MAGNETIC DENSITY SEPARATION
of Polyolefin Wastes

Bin HU
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Magnetic Density Separation of Polyolefin Wastes

Proefschrift

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To my beloved parents
献给我的父母
Polymer recycling has significantly positive impacts on the environment, economics and society. Using recycled polymer to replace virgins reduces the growing amount of post-consumer polymer wastes, decreases the demand for fossil fuel and creates local jobs. As the most used polymer types, polyolefins certainly have attracted attention of recycling.

Mechanical sorting has been pointed out as the preferred route for the recovery of relatively clean and homogeneous plastic waste streams. Whereas washing and compounding technologies for polymer scrap have dramatically improved over the last decade, there is as yet no sorting technology commercially available that is both cheap and effective. Consequently, the amount of recycled post-consumer mixed plastics remains low and the recycled polymer value is even worse. Innovative recycling technologies for post-consumer wastes should be extremely powerful, simple, cost-effective and accurate enough to create high quality products. Magnetic density separation (MDS) is such a technology. Early research showed that MDS has the potential to turn around the poor image of polyolefin recycling. However substantial research was needed to improve the separation process before high quality secondary polyolefins could be obtained from complex waste mixtures.

This thesis starts with the characterization of the main resources of secondary polyolefins to determine the proper input streams for MDS. Concerning the lifetimes and the total consumption of polyolefin applications, household packaging and car components are two interesting streams for MDS. To reach the necessary separation accuracy for gaining high quality secondary PP and PE in one single step, MDS needs to be sensitive to very small differences in the physical properties of the input materials. Therefore, three main aspects influencing the separation process were studied: wettability of polymers, quality of the process liquid and flow turbulence. For this thesis, a new wetting process to eliminate the effect of air bubbles on the apparent polymer density was developed, a simple method to avoid the segregation of the process liquid was provided, and the sources of turbulence were studied and reduced. Based on all of these achievements, two MDS setups were designed and their performance on the post-consumer polyolefins was evaluated. For both setups, over 98% of the polyolefin waste can be successfully recycled with sufficient separation accuracy: 10 kg/m³. The output products of the MDS were analysed to assess their quality: composition, rheological and mechanical properties. According to the analyses, the derived products from MDS were high purity PP and PE. Based on the mechanical analyses, the separated household wastes showed good and consistent properties, independently from their origin, which were comparable with neat polymers.

A model that simulates the MDS process was constructed on the basis of the fundamental principle of MDS and the major factors affecting the process. The study concluded that the separation accuracy of MDS in polymer recycling is determined by the flow turbulence and the uneven magnetic field. The combined effect of these two factors indicates that for most of the polymers turbulence is the major cause of an inaccurate separation.
Both the experiments and the simulations concluded that MDS can accurately separate polyolefin mixtures based on their density. With such high accuracies in density, polyolefin mixtures can be separated into different polymers according to their type by MDS in a single step.
De recycling van polymeren heeft een belangrijke positieve impact op het milieu, de economie en de samenleving. Het gebruik van gerecyclede polymeren ter vervanging van primaire grondstoffen vermindert de groeiende hoeveelheid plastic afval van gebruikers, vermindert de vraag naar minerale brandstof en creëert plaatselijke werkgelegenheid. Polyolefinen, als meest gebruikte polymeertypen, staan zeer zeker in de belangstelling van recycling.

Mechanisch sorteren is erkend de meest aangewezen weg voor het terugwinnen van relatief schone en homogene plastic afvalstromen. Hoewel de technologieën voor het schoonwassen en regranuleren van polymeerschroot de afgelopen 10 jaar beduidend zijn verbeterd is er tot op heden geen commercieel beschikbare sorteertechniek die zowel goedkoop als effectief is. Als resultaat hiervan blijft de hoeveelheid van het gerecyclede gemengd-plastic gebruikersafval laag en is de waarde van het herwonnen plastic zelfs nog teleurstellender. Innovatieve recyclingstechnologie voor gebruikersafval zou extreem krachtig, eenvoudig, kosteneffectief en nauwkeurig genoeg moeten zijn om producten van hoge kwaliteit te maken. Magnetische dichtheidsscheiding (MDS) is zo’n technologie. Eerder onderzoek liet zien dat MDS het potentieel heeft om het slechte imago van de recycling van polyolefinen te keren. Echter, er moest aan MDS nog substantieel onderzoek gedaan worden aan het verbeteren van het scheidingsproces voordat een hoge kwaliteit secundaire polyolefinen verkregen kon worden uit complexe afvalmengsels.

Dit proefschrift begint met het karakteriseren van de belangrijkste bronnen van secundaire polyolefinen, dit om de juiste invoerstromen voor MDS te bepalen. Gezien de levensduur en het totale verbruik van polyolefine producten zijn huishoudverpakkingen en auto-onderdelen twee interessante stromen voor MDS. Om in één enkele stap de benodigde nauwkeurigheid in scheidingsproces te bereiken voor de winning van hoogwaardige PP en PE moet de MDS gevoelig zijn voor zeer kleine verschillen in de fysische eigenschappen van het invoermateriaal. Om die reden werden drie hoofdaspecten bestudeerd die het scheidingsproces beïnvloeden: de benatting van polymeren, de kwaliteit van de procesvloeiostof en de turbulentie in de vloeistofstroom. In dit proefschrift wordt een nieuw benattingsproces ontwikkeld om het effect van luchtbelletjes op de schijnbare dichtheid van polymeren te elimineren. Verder wordt een eenvoudige methode gegeven om het ontmonden van de procesvloeiostof te voorkomen en worden de bronnen van turbulentie onderzocht en gereduceerd. Gebaseerd op al deze resultaten zijn twee MDS opstellingen ontworpen en hun prestaties op polyolefine gebruikersafval geëvalueerd. Beide opstellingen kunnen meer dan 98% van het polyolefine afval succesvol terugwinnen, met een voldoende scheidingsnauwkeurigheid: 10 kg/m³. De uitvoerproducten van de MDS werden geanalyseerd om de kwaliteit van de samenstelling en van de Rheologische en mechanische eigenschappen te beoordelen. Volgens deze analyses waren de door de MDS geproduceerde producten zeer zuivere PP en PE. De mechanische analyses laten zien dat de scheidings-
producten uit het huishoudafval goede en consistente eigenschappen bezitten onafhanke-
lijk van hun herkomst. Deze zijn vergelijkbaar met primaire polymeren.

Op basis van het fundamentele principe van MDS werd een model gebouwd inclusief de belangrijkste invloeden die het proces verstoren. Uit deze studie bleek dat, voor polymere, de nauwkeurigheid in scheiding van MDS bepaald wordt door de turbulentie in de vloeistofstroom en door onregelmatigheden in het magnetisch veld. Het gecombineerde effect van deze twee factoren laat zien dat voor de meeste polymeren turbulentie de hoofd-oorzaak is van een onnauwkeurige scheiding. Zowel de experimenten als de simulaties lieten zien dat MDS in staat is polyolefine mengsels op grond van hun dichtheid nauwkeu-
rig te scheiden. Met een dergelijke hoge nauwkeurigheid in dichtheid, kunnen polyolefine mengsels in één enkele stap gescheiden worden in verschillende types polymeer.
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Chapter 1

Introduction
1.1 POLYMERS AND EUROPE

Europe produced nearly 60 Mtons of polymers, approximately 20% of the world plastic production, with a turnover of 87 billion euros in 2013. The combined turnover of the plastic industry including plastics producers, plastics converters and the plastics machinery was above 300 billion euro (PlasticsEurope, 2013). Considering the generated value of plastics industry for public finance and welfare in EU27, 27.5 billion euros, the European plastics industry is an important part of economics and society (PlasticsEurope, 2012). A major part of the produced polymers are used in packaging materials, 39.4% of overall demand in Europe, followed by building & construction (20.5%), automotive (8.3%) and electrical & electronic equipment (5.4%). Of all the polymer resin applications, polyolefins, including both polypropylene (PP) and polyethylene (PE), are the most used as presented in Table 1.1 (PlasticsEurope, 2012; WRAP, 2013), particularly as packaging materials (shown in Fig. 1.1).

Table 1.1 European plastics demand in 2011 (PlasticsEurope, 2012).

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Demand (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP</td>
<td>19</td>
</tr>
<tr>
<td>HDPE</td>
<td>12</td>
</tr>
<tr>
<td>LDPE/LLDPE</td>
<td>17</td>
</tr>
<tr>
<td>PVC</td>
<td>11</td>
</tr>
<tr>
<td>PS/PS-E</td>
<td>7.5</td>
</tr>
<tr>
<td>PUR</td>
<td>7</td>
</tr>
<tr>
<td>PET</td>
<td>6.5</td>
</tr>
<tr>
<td>Others</td>
<td>20</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
</tr>
</tbody>
</table>

Fig. 1.1 European plastics demand by segment and resin type 2011 (PlasticsEurope, 2012).
There are three main sources of the polymer materials: fossil, biomass and plastic wastes. Fossil plastics are the most popular source of polymer materials because of their low price, high and stable qualities. For instance, 13 out of 14 million ton annually sold polyolefins in Europe are made of virgin materials (Di Maio et al., 2010). The main raw material of virgin polymers is petroleum. Around 4% of annual petroleum production is converted directly into plastics from petrochemical feedstock (British Plastics Federation). One litre of petroleum can produce about 0.54-0.72 kg polyolefins (Table 1.2). Since petroleum is a non-renewable resource, continuously producing and using virgin polymers is not a sustainable way. Using virgin plastics has other disadvantages next to the high cost, such as the high energy consumption. To produce 1 kg of PP or PE, the total consumed energy is about 70 MJ (Table 1.3). Another disadvantage is that virgin plastic production does not contribute to the green economy in terms of creating new jobs, although the turnover of the produced plastic raw materials is nearly 50% of the total plastic industry including plastic producing and converting (European Plastics Converters). The European plastic producing industry is composed of approximately 2000 companies and less than 200,000 employees, much less than those in polymer converting industry, nearly 50,000 companies and 1.3 million employees. The main reason is that most of the oil consumption in Europe relies on import from other countries.

Bio-plastics are made from plant matters, such as vegetable starch, cane sugar and cellulose: from biomass. In Europe, bio-plastic consumption is approximately 60,000-100,000 tonnes per year. Bio-plastics, particularly examples like polyethylene furanoate (PEF), are sustainable compared to fossil-based virgin plastics and partially or wholly renewable, and they consume less crude oil. But the polymer types are usually limited.

Table 1.2 Petrol consumption in plastic making for different kinds of plastics (Engelbeen; Gervet, 2007).

<table>
<thead>
<tr>
<th>Plastic</th>
<th>Min (kg/l)</th>
<th>Max (kg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVC</td>
<td>0.75</td>
<td>1.65</td>
</tr>
<tr>
<td>PE</td>
<td>0.57</td>
<td>0.72</td>
</tr>
<tr>
<td>PP</td>
<td>0.54</td>
<td>0.68</td>
</tr>
<tr>
<td>PS</td>
<td>0.50</td>
<td>0.72</td>
</tr>
<tr>
<td>PET</td>
<td>0.47</td>
<td>1.28</td>
</tr>
<tr>
<td>PC</td>
<td>0.37</td>
<td>1.10</td>
</tr>
</tbody>
</table>

Table 1.4 summarizes the status of the plastic production in Europe from the introduced three resources. Compared to virgins, using recycled polymers has several benefits. First of all, creating a solution for the increasing amount of polymer wastes. In 2011, the amount of collected post-consumer polymer wastes in EU27 reached 25.1 million tonnes, with an increase of 2.4% compared with in 2010. Using recycled polymer wastes to replace virgin materials in some applications, such as non-food packaging and automotive components,
Table 1.3 Energy consumption for different plastic making (Engelbeen; Gervet, 2007).

<table>
<thead>
<tr>
<th>Plastic</th>
<th>Energy consumption (MJ/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVC</td>
<td>53</td>
</tr>
<tr>
<td>PE</td>
<td>70</td>
</tr>
<tr>
<td>PP</td>
<td>73</td>
</tr>
<tr>
<td>PS</td>
<td>80</td>
</tr>
<tr>
<td>PET</td>
<td>84</td>
</tr>
<tr>
<td>PC</td>
<td>107</td>
</tr>
</tbody>
</table>

can effectively decrease the demand of the amount of import oil and conserve raw materials. Secondly, energy saving. The production of recycled polymer generally consumes less energy than the virgins, although the amount of saved energy varies very much in different references. (For instance, the research of Arena et al shows that the production of 1 kg of flakes of recycled PE requires a total amount of gross energy that is in the range of 40-49 MJ (Arena et al., 2003), but the study of Tempelman concluded about 1.69 MJ/kg (Tempelman, 1999)). Thirdly, creating new job positions. According to a EU report, the collection, treatment, sorting and materials recovery of lightweight packaging produces 2.4 jobs per 1,000 tons wastes (Snallfot et al., 2013), therefore the new jobs created thanks to polymer recycling is around 60,000 in Europe. Considering all these positive impacts of recycling polymer on environment, economy and society, it is encouraged to recover materials from polymer wastes. Nevertheless, only approximately six million tonnes out of 25 Mtons of plastic waste were collected for recycling in EU. Norway, which had the highest recycling rate of post-consumer polymer waste in Europe in 2011, recycled only 35%. In the UK only 24% of the consumed polymer packaging was recycled, and the amount of recycled post-consumer mixed plastics was even less: only 10% (WRAP, 2013). In terms of recycled value, the picture is even worse. According to EU statistics, only about 2% of the polymer value produced in Europe is from recycled polymers (European Environment Agency, 2011).

Table 1.4 Plastic production in Europe.

<table>
<thead>
<tr>
<th>Production in Europe (Mtons)</th>
<th>Gross energy consumption (MJ/kg)</th>
<th>Use of crude oil (L/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fossil-based</td>
<td>50-60</td>
<td>70</td>
</tr>
<tr>
<td>Bio-based</td>
<td>0.2-0.3$^5$</td>
<td>25-54$^4$</td>
</tr>
<tr>
<td>Recycled</td>
<td>$\approx 2^\ast$</td>
<td>1.7-49</td>
</tr>
</tbody>
</table>

$^5$ in 2011 (European-bioplastics, 2013).
$^4$ energy consumption for PHB, PLA and TPS (Momani, 2009).
$^\ast$ calculated based on the value (2 billion) and price of secondary polymers (half of the virgins).
1.2 CURRENT MECHANICAL RECYCLING TECHNOLOGIES

One reason of the low amount of value produced from polymer wastes is the lack of proper sorting technologies. Mechanical sorting has been pointed out as the preferred route for the recovery of relatively clean and homogeneous plastic waste streams. Whereas washing and compounding technologies for polymer scrap have been dramatically improved over the last decade, there is as yet no commercial sorting technology that is both cheap and effective. There are mainly four mechanical sorting techniques implemented in polymer recycling: near infrared sorting (NIR), triboelectrostatic separation, sink-float and flotation (Table 1.5).

Table 1.5 Mechanical sorting methods.

<table>
<thead>
<tr>
<th>Method</th>
<th>Sorting principle</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Near Infrared</td>
<td>Based on spectroscopic identification of polymer types</td>
<td>Fast, high resolution</td>
<td>Not applicable for black or small items, expensive, surface sensitive, low material recovery</td>
</tr>
<tr>
<td>(supported by hand-sorting)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Triboelectrostatic sorting</td>
<td>Based on electrostatic charge</td>
<td>High capacity</td>
<td>Only for clean, dry and non-surface-treated polymers, high requirement of operating conditions, the input materials should be a binary mix</td>
</tr>
<tr>
<td>Density sorting</td>
<td>Sorting on density</td>
<td>Low operating cost, no limitation on size or colour</td>
<td>Needs efficient difference in density (10-40 kg/m³)</td>
</tr>
<tr>
<td>Flotation</td>
<td>Different surface properties</td>
<td></td>
<td>Sensitive to reagents, condition temperature, time, particle sizes and roughness</td>
</tr>
</tbody>
</table>

Sensor-based near infrared spectroscopy is commonly used to sort household packaging wastes. Its sorting principle is based on the fact that NIR spectra of different types of polymers are quite distinct. NIR is able to provide rapid and reliable identification of various polymer types, for instance the polyolefin group, polyethylene (PE) and polypropylene (PP), and polyethylene terephthalate (PET). However, this technique has limitations on the size of the materials: too big or too small (< 5 cm) objects are not favourable for this technology. Another disadvantage is its incapacity to detect dark polymers, like automotive components and some of the electronics waste stream, as radiation is absorbed totally. In practice, for packaging polymers, around half of the total input materials end up in residuals. NIR is also a quite expensive sorting method. To sort one ton of input polymers from mixed polymers into three products, such as PP, PE and PET, the processing costs are about 120 €/ton of input.

A second way of sorting plastics is triboelectrostatic separation, which can distinguish small dry particles that have large differences in electric work function. Products with a
high grade in excess of 95% are feasible at proper operation conditions (Dodbiba et al., 2002b; Inculet et al., 1998), but with a typical recovery rate of 70% (Dodbiba et al., 2002b, 2003). Sometimes multiple-stage is necessary to obtain a product with high purity. This technique strongly depends on the surface potential of the components of the input mixtures, thus controlling air humidity and temperature is a prerequisite (Dodbiba et al., 2002b; Yanar and Kwetkus, 1995). Dirty, painted or labelled polymer items or flakes may be easily misidentified and end up in the wrong product.

