of recycled HDPE and ref. HDPE. Also, it defines the difference between melt flow index of ref.HDPE and powder made of that resin. For the practical part of this work items made of HDPE were sorted by the method of manufacturing, by the type of item and by the usage time. Items were recycled mechanically and dumbbell samples were produced by injection molding. Then mechanical properties were assessed from tensile tests and melt flow index was evaluated by melt flow index test. As a result, recycled materials initially manufactured by extrusion blow molding can't be used in rotational molding because MFI of these materials is too low for rotational molding. However, recycled materials that were manufactured by rotational molding and injection molding have enough value of MFI for rotational molding.

Keywords:	Rotational molding, HDPE, Recycling, rHDPE, MFI, Tensile Testing
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CONTENTS

Figures

Tables

Abbreviations

HDPE	High Density Polyethylene
MFI	Melt Flow Index
PE	Polyethylene
rHDPE	Recycled High Density Polyethylene

FOREWORD

I would like to express my sincere gratitude to my supervisor Valeria Poliakova for her support, expert guidance and inspiration throughout my research.

I wish to thank my examiner Mirja Andersson for counselling when I decided to start my thesis.

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I would like to acknowledge the valuable input to this research of company Motoral Oy Motoplast, which gave me HDPE powder, granules and rotomolded item for further testing.

Helsinki, April 2016

Maria Dvorak

1 INTRODUCTION

1.1 Background

Rotational molding is a thermoplastic process for producing hollow parts by placing powder or liquid resin into a hollow mold and then rotating it bi-axially in an oven until the resin melts and coats the inside of the mold cavity. Next the tool is cooled and the part is removed from the mold.

Rotational molding industry is developing rapidly since the 1950s especially compared with other types of plastics processing industry. Although, various factors inhibit the development, such as slower processing cycle and limitations of the materials used.

Nowadays approximately 90% of the materials used in rotational molding is polyethylene (Linear Low Density Polyethylene, Medium Density Polyethylene and High Density Polyethylene). HDPE is used widely in rotational molding because it has excellent chemical resistance, very high stiffness, good processability and low cost. HDPE is non-biodegradable and can take centuries to decompose, so it is imperative that products made of HDPE are recycled and used again. Moreover, HDPE can be easily recycled.

Recycling is important for many reasons and it is a part of global efforts to reduce plastic in the waste stream. It helps to reduce the high rates of plastic pollution and to conserve natural resources, specifically oil, which is a nonrenewable natural resource available only in limited supply. Also, the process of recycling plastic requires less energy and fossil fuels, it results in fewer greenhouse gas emissions including carbon dioxide, which contributes significantly to the global warming effect. Recycling plastic helps to conserve limited landfill space that can be used for other waste.

Moreover, it is forbidden to landfill packaging items since 1.1.2016 according to Finnish law for waste (Jätelaki 646/2011). Packaging plastic items are available, and recycling can be done easily.

It is clear from these observations that using recycled HDPE instead of raw HDPE can create significant environmental and economic benefits. Therefore, this work focuses on finding a way to use rHDPE instead of raw HDPE in rotational molding.

1.2 Aims

The main aim of this work is to find out applicability of recycled HDPE for rotational molding. It will be discussed after approaching three main objectives of this work:

- Define a requirements of the materials for rotational molding.
- Compare MFI and mechanical properties of recycled HDPE with properties of ref. HDPE.
- Find the most suitable rHDPE for further production of powder.

2 LITERATURE REVIEW

2.1 Rotational Molding

2.1.1 Overview of Rotational Molding

Rotational molding, also known as rotomoulding or rotocasting, is a low pressure, high temperature manufacturing method for producing hollow, one-piece plastic parts. The basic principle of forming a coating on the inside surface of a rotating mold dates back for many centuries, but the process did not gain recognition as a molding method for plastics until the 1940s. In the 1950s the use of the rotational molding process expanded more quickly due to the introduction of powdered grades of polyethylene specifically developed for the process.

There are many advantages associated with the rotational molding process. Firstly the molds are simple and relatively cheap. This is because rotational molding is a low-pressure process and therefore it is not necessary to manufacture the molds from expensive metal alloys, as in the injection molding process. The wall thickness of parts produced by rotational molding is more uniform in comparison to products from other processes and it is possible to alter the wall thickness of the part without altering the mold. Complex parts with undercuts and intricate contours can be manufactured relatively easily by rotational molding. It is also possible to produce double wall moldings. During rotational molding relatively little waste is produced since the required weight of the part is placed inside the mold.

One of the main disadvantages associated with rotational molding is that the number of materials suitable for the process is more limited than for other plastics manufacturing processes. Also, the cycle times are much longer than those of other processes because both the plastic and mold must be heated from room temperature to the molding temperature for the plastic and then subsequently cooled to room temperature during each cycle.

Common applications for rotomoulded products include:

- Material handling products- tanks, chemical drums, shipping containers, wheeled bins, hoppers and coal bunkers.
- Industrial products- pump housings, pipe fittings, effluent ducts, air ducts, sewer linings, safety helmets, paramedic stretchers and light fittings.
- Automotive products -truck mudguards, ducting, diesel fuel tanks, toolboxes and tractor dashboards.
- Environmental products- litter bins, sanitation bins and bottle banks.
- Leisure products- canoes, kayaks, windsurfing boards, boats, trailers, toys, playground furniture and mannequins.
- Marine products- floats, buoys, life belts and floating decks.
- Road signage- road barriers, road cones and road signs. (Crawford and Kearns, 2012)

2.1.2 Rotational Molding Process

The plastic powder is placed in one half of the mold portion. The mold is then closed and subjected to biaxial rotation in an oven with required processing temperature. The plastic powder inside the mold is melted by heat transferred through the mold wall. After all of the powder has melted, the mold is moved out of the oven, while biaxial rotation continues. Still air, a blowing fan, or a water shower is usually used to cool the mold. Once the product inside the mold is cooled to a state of sufficient rigidity, the mold opens and the product is removed.

During the tumbling process, the finer powder particles get sieved down closer to the wall and larger particles form layers on top. As the temperature rises in the mold, the powder softens and melts, adhering to the mold wall and forming a homogenous, bubble free melt pool along the entire inside surface of the mold. Biaxial rotation ensures that the powder is evenly distributed in the mold. After the heating cycle is completed, the mold is cooled resulting in solidification of the polymer. The amount of powder determines the wall thickness of the rotomolded part.

Rotational molding process involves four steps. They are:

1. In the separable cast or fabricated vented mold, pre-determined and weighed amount of powdered plastic material is charged;
2. The powdered material is heated in an oven with biaxial rotation and external heating without applying pressure or centrifugal force until powdered plastic melts and coats in the internal surface of the mold;
3. After all of the powder has melted, the mold is moved out of the oven, while biaxial rotation continues. Rotating mold is cooled externally with forced air or water mist to allow the molding to solidify;
4. Removing the part from the mold's cavity. (Subramanian, 2011)

Figure 1 illustrates the principle of rotational molding of plastics.