Both density sorting and flotation methods are wet separation techniques for sorting plastics. Density-based separation can be divided into static density separation and dynamic density separation. The sink-float technique is a typical static density separation. It works on the true density of a particle, regardless to the shape or size of it. Sink-float processes use a liquid medium with a density intermediate between the different polymers to be separated. Such separation is comparatively simple, easily automated, and flexible in operation. The dynamic density separation works on the differences in the settling velocity of particles (e.g. hydrocyclones, windsifting and kinetic gravity separator) or the differences of particle movement in a fluid flow (e.g. water table and fluidized bed). The dynamic density separation is not a purely density-dependent technique. Both particle size and shape determine the separation accuracy. For instance, a water table is able to obtain products with both high grade and recovery rate, but it is limited to narrowly size-classified particle sizes and shapes. Additionally, risks associated with organic solvents should be considered, for example the ethanol used for polyolefin separation. Moreover, sink-float is too difficult to separate mixed polymers with slight differences in density, since the medium density is not easily accurately controlled. Thus, in order to improve the grade of products, this technique is combined with flotation (Dodbiba et al., 2002a; Pongstabodee et al., 2008). Flotation utilizes the different surface properties of two or more types of polymers, for instance separating PET and PVC (Fraunholcz, 2004; Shent et al., 1999). By adding an appropriate agent, selective separation is achieved. But the efficiency of this separation is sensitive to reagents, condition temperature and residence time, particle sizes and roughness (Shent et al., 1999). Burat et al. using virgin polymer samples extracted all the PVC from a mixture of rigid virgin PET and PVC at 100% content and 98.8% recovery rate, while for post-consumer mixtures PET particles were obtained with a purity of 99.7% and 57.0% efficiency (Burat et al., 2009). Being wet separation techniques, both of these two methods have a common disadvantage: the treatment of the processing liquid from the processes.

All of these methodologies provide secondary polyolefins or other polymer types with an acceptable quality for reuse, but to effectively obtain more than one type of polymer from the mixture, multiple steps are necessary when using any of these three technologies. Due to the drawbacks of these current separation technologies for secondary polymer recycling, most of the potential polymer value in mixed wastes remains unused. The fact that recycled plastics have an extremely low turnover of plastic production in Europe indicates that recycling technologies that are to address post-consumer wastes should be extremely powerful, simple to be cost-effective and accurate enough to create high quality products.
1.3 MAGNETIC DENSITY SEPARATION

Magnetic density separation (MDS) is a density-based sorting technology, similar to the previously introduced sink-float method. But instead of using a medium with a single cut density, it uses a liquid separation medium with a density gradient. Such liquid contains magnetic iron oxide particles with a size about 10-20 nm suspended in water. By applying an artificial gravity, in the form of magnetic force, that varies exponentially in the vertical direction, the effective density of the liquid varies in this direction as well. Plastic particles with the same density will float in the liquid at the same level: where the effective density is equal to their own density. On the basis of this principle, plastic mixtures segregate into many different layers, and flakes with the same density stay at the same vertical height. Thanks to the advanced fluid dynamics, this process takes much less time compared to the sink-float technique: the processing time is the order of a few seconds (Bakker et al., 2009). By setting the separation cut densities at different positions, multiple products can be achieved in one single step (Muchova et al., 2009). Such advantages allow MDS to be more cost-effective than other recycling techniques.

MDS is utilized in the recycling and mineral processing industry since 2006. By changing the magnetization of the liquid, the MDS process provides a good solution for minerals and precious metals concentrating and for polymers separation. In PET recycling, PET flakes are still polluted with aluminium, glass and stone particles after using state of the art recycling processes. MDS can remove these contaminants with a higher density from the PET having a lower density (Bakker and Rem, 2006). In the recovery of non-ferrous metals from incinerated municipal solid waste (MSW), MDS is used to separate aluminium and copper (Bakker et al., 2007). Another important application is to recover precious metals (Hu et al., 2009; Muchova et al., 2009). MDS is also capable of improving germination quality by separating seeds continuously into multiple fractions in a single step, which saves a significant amount of time and work (De Koning et al., 2011).

It is challenging to achieve high-value secondary polyolefins with high recoveries as well by using MDS. Early trial separations showed that, in principle, MDS can be applied into recycling of PP and PE mixtures under carefully controlled situations. High quality recycled polymers is usually essential to polymer reproduction. The purity of many recycled polymers needs to be 95%, and for PET recycling a grade of more than 99% is necessary. In 2009, Bakker succeeded in obtaining high quality PP with a grade more than 97% from a mixture with an initial ratio of 70:30 between PP and PE (Bakker et al., 2009). However, the quality of PE was not good, moreover the recovery rates of both these two types of polyolefins were only about 70%. Therefore, to obtain high quality secondary polyolefins from complex waste mixtures, substantial research on the MDS process with a target of improving the polyolefin separation needed to be undertaken.
1.4 OUTLINE OF THE THESIS

This thesis studies the principle and key processes of the MDS system aiming to gain high quality secondary polyolefins. It includes in total ten chapters covering both the sources and properties of polyolefin wastes and the detailed research on MDS technique (Fig. 1.2). Chapter 2 presents the recycling-oriented characterization of polyolefins from two main waste streams: packaging and cars. The fundamental principle of MDS and the basic structure of an MDS setup are introduced in Chapter 3. Chapter 4, 5 and 6 discuss the essential factors that may cause separation errors in the MDS sorting process: the wettability of plastics, quality of the process liquid and flow turbulence. The sorting performance of both the lab-scale and pilot MDS plants is evaluated in Chapter 7. Chapter 8 assesses the quality of the output products of MDS. By investigating the factors that may contribute to the separation error, a model is constructed to simulate the polyolefin recycling process with MDS in Chapter 9. Conclusions and recommendations are given in Chapter 10 for future studies in this field.

Fig. 1.2 Schematic outline of the thesis.
Chapter 2

Characterization of Polyolefins
2.1 INTRODUCTION

Household packaging and car components are two of the major applications of rigid polyolefins (PO) that also produce significant amounts of waste to be recycled. The reason is that EU plastics consumption has been stable over the lifetimes of plastic products from these two applications. In contrast, construction and demolition waste (C&DW) originates from buildings of 50-60 years ago, when plastics consumption was more than an order of magnitude smaller than it is today. Therefore today’s C&DW contains relatively little PO waste. Electrical and electronic equipment is a fourth important use of polyolefins. Yet, the amount of polyolefin waste from this application is relatively small and so the interest in recycling PO from this resource is limited. Table 2.1 introduces the amounts of rigid PO wastes produced in EU27+2 (Switzerland and Norway) countries in 2009 and the recycling quota of PO for each waste resource (Lhôte, 2011). As shown, the amount of rigid PO wastes from three resources in 2009 was nearly 4.7 Mton, from which PO from WEEE only occupied 3.8%. Considering the short product lifespan and the amount of them, polyolefins from packaging and automotive wastes are important potential input materials for MDS. In order to optimize the MDS sorting process and to obtain good quality recycled PP and PE, it is fundamental to develop a new knowledge base, specifically addressed to perform a full characterization of the waste materials to recycle, and it is of primary importance to define and assess the relevant characteristics of these two plastic flow streams.

Table 2.1 Amount of rigid PO wastes and the recycling quota of PO in each waste resource in total EU27 countries, Switzerland and Norway in 2009 (Lhôte, 2011).

<table>
<thead>
<tr>
<th>Waste</th>
<th>Amount (kton)</th>
<th>Recycling quota</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rigid packaging</td>
<td>3968.0</td>
<td>85.0% 24.9%</td>
</tr>
<tr>
<td>Automotive</td>
<td>522.1</td>
<td>11.2% 17.9%</td>
</tr>
<tr>
<td>WEEE</td>
<td>175.5</td>
<td>3.8% 6.3%</td>
</tr>
<tr>
<td>Total (3 applications)</td>
<td>4665.6</td>
<td>100.0% 23.4%</td>
</tr>
</tbody>
</table>

The sorting principle of MDS is based on the fact that polypropylene in general has lower densities than polyethylene. To obtain high grade secondary polyolefins by using density separation, it is thus essential to determine the density distributions of both PP and PE from household packaging and automotive wastes. Additionally, attention should also be paid to the subtypes of polymers for different production processes, in particular injection moulding (IM) and blow moulding (BM). In fact, an important parameter that varies with the moulding methods is the melt viscosity, affecting the recycling for the reason that different viscosity resins do not mix homogeneously and, if they do, may result in resins with an off-spec viscosity. If different melt viscosity plastics are not separated, the properties will not be uniform in compounding.

This chapter discusses a recycling-oriented characterization addressed to household plastic packaging and automotive wastes, with the aim to recover high-value polyolefins. The results are meant to constitute the base to implement the MDS at an industrial level.
To reach this goal, investigations address: i) the composition of a polyolefin waste stream, ii) the polyolefin density distribution to define the optimal cut density and, finally, iii) the relation between manufacturing methods (i.e.: injection moulding and blow moulding) and flake physical properties.

2.2 MATERIALS AND METHODS

2.2.1 Polyolefin wastes

Household packaging wastes from both Eastern & Western European countries and dismantled car components were characterized for this study.

2.2.1.1 Packaging waste

Plastic samples from household wastes were collected in two countries: Romania and the Netherlands. The Romanian sample (RO HW) came from Valcea, a town with a population of approximately 80,000 in Romania. A 27 kg sample, including both polyolefin and non-polyolefin polymers, as well as a small amount of other wastes, e.g. food garbage, was hand-picked from raw household wastes. Foils and polymer objects smaller than 5 cm were not selected, due to the high cost of hand-sorting from raw HW. Different from the Romanian sample, the Dutch sample was separately collected from other household wastes according to the Dutch PlasticHero program launched by Nedvang in 2008. In this program, the citizens are encouraged to bring their plastic wastes into a separate trash bin near their houses. The desired wastes include plastic bags, food containers, lids of jars, bottles, etc., and exclude for instance, fast food packaging, meat packing materials or toys. The gathered polymer wastes from the trash bin are transported to a sorting plant for polymer recycling normally once a week. The analysed Dutch sample (NL HW) in this study was from Zeeland, a province in the south of the Netherlands. Compared to the Romanian plastic waste, the Dutch sample contains not only rigid plastics, but also plastic foils.

2.2.1.2 Car waste

The car polymer components used in this study were collected by a car recycling company, Alcufer, in Hungary. The selection of the polyolefin components was based on the instructions of the IDIS system (International Dismantling Information System), which contains data on the environmentally sound pre-treatment and dismantling of end-of-life vehicles, such as the used materials of each component in a car (Fig. 2.1). According to the instructions of the IDIS system, the polyolefin components of the vehicles were identified, and the > 500 g polyolefin components were dismantled and delivered to Delft for characterization (Fig. 2.2). In total, more than 100 kg of dismantled car components from ten brands and 14 series were characterized. The analysed vehicles and their components are listed in Table 2.2, and example components are shown in Fig. 2.3.
Fig. 2.1 International Dismantling Information System.

Fig. 2.2 Procedure of car polymer components selection.

Fig. 2.3 Polymer components of Fiat Croma.
Table 2.2 Received polymer components of cars.

<table>
<thead>
<tr>
<th>Cars</th>
<th>Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brand</td>
<td>Bumper</td>
</tr>
<tr>
<td>OPEL</td>
<td>VECTRA</td>
</tr>
<tr>
<td>FIAT</td>
<td>CROMA</td>
</tr>
<tr>
<td>FORD</td>
<td>ESCORT</td>
</tr>
<tr>
<td></td>
<td>ORION</td>
</tr>
<tr>
<td>DAEWOO</td>
<td>NEXIA</td>
</tr>
<tr>
<td>NISSAN</td>
<td>PRIMERA</td>
</tr>
<tr>
<td>MAZDA</td>
<td>323</td>
</tr>
<tr>
<td></td>
<td>626</td>
</tr>
<tr>
<td>VOLKSWAGEN</td>
<td>GOLF</td>
</tr>
<tr>
<td>MITSUBISH</td>
<td>LANCER</td>
</tr>
<tr>
<td></td>
<td>GLX</td>
</tr>
<tr>
<td></td>
<td>L300</td>
</tr>
<tr>
<td>SUZUKI</td>
<td>SWIFT</td>
</tr>
<tr>
<td>AUDI</td>
<td>80</td>
</tr>
</tbody>
</table>
2.2.2 Characterization methods

2.2.2.1 Classification

There are no accepted standards so far for the characterization of polymer waste for recycling purposes. The characterization methods applied in this chapter were performed on the basis that the characterized properties of the polyolefins can provide useful specifications to mechanical recycling processes, in particular for MDS.

The procedures of characterization conducted in this study for household wastes are introduced in Fig. 2.4. The household samples were first classified by inspecting the resin identification code on the objects into the following groups: rigid PP, LDPE, HDPE, non-polyolefins and films. The polyolefin groups were visually identified and divided, based on their shape (bottles versus the others), into BM and IM objects which were the two main manufacturing processes for making polyolefin parts.

Fig. 2.4 Flow diagram of characterizing procedures of household polymer wastes.

The grouped BM objects from each polyolefin type were divided further into subgroups based on their weight: >100 g, 70-100 g, 45-70 g, 35-45 g and <35 g; however, the IM objects were grouped mainly on the basis of their shapes and functions since IM were more widely applied such as caps, lids, boxes and buckets. An object was selected randomly for every 10-15 objects in each subgroup, the total number depending on the size of the
Characterization of Polyolefins

subgroups, to represent the subgroup for determining their physical properties, such as density distribution, wall thickness and rheological behaviour. These objects constitute Sample I. Examples of Sample I objects are shown in Fig. 2.5. The amounts of Sample I from both RO HW and NL HW are listed in Table 2.3. Flakes were cut from each object for density and viscosity analyses. An example of the selected flakes, with some of their related characteristics, is reported in Fig. 2.6.

<table>
<thead>
<tr>
<th>Sources</th>
<th>PP</th>
<th>LDPE</th>
<th>HDPE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blow mould</td>
<td>RO</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>NL</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Injection mould</td>
<td>RO</td>
<td>23</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>NL</td>
<td>36</td>
<td>4</td>
</tr>
</tbody>
</table>

Fig. 2.5 Example objects of Sample I from household wastes: container bottles (a, left) and caps (b, right), respectively.

The remaining material of each subgroup (Sample II) was shredded separately subgroup by subgroup to approximately 8 mm flakes for the analysis of their density distributions. This material constitutes Sample II.

Different from the packaging materials, which generally have clear polymer type marks on objects, the polymer types of the dismantled car components are not easily identified just on the basis of the components. One common method to identify polymer types is using Fourier transform infrared spectroscopy (FTIR). However, due to the fact that most of the polymer components from cars are black and often they contain lots of fillers or coating, no high quality spectrum of the samples was obtained with FTIR. Another solution is to recognize the polymer types according to the IDIS system. The received car components were first gathered on the basis of the brands and series, then by selecting the right functionality of them the materials of the components are provided by the IDIS database. A flake was sampled from each component (Sample III) in order to determine the polymer density.
Chapter 2

2.2.2.2 Density distribution
As the density distribution of the polymer types is the most critical parameter for PO recycling by using density separation, the main purpose of defining the density distribution was to establish the correct cut density. Since the density of polyolefins is in general lower than water, the density distribution of Sample I, II and III were determined using a series of water-ethanol mixtures with varying densities: from 880 to 1000 kg/m³ with 10 kg/m³ intervals. A flake of each object from Sample I and III and about 100 g split from each group of Sample II were used for these analyses.

2.2.2.3 Rheology characterization of packaging wastes
Rheological characterization on thermoplastic melts measures the material flow properties and provides vital information about polymer processing. BM materials are expected to be more viscous than would normally be employed for the IM process (Goodship, 2004). Tests were performed to study the rheological behaviour of the materials applied for BM and IM polyolefins especially at the melting temperature. The rheological properties of selected Sample I were tested in parallel plates with Ø8 mm by using the dynamic-mechanical rheometer SR-5000 from Rheometric Scientific which is stress controlled (Fig. 2.7). All the tests were performed on molten samples. The rheological properties were measured continuously as the sample underwent decreasing temperature-induced changes from molten to solid with a temperature gradient of 4 °C/min. The viscosity was determined within the linear range of the strain sweep and at a frequency of 1 Hz.
An example of the viscosity of PO flakes varying with temperature, measured with the Rheometer SR-5000, is given in Fig. 2.8. The viscosity of all polymers increases with decreasing temperature before the flake becomes solid again.

2.2.2.4 Wall thickness distribution of packaging wastes

The distribution of wall thickness, another mechanical property of plastic objects, has not yet been studied for any recycling purpose. The analysis of waste plastic flakes characterized by different thickness was carried out with the aim to find a correlation with the moulding process utilized for production: IM and BM. The possibility to identify the two typologies, that could be recycled separately, has been thus investigated. Because of the time-consuming analysis, only Sample I was measured for wall thickness by using a digital calliper.


2.3 RESULTS AND DISCUSSION

2.3.1 Types and moulds of polyolefins

2.3.1.1 Packaging waste

The household packaging sample from Romania consisted of 61.9% polyolefins (including PP, LDPE and HDPE), followed by 37.3% of PET, which were mainly beverage bottles, and a single bottle made of PVC, which is rarely used as packaging material (demonstrated in Table 2.4). The foils in this stream were labels from the beverage bottles. The Dutch packaging waste stream is more complex. The biggest fraction of the sample is plastic foils accounting for 39.0%. Similar to the RO HW, following polyolefins, PET is the second largest defined polymer type in the Dutch sample. In addition, a small amount of PS (2.8%) was found in this sample as well.

Table 2.4: Compositions of the household packaging plastic waste.

<table>
<thead>
<tr>
<th>Type of polymer</th>
<th>RO HW</th>
<th>Mass (kg)</th>
<th>Mass %</th>
<th>NL HW</th>
<th>Mass (kg)</th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP (rigid)</td>
<td>5.6</td>
<td>20.8</td>
<td></td>
<td>8.3</td>
<td>15.3</td>
<td></td>
</tr>
<tr>
<td>LDPE (rigid)</td>
<td>0.27</td>
<td>1.0</td>
<td></td>
<td>0.1</td>
<td>0.1</td>
<td></td>
</tr>
<tr>
<td>HDPE (rigid)</td>
<td>10.8</td>
<td>40.2</td>
<td></td>
<td>9.6</td>
<td>17.5</td>
<td></td>
</tr>
<tr>
<td>PET (rigid)</td>
<td>10.0</td>
<td>37.2</td>
<td></td>
<td>12.6</td>
<td>23.0</td>
<td></td>
</tr>
<tr>
<td>PS (rigid)</td>
<td>0.0</td>
<td>0.0</td>
<td></td>
<td>1.5</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>PVC (rigid)</td>
<td>0.04</td>
<td>0.1</td>
<td></td>
<td>0.0</td>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>Others (rigid)</td>
<td>-</td>
<td>-</td>
<td></td>
<td>0.7</td>
<td>1.3</td>
<td></td>
</tr>
<tr>
<td>Foils</td>
<td>0.18</td>
<td>0.7</td>
<td></td>
<td>21.3</td>
<td>39.0</td>
<td></td>
</tr>
<tr>
<td>Non-plastic</td>
<td></td>
<td></td>
<td>0.6</td>
<td>1.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>26.9</td>
<td>100</td>
<td></td>
<td>54.7</td>
<td>100</td>
<td></td>
</tr>
</tbody>
</table>

The concentrations of different polymer types are comparable for the two samples, if only rigid polymers are concerned (Fig. 2.9). In both samples, more than half of the objects are made of polyolefins, mainly PP and HDPE. PET is another commonly used material in packaging. Rigid LDPE is not popular in either case.

![Fig. 2.9 Composition of the rigid polymers from RO HW (left) and NL HW (right).](image)
In the polyolefin stream (see in Table 2.5), HDPE occupied more than half the amount of weight and > 94% of that was blow moulded used as bottles for shampoo, juice and laundry detergent for instance. In contrast, PP is generally found to be injection moulded. 78.0% and 95.8% in mass of PP in RO HW and NL HW respectively was injection moulded. Only a few LDPE objects were discovered for both mould types.

Table 2.5: Mass of blow and injection polyolefin moulds from packaging wastes.

<table>
<thead>
<tr>
<th>Resource</th>
<th>Polymer</th>
<th>Mass (kg)</th>
<th>Mass (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Injection</td>
<td>Blow</td>
</tr>
<tr>
<td>RO HW</td>
<td>PP</td>
<td>4.38</td>
<td>1.24</td>
</tr>
<tr>
<td></td>
<td>LDPE</td>
<td>0.02</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>HDPE</td>
<td>0.6</td>
<td>10.15</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>5.00</td>
<td>11.64</td>
</tr>
<tr>
<td>NL HW</td>
<td>PP</td>
<td>8.0</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>LDPE</td>
<td>0.1</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>HDPE</td>
<td>1.5</td>
<td>7.9</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>9.6</td>
<td>8.3</td>
</tr>
</tbody>
</table>

*Number with high statistic error.

The injection moulded polyolefin objects represent a wide variety of applications: buckets, caps, yoghurt jugs and so on (Fig. 2.10). Most of the caps in these two waste streams were originally connected with the bottles, but they were removed for characterizing. That explains why these small objects are also found in the RO HW which targets on > 5 cm objects. The NL HW consists of considerable amounts of fast food and meat boxes, although they were not preferred to be collected. However, similar items were not in RO HW. One possible reason is that they are not worth hand-picking, since they weigh little in comparison with the others.

Fig. 2.10 Functionality distribution of the injection moulded polyolefins from RO HW (left) and NL HW (right).
From the point of the manufacturing method, more than 80% in mass of IM products was PP; on opposite, the most popular material for blow moulds was HDPE (Fig. 2.11). These statistics imply that the sorting process on polyolefin types can also separate them by mould types.

![Fig. 2.11 Distribution of polyolefins in blow and injection moulds in RO HW (left) and NL HW (right).](image)

In addition to the rigid polymers, the foils in NL HW were also identified for their types. However, most of the foils, > 90%, did not contain a valid polymer type mark. According to the analysis on the marked polyolefin foils, LDPE is the most favourable material for packaging foils (Table 2.6). Further FTIR analysis on a sample of the unmarked foils shows that 34.0% of these foils are made of PE, and 19.5% of them are PP.

<table>
<thead>
<tr>
<th>Marked foils</th>
<th>Mass kg</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP</td>
<td>0.23</td>
<td>11.6</td>
</tr>
<tr>
<td>LDPE</td>
<td>1.51</td>
<td>76.2</td>
</tr>
<tr>
<td>HDPE</td>
<td>0.24</td>
<td>12.2</td>
</tr>
<tr>
<td>Total</td>
<td>1.98</td>
<td>100</td>
</tr>
</tbody>
</table>

**Table 2.6 Composition of the marked polyolefin foils in NL HW.**

### 2.3.1.2 Car waste

The characterization of the car wastes demonstrates that most of the analysed components are PP (see in Table 2.7). Except for the American Ford series, PP was the only chosen material for bumpers. Dashboard was another application of PP. PE is only used to produce water tanks based on this investigation. ABS was the most prevalent polymer for the inner components in a car, especially the air conditioning related parts. Concerning the mass ratio of PP and PE according to the samples investigated in this analysis, it is questionable whether car polyolefin wastes are worth separating. It is known from industry
practice, however, that there is a real interest in separating PE and PP from automotive shredded residues (ASR) which contains considerable amounts of polyolefins, and the ratio between PP and PE may reach 70:30 (Bakker et al., 2009). Further research on the composition of the car components on the basis of the IDIS system shows that most of the existing PE products in cars were less than 500 g. For instance, the air ducts in Audi 80 and Fiat Croma were all less than 400 g. Some of the cars even did not contain PE parts: e.g. Daewoo.

2.3.2 Density distribution

2.3.2.1 Packaging waste

The density distribution of objects in Sample I is presented in Table 2.8. In both streams, the density of PP mainly ranged between 890 kg/m³ and 920 kg/m³, which was lower than that of most HDPE covering from 940 kg/m³ to 980 kg/m³. Different from the RO HW, there were high density PP objects (> 920 kg/m³) in the NL HW. These high density PP were mostly from lids and boxes that were collected only in the NL HW. The density of LDPE distributing between 910 kg/m³ and 950 kg/m³ overlapped with both PP and HDPE. However, considering the small amount of the rigid LDPE in the packaging wastes and the fact that LDPE is often used as compatibiliser to make PP/HDPE mixed blends, it is not suggested to collect it in a separated product, but instead mixing with either PP or HDPE product.

A similar distribution is also observed in Sample II (see in Fig. 2.12). The peaks of the density distributions of PP and HDPE from RO HW were at a density of 890-900 kg/m³ and 940-950 kg/m³, respectively. For PP in NL HW, the peak shifted to 900-910 kg/m³. For both cases, the range of overlap density of all the three polyolefins was 920-930 kg/m³. Nevertheless, the sharp density distinction between PP and HDPE indicates that the appropriate cut density for recycling polyolefins using density separation should be between 920 kg/m³ and 930 kg/m³.

As expected, PP and HDPE were found respectively in the high and low density ranges in Sample II. For instance, 1% of HDPE in RO HW was discovered in the density range of 910-920 kg/m³. Considering the small amounts of polymers present in the “wrong” part of the distribution of Sample II, one possible reason is that some of the samples mixed with those from other groups during shredding, even though careful attention was paid to cleaning after each group. The second reason can be that it is practically difficult to have an absolutely accurate density analysis on small amount of shredded samples. Finally, it is known from experience in the field that the resin ID code on packaging objects is not always correct: apparently packaging manufacturers occasionally change resin type without changing the identification code. The existence of a small amount of high density PP, in particular in NL HW could be real since fillers may substantially increase the density of PO. The particles with a density of more than 1000 kg/m³ were contaminants from paper and labels.
## Table 2.7 Polymer type of the car components.

<table>
<thead>
<tr>
<th>Car</th>
<th>Components</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Air conditioner</td>
</tr>
<tr>
<td>OPEL</td>
<td>VECTRA</td>
</tr>
<tr>
<td>FIAT</td>
<td>CROMA</td>
</tr>
<tr>
<td>FORD</td>
<td>ESCORT</td>
</tr>
<tr>
<td></td>
<td>ORION</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>DAEWOO</td>
<td>NEXIA</td>
</tr>
<tr>
<td>NISSAN</td>
<td>PRIMERA</td>
</tr>
<tr>
<td>MAZDA</td>
<td>323</td>
</tr>
<tr>
<td></td>
<td>623</td>
</tr>
<tr>
<td>VOLKSWAGEN</td>
<td>GOLF</td>
</tr>
<tr>
<td>MITSUBISH</td>
<td>LANCER</td>
</tr>
<tr>
<td></td>
<td>GLX</td>
</tr>
<tr>
<td></td>
<td>L300</td>
</tr>
<tr>
<td>SUZUKI</td>
<td>SWIFT</td>
</tr>
<tr>
<td>AUDI</td>
<td>80</td>
</tr>
</tbody>
</table>


Table 2.8 Number of polyolefin objects in household wastes distributed in different density classes (Sample I).

<table>
<thead>
<tr>
<th>Resource</th>
<th>Polymer type</th>
<th>Density classes (kg/m³)</th>
<th>&lt;880</th>
<th>880-890</th>
<th>890-900</th>
<th>900-910</th>
<th>910-920</th>
<th>920-930</th>
<th>930-940</th>
<th>940-950</th>
<th>950-960</th>
<th>960-970</th>
<th>970-980</th>
<th>980-990</th>
<th>990-1000</th>
<th>&gt;1000</th>
</tr>
</thead>
<tbody>
<tr>
<td>RO</td>
<td>PP</td>
<td></td>
<td>1</td>
<td>10</td>
<td>11</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>LDPE</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>3</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>HDPE</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>1</td>
<td>12</td>
<td>8</td>
<td>4</td>
<td>3</td>
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<td></td>
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<tr>
<td>NL</td>
<td>PP</td>
<td></td>
<td>2</td>
<td>8</td>
<td>14</td>
<td>7</td>
<td>4</td>
<td>1</td>
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<tr>
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<td></td>
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<td>1</td>
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<td></td>
<td></td>
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<tr>
<td></td>
<td>HDPE</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>1</td>
<td>10</td>
<td>2</td>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
For the RO HW, there was no significant density difference for BM and IM from the same polymer type. This is true for PP, HDPE and LDPE (see in Fig. 2.13 a, b and c). The amounts of BM PP and IM HDPE were too small to make a similar conclusion for NL HW (Fig. 2.13 d, e and f). In Fig. 2.13 f, 30% of the HDPE IM has a density smaller than 920 kg/m³, which should be the density range of PP. However, similar phenomenon was not observed in the density analysis on Sample I of NL HW. Hence the HDPE IM could be contaminated with some materials from other groups during shredding.

Fig. 2.12 Density distribution of different rigid (with thickness > 0.2-0.3 mm) polyolefins of Sample II (above: RO HW; below: NL HW).
Fig. 2.13 Density distribution of Romanian BM and IM: PP (a), LDPE (b) & HDPE (c) and Dutch BM and IM PP (d), LDPE (e) & HDPE (f) of Sample II (continued).
Fig. 2.13 Density distribution of Romanian BM and IM: PP (a), LDPE (b) & HDPE (c) and Dutch BM and IM PP (d), LDPE (e) & HDPE (f) of Sample II (continued).
Fig. 2.13 Density distribution of Romanian BM and IM: PP (a), LDPE (b) & HDPE (c) and Dutch BM and IM PP (d), LDPE (e) & HDPE (f) of Sample II.
Fig. 2.14 shows the density features of BM and IM polyolefins. Most of the injection moulds concentrated in the low density range, and the blown ones in the high density area. This result suggests the possibility of sorting blow and injection moulded polyolefins by density separation.

![Density distribution of BM and IM of Sample II (above: RO HW; below: NL HW).](image)

2.3.2.2 Car waste

The density distribution of the polymers from the car waste sample is more complex than those from packaging (Fig. 2.15). PP, the major material in Sample III, covers almost the entire density range. The density range 880-920 kg/m³, where the packaging PP concen-
trates, contains not only PP but also the so-called PC-PBT blend (marked with white dots in Fig. 2.15). However, it is not possible to obtain PC-PBT blend with such low density, as the typical density of PC and PBT is well above 1000 kg/m³. It is thus concluded that the information of the bumpers from Ford Escort provided by the IDIS system may be incorrect. 13.6% of PP has a density larger than 1000 kg/m³, which is not the suitable density for the MDS process of polyolefin separation. PP with such high densities shows that in car applications PP is probably reinforced. The PE, from the two blow moulded water tanks, have a typical PE density: 930-950 kg/m³. However, it is not clear whether the < 500 g PE components are also within the same density range as these two collected PE samples. Nevertheless, we can conclude that by dismantling the reinforced PP before any further sorting processes, a pure PP product can be obtained and also can avoid mixing with other types of polymers. The other PP, with a density less than 1000 kg/m³, can be recycled with MDS: extracting the particles with a density between 930 kg/m³ and 950 kg/m³ as one product, and the rests as other products.

Fig. 2.15 Density distribution of polymers from the car wastes.

2.3.3 Rheology characterization of packaging wastes

As shown in Fig. 2.16, polyolefins of packaging are easily identified by their melting temperature. LDPE, especially the blow mould types, has low melting temperature compared to the other two types of polyolefins. PP starts to melt from nearly 125 °C but some can stay solid even at a temperature higher than 140 °C. HDPE has a relatively narrow melting temperature range, in general between 118 °C and 125 °C.

The rheological behaviour of Sample I demonstrates that BM products are generally more viscous than the injection ones. Almost all the BM polyolefins have higher viscosity
than the IM of the same type. Hence, apart from sorting polyolefins on types for improving the mechanical properties, it makes sense to separate BM and IM from each other to avoid differences in viscosity that reduces homogeneity of the new products made from secondary polyolefins and results in polymers with an off-spec viscosity.

Fig. 2.16 Viscosity of selected Sample I (RO HW) at melting temperature.

### 2.3.4 Wall thickness distribution of packaging wastes

The wall thickness distributions of PP, LDPE and HDPE in both waste streams are similar (Fig. 2.17). In both wastes, almost all of the rigid packaging objects have a wall thickness above 0.4 mm. A small amount of PP from NL HW is thinner than 0.4 mm. Further investigation shows that they were all with a form of box or lids. This indicates that polyolefin flakes from rigid packaging materials thinner than 0.4 mm are very likely to be PP.

The material wall thickness distributions of both blow and injection polyolefin objects are shown in Fig. 2.18. The thickness of BM polyolefin objects was in general around 0.5-1 mm, and all the sampled BM polyolefins had a thickness less than 1.1 mm; the IM, however, had a wider range: from 0.2 mm up to 3 mm. This difference of these two types in thickness allows some sorting by flake thickness to distinguish blown and injected polyolefins: the flakes thicker than 1.1 mm are from IM products, but the fraction thinner than 1.1 mm remains as a mixture of blow and injection moulds.
Although injected polyolefin items were found in the less than 1.1 mm fraction as well, the amount of them in mass percentage is low since they are relatively small and light compared to these blown bottles. Fig. 2.19 illustrates the mass distribution of the samples correlated with their wall thicknesses. Most of the IM objects were lighter in mass than the BM ones. As a result, the Mass percentage of these IM objects in the < 1.1 mm fraction is small. For HDPE in particular, in addition to a pure IM product, sorting by flake thickness at 1.1 mm increased the grade of BM in mass in the < 1.1 mm fraction (Fig. 2.20).
Fig. 2.19 Mass distribution of the objects in sample I (from both RO and NL HW) correlated with wall thicknesses.

Fig. 2.20 Mass percentage of BM and IM polyolefins of RO HW Sample I before (up), after sorting by flake thickness in the < 1.1 mm fraction (below left) and > 1.1 mm fraction (below right), respectively.
2.4 CONCLUSIONS

Characterization of waste streams should reflect the parameters that are important for predicting the result of recycling processes and the quality of the recycled products. In the polyolefin packaging stream, rigid PP and HDPE are in general found to be injection and blow moulded, respectively. The density difference between PP and HDPE indicates that density separation can sort these two types of polyolefins and the appropriate cut density should be between 920 kg/m³ and 930 kg/m³. In addition, the separation inaccuracy for such technologies, e.g. MDS, is recommended to be lower than 10 kg/m³ to obtain products with both high grade and high recovery. Packaging polyolefins can be identified by their melting temperatures, and the rheological properties distinguish their moulding procedures and moreover indicate the necessity of sorting not only on types but also on manufacture processes (blow moulding and injection moulding) for the purpose of reaching secondary polyolefin products with good mechanical properties. The difference of BM and IM in terms of wall thickness distribution allows some sorting by flake thickness, for instance by using ballistic separation.