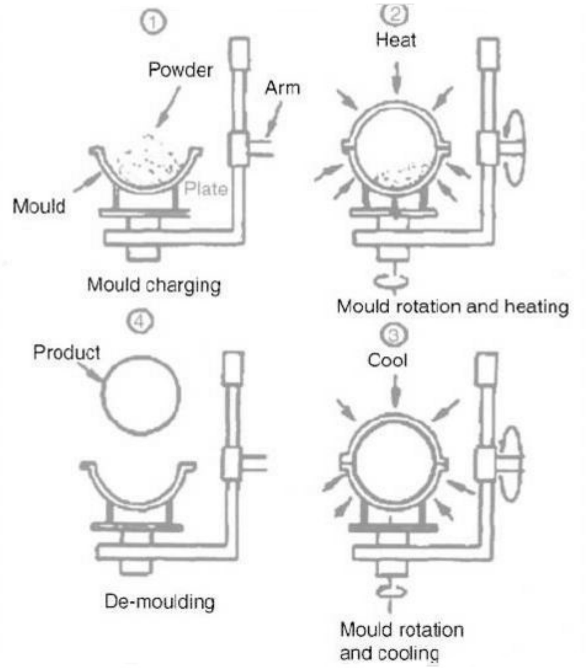


Figure 1. Principles of rotational molding of plastics (Subramanian, 2011)

2.2 Materials for Rotational Molding

Currently polyethylene represents 90% of all polymers that are rotationally molded. The most obvious reasons for this are the ability of PE to withstand the long time-temperature environment of the process, and it is relatively low cost. Polyethylene is generally resistant to water, vegetable oils, alkalis and most concentrated acids at room temperature. With the proper precautions it can be re-ground and re-molded. PE is compatible with a full range of pigments to produce colored parts. It can be readily pulverized into a free-flowing powder at room temperature. Moreover, PE is an easy flow material when it is in the melt form.

PVC plastisols are also used, and other materials such as polycarbonate, nylon, polypropylene, unsaturated polyesters, ABS, acetal, acrylics, cellulosics, epoxies, fluorocarbons, phenolics, polybutylenes, polystyrenes, polyurethanes and silicones account for less than 3% of the market.

The powder form of polymers is used in rotational molding process. Some materials, such as plastisols, can be used as liquids, others, such as nylons, can be used as granules due to their high flowability once molten. (Crawford and Kearns, 2003)

2.2.1 High Density Polyethylene

Polyethylene (PE) was discovered in 1933 by Reginald Gibson and Eric Fawcett at the British industrial giant, Imperial Chemical Industries. This widely used plastic is a polymer of ethylene, CH2 =CH2, having the formula (–CH2 CH 2–) n. It is produced at high pressures and temperatures in the presence of any one of several catalysts, depending on the desired properties of the end-use product.

HDPE is more rigid and harder than lower density materials with a molecular weight below 300,000 g/mol. The extremely high molecular weight of HDPE combined with its very low coefficient of friction produces an excellent abrasion-resistant product which is resistant to gouging, scuffing, and scraping.

It is also more prone to warpage due to its higher crystallinity, which makes it very sensitive to differential cooling rates across the walls of rotomoulded products. HDPE also has higher shrinkage than LDPE. HDPE is also non-toxic and non-staining.

Moreover, HDPE is a low cost material with an excellent balance between stiffness and toughness, over a wide temperature range. In its natural state it is a translucent, milky white material. This can be a drawback in applications where absolute transparency is important. It is easily pigmented and thus is available in a wide range of colors.

HDPE can be processed very easily by injection molding, extrusion, blow molding, rotational molding, and so on.

Table 1 shows a common engineering properties of HDPE. (Vasile and Pascu, 2005)

Table 1. Common engineering properties of HDPE (Vasile and Pascu, 2005)

Property	**Value**
Density (g/cm^3)	0.941-0.965
Tensile Strength (MPa)	20-35
Tensile Modulus (MPa)	413-1241
Flexural modulus (GPa)	0.75-1.575
Strain at Yield (%)	15
Melting temperature (˚C)	120-130
Max. operating temperature (°C)	82
Specific heat (kJ/kg/ K at 25 °C)	2.22– 2.3
Thermal conductivity (W/m/°C)	$4.63– 5.22 \times 10^{-3}$
Cristallinity (%)	60-90

2.2.2 Grinding

Powder is produced by grinding or pulverization, sometimes also called attrition. The basic stages in the grinding of polymers for rotational molding are illustrated in Figure 2. Pellets are fed into the throat of the mill from a feed hopper by means of a vibratory feeder (or auger) at a uniform and controlled rate. As these pellets enter the mill, along with a flow of air, they pass between two metal cutting plates, each with a series of radial cutting teeth.

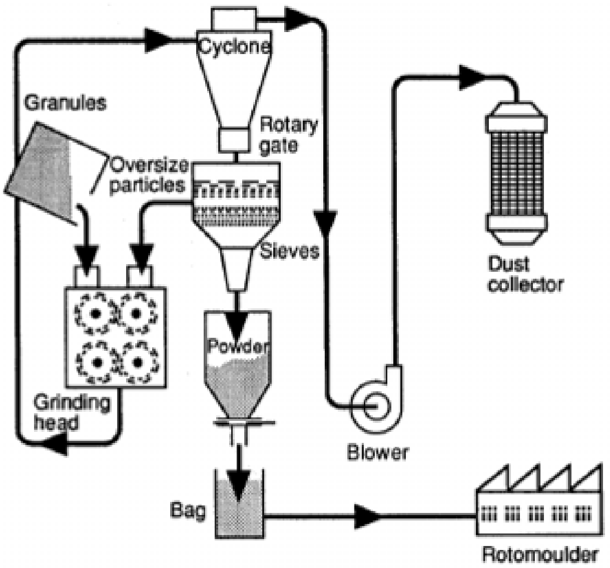


Figure 2. Stages in the grinding of powders (Crawford and Kearns, 2003)

Each pellet is slowly reduced in size as particles are shaved off it and it moves outwards into the narrowing gap between the two cutting faces. The particles remain between the plates until they are of a size that allows them to escape from the gap at the periphery.

In the grinding process, frictional heat increases the temperature of the metal cutting faces, as well as the individual polyethylene particles and the surrounding air. As a consequence, the temperature must be controlled so that it does not rise beyond the melting point of the polyethylene or to a critical softening temperature, prior to melting, when the particles begin to adhere to each other. This can cause blockages in the passage of new material entering the mill.

Once the particles exit the mill they go into an air stream, which carries them to a screening unit containing a number of sieves of a standard mesh size. Particles that pass through the screens are taken out of the system and collected as usable powder. Those particles that do not pass through are conveyed back to the mill and reground.

There are factors affecting powder quality: gap between the discs, feed rate of granules, system pressure, disc design, disc speed, choice and type of feeder, cooling efficiency, operating temperature, moisture control, air velocity, amount of recycle, type of auxiliary equipment used, amperage of the mill and sieve aperture in the screen unit. (Crawford and Kearns, 2003)

2.2.3 Requirements of the materials

Due to the importance of grindability, particle size distribution, particle shape, pourability, bulk density, thermal stability, MFI and shear viscosity to successful rotational molding, these aspects are considered in detail in the following sections.