The second main source of polyolefin wastes is car components. More than 100 kg of the dismantled car components from ten brands and 14 series were studied for their properties. The characterization of the car waste demonstrates that most of the analysed components are PP. The density distribution of the PP components covered from 880 kg/m³ to even > 1000 kg/m³. That is probably because some of the PP is reinforced for certain applications. By dismantling the reinforced PP before any further sorting processes, a pure PP product can be obtained and also can avoid mixing with other types of polymers. The PE, from the two blow moulded water tanks, have a typical PE density: 930-950 kg/m³. According to the density distribution, it is possible to remove PE from the PP mixtures less than 1000 kg/m³: extracting the particles with a density between 930 kg/m³ and 950 kg/m³ as one product, and the rests as other products.
Chapter 3

Magnetic Density Separation
3.1 PRINCIPLE OF MDS

The basic principle of MDS is to use a magnetic liquid as the separation medium. The magnetic liquid is a colloidal liquid made of nanometre-size ferromagnetic particles suspended in a carrier liquid, in this case water. It has a material density similar to water. However, when such a liquid is in a magnetic field, the weight of the liquid becomes the sum of gravity and the vertical (z) component of the magnetic force. In such way, the separation medium can be artificially lighter or heavier than would be expected on the basis of its material density ($\rho_l$). In a gradient magnetic field ($B$), the total weight ($F$) of a volume of magnetic liquid ($V_l$) with magnetization $M$ is:

$$F = \rho_l g V_l + M V_l \nabla |B| = \left( \rho_l + \frac{MB}{g} \right) g V_l$$

When particles made of a non-magnetic material of material density $\rho_p$ are introduced into the liquid, their weight will be equal to their gravity minus the weight of the same volume of liquid (Archimedes’ Law):

$$F = \rho_p g V_i - \left( \rho_l g V_i + M V \nabla |B| \right)$$

In particular, the particles will be suspended (weightless) if (see Fig. 3.1)

$$\rho_p = \rho_{eff} = \rho_l \pm \frac{M}{g} \frac{d|B|}{dz}$$

The magnetic liquid therefore acts on non-magnetic particles in the same way that an ordinary liquid would act with density $\rho_{eff}$. In situations like in Fig. 3.1.a, where the gravity and the magnetic force work in opposite directions, the effective density ($\rho_{eff}$) becomes less than $\rho_l$:

$$\rho_{eff} = \rho_l + \frac{M}{g} \frac{d|B|}{dz}$$

The gradient of $|B|$ decreases in size with the distance to the magnet that produces the field. Therefore particles of different densities are suspended at different heights. In this way, the MDS can be used to sort light polymers (polymers with different densities less than that of $\rho_l$), particularly polyolefins. Furthermore, the effective medium density becomes larger than $\rho_l$ when the gravity and the magnet force are in the same direction (Fig. 3.1, b). Heavy polymers (polymers with a density higher than that of $\rho_l$), such as PVC and PS, can be separated in this way.
Magnetic Density Separation

Fig. 3.1 Forces on magnetic liquid in a magnetic field. Yellow arrow is magnetic force; blue arrow is gravity; red arrow is total force.

MDS uses magnets with a specially designed field that is constant in size in planes parallel to the magnet surface (x-y plane) and decreases exponentially away from the surface (z-axis). If the magnetic induction is equal to $B_0$ at the surface of the magnet, and $p$ is the pole-size:

$$|B(x, y, z)| = B_0 e^{-\pi z/p}.$$  

Therefore, the variation of $\rho_{\text{eff}}$ can be described with Eq.(3.3) and Eq.(3.4) for light and heavy polymers respectively.

$$\rho_{\text{eff}} = \rho_l - \frac{\pi MB_0}{gp} e^{-\pi z/p}, \quad (3.3)$$

$$\rho_{\text{eff}} = \rho_l + \frac{\pi MB_0}{gp} e^{-\pi z/p}. \quad (3.4)$$

Such magnets can be built in two ways. The first method invented by Rem and Berkhout in 2007 is able to produce a magnet with a $B_0$ of 0.6 T (Rem and Berkhout, 2007). In 2011, the other design uses only one third of FeNdB of the former (Lahaye et al., 2011), and such type of magnet has a $B_0$ of 0.21T. More detailed comparison between these two magnets is introduced in Chapter 7.

The MDS approach to density separation is by utilizing such feature of magnetic liquid to create a medium with an artificial density that varies only in the vertical direction. Fig. 3.2 presents an example of the variation of the effective density of magnetic liquid with $z$. Particles in such liquid will suspend at the location where the effective density of the liquid is equal to that of the particles (equilibrium position). As a result, particles with the same density float at the same height as shown in Fig. 3.3.
Fig. 3.2 Variation of the effective density of magnetic liquid ($\rho = 1006 \text{ kg/m}^3$, $M = 500 \text{ A/m}$) with $z$, in a magnetic field with $B_0 = 0.6 \text{ T}$ and $p = 0.12 \text{ m}$.

Fig. 3.3 Polymer balls with different densities suspended in magnetic fluid.

### 3.2 POLYMERS IN MDS

In magnetic density separation, the polymer particles in the magnetic liquid take some time to reach the equilibrium height where their density is the same as the effective density of the liquid. The processing time is a result of the terminal velocity of particles which...
changes by the exponential gradient of the $\rho_{eff}$ in MDS. Typical Reynolds numbers for the particle motion in the liquid are between 10 and 300. The terminal velocity of a polymer particle with a thickness $d$ at a vertical coordinate $z$ is then determined by Eq.(3.5) (Bakker et al., 2009):

$$v_t = \sqrt{\frac{2(\rho_p - \rho_{eff})dg}{C_d \rho_l}}, \quad (3.5)$$

$C_d$ is the drag coefficient correlated to the Reynolds number ($Re$).

The change of terminal velocity and the processing time in magnetic liquid can be illustrated with the experimental movement of a polymer particle, which is made visible with ultrasound technology. A setup including a Mylab medical imager system for real-time ultrasound monitoring was constructed (Fig. 3.4). A glass tank of magnetic liquid (600 A/m) was placed on top of the magnet with a flat magnetic field. Polymer particles were released in a PVC tube with a diameter of 50 mm. The medical ultrasound sensor array probe (5 MHz, 128 elements) was placed on the top of the tube and the Mylab system was able to record the whole process with a rate of 29 frames per second. Three polystyrene (PS) flakes used for the tests had similar densities around 1033 kg/m$^3$ (Table 3.1).

**Table 3.1 Properties of samples used in the test of polymer movement in MDS.**

<table>
<thead>
<tr>
<th>Test</th>
<th>Polymer</th>
<th>Shape</th>
<th>Size (mm)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PS1</td>
<td>Square</td>
<td>10×10</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>PS2</td>
<td>Square</td>
<td>5×5</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>PS3</td>
<td>Square</td>
<td>5×5</td>
<td>2</td>
</tr>
</tbody>
</table>

As shown in the first graph in Fig. 3.5, the particle was clearly observed by using the Mylab system during the process. By stacking the pictures of each frame from the video, an MIP (maximum intensity projection) image of the particle trajectory in time was obtained and is presented in Fig. 3.5. b. Although the ultrasound noise in the picture is quite disturbing, the trajectory of the particle is clearly visible and shows that the flake was rising and its speed was decreasing during the process.
Chapter 3

Fig. 3.5 Snapshot of flake rising up after being released for about 1 second (a) and flake trajectory in time (b).

The trajectories of the three PS particles in the MDS were successfully predicted by the simulation based on Eq. (3.5) (Fig. 3.6). At the beginning of the process, the polymers were moving with a high speed of around 30 mm/s. They slowed down gradually when close to their equilibrium heights. Compared to the particles of 1 mm thick (PS1 and PS2), the 2 mm thick particle (PS3) rose faster as shown by both the experiments and the simulation. For PS1 and PS2, although their sizes differed, their movements were quite similar. For all the particles, four seconds was sufficient to complete the procedure, and the processing time can be less if the initial release height is closer to the equilibrium height. The three PS particles stopped at a similar height at the end of the process, because of their almost identical densities.

The separation principle and the polymer movement reveal that MDS is able to sort polymer mixtures based on density and to produce multiple products within a few seconds. In order to apply this technique in the market of polymer recycling, a continuous sorting process should be achieved with an MDS plant.
Magnetic Density Separation

3.3 MDS PLANT

An MDS plant for polymer or polyolefin recycling should comprise the following steps:

1. wetting and injecting polymer flakes to avoid air into the magnetic liquid;
2. flow in the separation channel of MDS where the separation is carried out should be at low turbulence;
3. products reaching the end of the separation channel should be split according to their floating height;
4. the entire system should be free of air bubbles and there should be no gravity waves affecting the flow in the separation channel;
5. clean the products and lose as little as possible the process liquid;
6. reuse of process liquid to avoid unnecessary discharge.

Considering all the above-mentioned points, an MDS plant should mainly consist of the sections of wetting & injection, flow laminarization, separation and product collection & washing/centrifuging.

3.3.1 Wetting and injecting particles

Wetting is an important first step in an MDS process for polymer recycling, because of the hydrophobicity of polymers (Hu et al., 2010). Therefore, the wettability of the polymers should be improved before they are injected into MDS. Thereafter, wetted polymers can be fed into the system by taking advantage of their natural tendency to float or sink in the
liquid medium. Hence, light polymers are injected from the bottom of the separation channel (Fig. 3.7, a), and heavy polymers enter from the top (Fig. 3.7, b).

![Fig. 3.7 Injection methods for light polymers (a) and heavy polymers (b).](image)

### 3.3.2 Turbulence reduction

As shown in Fig. 3.3, polymers with different densities intend to float at different vertical positions. Thus, ideally, the flow should have no vertical movement, and the injected polymers should move up and down only on the basis of their density. However, flow is often turbulent. To minimize turbulence in the separation channel, the flow can be fed through a series of parallel tubes with openings at the beginning of the channel to reduce the turbulence level. These tubes are recommended to cover the entire cross-section of the separation channel, and they should have the same resistance to avoid uneven flow.

![Fig. 3.8 Injection unit for light polymers (a) and heavy polymers (b) covered with laminators (the direction of the process liquid is marked with the solid arrows).](image)

### 3.3.3 Separation channel of MDS

The separation channel is the place where the polymer mixture is sorted. It should be a closed channel so the process liquid cannot move out of or into the channel from the sides, to avoid generating turbulence. The length of the channel is rather important. It should not be too short that particles have no sufficient time to reach their equilibrium position, and should be not too long that the flow structure from the laminators may disappear. Since fixed walls may create turbulence, both the top and the bottom of the channel can be covered with belts that are running with the same speed as the flow, in order to avoid turbulence. The top belt, in the case of MDS for light polymers, has also another function: to remove ferrous particles in the feed if there is any, to protect the magnet and to avoid material obstructing the flow. Hence, the surface of the top conveyor belt needs to contain a series of riffles, and the riffles can take the top flow moving the same speed as the rest. The sides of the channel, however, can in general be closed with two fixed plates. Since polyolefins are generally lighter than the density of the process magfluid, the magnet is placed on
the top of the channel and it should cover the complete channel. The process liquid introduced through the laminarization section flows along the length of the channel from left to right, carrying the polymers while they are reaching their equilibrium height.

### 3.3.4 Product collection

Polymers should be split at the end of the separation channel and collected in such way that the action does not destroy the existing product layers. Multiple splitters can be installed to obtain more than two products in one separation step. The polymer products are pumped out separately, together with process liquid.

To avoid undesired air bubbles and gravity wave, the entire system is immersed under liquid level in a tank. The tank is filled up with process liquid, which circulates within the system.

On the basis of the above discussion, the basic scheme of a MDS plant is given in Fig. 3.9.

![Fig. 3.9 Side view of MDS for light polymers (wetting section is not indicated).](image)

### 3.4 CONCLUSIONS

Chapter 3 presents the fundamental principle of MDS separation processes. MDS is similar to conventional sink-float processes. However, it uses a magnetic liquid that has an effective density gradient in a magnetic field, as the separation medium. By applying an upward or downward magnetic field into such liquid, the effective density of the liquid can be heavier or lighter than the liquid density, which is close to that of water. The MDS approach to density separation is by utilizing such feature of magnetic liquid to create a medium with an artificial density that varies only in the vertical direction. Particles in such liquid will float at the height where the effective density of the liquid is equal to that of the particles. Because of the optimal fluid dynamics, polymer particles can reach their equilibrium position within a few seconds.

An MDS plant should start by improving the wettability of the polymer feeding materials, and should include the injection methodology, control of the flow turbulence, process
liquid reuse and product collection and cleaning. On the basis of these discussions, a scheme of an MDS system is provided for continuous sorting processes.

To reach the necessary separation accuracy of 10 kg/m³ for gaining high quality secondary PP and PE in one single step, the separation technology needs to be sensitive to very small differences in the physical properties of the input materials. Moreover, in order to be economical and ecological, processes should recover most of the polyolefins in a useful product and minimize process residues. Therefore, further research on improving the separation accuracy of the MDS process was conducted. Three main aspects deserve attention: improvement of polymer wettability, magnetization control of the process liquid and minimization of turbulence.
Chapter 4

Wetting Technologies for MDS
4.1 INTRODUCTION

In order to become a practical method for polyolefin separation, MDS technology must reach an accuracy of 10 kg/m³ at commercially interesting production rates. A major factor in separation quality, next to minimization of turbulence and quality control of the process liquid, is the entrainment of air bubbles in the feed. The focus of this chapter is therefore on the efficiency of the wetting process, with the aim to eliminate air bubbles to less than 1% (contributing to roughly 10 kg/m³) of the volume of the flakes, in order to avoid flotation of particles or variation of effective material density, both of which would affect the recovery of the polyolefins or introduce contaminants into the products.

Traditional methods for wetting plastic flakes prior to sink-float separation are wet grinding or friction washing. Practice with industrial wetting steps based on these methods show that a significant fraction of the heavy materials still end up in the float fraction. For example, in the separation of the polyolefins from PET (1350 kg/m³), up to 2-5% of the input PET is found in the floats, showing that air bubbles may account for 35% of the volume of the thin flake. Experiments, in which the shear at the flake surface is increased by means of passing a high speed water flow (1 m/s) through a bed of plastics in between screens show a more efficient removal of air, but such a procedure is difficult to implement as a continuous process. An alternative to mechanical methods is to add surfactants to the process water or mix in compounds of intermediate hydrophilicity, such as alcohol. Such additives are known to effectively wet the plastics, but a disadvantage is that surfactants end up in the process liquid. In the case of MDS, the process liquid already contains a surface active component to stabilize the suspension of nanometer-size magnetite particles that make the liquid magnetic. The addition of surfactants for wetting is therefore compromising the quality of the process liquid.

A third route to wet the surface of hydrophobic materials is by means of contacting with steam or boiling water. In these methods, the air is carried away with the steam or water, and the surface of the particles is completely covered with water. The major issue with this route is that the magnetic process liquid degrades at high temperatures and the separation depends on the degree of dilution. This means that in carrying the plastics from the steam or hot water to the process liquid, a minimum of heat and water should be carried with it.

4.2 EFFECT OF AIR BUBBLES

The most critical property of the polyolefin concentrates for density separation process is the distribution of density of the various PP and PE components (Table 4.1). These distributions relate directly to the grade and recovery that can be accomplished with a certain cut density. About 94% of the PP from RO HW has a density lower than 920 kg/m³, whereas more than 85% of the LDPE and more than 98% of HDPE have a density higher than 930 kg/m³. A sink-float process with a medium density of 920-930 kg/m³ would therefore give good results for both the PP and the PE products, as concluded in Chapter 2. With a separation accuracy of 10 kg/m³, both PP and PE recovery can reach 94% with
an acceptable contamination of PE and PP respectively. For an accuracy of 20 kg/m³, the PP recovery hardly changes, but the grade of the PP product will be lower, since 10% of the total amount of HDPE now ends up in the wrong product. On the basis of these analyses, the allowable amount of air on a polyolefin flake is approximately 1% in volume of the total volume of the flake.

Table 4.1 Composition of PP, LDPE and HDPE in the different density fractions of RO HW.

<table>
<thead>
<tr>
<th>Density Fraction (kg/m³)</th>
<th>PP (mass%)</th>
<th>LDPE (mass%)</th>
<th>HDPE (mass%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 900</td>
<td>64.9</td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>900-910</td>
<td>21.7</td>
<td>0.8</td>
<td>0.7</td>
</tr>
<tr>
<td>910-920</td>
<td>7.6</td>
<td>4.0</td>
<td>0.6</td>
</tr>
<tr>
<td>920-930</td>
<td>2.5</td>
<td>10.0</td>
<td>0.4</td>
</tr>
<tr>
<td>930-940</td>
<td>0.3</td>
<td>48.0</td>
<td>10.4</td>
</tr>
<tr>
<td>940-950</td>
<td>1.9</td>
<td>1.5</td>
<td>51.5</td>
</tr>
<tr>
<td>950-960</td>
<td>0.9</td>
<td>4.7</td>
<td>30.8</td>
</tr>
<tr>
<td>960-1000</td>
<td>0.2</td>
<td>0.0</td>
<td>5.4</td>
</tr>
<tr>
<td>Total</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
</tr>
</tbody>
</table>

The effective density (or apparent density) of a flake covered with air bubbles (ρ_p_eff) is determined by the volume of air as a function of the volume of the flake (ζ_air):

\[ \zeta_{air} = \frac{V_{air}}{V_{flake} + V_{air}}, \]

and

\[ \rho_{p, eff} = \rho_p \cdot (1 - \zeta_{air}). \]

As shown in Fig. 4.1, the effective density of the flakes decreases quickly with the number of air bubbles. As little as two air bubbles with a total volume of 1 mm³ is able to create a separation inaccuracy of 10 kg/m³. Polymer particles with a density between 930-940 kg/m³ are then likely to mix with the PP product.

4.3 **WETTABILITy IMPROVEMENT**

Contacting with steam or boiling water may affect the wettability of plastic flakes in several ways. If the flakes are passed from an air atmosphere into pure steam and contacted with the steam for several seconds, a thin but continuous layer of condensate may form on the surface. If such a thin film proves sufficiently stable against breaking up into individual droplets, the treated plastics may be mixed with the magnetic liquid straight away. Such a process would transfer a minimal amount of heat and water to the flakes and therefore to the magnetic liquid. At the same time, it would be a fast and relatively simple process. Another possibility is to allow for a longer contact of the flakes with steam or boiling water.
Fig. 4.1 Effective separation density of typical plastic flakes (100 mm$^3$) covered with air bubbles with a diameter of 1 mm.

In this case, the plastics will be heated and a significant amount of water and heat is transferred to the flakes. In order to avoid heating and diluting the magnetic liquid, the flakes would then need to be cooled and at least partially dewatered before mixing them with the magnetic liquid. The most interesting issue in such processes is to see whether it is possible to create permanent or semi-permanent changes of the physical or chemical state of the flake surface, so that the wettability of the flakes remains high even after cooling and dewatering.