Grindability

The grindability of a material means that the resin can be ground to a fine powder. Resin grades that have very low melting points may not be easy to grind in the high-speed impact mills that have proven to give the most consistently good powder because they melt. In some cases a low melting resin can be ground under an atmosphere of liquid nitrogen or some other cooling method so that the material will not become too hot during melting operation. (Strong, 2005)

Particle shape

Particle shape will have major effects on heat transfer and flow characteristics of the powder mass. The particle should be ovoid in side projection but rectangular or square, with generous radii, in end projection. Spherical particles should be avoided since their packing density is low and the particle-to-particle contact is point-like rather than areal. Acicular particles should also be avoided due to excessive porosity and bridging in the formed part. Defects such as tails and distorted particles can be indications of a powder with poor mouldability.

For rotational molding grade polymers, the particle sizes are easily seen and photographed through 30× magnifiers using Scanning Electron Microscope.

Figure 3 shows a good particle shape for rotational molding powders. (Crawford and Throne, 2002)

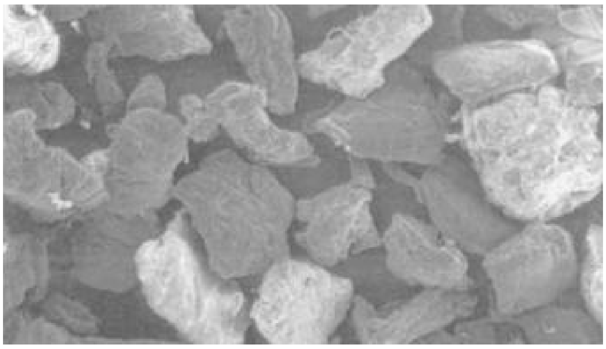


Figure 3. Good particle shapes for rotational molding powders (Crawford and Throne, 2002)

Particle size distribution

High-speed attrition mills grind pellets approximately 5-6 mm in diameter down to the required particle size-distribution. The mesh size is a measure of the size of the screen mesh through which 95% of the particles will pass. The meshes are defined by standards adopted by each country. (Strong, 2005)

While particles are still characterized widely by mesh size, the current trend is to characterize them by the size of the opening in millimeters. Normally powder particles for rotational molding vary from less than 150 microns to about 500 microns (35 mesh) which correspond to hole openings of 0,15 mm to 0,5 mm. It affords a compromise between grinding rates and the fusion characteristics of the polymer. For successful rotational molding particle size distribution should be 95% < 500 micron with maximum 15% < 150 micron. (Nugent, 1990)

The particle size distribution of rotational molding powders is measured according to ASTM test method D-1921. A set of nested, stacked, welded wire sieves, with mesh sizes ranging from about 35 mesh to 200 mesh is used for this determination, which correspond to hole openings of 0,5 mm to 0,074 mm. Basically a thief of powder is taken, weighed, and placed in the top sieve of the sieve stack. The shaker is covered and mounted in a device that rotates, shakes, and vibrates. After a predetermined period of time, the sieves are separated and the amount of powder retained on each sieve is weighed.

Although vibratory sieves of the type described above are the most commonly used in the rotational molding industry, there are other ways of measuring particle size distribution: elutriation, streaming, sedimentation and fluidization. (Crawford and Throne, 2002)

Figure 4 shows a typical particle size distribution for polyethylene used successfully by rotomolders. This particle size distribution skewed towards the larger particles below 500 microns produce moldings of good quality. (Crawford and Kearns, 2003)

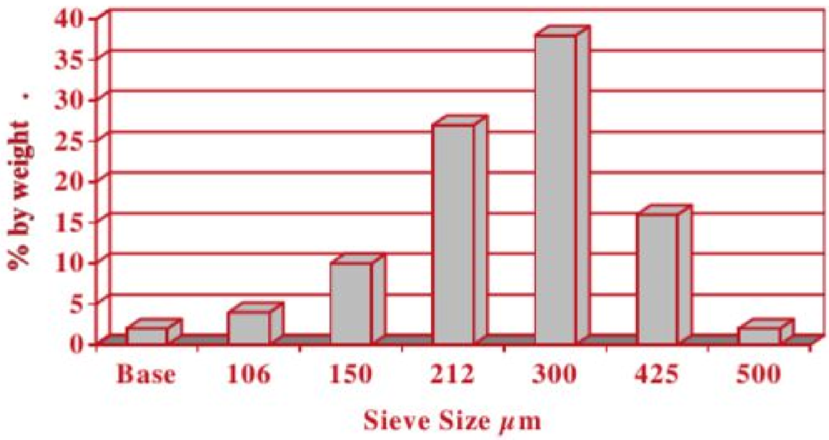


Figure 4. Typical particle size distributions for polyethylene (Crawford and Kearns, 2003)

Pourability

Powder dry flow properties are important during rotational molding as they determine how the polymer distributes itself within the mold and how well the polymer melt flows into complex shapes. Dry flow depends mainly on particle size and particle shape. Since the particle size distribution of a 35 mesh powder tends not to vary greatly, it is the particle shape that has the greatest effect on dry flow. The presence of tails on powder particles reduces dry flow properties, leading to detrimental part properties such as bridging across narrow recesses in the mold and high void content within the part wall.

The standard method for measuring the dry flow of a powder is described in ASTM D-1895. It is the time taken for 100 g of powder to flow through a standard funnel. The dry flow is quoted in seconds.

When the funnel that is defined by ASTM Test Procedure 1895-69 is used, a minimum flow rate of 185 g/min characterizes acceptable rotational molding powders. (Crawford and Throne, 2002)

Bulk density

Bulk density is a measure of the efficiency with which the powder particles pack together. A good quality powder having "clean" particles with no tails will have a high bulk density. Bulk density and dry flow are dependent on the particle shape, particle size, and particle size distribution of the powder. These two properties are inversely related, in that an increase in the bulk density corresponds to a faster dry flow rate, as shown in Figure 5. Bulk density should be more than 320 kg/m3 for successful rotational molding. (Crawford and Throne, 2002)

Bulk density (kg/m^3) = Dry weight (kg) / volume (m^3)

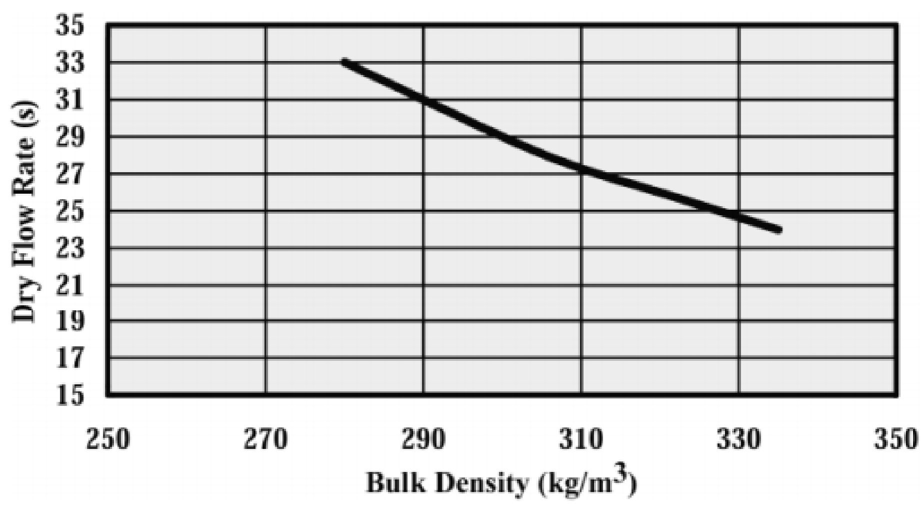


Figure 5. Variation of dry flow rate with bulk density for rotomolding powders (Crawford and Throne, 2002)