If a cold flake is introduced into a pure steam atmosphere, the so-called laminar boundary layer of air will mix with the steam in about 0.01 s. As the flakes pass from the air into the steam, this thin boundary layer of air (typically $10^{-4}$ m, estimated based on the Blasius solution for laminar boundary layer thickness) will move with the flake surface. The time of diffusion between air and water vapour is (Wesselingh and Krishna, 1990):

$$t = \frac{\delta_{\text{air}}^2}{D_{\text{air-vapour}}},$$

in which $\delta_{\text{air}}$ is the thickness of the laminar air layer and $D_{\text{air-vapour}}$ is the air-vapour diffusion coefficient ($10^{-5}$ to $10^{-6}$ m$^2$s$^{-1}$) (Nellis and Klein, 2008). The equation shows that air is able to diffuse so fast into the surrounding steam that the air will not essentially influence the condensation process.

After the air has been dissolved into the steam, the flake will be covered by a film of water which is caused by water vapour condensation. The vapour will condense on the surface of the flake at a rate which is controlled by the flux of heat into the interior of the flake. For the computation of the film thickness, the thermal capacity of the condensate
film can be neglected. The water film thickness may be computed by numerical or analytical methods, using the equation for heat transfer in the interior of the flake:

\[
\frac{dT}{dt} = \frac{\lambda_p}{C_p \rho_p} \frac{\partial^2 T}{\partial x^2}.
\]  

(4.1)

The parameters of this equation are the polymer thermal conductivity \( \lambda_p \), the heat capacity \( C_p \) and the density \( \rho_p \). The boundary condition at the surface is assumed to be the condensation temperature of the steam, i.e., \( T = 100 \) °C. The rate of condensation is controlled by the flux of heat (\( \Phi \)) at the surface:

\[
\Phi = -\lambda_p \frac{\partial T}{\partial x}.
\]

In the simulation presented in Fig. 4.2, the PP and PE flakes were 1 mm thick, which is close to the flakes used in the experiments, and the initial temperature of the polymer was 20 °C. The properties of PP and PE and the detailed information for the simulation are presented in Table 4.2.

<table>
<thead>
<tr>
<th>Table 4.2 Parameters in the simulation.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parameter</td>
</tr>
<tr>
<td>----------------------------------------</td>
</tr>
<tr>
<td>Thermal conductivity (J/s-1K-1m-1)</td>
</tr>
<tr>
<td>Density (kg/m³)</td>
</tr>
<tr>
<td>Heat capacity (J/kg·K⁻¹)</td>
</tr>
<tr>
<td>Area (m²)</td>
</tr>
<tr>
<td>Heat of water vapour (kJ/kg)</td>
</tr>
<tr>
<td>Temperature (°C)</td>
</tr>
<tr>
<td>Thickness (mm)</td>
</tr>
</tbody>
</table>

The numerical analysis shows that the temperature of PE rises faster than PP, as a result of the higher thermal conductivity and the slightly lower heat capacity. The temperature of PE reaches the maximum in about 1 s.

The total heat content of the flake \( H_t \) at time \( t \) relates to the thickness of water vapour (\( \delta_w \)) on the polymer flake with unit area:

\[
\delta_w = \frac{H_t - H_0}{\rho_w \cdot Q_w},
\]

in which \( \rho_w \) is the density of water and \( Q_w \) is the heat of evaporation.
The final water film for both PP and PE flakes is around 60 μm as predicted numerically (Fig. 4.3), which is 6.5% of the polymer mass, and a thin film of 10 μm could be established within less than a second. The ultimate film thickness is the same for PP and PE because of the similar heat capacities of both polymers.

Analytically, the initial development of the water film thickness may be estimated by assuming that the heat will flow into the polymer surface to some depth $x$. It then follows, on the basis of Eq.(4.1), that:

$$\frac{dT}{dt} = \frac{\Delta T}{\Delta t} = \frac{\alpha \cdot \lambda_p}{C_p \rho_p \cdot x^2} \cdot \Delta T.$$
\[ x = \sqrt{\frac{\alpha \cdot \frac{\lambda_p}{C_p} \cdot \Delta t}{\rho_p \cdot T}} \]

where \( \alpha \) is a geometrical factor, depending on the dimension of the particle (\( \alpha \approx 6 \) for sheet). The heat \( H_t \) absorbed by the plastic flake is a function of \( x \) and \( \Delta T \):

\[ H_t = x \cdot \rho_p \cdot C_p \cdot \Delta T \cdot A. \]

Since the heat released by the water vapour due to the condensation is the same as the energy absorbed by the polymer flakes (with area \( A \)) the water film thickness \( \delta_w \) is given by:

\[ \delta_w = \sqrt{\frac{\rho_w \cdot H_{\text{vapour}}}{\rho_p \cdot C_p \cdot \Delta T \cdot A}} \cdot \sqrt{\frac{\alpha \cdot \frac{\lambda_p}{C_p} \cdot \Delta t}{\rho_p \cdot T}}, \]

in which \( H_{\text{vapour}} \) is enthalpy of water vaporization (2400 kJ/kg). To a good approximation, the film thickness in the initial growth phase of the condensate film is (see Fig. 4.3):

\[ \delta_w = \sqrt{\frac{\alpha \cdot \lambda_p}{\rho_w \cdot H_{\text{vapour}}} \cdot \frac{\Delta t}{\rho_p \cdot C_p \cdot A}}. \quad (4.2) \]

Note that \( \Delta T \) here is assumed with the temperature difference between the water vapour and the initial temperature of the flake, thus the estimation of \( \delta_w \) is only true at the beginning when the temperature of the flake has not raised too high. After a short while, a few seconds, the temperature difference between the flake and water vapour becomes less than \( \Delta T \), so Eq.(4.2) is no longer valid for such situation. Both of the numerical and analytical calculation shows that a thin film of water should form on flake surfaces in less than a second.

### 4.4 MATERIALS AND EXPERIMENTS

A series of experiments were conducted to observe the formation of the water film on flake surfaces. The materials used in the experiments included virgin PS, PP, LDPE & HDPE, shredded PET, HDPE and two types of shredded PS. Examples of the shredded samples are given in Fig. 4.4. Complete descriptions of these samples are given in Table 4.3. One of the shredded PS samples (PS1) consisted of simple flakes of 10-15 mm diameter and 0.5-1 mm thickness. Without wetting, 15% of this material floats in cold water. The other PS sample (PS2), with the same particle size range, contained a large proportion of twin-layer material of which some particles were partially split, forming a pocket of air in between the two layers. As a result, the floats of this sample reached 32%. The PET sample had a particle size between 5 and 10 mm and a thickness of 0.5 mm. The amount of floats of PET is 4%, much lower than those of PS1 and PS2. But considering the fact that PET is hydrophilic (with water contact angle < 90º), the wettability of it is still worrying.
Table 4.3 Basic data of the shredded polymer materials.

<table>
<thead>
<tr>
<th>Source</th>
<th>PET</th>
<th>PS</th>
<th>HDPE</th>
<th>LDPE</th>
<th>PP</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Secondary</td>
<td>Virgin</td>
<td>PS1</td>
<td>PS2</td>
<td>Secondary</td>
</tr>
<tr>
<td>Waste bottles</td>
<td>-</td>
<td>Thermoforming PS-trays</td>
<td>Extrusion grade</td>
<td>Household wastes</td>
<td>-</td>
</tr>
<tr>
<td>Density (kg/m³)</td>
<td>1350</td>
<td>1050</td>
<td>1050</td>
<td>1050</td>
<td>940</td>
</tr>
<tr>
<td>Float (mass%)</td>
<td>4</td>
<td>41</td>
<td>15</td>
<td>32</td>
<td>-</td>
</tr>
<tr>
<td>Water contact angle (°)</td>
<td>80</td>
<td>96</td>
<td>96</td>
<td>97</td>
<td>96</td>
</tr>
</tbody>
</table>


Two types of process equipment were tested: a steam tube and a screw conveyor with steam injection. The steam tube consisted of a vertical section of transparent pipe, 0.4 m high and 12 cm diameter, in which the particles were fed from the top by a screw conveyor. The falling particles were contacted with a counter-current flow of low-pressure steam (see Fig. 4.5). After passing through the steam in about 0.3 s, the particles were collected in a stirred reservoir with cold water directly below the tube.

A second type of equipment, an inclined screw conveyor with steam injection, is shown in Fig. 4.6. The feed was introduced in the middle, together with a co-current flow of steam, and the material was again collected in a stirred reservoir of cold water. Part of the condensate was collected at the lower end of the conveyor.

The tests with the steam tube and steam screw were done with the shredded PS samples and the results were analysed in terms of the fraction of floats. The reason of choosing PS is because their density is about 1050 kg/m³, so even a minor amount of air of 5 vol% will result in floating of these flakes. The tests with single particles were done on all sam-
samples. The results of these tests were analysed in terms of the volume of air bubbles, the water contact angles and the number of bubbles attached on a flake. Apart from the tests with the two types of process equipment, single particles were tested for wettability by contacting them with steam or boiling de-mineralized or tap water for periods of 10-60 s.

4.5 RESULTS AND DISCUSSION

4.5.1 Wetting with steam

Introductory tests were performed with the steaming tube on samples of shredded PS. It was very difficult to feed the flakes into the flow of steam individually because of the formation of sticky agglomerates of flakes due to condensing water near the exit of the screw. Flakes that were collected in the stirred reservoir showed very poor wetting. It was therefore suspected that the thin film of water produced by the contact with the steam is not stable. Later experiments with the steaming of individual flakes showed that, indeed, the water film quickly breaks up into droplets.

A second series of experiments were performed with both types of shredded PS (PS1 and PS2) by using the setup shown in Fig. 4.6. Generally, the mass percentage of the sink fraction increased with the residence time. More than 90 mass% of PS1 was well wetted after one minute, and 96%-99% after 2 to 3 min (Table 4.4). As expected, higher steam power compensates for shorter residence time. However, wetting was not much affected by the angle of inclination or by pre-heating of the steam screw. PS2 was significantly harder to be wetted compared to PS1. 67% PS2 was wetted after the stream screw, only 7 % more than without any pre-wetting.

<table>
<thead>
<tr>
<th>Inclined angle (θ)</th>
<th>Without pre-heating</th>
<th>With pre-heating</th>
<th>With pre-heating</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Residence time</td>
<td>Sink</td>
<td>Float</td>
</tr>
<tr>
<td>0°</td>
<td>1’09”</td>
<td>91 %</td>
<td>8 %</td>
</tr>
<tr>
<td>1’20”</td>
<td>93 %</td>
<td>7 %</td>
<td></td>
</tr>
<tr>
<td>12.5°</td>
<td>1’35”</td>
<td>93 %</td>
<td>7 %</td>
</tr>
<tr>
<td>2’19”</td>
<td>96 %</td>
<td>4 %</td>
<td></td>
</tr>
<tr>
<td>18.5°</td>
<td>2’26”</td>
<td>99 %</td>
<td>1 %</td>
</tr>
<tr>
<td>2’51”</td>
<td>96 %</td>
<td>4 %</td>
<td></td>
</tr>
</tbody>
</table>

It was concluded from the steaming tests that condensation of a water film on the feed is not a robust wetting process, except for not too hydrophobic materials with a simple particle geometry, which excludes pockets of air.
4.5.2 Wetting with boiling

The alternative of contacting the plastics with boiling water was studied. In a first experiment, a reservoir with plastic flakes from the PS sample immersed in water was heated to study the behaviour of the air bubbles (Fig. 4.7). The results in Fig. 4.8 show that both the volume of the bubble and the air contact angle increase with temperature. A simple explanation for the increasing bubble size can be that with increasing temperature the original content of air in the bubble expands and at the same time is diluted with water vapour. At 293 K (20 °C), a bubble attached on an immersed polymer flake consists mainly of air with a volume of $V_{293, \text{air}}$; at 373 K (100 °C), it contains mainly water vapour. Suppose that at T (Kelvin temperature) the volume of an attached air bubble is $V_{T, \text{bubble}}$. According to the ideal gas law, for isobaric expansion,

$$V_{T, \text{air}} = V_{293, \text{air}} \cdot \frac{T}{293}.$$  

Gradually the temperature increases, the bubble, with a volume of $V_{T, \text{bubble}}$, consists of both air and water vapour at temperature T:

$$V_{T, \text{bubble}} = V_{T, \text{air}} + V_{T, \text{vapour}},$$

in which $V_{T, \text{vapour}}$ is the vapour volume at T. Furthermore, the water vapour pressure ($P_{T, \text{sat}}$) in the bubble increases with T so more vapour is contained in the bubble. In contrast, the air pressure in the bubble ($P_{T, \text{air}}$) decreases, and $P_{T, \text{air}} = 1 - P_{T, \text{sat}}$, since the total pressure of these two should be 1 atm ($P_0$). Therefore,

$$\frac{V_{T, \text{vapour}}}{V_{T, \text{air}}} = \frac{P_{T, \text{sat}}}{P_{T, \text{air}}},$$

and then

$$V_{T, \text{bubble}} = V_{T, \text{air}} \left( \frac{P_0}{P_0 - P_{T, \text{sat}}} \right).$$ \hspace{1cm} (4.3)

The growth of the bubble under this hypothesis is presented in Fig. 4.8. The volume of the bubble indeed grows with T, but the predicted growth rate is much smaller than the actual increase of the bubble diameter. So air that was initially dissolved in the liquid around the bubble must also add to the volume of the bubble and this is in fact the major contribution to the bubble growth. As a result, the bubble expands, lifts up and finally floats to the water surface. The fact that the air contact angle, 52.8° at 293 K, almost doubled at 343 K indicates that the plastic surface gradually becomes hydrophilic.
Fig. 4.7 PS flake immersed in hot water.

Fig. 4.8 Bubble diameter and air contact angle as a function of temperature during continuous heating.

- Air bubble diameter
- Predicted air bubble diameter on the basis of expansion and water vapor
- Air contact angle = 180°C-water contact angle
**Table 4.5 Predicted air bubble size as a function of temperature.**

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Water vapour pressure (bar)</th>
<th>Air pressure (bar)</th>
<th>Relative bubble volume</th>
<th>Relative bubble diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>293</td>
<td>0.025</td>
<td>0.975</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>313</td>
<td>0.08</td>
<td>0.92</td>
<td>1.13</td>
<td>1.04</td>
</tr>
<tr>
<td>333</td>
<td>0.21</td>
<td>0.79</td>
<td>1.4</td>
<td>1.12</td>
</tr>
<tr>
<td>353</td>
<td>0.50</td>
<td>0.50</td>
<td>2.3</td>
<td>1.32</td>
</tr>
</tbody>
</table>

In a second experiment the improvement of the wettability of the plastics was studied before and after contacting with boiling tap water. As a first measure for the wettability, the water contact angles on the plastics before and after wetting were compared. As shown in Fig. 4.9, the water contact angles of PS1, PS2, and PET decreased significantly after ten seconds of boiling while virgin PS and shredded HDPE showed a slower and less pronounced difference. Since a large water contact angle corresponds to a hydrophobic surface, whereas a small water contact angle implies a hydrophilic surface, the PS1, PS2, and PET evidently become hydrophilic after contact with boiling tap water. This is confirmed by recording the number of air bubbles collecting on the upper-surface of the polymer flakes when immersed in water before and after contact with boiling water. It is clear that boiling tap water can effectively wet PS1, PS2 and PET, and improve the wettability of virgin PS and shredded HDPE (Fig. 4.10). Moreover, at least half a minute of boiling is necessary for a robust process. It was concluded from the correspondence between the results for the water contact angle and the number of air bubbles per unit surface area that the contact angle is a reliable and practical measure for the wettability of the flakes.

**Fig. 4.9 The contact angles of the plastics as a function of contact time with boiling tap water.**

**Fig. 4.10 The number of air bubbles on the upper-surface of the polymers as a function of contact time.**

In order to understand the effect of contact with boiling tap water, the polymers were also wetted by steam and by contact with boiling de-mineralized water. The de-mineralized water was obtained by an ion-exchange de-mineralizing device and the conductivity of the
water was 0.08 μs/cm. The wettability of most of the polymers, after exposure to steam for 10-60 seconds, were not strongly improved, except for the shredded PET, which is the least hydrophobic material to start with (Fig. 4.11). For some materials, the water contact angle decreased initially; however, different from the boiling tap water results, there was no permanent effect on the wettability. The experiments with de-mineralized water present slightly better wetting on PS2: the contact angles of PS2 and PET were below 60°, but again, the results were significantly less positive than for boiling tap water (Fig. 4.12).

Tap water contains a mixture of minerals. It was therefore suspected that the main difference of the wetting with tap water versus steam and de-mineralized water is due to the minerals, such as lime-scale created in the boiling process. Observation of a sample of virgin PS by a microscope showed that the contact angle was much smaller where a stain of scale had been left by contact with tap water (Fig. 4.13), and water film can cover the entire shredded PS flake after boiling in tap water. The formed scale on the flakes, for instance lime-scale (but not limited to Ca), calcium carbonate (CaCO₃) is produced from calcium hydrogen carbonate (Ca(HCO₃)₂) which is soluble in water:

\[ Ca(HCO_3)_2 \rightarrow CaCO_3 + H_2O + CO_2. \]

Further study proved that virgin PS was completely wetted, after adding a calcium carbonate layer artificially without extra heating. The probable reason for the increased wettability of flakes treated with boiling water is therefore that a very thin layer of e.g. lime-scale was deposited on the plastic surfaces.
Fig. 4.13 Virgin PS wetted with water because of a stain of lime-scale (a, left) and water film on a shredded PS flake after boiling in tap water (b, right).

A further wetting test was carried out for virgin plastics. The results show that the wettability of virgin plastics hardly improve even after 1 minute of tap water boiling (Fig. 4.14). Therefore, virgin polymers are harder to be wetted than post-consumer polymers. A possible reason is that virgin plastics normally have a smoother surface or a surface coating. Since all the waste polymers have been used and very often they have been shredded, they rather have a rougher surface which affects the contact angle as well.