Thermal stability

Good heat stabilization is needed to survive prolonged exposure to heat in an oxidative atmosphere. (Strong, 2005)

Melt Flow Index

MFI test is a method of determining and comparing the flow of melts under standard conditions. A vertical load is applied to a piston and the polymer melt is squeezed through a die. The amount of polymer that is extruded in a fixed time gives a measure of the flow as a MFI. An ‘easy flow’ grade of plastic will have a high MFI, which corresponds to a low viscosity.

Different MFI values are preferred for different plastics manufacturing methods. The low MFI material is useful in manufacturing method where the strength of the resin in the melt phase is important, such as in blow molding. High MFI indicates a low molecular weight resin which is useful in achieving high throughputs in manufacturing method, such as injection molding. (Subramanian, 2013)

In rotational molding void-free parts are usually achieved with HDPE melt indexes in the range of 3 to 8 g/10 min (Crawford and Throne, 2002). Resins with high melt index numbers are chosen when the part is very complex and good flow into complicated areas is required (Strong, 2005). However, a higher MFI is associated with lower impact strength, stress crack resistance, chemical resistance and weatherability. These effects of increasing of MFI on properties of PE are summarized in Table 2 (Crawford and Kearns, 2003).

Resins with low melt index numbers are chosen when improved stress crack resistance, impact toughness, or creep resistance is needed. From a physical performance viewpoint, the low-melt index resins would always be preferred, but they are difficult to mold and cannot be used for some parts. (Strong, 2005)

Table 2. Property changes of PE with increasing melt index (Crawford and Kearns, 2012)

Property	Change
Barrier properties	No trend
Bulk viscosity	Decreasing
Chemical resistance	Decreasing
Creep resistance	No trend
Ductility	Decreasing
Ease of flow	Increasing
Environmental stress cracking resistance (ESCR)	Decreasing
Flexural modulus	Decreasing
Hardness	No trend
Impact strength	Decreasing
Molecular weight	Decreasing
Stiffness	No trend
Tensile strength	Decreasing
Weatherability	Decreasing

On the other hand, in the MFI test the shear rates of the melt are considerably higher than are experienced during rotational molding. As a result, it is possible to have 2 materials that have the same MFI but behave differently during rotational molding. (Crawford and Throne, 2002) Nevertheless MFI test is widely used in the industry to rank materials in terms of flowing too easily, or not flowing sufficiently well, for specific rotomoulded parts. (Crawford and Kearns, 2012)

Shear viscosity

Also, in order for a plastic to perform well in rotational molding it should have a low zero shear viscosity. The test to measure this property is more expensive than the MFI test but it represents a much more useful way to rank resins for rotational molding. (Crawford and Throne, 2002)

2.3 Mechanical recycling

Mechanical recycling is the process of converting discarded plastic into new products, principally by melting and molding. The waste plastic used may come from the manufacturing process or from post-consumer products. This is the simplest way of recycling plastic waste, demanding the lowest initial investments.

In this form of recycling, the macromolecular nature of the polymer is not destroyed, so that the degradation reactions that directly affect the physical and chemical properties of the polymer are minimized and controlled. Nevertheless, chemical changes that occurred during the original processing and in-service use may have a negative effect on the quality of products reprocessed by mechanical recycling, in comparison with those manufactured from virgin resin.

Mechanical recycling involves several steps: collection, separation and sorting, shredding, cleaning of the plastic to eliminate organic matter, drying (particularly important for polymers that are hydrolyzed) and reprocessing. These aspects are considered in detail in the following sections.

Collection

Waste collection is the starting point for any recycling process. This stage is often done to gather all kind of plastics into the single place for further processing. There are two main sources in which plastic wastes can pollute the environment: post-consumer plastics and post-industrial plastics. Post-consumer plastics can be easily collected for further recycling in residential areas where people put plastic in waste bins, and also it can be collected from the roadside. Post-industrial plastics can be collected from the industry, for example, it can be plastics waste and defected products.

Separation and sorting

Separation of the different types of plastic and sorting of plastic material from mixed waste are very important stages in mechanical recycling because quite different plastics cannot be used for the same end. If plastic is separated and sorted improperly, it could affect the quality of the produced resin.

Separation and sorting consist of three steps: the identification is made directly by resin identification codes, then the identified product is correlated with the most likely material, and certain properties specific to each material are determined.

Shredding

The stage of breaking into flakes or binding together is mainly done to reduce the overall volume taken up by the plastic residues. Also, it is done to promote the interaction of those residues with the cleaning solution. It normally occurs between separation and cleaning of the plastics.

However, the size of the flakes produced at this stage may interfere with the cleaning efficiency. Also, during extrusion of the plastic, depending on the system used, the size of flakes in the feed can be a hindrance to the feeding process.

Cleaning

Plastics during their use and disposal come into contact with other compounds, and their composition may be changed by contaminants permeating through and impregnating the material. Therefore, cleaning should be done in order to remove contaminants from the material like dust, oil, labels, etc.

The cleaning, in most cases, takes between 5 and 20 minutes at temperatures up to 88 °C. Short times do not suffice to remove adhesives, while the use of baths at high temperatures facilitates the removal of glue. Plastics can be washed with surfactants (detergents) or sodium hydroxide ($Na0H$) solution.

Drying

The drying stage should be done in order to reduce the water content in plastics. The conditions chosen for drying depend on the type of humidity and the way it is bound to the material, on the size and shape of the particles, and on the degree of crystallinity. Usually, it is done using a drying machine at recommended drying temperature of the material.

Reprocessing

After the water content has been reduced, the material proceeds to the reprocessing step, which directly affects the eventual quality of the end-product. At this point, the polymer mixture is formulated in accordance with the target application. Stabilizers, reinforcements, other types of polymer, coupling agents, flame retardants, foaming agents may be added to the polymer.

Extrusion and pelletizing are done in order to obtain a pellets. The flakes are fed into an extruder where they are heated to melting state and forced through the die converting into a continuous polymer product (strand). Then the strands are cooled by water and cut into pellets, which may be used for new polymer products manufacturing. Extrusion is done according to the material data sheet of the specific material. For example, for PE the temperature zones of the extruder have range between 190 - 200°C. (Manrich and S.F. Santos, 2009)

3 METHODS

3.1 Materials and equipment for further recycling and testing

3.1.1 Materials

In the experiment were used items made of HDPE that were sorted into different categories: by the method of manufacturing, by the type of item and by the usage time.