![Graph showing wetting test results](image)

**Fig. 4.14 Virgin plastic wettability after boiled in tap water.**

The roughness ($r$) of the surface is defined as:

$$r = \frac{A_{true}}{A_{projected}},$$

in which $A_{true}$ and $A_{projected}$ are true and projected area of the surface, respectively. For a rough surface, the water contact angle ($\phi$) relates to the contact angle of a smooth surface of the same material by Wenzel’s equation (Cherry, 1981):
\[ \cos \varphi_{\text{apparent}} = r \cdot \cos \varphi_{\text{true}}. \]

According to the equation, the roughness of a surface further decreases the contact angle if the contact angle is \(< 90^\circ\), whereas the roughness further increases the contact angle if the contact angle is \(> 90^\circ\). For this reason, a water layer is easier to be created on shredded plastics than on virgins.

Tests with the wetting procedure based on contacting plastics mixtures with boiling tap water showed that directly after taking the flakes from the water, the moisture content is typically about 10\%, which reduces to about 7\% after cooling. Such an amount of water in the feed to the MDS process results in acceptable rates of dilution, both economically and in terms of process liquid control.

### 4.6 CONCLUSIONS

Good wettability of polymers is essential for high-accuracy sink-float separation in water-based media such as MDS, because the grade and recovery of the products are critically influenced by even a small percentage of air bubbles. Both shredded waste and virgin polymers were contacted with steam and boiling water and the resulting wettability of the polymers was analysed by means of measuring contact angles and counting the number of bubbles on the polymer surfaces.

The tests show that the wettability of most polymers improve slightly by steaming for about 1 minute, but the best wetting results are obtained with immersion in boiling tap water. The success of wetting in boiling water can be explained by the deposition of a thin layer of e.g. calcium carbonate. Shredded plastics are easier to be wetted than virgin polymers, probably because a relatively rougher surface is caused by the shredder process. A new wetting process to eliminate air bubbles was developed on the basis of these results. That carries just a small amount of heat and water with the feed to the MDS process liquid.
Chapter 5

Magnetization Control of Magnetic Liquid
5.1 INTRODUCTION

In MDS, the magnetic liquid flows above or beneath a flat magnet and its cut-density as a separation medium varies strictly in the vertical direction. As a result, the system separates a complex mixture into many different materials in a single process step, using one and the same process liquid. By simply changing the magnetization of the fluid, the MDS can be applied in metal-metal, metal-polymer and polymer-polymer separations (Bakker and Rem, 2006; Bakker et al., 2009; Muchova et al., 2009). This is one of the advantages of the MDS technology. However, the other side of this is that the magnetization of the process liquid needs to be tightly controlled to reach and keep stable cut densities during processes. Preferably, for polyolefin wastes, the variation of the magnetization should be within 5%.

Two important mechanisms tend to drive the magnetization of the process liquid away from its initial specification. In general, the first step of the MDS process is wetting, which is important especially for polymer-polymer applications as introduced in Chapter 4. If the particles are not wetted properly, air attached to the particles will decrease their effective densities and the separation will be imperfect. If plain water is used for wetting, the introduction of the wet particles into the process liquid will gradually lower its magnetization. After separation, the particles need to be washed with water for some applications or because the magnetic liquid is too expensive to lose with the particles (the concentrated liquid costs about 30 €/litre). The rinsing water can be concentrated again by means of a membrane (Agterhuis et al., 2007) and the concentrate is then recycled into the process liquid. As a result of wetting and washing, the magnetization of the process liquid may become off-spec so that the cut-point of the separation process is affected. For this reason, the quality of the liquid is checked by a magnetization measurement tool and controlled by adding either water or concentrated magnetic liquid.

There are several options to correct the quality of the liquid in industrial processes. If the liquid drifts away from the spec relatively slowly, the quality can be corrected once per day by adding some amount of water or concentrated magnetic liquid during start-up. This strategy has a disadvantage that the added liquid may distribute in an inhomogeneous way over the process liquid and create variations of the magnetization at the scale of the system as a whole or across the height of the separation channel, as a result of segregation of ill-mixed liquid. The first problem can be avoided by adding the correction liquid to a large well-stirred tank that is usually part of the system. The second problem can only be solved by intensive on-line mixing. The alternative process control option is to correct the process liquid on-line, continuously. In this latter case, less mixing energy is needed to avoid segregation. In this chapter, three mixing options on a simplified process line are studied and the theoretical and experimental results on the mixing behaviour are reported. In order to keep track of fluctuations of the magnetization in an industrial process, a magnetization measurement tool was developed based on a magneto-gravimetric principle and tested for sensitivity in the laboratory and in an industrial environment.
5.2 MIXING OF MAGNETIC LIQUID

Water-based magnetic fluids have a relatively low density $\rho_l$ (typically 1000-1200 kg/m$^3$), comparable to that of water, but in a gradient magnetic field $B$ the weight of the fluid may be artificially increased or decreased, because the force on the fluid is the sum of gravity and the magnetic force. By a clever arrangement of the magnetic induction $B$, it is possible to create an effective density $\rho_{\text{eff}}$ of the medium which varies only with the vertical coordinate $z$. If the fluid has a constant magnetization $M$, the effective medium density varies exponentially with $z$ (Eq.(3.3) and (3.4)).

![Diagram showing the effective density of the process fluid in the separation channel before and after correction by adding concentrated magnetic liquid. The example shows the effect of correcting the magnetization from an original 900 A/m to an average of 1000 A/m by dispersing 1 vol% of concentrate. The resulting variations of density were calculated for a magnetic field amplitude of 0.6 T and a pole size of 120 mm.]

Fig. 5.1 Effect of segregation on the effective density of the process liquid in the separation channel.

Fig. 5.1 shows an example of the effective density of a process fluid in the separation channel before and after correction by adding concentrated magnetic liquid. The example shows the effect of correcting the magnetization from an original 900 A/m to an average of 1000 A/m by dispersing 1 vol% of concentrate. The resulting variations of density were calculated for a magnetic field amplitude of 0.6 T and a pole size of 120 mm. If the concentrate is not well-mixed and bodies of concentrated liquid are large enough to segregate towards the surface of the magnet, the magnetization will be off-spec in both the top and bottom part of the channel.

One factor affecting the size of bodies of concentrated liquid in the process fluid is diffusion. The diffusion of magnetite nano-particles in a magnetic fluid can be determined by the Einstein diffusion equation (Islam, 2004):

$$S^2 = 2D \tau ,$$
in which \( \tau \) is the elapsed time, \( S \) is the diffusion length scale, and \( D' \) is the diffusion coefficient of the magnetite particle in water given by the Einstein-stokes equation (Wesselingh and Krishna, 1990):

\[
D' = \frac{RT}{L3\pi \eta d_{nano}}.
\] (5.1)

Here \( R \) is the gas constant, \( T \) is the absolute temperature, \( L \) is Avogadro’s number, \( \eta \) is the dynamic viscosity of the magnetic fluid and \( d_{nano} \) is the diameter of the nano-size iron particles suspended in the fluid.

The diffusion coefficient determined by Eq. (5.1) is about \( 10^{-10} \) m\(^2\)/s. If the time between injection of the water or concentrated liquid and the arrival of the process fluid in the separation channel is 3–4 seconds, the diffusion scale is approximately 25 \( \mu \)m (Fig. 5.2), which means that bodies of water or concentrate with a size substantially smaller than 25 \( \mu \)m will have effectively diffused into the surrounding process fluid before they get to the magnetic field. Larger droplets, which exceed the critical diffusion size, may survive to the separation channel and cause segregation of the process fluid.

*Fig. 5.2 Diffusion distance of magnetic fluid as a function of elapse time.*

A second issue is whether droplets bigger than the critical diffusion size will actually segregate in the magnetic field. Any droplet of water or concentrated magnetic fluid which does not diffuse into the process liquid before reaching the magnetic field behaves as a particle with a density lower or higher than the effective density of the process liquid, and so it will float or sink in the separation channel.

A small water or magnetic liquid droplet of diameter \( d \), volume \( V_d \) and material density \( \rho_d \) immersed in a magnetic fluid of effective density \( \rho_{eff} \) and dynamic viscosity \( \eta \) will
experience a gravity force \((G)\), a drag force \((F_{\text{drag}})\) and a buoyancy force \((F_B)\) (Murariu et al., 2005):

Gravity force:

\[
G = \rho_d V_d g ,
\]

Drag force:

\[
F_{\text{drag}} = 3\pi \eta d_a v_t ,
\]

Buoyancy force:

\[
F_B = \rho_{\text{eff}} V_d g .
\]

Therefore, the terminal velocity of the droplets, \(v_t\), is given by (see Fig. 5.3):

\[
v_t = \frac{\left(\rho_d - \rho_{\text{eff}}\right) d_a^2 g}{18\eta} .
\] (5.2)

For a droplet of concentrated magnetic liquid, however, in addition to the gravity, there is also magnetic force on the droplet. Therefore, Eq. (5.2) becomes

\[
v_t = \frac{\left(\rho_d' - \rho_{\text{eff}}\right) d_a^2 g}{18\eta} ,
\]

in which \(\rho_{\text{d}}'\) is the effective density of the magnetic droplets.

---

**Fig. 5.3** Simulated trajectories of droplets of water and concentrated magnetic fluid \((d_a = 200 \mu m, \text{left}; d_a = 60 \mu m, \text{right})\). The magnetization of the process liquid is 900 A/m. The straight lines are water droplets; the dashed lines are for concentrated magnetic fluid droplets \((12000 \text{ A/m})\). Magnet is at the bottom in these graphs. The distance between the bottom of the separation channel and the surface of the magnet \((B_0 = 0.6 \text{ T})\) is 11 cm.
In the simulation shown in the first picture of Fig. 5.3, water droplets of diameter 200 μm fed into the magnetic field from different heights float upward in the process liquid relatively slowly. The concentrated magnetic fluid droplets are attracted by the magnet and produce a flow of concentrated magnetic fluid (effective density 2260 kg/m³) on the bottom of the separation channel within several seconds. It is even possible that the magnetic fluid will remain on the bottom of the separation channel, due to the attraction by the field, if the magnet is sufficiently strong. As a result, the effective densities of the magnetic fluid in the magnetic field become lighter and heavier along the top and bottom respectively.

The distribution is significantly improved by reducing the sizes of droplets. In the second picture of Fig. 5.3, droplets of both water and concentrated magnetic fluids (d = 60 μm) distribute homogeneously in the magnetic field. There is a slight settling for the magnetic fluid droplets, and the settling distance is smaller than 0.5 cm in three seconds. Taking into consideration that the height of each layer in sampling is 2 cm, the settling distance is acceptable to avoid segregation. Hence, any water or magnetic droplet smaller than 60 μm will not create substantial segregation of the process liquid. As a result, the liquid does not segregate and the separation will be not influenced.

Based on the theory presented above, the distribution of droplets essentially depends on the sizes of droplets of water or concentrated magnetic fluid, which on the other hand may cause segregation. By proper mixing, it is possible to reduce the sizes of the droplets and avoid segregation.

### 5.3 EXPERIMENTAL METHODS

#### 5.3.1 MDS setup and mixers

The experimental setup used to measure the segregation of magnetic liquid was made of a feeding zone, mixing area, separation channel and sampling area as shown in Fig. 5.4. As shown, the magnet is placed below the separation channel. The $B_0$ of the magnet was 0.6 T. At the end of the channel, four splitters divided the stream into five layers, with a 2 cm opening for each one. The flow rate was 8.6 m³/h. About 1 kg of water or concentrated magnetic fluid (12000 A/m) was pumped into the feeding zone in approximately 1 minute for each single experiment. Two types of mixers were tested: a static mixer and an impeller mixer (Fig. 5.5). The flow speed in the separation area was 0.08 m/s and the liquid head was 0.1 m. The magnetization of the process liquids ranged from 800 to 1000 A/m. In this situation, 1 kg of concentrated magnetic fluid injected in the system produced approximately 10% increase of the magnetization in each layer of the separation channel. The pressure drop across the static mixer was 9 mbar. The speed of the impeller mixer was 300 RPM. The sampling started after 30 to 45 seconds from the injection of the water or concentrated magnetic fluid to the system. Five samples were taken from each layer in the sampling area and their magnetization was analysed. Apart from the tests with the two types of mixers, one experiment was done without mixing as a reference.
5.3.2 Nano-filtration membrane

A nano-filtration membrane was utilized to imitate the online control of the concentration of the process liquid. Extra water may be brought into the MDS system because of wetting as mentioned in Chapter 4. In addition, after separation, the process liquid sticking on the particles is reclaimed so that water and concentrated magnetic fluid can be produced continuously for the maximum reuse of magnetic liquid and water. It is consequently essential to keep the magnetization of the process liquid in a stable level by means of a nano-filtration membrane, especially for a long run. The water extracted by the membrane may be used for washing. Meanwhile the concentrate flow from the membrane may be added into the system to maintain the right magnetization of the process liquid. In this way, water or concentrated magnetic fluid may accumulate as segregation layers in the separation channel if mixing does not work as expected.

The pilot nano-filtration unit used in the experiment consisted of a spiral wound nano-filtration element with a 62 mm diameter. A TFC© polyamide membrane with a surface of 1.1 m² was used to treat the process liquid (shown in Fig. 5.6). During the test, the membrane produced 24 l/h of water with a total feed of 250 l/h. The test was performed in a closed loop: the concentrate (magnetic fluid) and permeate (water) produced by the mem-
brane were fed to the feeding zone simultaneously, and mixed with the process liquid by the static mixer. Samples were taken from each layer in the separation channel for magnetization measurement. After sampling, the main stream left in each layer were pumped out and finally mixed in a storage vessel, from which a small amount of magnetic fluid was extracted and fed into the nano-filtration device. The system was running for more than 70 minutes. Samples were taken at the 15th and 70th minute in order to find out whether there was accumulation of water or concentrated magnetic fluid in the long run.

Fig. 5.6 Nano-filtration test setup.

5.3.3 Magnetization measurement

All the samples from the tests were analysed for their magnetization by using a simple setup (Fig. 5.7). A cup filled with magnetic fluid of certain volume \( V_l \) is placed in the magnetic field \( B \). The distance between the cup and the magnet is \( z \). The magnetic force \( F_{mag} \) acting on the \( V_l \) is measured using a balance. The magnetization \( M \) is determined by:

\[
M = \frac{F_{mag}}{V_l \cdot \nabla B}.
\]

Fig. 5.7 Magnetization measurement setup.
Based on the simplified magnetization measurement setup described above, a magnetization sensor was developed (Fig. 5.8). The advantage of the tool is the continuous measurement of the magnetization of the liquid. The output of the tool can be transferred to a PLC (programmable logic controller), which is a common system unit to control industrial processes. The magnetization measurement tool can control both the reclamation process with nano-filtration and the quality of the process liquid. Such sensor can be used to monitor any significant change in the magnetization when this change is more than 0.5%, although it does not provide directly the absolute magnetization of magnetic liquids.

![Fig. 5.8 An example of the magnetization sensor.](image)

### 5.4 RESULTS AND DISCUSSION

#### 5.4.1 MDS and mixing

The first tests were performed by adding water to the system (Fig. 5.4). It was found that the magnetization of the magnetic fluid did not appreciably vary after 1 kg water was fed within one minute (see in Fig. 5.9). The differences of the magnetizations in Layer 2 to 4 were generally less than 1%, when no mixer or the static mixer was applied. However, the magnetization in Layer 5 decreased by 2%. The test with the impeller mixer is not introduced here since a sampling mistake occurred in Layer 3 which caused the big change in magnetization before and after water was introduced. Another issue which may have introduced error is the inaccuracy of the magnetization measurement itself: the sensitivity of the instrument seems not sufficient to measure such small differences. Consequently, there is not enough information to estimate the sizes of the added droplets of water in the separation channel.

In the second experiment, concentrated magnetic fluid was fed to the system. The corresponding variations of the magnetization of the process liquid in each layer were compared. As shown in Fig. 5.10, without extra mixing, the magnetization of the liquid in Layer 2 to 4 increased 10% while the magnetization in layers 1 and 5 was higher and lower...
respectively. The reason is that without mixer, the added droplets of concentrated magnetic fluid were too big to be uniformly distributed when in the separation channel. Therefore, because of the combined effect of both the gravity and magnetic fields, they settled to the bottom of the separation channel so that the magnetization in layer 1 was increased. In contrast, the increase of magnetization of the flow in the top layer (Layer 5) was less. This test demonstrates that within the diffusion time (3-4 seconds) both the concentration and the size of the droplets are above the minimal values which cause segregation. Moreover, according to the previous simulation results, the size of the droplets was larger than 60 μm which is the upper limit for avoiding segregation on theoretical ground.

Fig. 5.9 Magnetization differences of the magnetic fluid in various layers before and after adding water by using the static mixer.

By using the static mixer, the measured increase of the magnetization of the magnetic fluid was 10% in each layer. This means that the added concentrated magnetic fluid has been uniformly distributed in the process fluid due to the action of the static mixer, which apparently broke down the droplets of magnetic fluid to a size less than 60 μm. Calculation showed that the power dissipated by the static mixer was 14 W, while the one of the impeller mixer was only 0.6 W.

It is concluded that the process liquid may become inhomogeneous in the separation channel if an amount of water or concentrated magnetic fluid is injected into the system. A static mixer may be applied to avoid such in homogeneities.
5.4.2 Nano-filtration

The experiment layout described in 5.3.2 shows that the magnetization of the process liquid was well controlled through the simultaneous operation of both the nano-filtration membrane and the static mixer. Therefore, there was no variation of magnetization in each layer following the combined injection of both water and reclaimed magnetic fluid to the system (shown in Fig. 5.11). It implies that neither water nor concentrated magnetic fluid accumulated within 70 minutes of continuous operation of the system. Thus the static mixer was proved to work effectively also in a long run.
5.5 CONCLUSIONS

The cut-density of sink-float separations in magnetic liquids depends on the magnetization of the process liquid. The control of the magnetization of the liquid is therefore a critical part of the MDS technology. There are several options to correct the quality of the liquid in industrial processes. However, a potential problem is that the process liquid may become inhomogeneous and will segregate in the magnetic field region.