Items that were manufactured by injection molding, extrusion blow molding and rotational molding were chosen because materials manufactured by different manufacturing methods have different MFI values. Also, packaging and non-packaging items were chosen according to the information of Finnish laws about waste (Jätelaki 646/2011, Valtioneuvoston asetus pakkauksista ja pakkausjätteistä 518/2014).

Table 3 shows a classification of used materials in the experiment.

Table 3. Classification of used materials in the experiment

Number	Material	Manufacturing Method	Type	Items
1	HDPE	Injection Molding	Non-packaging	Bucket, sledges
2	HDPE	Injection Molding	Packaging	Pallet
3	HDPE	Extrusion Blow molding	Non-packaging	Watering pots
4	HDPE	Extrusion Blow molding	Packaging	New canisters
5	HDPE	Extrusion Blow Molding	Packaging	Old Canister
6	HDPE	Extrusion Blow Molding	Packaging	Bottles
7	HDPE	Rotational Molding	Non-packaging	Panel
8	Ref. HDPE granules	-	-	Pellets
9	HDPE Powder	.	-	Powder

Table 4 shows description of each material used in the research.

Table 4. Data about materials that were used

Item	Picture	Method of Manufacturing	Manufacturers	Size	Colour
Bucket and sledges		Injection Molding	Bucket- Orthex, sledges- unknown manufacturer	Bucket- diameter 56 cm, height 41 cm, sledges- 31×37 cm	Green and red
Pallet		Injection Molding	Satamuovi	30×43 cm, height 22 cm	Blue
Watering pots		Extrusion Blow Molding	Plastex	47×34 cm	Green
New canisters		Extrusion Blow Molding	Europak, Greiner, Tehnoplast, Promens	19×25 cm	White
Old canister		Extrusion Blow Molding	Unknown manufacturer, time of manufacturing: September 1995	34×34 cm, height 70 cm	White, in some places painted into rust color

Bottles		Extrusion Blow Molding	Foxtel, Henkel, Cederroth, Proctel & Gamble	Different sizes, but not more than 26 cm in height	Different colours
Panel		Rotational Molding	Motoplast	56×23 cm	Dark brown
Ref. HDPE Pellets (Information about HDPE Resin DOWLEX™ 2631UE can be found in Appendix 1.)		-	-	5 mm	White
HDPE Powder		Grinding	-	Particle size distribution of powder produced from ref. HDPE Pellets can be found in Appendix 2.	White

3.1.2 Equipment

Following laboratory equipments were used:

- Rapid Shredder- to produce flakes.

- Nova BS 400 Heavy duty band saw- to cut pieces of plastic.
- Labotek Flexible Modular Drying Unit FMD-MM-25-40-v- to dry flakes.
- KFM Eco Ex Extruder with a screw L/D ratio of 25/D and a screw diameter of 18 mm- to produce pellets.
- ENGEL ES 200/50HL CC90 Injection molding machine- to produce dumbbell samples.
- Testometric M 350- 5CT Material testing machine with wedge grips- to test mechanical properties.
- Mitaten MEP 2/PC Extrusion plastometer- to test MFI.

3.2 Preparation of samples for testing

Pelletizing from post-consumer materials included collection, sorting, washing, drying, cutting, shredding, extrusion and pelletizing. Then dumbbell samples were produced using ENGEL Injection molding machine.

3.2.1 Recycling process

Collection

Plastic item made of HDPE has a resin identification code "2" on its surface or label (see Fig. 6). In that way post-consumer HDPE items were collected. Most of these collected materials were from domestic products such as used different containers, cleaning agent bottles and canisters.

Figure 6. HDPE identification code

Sorting

The collected plastics were sorted and separated into different categories: by the method of manufacturing, by the type of item and by the usage time.

Rotational molding, injection molding and extrusion blow molding processes were involved in research. A clear indication as to whether an object has been injection molded were signs of feed points and ejector pin marks. Also, evidence of the extrusion blow molding process was a thin rim running vertically around the surface of the object left by the split mold (see Fig. 7).

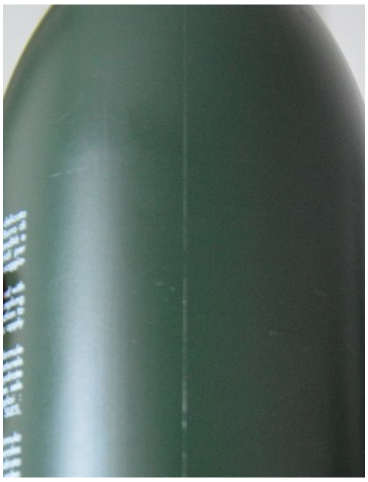


Figure 7. Thin rim left by the split mold

Packaging and Non-packaging items were separated according to the information of Finnish laws about waste (Jätelaki 646/2011, Valtioneuvoston asetus pakkauksista ja pakkausjätteistä 518/2014).

Cutting

Sizes of plastic items were reduced approximately to 10×10 cm using Nova Heavy duty band saw in order to wash large items and enable the fitting in the shredder.

Washing

Water was heated once to 60˚C and then gradually cooled during washing time. The plastic was soaked in this water for approximately 5 hours in order to remove the labels and the glue while washing at the same time (see Fig. 8). Then labels and glue were removed manually.

Figure 8. Washing process

Glue of some canisters was impossible to remove, so this area of canisters was cut by Nova Heavy duty band saw. Also, for cleaning items such as watering pots and old canister was used cleaning agent Abnet (information about cleaning agent Abnet can be found in Appendix 3).

Drying

Washed HDPE parts of items were dried in ambient air for 24 hours on a surface of table as shown on Fig. 9.

Figure 9. Drying process

Shredding

Then flakes from plastic parts were produced using Rapid shredder (see Fig.10). Shredder works by throwing the plastic through a hopper into the shredder that cuts the plastic into flakes. The flakes are collected through a 5 mm filter located at the bottom of the shredder to get an approximate uniform flake size.

Figure 11 shows a flakes of bottles that will be used in further extrusion process.

Figure 10. Rapid shredder

Figure 11. Flakes of bottles

Extrusion

The flakes were fed into a KFM Eco Ex Extruder where they were heated to melting state and forced through the die converting into a continuous polymer product (strand) as shown in Fig. 12.

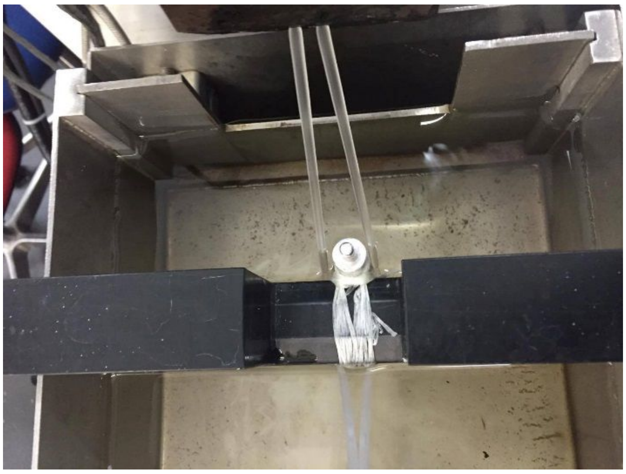


Figure 12. Continuous polymer product (strand)

The materials behaved differently during extrusion process, so temperature profiles from die to hopper also differed. The temperature range was 185- 215 ˚C.

Pelletizing

The strands were cooled by water and cut into pellets of size 3 mm, which will be used for dumbbell samples manufacturing (see Fig.13).

Figure 13. Pellets of recycled watering pots

In the case of watering pots, bucket and sledges, some pellets were with holes inside. So flakes were once again dried in Labotek Flexible Modular Drying Unit at 80 ˚C for 120 minutes to avoid extra moisture, but subsequently, it had no effect on holes in pellets.

Also, it was impossible to do an extrusion process of recycled pallet, because this material was too viscous and liquid (see Fig. 14). A small amount of pellets was produced just for MFI test.

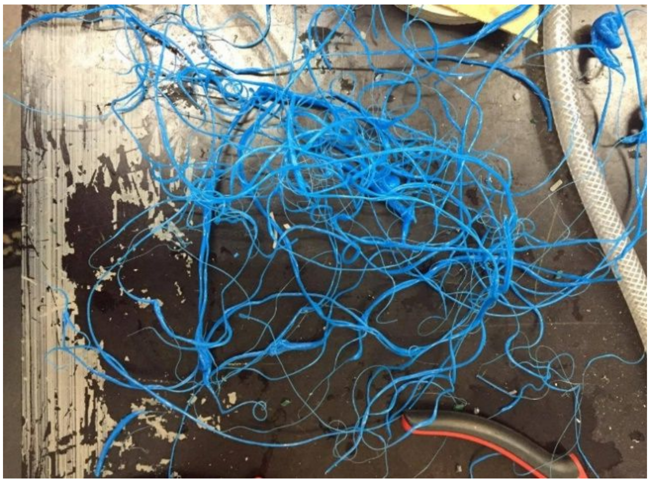


Figure 14. Extrusion of recycled pallet

3.2.2 Injection Molding

Engel Injection Molding machine was used in order to produce dumbbell samples for further tensile testing (see Fig. 15).

Figure 15. Engel Injection Molding machine

HDPE pellets for the dumbbell samples were fed via a hopper into a heated barrel, melted using heater bands and the frictional action of a reciprocating screw barrel. The plastic was then injection through a nozzle into a mold cavity where it cooled and hardened to the configuration of the cavity. The mold tool was mounted on a moveable platen – when the part has solidified, the platen opened and the dumbbell samples were ejected out using ejector pins. As a result got dumbbell samples with dimensions: length 57 mm, width 12,8 mm and thickness 3,1 mm.

Injection molding was done after MFI test, so molding parameters were optimized depending on MFI values. There were two main sets of parameters: for rHDPE with MFI 6-8 g/10 min and for rHDPE with MFI<1 g/10 min. Table 5 shows optimized parameters that were used in injection molding. Only pressure and injection speed were changed every time.

However, these parameters didn't approach perfectly to all materials, because of the small amount of material. Some dumbbell samples were produced with minor sink marks and flash.

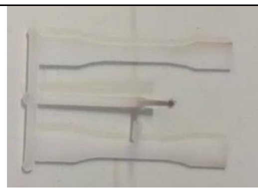
Table 5. Optimization of parameters for Injection Molding

Parameter	Value for materials with MFI 6-8 g/10 min	Value for materials with MFI<1
Nozzle temperature	230 ˚C	235 ˚C
Cylinder 2 temperature	225 ˚C	230 ˚C
Cylinder 3 temperature	220 ˚C	225 ˚C
Cylinder 4 temperature	220 ˚C	220 ˚C
Hopper temperature	70 ˚C	70 ˚C
Mold temperature	20 ˚C	20 ˚C
Clamping force	350 kN	350 kN
Cooling time	24 s	24 s
Injection speed	30/35 mm/s	55/60 mm/s
Limitation of injection pressure	60 bar	90 bar
Injection time	2 s	2 s
Holding pressure	40 bar	55 bar
Holding time	11 s	11 s
Cushion	2 mm	2 mm

Table 6 shows a process of recycling and preparation of samples in pictures.

Table 6. A process of recycling and preparation of samples in pictures

Item		Flakes	Pellets	Dumbbell samples
Bucket, sledges	 			
Pallet				-
Watering pots				
New canisters				
Old canisters				
Bottles				
Panel				

Ref. HDPE	-	-		

3.3 Testing

3.3.1 Melt Flow Index

Mitaten Extrusion plastometer was used in order to measure the ease of flow of the melt rHDPE, HDPE and HDPE powder (see Fig.16). The test was done according to ISO 1133 Standard for Polyethylene. It was defined as the mass of polymer, in grams, flowing in 10 minutes through a capillary by a load applied via weights of 2.16 kg for temperature 190 ˚C. Interval time was 30 seconds, then calculations were done and values of MFI in g/10 min obtained.

Figure 16. Mitaten Extrusion plastometer

Initially extrusion plastometer was heated to 190 °C. Then 5 grams of polymer sample were taken inside the barrel and preheated for a 5 min at 190 °C. A piston was also inserted inside the barrel. After the preheating a weight of 2.16 kg was introduced onto the piston according to ISO1133 Standard. Molten polymer immediately started to flow through the die. Then 5 samples of the melt were taken after 30 seconds and were weighed accurately. Average MFI was expressed in grams of polymer per 10 minutes of duration of the test. Standard deviation was also calculated using values such as average of MFI and MFI of each sample.

3.3.2 Tensile Testing

In order to obtain mechanical properties of rHDPE and ref. HDPE tensile testing was performed. Mechanical properties include young's modulus, tensile strength, maximum elongation, reduction in area, Poisson's ratio, yield strength and strain-hardening characteristics.

10 dumbbell samples of each material produced by Injection Molding machine were tested using Testometric Material testing machine with wedge grips. Dimensions of 1 dumbbell sample: thickness 3.1 mm, width 12.8 mm and length 57 mm. Pulling force was 5 kN and testing speed was 51 mm/min according to Standard ASTM D638 – 14. Figure 17 shows a tensile testing of rHDPE panel where the sample stretched without breaking.

Figure 17. Tensile testing of rHDPE panel

4 RESULTS

4.1 Melt Flow Index

Table 7 shows MFI values and standard deviation obtained during MFI testing of 7 different rHDPE, ref. HDPE and powder.

Table 7. Results of MFI Test

Item	Manufacturing Method	MFI	Standard Deviation
Bucket, sledges	Injection Molding	6,3 g/10 min	2,9 %
Pallet	Injection Molding	7,8 g/10 min	1,2 %
Watering pots	Extr. Blow Molding	0,2 g/10 min	2,2 %
New canisters	Extr. Blow Molding	0,15 g/10 min	3 %
Old canister	Extr. Blow Molding	MFI<1 Impossible to test with temperature 190 ˚C, material got stuck and didn’t come from die.	-
Bottles	Extr. Blow Molding	0,37 g/10 min	2,75 %
Panel	Rotational Molding	7 g/10 min	2,84 %
Ref. HDPE	-	8 g/10 min	2,3 %
HDPE Powder	-	7, 6 g/10 min	1,5 %

4.2 Tensile Testing

Table 8 shows tensile strength, young's modulus and strain at yield obtained during tensile testing of 6 different rHDPE and ref. HDPE.