In this chapter, three mixing options on a simplified process line were studied and theoretical and experimental results on the mixing behaviour were reported. There are two parameters governing the distribution of the droplets in the process liquid: the size of the droplets and the value of the magnetic field. For a magnetic field amplitude of 0.6 T and pole size of 120 mm, the critical size of the droplets of concentrated magnetic liquid (12000 A/m) is 60 μm. The experiments show that segregation of the process liquid due to incomplete mixing can be avoided by utilizing a static mixer which is able to reduce the size of droplets of concentrated magnetic fluid down to below 60 μm. By combining a nano-filtration membrane and a static mixer, process liquid is properly controlled online as well. Based on a magneto-gravimetric principle, a magnetization measurement tool was designed to detect and control fluctuations of the magnetization of the process liquid in an industrial environment.
Chapter 6

Turbulence Control in MDS
6.1 INTRODUCTION

Turbulence is a major factor in sink-float separation of polymers, in addition to wetting technology and the control of cut density of the process liquid. In order to obtain valuable secondary polyolefins, and since the density differences between PP and PE are small, the separation accuracy of the MDS process should reach 10 kg/m³ in density, as introduced in Chapter 2. Such separation accuracy in density can be translated into position error of flakes in the vertical direction. The density of a polyolefin particle (\(\rho_p\)) can be expressed on the basis of Eq. (3.3) as:

\[
\rho_p = \rho_l - C_1 e^{-\pi z_{eq}/p} \\
C_1 = \frac{\pi MB_0}{gp}
\]

in which \(z_{eq}\) is the height at which the \(\rho_{eff}\) is the same as \(\rho_p\). Hence, the difference between the densities of the process liquid and the polymer is

\[
\Delta \rho_{l-p} = \rho_l - \rho_p = C_1 e^{-\pi z_{eq}/p}.
\]

When the particle floats at \(z\), the local effective density of the liquid is

\[
\rho_{eff} = \rho_l - \Delta \rho_{l-p} \cdot e^{-\pi z_{error}/p}
\]

According to Taylor’s theorem, Eq. (6.1) becomes

\[
\rho_{eff} = \rho_p + \Delta \rho_{l-p} \cdot \left(\frac{\pi \Delta z_{error}}{p}\right)
\]

Therefore, the separation error in density (\(\Delta \rho_{error}\)) can be related with that in distance (\(\Delta z_{error}\)) with:

\[
\Delta \rho_{error} = \Delta \rho_{l-p} \cdot \frac{\pi \Delta z_{error}}{p}.
\]

The separation accuracy in millimetres calculated based on Eq.(6.2) for polyolefin recycling using MDS is listed in Table 6.1. As seen, the required separation accuracy is small, approximately 4-5 mm. If the flow in the separation channel of MDS is too turbulent, the particles may be driven away from their equilibrium position and mix with other layers of products. As a result, the separation process is destroyed. This chapter studies three sources of flow turbulence and presents solutions to prevent or reduce it.
Table 6.1 Desirable separation accuracy for polyolefin recycling at a cut density of 920 kg/m³ (pole size of magnet, \( p \), 0.12 m).

<table>
<thead>
<tr>
<th>Unit</th>
<th>PP /PE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (( \Delta \rho_{\text{true}} ))</td>
<td>kg/m³</td>
</tr>
<tr>
<td>Position (( \Delta z_{\text{true}} ))</td>
<td>mm</td>
</tr>
<tr>
<td>10</td>
<td>4.7</td>
</tr>
</tbody>
</table>

6.2 SOURCES OF TURBULENCE

6.2.1 Turbulence from static boundaries

There are three distinct types of sources of turbulence in the MDS setup. First of all, turbulence is produced near static boundaries. Fig. 6.1 shows the parts of the separation channel where this type of turbulence is created. Of these parts, the side walls are least important, because their effect is marginal for wider, industrial installations, and, for the prototype studied here, their effect can be largely avoided by feeding polymers at low capacities away from the walls. Turbulence from the interaction of the flow with the top and bottom of the channel is avoided by covering these walls with two conveyor belts, running at the same speed as the flow. The only static walls for which turbulence cannot be avoided are the floor and ceiling of the middle injection channel.

![Fig. 6.1 Turbulence sources in MDS setup.](image)

6.2.2 Turbulence from laminators

A second source of turbulence is at the outlet of the laminators. The laminator structures with their small tubes effectively prevent big eddies entering the channel. Small eddies with a maximum size of the tubes, i.e. 3.5 × 4 mm², are allowed to remain in the flow and move into the channel. These small eddies could be reduced further by implementing even smaller diameter tubes. However, their lifetime is less than a few second and small diameter tubes can be blocked by dirt in the liquid. Fig. 6.2 shows the transition of the approximately piece-wise parabolic profile of the flow formed in the tubes to a turbulent flow with small eddies.
Chapter 6

Fig. 6.2 Transition from laminar to turbulent flow at the outlet of the laminator (far left).

The laminator does produce also eddies at larger scales due to an unexpected effect. It is found experimentally that the flow through individual tubes is itself stochastic in nature, probably as a result of end effects. Four identical parallel tubes were placed in a box of water (Fig. 6.3). Water was flowing continuously due to the pressure difference between the right and the left side of the box, similar to the MDS setup. When the flow was stable, a few droplets of blue ink were added to the water, and the motion of the blue ink in each tube indicated the flow speed. Diffusion of ink in water was not a concern, since the traveling time of the flow in these tubes was short, less than one second. Due to the velocity profile within each tube, the ink tracer front quickly transforms into a thin line in the middle of the tube, so that the speed that is measured is the maximum of the approximate parabolic profile. The complete process was recorded with a camera with a filming speed of 25 frames/s, and the data was processed with Matlab to calculate the central velocity in the tubes at a given moment in time. Table 6.2 shows the velocity measured for each tube. It is clear that the velocity ($v_{av}$) in a single tube is a random function of the type $128 \pm 8$ mm/s, i.e., $v_{av} \pm 6\%$. Assuming the throughput of neighbouring tubes in the laminator is uncorrelated, the average velocity over a block of $N$ times $N$ tubes at any given time will be $v_{av} \pm 6.0\%/N$. Therefore, eddies with a size larger than the tube diameter will be formed, but with increasingly smaller intensity. Since eddies of larger diameter have longer decay times, and it is the turbulence near the splitters that causes the separation error, it is not clear at first which eddies are the most detrimental to the separation.

Fig. 6.3 Setup to measure flow velocity in individual parallel tubes.
Table 6.2 Average velocity measured for individual parallel tubes.

<table>
<thead>
<tr>
<th>Tube no.</th>
<th>Speed (mm/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>123.0</td>
</tr>
<tr>
<td>2</td>
<td>135.5</td>
</tr>
<tr>
<td>3</td>
<td>128.6</td>
</tr>
<tr>
<td>4</td>
<td>124.4</td>
</tr>
</tbody>
</table>

Fig. 6.4 Approximately parabolic profile of the flow from the tubes.

In order to estimate the decay of the primary small size eddies from the laminator, the profile of the periodic flow coming from the tubes of the laminator (Fig. 6.4) is approximated by a simple Fourier representation:

\[ u_x = \frac{1}{2} \psi_{aw} + \frac{1}{2} \psi_{aw} \cdot \cos \left( \frac{2\pi y}{\lambda} \right) \cdot \cos \left( \frac{2\pi z}{\lambda} \right) \cdot e^{-\alpha x}, \]

where \( \lambda \) is the size of the tube (4 mm) and \( x \) runs from 0 (laminator outlet) to 600 mm (splitter). Downstream the channel, \( u_x \) satisfies the Navier-Stokes equation (Kundu and Cohen, 2004):

\[ u_x \frac{\partial u_x}{\partial x} = \nu \left( \frac{\partial^2 u_x}{\partial y^2} + \frac{\partial^2 u_x}{\partial z^2} \right), \]

where \( \nu \) is the kinematic viscosity. Therefore, \( \alpha \) is

\[ \alpha = 2\nu \cdot \left( \frac{2\pi}{\lambda} \right)^2 \cdot \frac{1}{u_x}. \quad (6.3) \]

The analysis above can also be applied to the larger eddies originating from the stochastic behaviour of the tubes, assuming always that their decay is due to molecular viscosity. Then the decay of fluctuations, \( \Delta u \), for larger wavelengths can be estimated with:

\[ \Delta u(x) = u_x(0) \cdot e^{-\alpha \left( \frac{2\pi^2 x^2}{\lambda^2} \right) \frac{1}{u_x}}. \]

Table 6.3 gives a straightforward estimate of the random velocities for different scales. Fig. 6.5 presents the time scales of the corresponding type of turbulence. As shown, turbu-
lence with a small wavelength, e.g. 4 mm, has big fluctuation on velocity at the beginning, but it decays fast. Turbulence with a big wavelength, on average has small fluctuation, but it takes longer to decay.

Table 6.3 Estimate of random velocities at the splitter ($v_x = 0.15 \text{m/s}$, $\nu = 10^{-6} \text{m}^2/\text{s}$) for different scales.

<table>
<thead>
<tr>
<th>N</th>
<th>$\Delta \nu(0)$ (mm/s)</th>
<th>$\lambda$ (mm)</th>
<th>$-2\nu \cdot \left(\frac{2\pi}{\lambda}\right)^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>4</td>
<td>-4.9</td>
</tr>
<tr>
<td>2</td>
<td>4.5</td>
<td>8</td>
<td>-1.2</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>12</td>
<td>-0.5</td>
</tr>
<tr>
<td>4</td>
<td>2.25</td>
<td>16</td>
<td>-0.3</td>
</tr>
</tbody>
</table>

Fig. 6.5 Decay of fluctuations with different scales.

### 6.2.3 Turbulence from shear instability

A third source of turbulence is the shear instability produced at the interface between the flow from the Laminator 2 and the static liquid in the injection tube. Generally, shear instabilities develop as ripples at the interface between two parallel flows with a difference in speed. In order to analyse the instability for the MDS geometry, we consider a volume
of liquid as it moves out of the middle laminator with a velocity $v_x$ and interacts with the liquid in the injection tube. The analysis will be from a frame of coordinates moving with the flow from the laminator, and the interaction with the liquid in the injection tube starts at time $t = 0$ and stops at $t = D/v_x$, where $D$ is the diameter of the injection tube along the flow direction. This approach is viable only if the wavelength of the instability is essentially smaller than $D$. This will be verified afterwards.

6.2.3.1 Analytical study of shear instability

For the simple two-dimensional case of a horizontal flow of liquid with velocity $v(x, z)$ over a static layer of liquid (see Fig. 6.6) starting at time $t = 0$, such a ripple is described as a small perturbation $u'(x, z, t)$ of the flow on top of the main flow:

$$u'(x, z, t) = A(k, z) e^{ikx - i\omega t} , \quad (6.4)$$

$$A(k, z) = A e^{-\zeta} , \quad (6.5)$$

where $A(k, z)$ is the profile of the amplitude in the vertical direction. $k$ is the wavenumber, $\omega(k)$ is the corresponding frequency of the perturbation, and the interface between the two layers is chosen at $z = 0$. $A$ is a constant depending on boundary conditions. $u = u' + v$ satisfies the Navier-Stokes equation, where the viscous term is left out because the instability is essentially a non-viscous phenomenon:

$$\frac{\partial u'}{\partial t} + v_z \frac{\partial u'}{\partial x} = -\frac{\nabla p}{\rho} , \quad v_x = \begin{cases} v_z, z > 0 \\ 0, z < 0 \end{cases} , \quad (6.6)$$

$\omega(k)$ is related to $k$ through the equations of motion of the liquid, i.e., the Navier-Stokes equation and the continuity equation:

$$\nabla u = 0 , \quad (6.7)$$

when $\omega(k)$ has a negative imaginary component, the flow is stable with respect to perturbations with wavenumber $k$; when the imaginary component of $\omega$ is positive, the flow is unstable and perturbations with wavenumber $k$ will grow exponentially unto the state of turbulence.

For the case of two layers of a truly inviscid liquid, the flow is called a tangentially discontinuous flow and the $x$-component of the flow is discontinuous at the interface. Let $\zeta = \zeta e^{ikx - i\omega t}$ be the displacement in the $z$-direction of points at the interface, due to the perturbation, then

$$\frac{\partial \zeta}{\partial t} = u'_z - v_z \frac{\partial \zeta}{\partial x} , \quad v_z = \begin{cases} v_z, z > 0 \\ 0, z < 0 \end{cases} . \quad (6.8)$$
Scenario I:

For the case in which both layers develop in infinite space, the pressure perturbations at Layer 1 \( (p_1) \) and Layer 2 \( (p_2) \) can be expressed as:

\[
p_1(x, z, t) = P_1 e^{-kz} e^{ikx - i\omega t}, \quad \text{(Layer 1)}
\]

\[
p_2(x, z, t) = P_2 e^{kz} e^{ikx - i\omega t}, \quad \text{(Layer 2)}
\]

in which \( P_1 \) and \( P_2 \) are two constants, and \( p_1 = p_2 \) at the interface. This flow was analyzed by Landau and Lifshitz (Landau and Lifshitz, 1959), and the relation between \( k \) and \( \omega \) is found to be

\[
\omega = \frac{1}{2} kv_z \left( 1 \pm i \right). \tag{6.9}
\]

Since \( \omega \) has a positive imaginary part for all values of \( k \), thus the flow is unstable with respect to perturbations of any wavenumber.

Scenario II:

The actual MDS geometry (Fig. 3.6), at the point where the shear instability occurs, has a relatively shallow duct, with a lower wall at \( z = -30 \) mm. By adding a wall at the lower layer (Fig. 6.7) into the tangential discontinuity model, the pressure perturbation at the lower layer (Layer 2) becomes:

\[
p_2(x, z, t) = \left( P_2^+ e^{kz} + P_2^- e^{-kz} \right) e^{ikx - i\omega t}.
\]

\( P_2^+ \) and \( P_2^- \) are two different constants to match also the boundary condition \( u_z = 0 \) at the solid boundary \( (z = -H) \). The pressure perturbation at Layer 1 \( (p_1) \) does not change and \( p_1 = p_2 \) at the interface, consequently,
Turbulence Control in MDS

\[ P_1 = P_2^+ + P_2^- . \]  

(6.10)

\[
\begin{align*}
\zeta_1 &= \frac{-kP_1}{\rho_1 (kv_x - \omega)^2} = \frac{-k(P_2^+ + P_2^-)}{\rho_1 (kv_x - \omega)^2}; \\
\zeta_2 &= \frac{k(P_2^+ - P_2^-)}{\rho_1 \omega^2} .
\end{align*}
\]

Since these two results should be the same, therefore

By solving the Eqs.(6.6) and (6.7), the perturbation in the \( z \)-direction can be written

\[
u_{1,z} = \frac{kP_1}{i\rho_1 (kv_x - \omega)} e^{-kz} e^{ikz - kv_x t} , \quad \text{(Layer 1)}
\]

\[
u_{2,z} = \frac{k}{i\rho_1 \omega} (P_2^+ e^{kz} - P_2^- e^{-kz}) e^{ikz - kv_x t} . \quad \text{(Layer 2)}
\]

At \( z = -H \), \( \nu_{2,z} \) should be zero. Therefore,

\[
P_2^+ = P' e^{kH} \\
P_2^- = P' e^{-kH} ,
\]

\( P' \) is a constant. According to Eq. (6.10),

\[
P_1 = P' \left( e^{kH} + e^{-kH} \right).
\]

Therefore, from Layer 1, \( \zeta \) is given by:

\[
\zeta_1 = \frac{-kP_1}{\rho_1 (kv_x - \omega)^2} = \frac{-k(P_2^+ + P_2^-)}{\rho_1 (kv_x - \omega)^2};
\]

From Layer 2, \( \zeta \) is

\[
\zeta_2 = \frac{k(P_2^+ - P_2^-)}{\rho_1 \omega^2} .
\]

Fig. 6.7 Horizontal flow with velocity \( v_x \) over a static layer of liquid with finite depth \( H \) (shear instability study, Scenario II).
\[ \omega^2 = -\tanh\left(kH\right) \cdot \left(kv_x - \omega\right)^2. \] 

(6.11)

Then

\[ \omega = kv_x \cdot \frac{\tanh\left(kH\right) \pm i\sqrt{\tanh\left(kH\right)}}{1 + \tanh\left(kH\right)}. \]

For \( H = 30 \) mm, the result for \( \omega \) is different from the result of Landau and Lifshitz only for very small values of the wavenumber (Fig. 6.8), and the perturbation remains instable for any value of \( k \). In order to get a more realistic view of the shear instability for the MDS geometry, a finite layer is added, around \( z = 0 \), where the horizontal velocity changes from 0 (lower layer) to \( v_x \) (top layer), to account for the effect of the finite viscous fluid.