Table 8. Results of Tensile Testing

Item	Manufacturing Method	Tensile Strength	Young's Modulus	Strain at Yield	Elong. Break
Bucket, sledges	Injection Molding	9,67 MPa	468,29 MPa	15,61 %	566,38 mm
Pallet	Injection Molding	-	-	-	-
Watering pots	Extr. Blow Molding	12,98 MPa	413,46 MPa	19,86 %	35,87 mm
New canisters	Extr. Blow Molding	11,75 MPa	378,58 MPa	19,15 %	34,28 mm
Old canister	Extr. Blow Molding	40,08 MPa	477,39 MPa	19,38 %	12,29 mm
Bottles	Extr. Blow Molding	11,1 MPa	411,38 MPa	17,64 %	47,69 mm
Panel	Rotational Molding	19,16 MPa	205,46 MPa	21,23 %	620 mm
Ref. HDPE	-	19,12 MPa	191,52 MPa	20,28 %	622 mm
HDPE Powder	-	-	-	-	-

5 DISCUSSION

5.1 MFI

Tested MFI of ref. HDPE gives 8 g/10min and HDPE powder gives 7,6 g/10min. Their difference of 0,4 g/10min indicates a decrease of 5%. This decrease in value of HDPE powder can be evaluated as a result of grinding process.

However, it was noticed that there is a difference between experimentally obtained MFI of ref. HDPE and MFI of the same resin from data sheet Polyethylene resin DOWLEX™ 2631 UE (in Appendix 1). The difference is 1 g/10 min, which corresponds to 12,5 %. It can be assumed, the factor that caused a mismatch was a low reliability of Mitaten Extrusion plastometer which worked not always properly during an experiment.

MFI of all recycled materials were lower than MFI of ref. HDPE. Standard deviation didn't exceed 3%.

Half of samples were with MFI lower than 1 g/10 min: new canisters, watering pots and bottles. Moreover, pellets of old canister got stuck and didn't come from the die, so it can be concluded that old canister has also MFI<1. All recycled materials with MFI<1 were manufactured initially by extrusion blow molding. It means that materials are very viscous and resistant to flow. Also, a low MFI value indicates a high molecular weight polymer.

MFI of recycled materials manufactured by injection molding were 6,3 g/10 min (bucket and sledges) and 7,8 g/10 min (pallet). Panel that was manufactured by rotational molding had MFI 7 g/10 min and in comparison with ref. HDPE difference is 1 g/10 min, which corresponds to 12,5 %.

5.2 Tensile Testing

All recycled materials initially manufactured by extrusion blow molding had elongation at break in the range 12,29-47,69 mm and were broken quickly in comparison with other materials. However, these materials are not similar, because bottles, watering pots, old canister and new canisters have different values of tensile strength, young's modulus, strain at yield and elongation at break. Old canister has the highest tensile strength and young's modulus, and the lowest elongation at break in comparison to all other materials. Watering pots, bottles and new canisters have quite similar properties with slight differences in young's modulus.

In contrast, panel and ref. HDPE didn't break during tensile testing. Recycled panel that was manufactured by rotational molding is the most similar to ref. HDPE. Their difference is just 1 % of strain at yield and 14 MPa in young's modulus, which corresponds to 4,7% of strain at yield and 6,7 % in young's modulus.

Material made of recycled bucket and sledges had fracture, but was stretched for a long time because the material was very viscous. It has a lower strain at yield and tensile strength in comparison with ref. HDPE. Young's modulus of bucket and sledges is 468,29 MPa, it is the highest value of young's modulus in tensile testing.

However, bottles, new canisters, watering pots, bucket and sledges had unexpectedly low values of tensile strength. Their range is 9,67-12,96 MPa. According to Table 1, for HDPE standard values of tensile strength are in the range 20-35 MPa. Low tensile strength values of rHDPE materials can be evaluated as a result of degradation occurred during the recycling process.

6 CONCLUSION

The purpose of this work was to find out applicability of recycled HDPE in rotational molding. Requirements of the materials for rotational molding were defined as MFI 3-8 g/10 min, Particle size distribution 95% < 500 micron with maximum 15% < 150 micron, dry flow rate minimum 185 g/min and bulk density >320 kg/m3 (p. 17-21).

In order to find the most suitable material for further production of powder, different HDPE materials were mechanically recycled. Items made of HDPE were sorted into different categories: by the method of manufacturing, by the type of item and by the usage time. Then MFI and tensile testing were performed in order to obtain values of MFI, tensile strength, young's modulus, strain at yield and elongation at break.

As a result, tested MFI of ref. HDPE gives 8 g/10min and HDPE powder gives 7,6 g/10min. Their difference of 0,4 g/10min indicates a decrease of 5%. This decrease in value of HDPE powder can be evaluated as a result of grinding process.

Recycled materials initially manufactured by extrusion blow molding can't be used in rotational molding because of the material requirements of MFI. But it is possible to use recycled bottles and canisters in another manufacturing processes, for example in film extrusion.

Recycled materials that were manufactured by rotational molding and injection molding have enough value of MFI to be used in rotational molding. However, MFI is not the only one property which is important, so further testing of other properties such as viscosity, dry flow rate, bulk density and particle size distribution should be done in order to be sure that materials are suitable.

7 SUGGESTIONS FOR FURTHER WORK

Further research could be done on grinding and testing of rHDPE powder for rotational molding. Materials chosen for recycling should be manufactured by rotational molding and injection molding.

Also, further research could be focused on studying the differences between properties of HDPE and powder made of that resin. One measurement of MFI already showed that when the powder is produced from HDPE pellets, MFI decreases for 5%. It is possible to test other properties in order to see what is the difference.

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APPENDICES

Appendix 1: Polyethylene resin DOWLEX™ 2631 UE- The Dow Chemical Company

Distributed by RESINEX

DOWLEX™ 2631UE

The Dow Chemical Company - Polyethylene Resin

Monday, February 11, 2013

General Information

Product Description

DOWLEX™ 2631UE Polyethylene Resin for rotational and injection moulding is specifically designed for applications requiring excellent processability and aesthetics combined with low warpage and good mechanical properties. Processing and Stabilisation: DOWLEX™ 2631UE Polyethylene Resin is fully heat and UV stabilised resulting in a wide processing latitude, good colour retention and long life expectancy. The powder version is named DOWLEX™ 2631.10UE Polyethylene Resin.

Applications:
- Toys
- Technical mouldings
- Flat surface containers
- Caravan tanks

Complies with:
- EU, No 10/2011
- U.S. FDA 21 CFR 177.1520(c)3.1a

Consult the regulations for complete details.

General

Material Status	• Commercial: Active	
Availability	• Europe	
Agency Ratings	• EU No 10/2011	• FDA 21 CFR 177.1520(c) 3.1a
Forms	• Pellets	
Processing Method	• Injection Molding	• Rotational Molding

ASTM & ISO Properties [1]

Physical	Nominal Value	Unit	Test Method
Specific Gravity	0.935	g/cm³	ASTM D792
Melt Mass-Flow Rate (MFR) (190°C/2.16 kg)	7.0	g/10 min	ISO 1133
Environmental Stress-Cracking Resistance			ASTM D1693
50°C, 100% Antarox, Compression Molded	> 1000	hr	
Mechanical	**Nominal Value**	**Unit**	**Test Method**
Tensile Stress (Yield, Compression Molded)	17.8	MPa	ISO 527-2
Tensile Strain (Break, Compression Molded)	420	%	ISO 527-2
Flexural Modulus (Compression Molded)	628	MPa	ISO 178
Impact	**Nominal Value**	**Unit**	**Test Method**
Multi-Axial Instrumented Impact Energy [2]			ISO 6603-2
-20°C, Rotational Molded	59.7 to 79.6	J	
23°C, Rotational Molded	53.1 to 70.8	J	
Hardness	**Nominal Value**	**Unit**	**Test Method**
Shore Hardness (Shore D, Compression Molded)	56		ISO 868
Thermal	**Nominal Value**	**Unit**	**Test Method**
Heat Deflection Temperature (0.45 MPa, Unannealed)	52.0	°C	ISO 75-2/B
Vicat Softening Temperature	115	°C	ASTM D1525 [3]
Melting Temperature	124	°C	DSC
Peak Crystallization Temperature (DSC)	110	°C	DSC

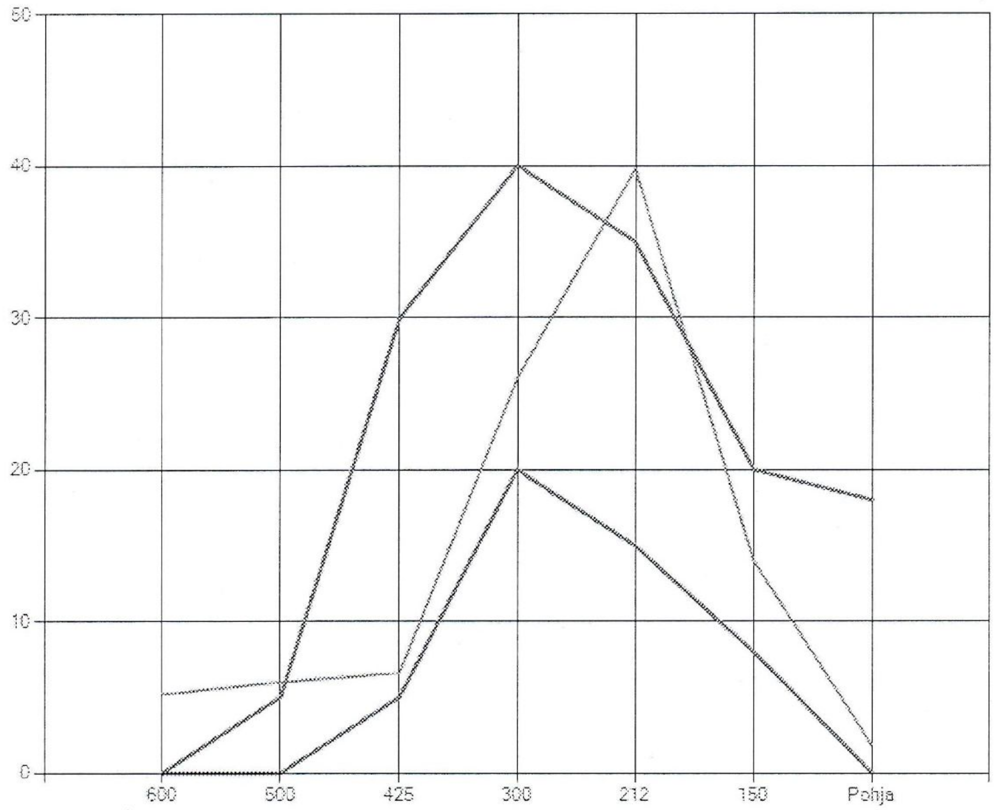
Appendix 2: Particle size distribution of HDPE powder (Motoplast)

Raaka-aineanalyysi

Materiaali NG2631 (RX102 nat)

Näyte otettu	1/22/2015 12:00:00 AM	
Mittaus tehty	1/22/2015 12:00:00 AM	
Juoksevuus	34	s/?
Kopautuksia	2	kopautusta
Tiheys	0,288	g/cm3
Sulaindeksi	-	g/10min
Terävälys	0,35-0,6	
Terät	360	
Muuta		

Seula	Mitattu %	Ref % ALA	Ref % YLÄ
600	5,2	0	0
500	6	0	5
425	6,6	5	30
300	26	20	40
212	39,8	15	35
150	14	8	20
Pohja	1,8	0	18



Appendix 3: ABNET Professional Concentrate Multipurpose cleaner and degreaser

ABNET Professional 1L Concentrate Multipurpose cleaner and degreaser

- Harmless, very effective and versatile detergent.
- Based on surfactants that clean effectively but gently.
- For all types of cleaning and stain removal.
- Sufficient for 5-50 liters of ready cleaning solution, depending on target difficulty.
- Excellent for manual and automated cleaning.
- Well suitable for cleaning any surface including textiles.
- Makes surface antistatic.
- The product can be used on all surfaces and objects that can withstand water.

ABNET Professional provides a clean and dirt-resistant surface. Eco-friendly cleaning and stain removal on all water resistant surfaces. Suitable for all plastics, wood, textiles, stone, metals, painted and varnished surfaces. Gently removes ingrained dirt such as: oil, grease, mold, algae, soot, oxidation, coffee, wine, ink, cigarrette smoke stains, blood, fruit, ketchup, etc. ABNET Professional is a highly concentrated cleaner which is easily diluted with water. ABNET is easy to dose for both light cleaning of large surfaces and removal of difficult stains.

DOSAGE

- Light, everyday cleaning, 20-50 ml/ 1l water (2-5 %)
- Heavy cleaning, 100 ml/ 1l water (10 %)
- Extremely difficult stains, 200 ml/1l water (20%)

CHARACTERISTICS

Clear, yellowish, mildly fragrant liquid. User and environmentally friendly.

COMPOSITION

5-15% non-ionic surfactants. Less than 5% of phosphates, phosphonates, cationic surfactants, perfume. pH 12.75 in Concentrate.

SAFETY

Irritating to eyes upon contact. Keep out of reach of children. In case of contact with eyes, rinse immediately with plenty of water and visit doctor if necessary. If swallowed, seek medical advice immediately and show the bottle or label of the product. When the product is used as spray, avoid inhalation of mist as well as mist in the eyes.

ENVIRONMENT

Minimal environmental impact, biodegradable. The product meets European Parliament and Council Regulations (EC) No 648/2004 of 31 March 2004 on detergents criteria for biodegradability.