**Fig. 6.8 Correlation between imaginary component of \( \omega \) and small values of \( k \) at Scenario I and II.**

**Scenario III:**

A thin boundary layer with a thickness \( \delta \) is now assumed to be in between Layer 1 and 2, as a result of viscosity (Fig. 6.9). The velocity profile in the boundary layer \( (v_b) \) is simplified with a linear profile:

\[ v_b = v_x \cdot \frac{z}{\delta}. \]
The general solutions of $u_x$, $u_z$, and $p$ are:

$$
\begin{align*}
    u_x' &= X(z) \cdot e^{i(kz - \omega t)} \\
    u_z' &= Z(z) \cdot e^{i(kz - \omega t)} \\
    p &= P''(z) \cdot e^{i(kz - \omega t)}
\end{align*}
$$

in which $X(z)$, $Z(z)$ and $P''(z)$ are found from the Navier-Stokes equation and the continuity equation, Eqs. (6.6) and (6.7). By solving the equations above, the general correlation between $\omega$ and $k$ is found to be:

$$
\left(1 + \frac{1}{\chi}\right) \cdot \left(\frac{\omega}{kv_x}\right)^2 + \left[\left(1 - \frac{1}{\chi}\right) \cdot e^{-2k\delta} - 1 \cdot \left(1 + \frac{1}{\chi}\right)\right] \cdot \frac{\omega}{kv_x} + \frac{e^{-2k\delta} - 1 + 2k\delta}{2k^2\delta^2} = 0 \quad (6.12)
$$

$$
\chi = \tanh(kH).
$$

When $H$ is infinite and $\delta = 0$, Eq.(6.12) becomes Eq.(6.9) (Scenario I). When $\delta$ is nearly zero, Eq.(6.12) converges to Eq.(6.11) (Scenario II).

The zone of flow establishment (Fig. 6.10), is defined as the length of the jet for which the boundary layer has not reached the centre. This length $x_0$ is found experimentally to be equal to 5.2 times the slot height ($H$) for two-dimensional flow (Albertson et al., 1948). For the MDS geometry, the shear instability is always within this zone. Within the flow establishment, the boundary layer thickness $\delta$ of the submerged flow was determined by Albertson et al (Albertson et al., 1948):

$$
\delta = \frac{Hx}{\sqrt{\pi x_0}}
$$

$x$ is the distance downstream from the slot. For the first version of the MDS, $\delta$ is 13 mm at $x = 120$ mm which is the end of the injection tube ($D$). Assuming $\delta$ is 2 mm (half the size of the laminator tubes) at $x = 0$ as a result of the parabolic profiles from the top tubes of the
laminator at the point where flow is just discharged from the middle injection zone, thus
the average value of $\delta$ across the injection tube is 7.5 mm.

![Definition sketch for zone of flow establishment (Albertson et al., 1948).](image)

The correlation between $k$ and the imaginary part of $\omega$ is presented in Fig. 6.11, for
Scenario I (reference) and III with a range of values of the boundary layer thickness $\delta$. For
viscous flow (Scenario III), high frequency perturbations (with large $k$) die out (the imagi-
inary component of $\omega$ is negative). Therefore, the flow is stable with respect to high wave-
number perturbations, $k > k_0 = 1.3/\delta$. However, the graph shows a maximum for the imag-
inary component of $\omega$, corresponding to the perturbation with the fastest growth rate. For
the average thickness of the boundary layer, 7.5 mm when $D=120$ mm, perturbations with
a wavenumber of approximately $90/m$ explode the fastest. When $D$ reduces to 50 mm,
with an average $\delta$ of 3.8 mm, the fastest explosion is predicted for a higher frequency, $k =
190/m$. In practice, the perturbation with the fastest explosion rate is expected to be visible
and dominant.

### 6.2.3.2 Numerical simulation of shear instability

The instability of the flow in the separation channel was also studied by numerical simula-
tions by Barcelona Supercomputing Centre (Rem et al., 2013). The Computational Fluid
Dynamics (CFD) simulations were carried out using the in-house code Alya. The physical
module of Alya considered in this project solves for the incompressible Navier-Stokes equa-
tions. The numerical technique employed in Alya is based on a variational multi-scale
finite element method, extensively detailed in (Houzeaux and Principe, 2008). The alge-
braic system of equations is solved using a fractional step like technique, based on the solu-
tion of the pressure Schur complement, which converges to the monolithic solution
(Houzeaux et al., 2011) so that no fractional errors are introduced. The code is parallelized
with MPI (Houzeaux et al., 2009) and exhibits near to linear speedups up to 22000
CPU’s. The meshes considered in this worked ranged from 5 to 20 million of elements,
and the simulations were run on up to 520 CPU’s of the MareNostrum Supercomputer.
In the context of this work, the code solves for the Navier-Stokes equations to compare
with experimental results. The complete strategy has been explained in detail by Houzeaux
et al (Houzeaux et al., 2010).
Fig. 6.11 Correlation between $k$ and $\omega$ for Scenario I and III and $D = 120$ mm and 50 mm ($v_x = 0.15$m/s).

The CFD simulations were conducted for the MDS prototype with both $D = 120$ mm and $D = 50$ mm at $v_x = 0.15$ m/s. The detailed flow profile from the laminator tubes was not included in these simulations, hence the profile of $v_x$ coming from all the three injections was assumed perfectly square-shape. The floor and the ceiling of the middle injection of the MDS prototype were described realistically with a stick boundary condition. Therefore the simulated turbulence levels in the separation channel relate to the instability in the feeding tube and to the stick walls in the middle injection, but not to the stochastic flow profile of the laminator tubes. Fig. 6.12 shows a snapshot of the velocity components $v_x$ and $v_z$ after the flow has developed.

The simulation reveals that the instability in the injection tube is growing exponentially to the right, as predicted by the analytical study. It also shows that the disturbed flow is transported to the separation channel through the middle injection, and consequently the main flow in the channel becomes turbulent. A second simulation for $D = 50$ mm (Fig. 6.13) shows strongly decreased levels of turbulence in the separation channel, indicating the stick walls are a minor cause of turbulence. Fig. 6.14 zooms in on the explosion of the instability along the width of the injection tube. It shows that the maximum amplitude of $v_z$ at $D = 120$ mm is slightly higher than that at $D = 50$ mm, but at a lower wave number. This fact indicates that the frequency of the developed turbulence in the tube with $D = 50$ mm is essentially higher than that for $D = 120$ mm.
Fig. 6.12 Development of $v_x$ (a) and $v_z$ (b) of the flow ($D = 120$ mm), provided by BSC.
Fig. 6.13 Development of $v_x$ (a) and $v_z$ (b) of the flow ($D = 50$ mm), provided by BSC.
Fig. 6.14 Explosion of the instability in the injection tube with a width of 120 mm (a) and 50 mm (b), provided by BSC.
The average wavelength ($\lambda$) and corresponding wavenumber ($k$), derived from Fig. 6.14, are presented in Table 6.4. At $D = 50$ mm, the wavelength of the instable flow was changing during its movement, from 17 mm to 26 mm. Correspondingly, $k$ varies between 243 and 372. However, similar phenomenon was not observed for $D = 120$ mm, since only one datum point was available.

<table>
<thead>
<tr>
<th>$D$ (mm)</th>
<th>$\lambda$ (mm)</th>
<th>$k$</th>
<th>Imaginary component of $\omega$</th>
</tr>
</thead>
<tbody>
<tr>
<td>120</td>
<td>75</td>
<td>84</td>
<td>2.6</td>
</tr>
<tr>
<td>50</td>
<td>17</td>
<td>372</td>
<td>11.03</td>
</tr>
<tr>
<td></td>
<td>26</td>
<td>243</td>
<td>10.35</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>253</td>
<td>7.05</td>
</tr>
<tr>
<td></td>
<td>18</td>
<td>352</td>
<td>7.05</td>
</tr>
</tbody>
</table>

Fig. 6.15 compares the analytical and numerical results for the two injection tubes. As shown, the observed perturbations in the CFD simulations have a wavenumber near the maximum of the imaginary part of $\omega$ according to the analytical prediction, and the growth rates computed from the simulation agree reasonably well with the theoretical values for $\omega$.

Fig. 6.15 Comparison between the analytical and numerical results of $k$ and the corresponding imaginary component of $\omega$ for $D = 120$ mm (square) and $D = 50$ mm (circle).

According to both the analytical expectation and the numerical simulation, high frequency perturbations decay faster. As a result, an injection tube with $D = 50$ mm is better than a one with $D = 120$ mm. In addition, based on the study of the sources of turbulence in MDS, it is clear that totally avoiding turbulence is not possible. However, by under-
standing the effect of turbulence on individual polymer particles, we can estimate the influence of it in the separation process.

### 6.2.4 Turbulence on polymer flakes

To evaluate the effect of the turbulence on polyolefin particles and estimate the separation error of MDS, the flow fluctuation is simplified with a square-wave model, which has an amplitude equal to the root-mean-square of $v_z (v_{z, \text{rms}})$, and a period $T$ estimated on the basis of the autocorrelation function. As shown in Fig. 6.16, the autocorrelation coefficient of a square wave $\tau$ is equal to a quarter of $T$.

![Fig. 6.16 Square-wave (a, Left) and its autocorrelation function (b, right).](image)

The response of a flake to a square wave of the vertical velocity in the MDS channel can be calculated analytically. Suppose a flake of thickness $d$ and cross-sectional area $A$ is floating at a position $z$, which is slightly off from its equilibrium position ($z_{eq}$) due to the turbulent flow, then the effective density $\rho_{\text{eff}}$ from Eq. (3.3) at $z$ can also be expressed as:

$$\rho_{\text{eff}} = \rho_p + \frac{d\rho_{\text{eff}}}{dz} (z - z_{eq})$$

and

$$\frac{d\rho_{\text{eff}}}{dz} = \frac{\pi}{p} (\rho_{\text{eff}} - \rho_l).$$

Therefore,

$$\rho_{\text{eff}} = \rho_p - \frac{\pi}{p} (\rho_p - \rho_l) \cdot (z - z_{eq}).$$

Accordingly, the force on the flake ($\mathbf{F}_{\text{buoyancy+gravity}}$), with a volume $V_p$, can be described as:

$$\mathbf{F}_{\text{buoyancy+gravity}} = \frac{\pi}{p} \cdot g \cdot V_p \left(\rho_p - \rho_l\right) \cdot (z - z_{eq}).$$
Because of the vertical flake movement, drag ($F_{\text{drag}}$), in addition to gravity and buoyancy, applies on the flake as well ($C_d$ is the drag coefficient):

$$F_{\text{drag}} = -A C_d \cdot \frac{1}{2} \rho \left( v_p - v_z \right)^2,$$

$$F_{\text{buoyancy+gravity}} + F_{\text{drag}} = 0.$$ 

Assuming that the total force on the flake is balanced at any moment, then the flake motion (velocity $v_p$), caused by the square-wave fluctuation, is dependent on both the $v_{z,\text{rms}}$ and the flake position.

**Case I:**

When $z > z_{eq}$, $v_p - v_{z,\text{rms}} < 0$, therefore

$$v_p = v_{z,\text{rms}} - \sqrt{\frac{\rho_l - \rho_p}{2 \rho_l C_d} \cdot \frac{\pi g d}{p} \cdot \sqrt{z - z_{eq}}} \quad (z > z_{eq}). \tag{6.13}$$

**Case II:**

When $z < z_{eq}$, $v_p - v_{z,\text{rms}} > 0$,

$$v_p = v_{z,\text{rms}} + \sqrt{\frac{\rho_l - \rho_p}{2 \rho_l C_d} \cdot \frac{\pi g d}{p} \cdot \sqrt{z - z_{eq}}} \quad (z < z_{eq}) \tag{6.14}$$

As shown in Case I and II, for a polyolefin flake, $v_p$ at $z > z_{eq}$ is smaller than $v_p$ at $z < z_{eq}$. The $v_p$ changes also with $d$. As a result, the maximum $v_p$ of a thin flake is less than that of a thick one, and the $v_p$ of a thick flake drops faster than a thin one (indicated in Fig. 6.17, left figure).

Assuming a flake at $\Delta z = z - z_{eq}$ when $t = t_0$, because $v_p = v_{z,\text{rms}}$ and at $t = 0$ or $t = T$, $\Delta z$ reaches to its minimum value ($-\Delta z_{\text{max}}$). Let

$$A = v_{z,\text{rms}},$$

and
therefore, by solving \( \frac{dz}{dt} = v_p = A + B \sqrt{\Delta z} \), the absolute value of the maximum displacement of a flake from its equilibrium position \( z_{eq} \) can be expressed with

\[
\Delta z_{\text{max}} = v_{z_{\text{rms}}} \cdot \tau \cdot \left( 1 - e^{-T/4\tau} \right),
\]

(6.15)

\[
\tau = \frac{v_{z_{\text{rms}}}}{2} \frac{\rho_i C_d}{\rho_f} \cdot \frac{\pi}{g d}
\]

As shown by Eq.(6.15), the displacement caused by the turbulent flow depends on the properties of both the flow and the polymer flake. The displacement results in the separation error. Fig. 6.18 gives an example of the \( \Delta z_{\text{max}} \) of polyolefins with different densities and thicknesses. The properties of the turbulent flow were set at \( v_{z_{\text{rms}}} = 9.4 \, \text{mm} \) and \( T = 5 \, \text{seconds} \) in accordance with experimental data reported in Chapter 9. As shown, \( \Delta z_{\text{max}} \) increases with decreasing values of polymer thickness. At the cut density of 920 kg/m\(^3\), a typical cut density for PP and PE separation, the minimum thickness of the input materials needs to be not essentially below 0.5 mm if an average accuracy of 10 kg/m\(^3\) is necessarily to be achieved. For polyolefins with the same thickness, the turbulence has more influence on those flakes having less density difference with the process liquid.
6.3 CONCLUSIONS

Turbulence is a major factor that affects MDS processes for polyolefin recycling. To obtain valuable secondary polyolefins using MDS, the separation accuracy should reach 4-5 mm in terms of the flake’s vertical position near the splitters. It is thus critical to prevent turbulent flow. Three types of sources of turbulence in the MDS setup were studied: turbulence produced near static boundaries, turbulence generated by the laminator tubes, and the shear instability created at the interface between the flow from the laminator 2 and the static liquid in the injection tube. The laminator tubes in the MDS system can effectively prevent big eddies entering the separation channel, but they may also produce eddies due to the fact that flow through individual tubes is itself stochastic in nature. Both the analytical and numerical studies reveal that the shear instability in the injection tube is growing exponentially along the flow direction. By narrowing the width of the injection tube, the frequency of the developed turbulence increases, resulting in a faster decay of the eddies transported to the separation channel. The effect of the turbulence on the separation for individual flakes can be estimated by simplifying the turbulence into a square wave. For individual particles, the turbulence effect increases with the reduction of the polymer thickness.

Fig. 6.18 Change of $\Delta z_{\text{max}}$ with densities and thicknesses of polymer for a magnetic liquid with a density $\rho_l = 1002$ kg/m$^3$ and a magnet pole size $p = 120$ mm ($v_{z_{\text{rms}}} = 9.4$ mm/s).
Chapter 7

MDS Polyolefin Recycling
7.1 INTRODUCTION

The fundamental principle and the basic structure of MDS are introduced in Chapter 3. The three main relevant technologies, wetting, quality control of magnetic liquid and turbulence control in MDS, are discussed in Chapter 4, 5 and 6, respectively. Based on these studies, two MDS setups, a lab-scale and a pilot scale, have been constructed. The lab-scale MDS was designed to separate small amounts of polymers, and the pilot MDS aims to work at high throughput in an industrial environment. This chapter evaluates the sorting performance of both MDS system on polyolefin packaging wastes.

7.2 MDS SETUPS

7.2.1 Lab-scale MDS

The lab-scale MDS was designed to further understand the separation process as well as to determine the proper input materials for MDS by sorting small amounts of polyolefin wastes. Fig. 7.1 shows the scheme of the lab-scale MDS setup. All the components of the MDS setup are submerged under the liquid surface. The process liquid, magnetic fluid, circulates in the system, as proposed in Chapter 3: it moves from the left chamber to the right side of the MDS by the pressure difference generated by the pumps, and then flows back to the left side.

![Fig. 7.1 Scheme of the lab-scale MDS for polyolefin recycling (the wetting section is not shown).](image)

In the lab-scale MDS system, the wetting process is carried out simply in a pan of boiling water, which is not a part of the MDS system. The wetted samples are fed into a box made of stainless steel wire gauze with openings of 1 mm. Air in the feeding box is first discharged before the box is placed in position, to avoid air caused turbulence. As introduced in Chapter 3, the light polymers (< 1000 kg/m³) are fed into the separation channel by taking advantage of their buoyancy. When the lid of the box is open, the polyolefin particles rise up and then flow into the separation channel with the main flow stream.

The laminar flow in the separation channel is created by forcing most of the process liquid to pass stacked layers of tubes. These tubes are 250 mm long and have a cross-section area of 3.5 × 4 mm². In this way, big eddies are stopped by the tubes, only small
eddies, smaller than the cross-section area, can pass. The study of the function of these tubes has been introduced in Chapter 6.

The separation channel of the lab-scale MDS is 600 mm long and 400 mm wide. Its top and bottom are covered by two belts. On the upper belt, there are a series of 10 mm high riffles. One function of this belt is to remove the magnetic particles, if there is any in the feed. The other use is to remove contaminants lighter than PP, e.g. wood and foam, and not well-wetted polymers. Both belts cover the entire channel, and they move with the same speed as the flow in the channel to avoid generating turbulence.

The magnet is placed on the top of the separation channel to create effective densities of the magnetic liquid less than 1000 kg/m³. It has a magnetic field \( (B_0) \) of 0.6 T on its surface and a pole size of 0.12 m. Its detailed design has been described in the patent of Rem et al.\textsuperscript{(Rem and Berkhout, 2007)}. The strength of the magnetic field changes only in the vertical direction \( z \). However, the strength drops dramatically near the edges which results in a change of the effective density of the magnetic fluid. Fig. 7.2 presents an example of the effective density distribution of the magnetic liquid in a magnetic field for polyolefin recycling. As shown, the effective density of the magnetic liquid increases and gets close to the material density of the liquid near the edge of the magnet. Therefore, to prevent this effect, the separation process should be carried out in the centre area, where the magnetic effect is not influenced: the middle \( 0.6 \times 0.4 \text{ m}^2 \).

![Magnet](image)

**Fig. 7.2 Effective medium density in the magnetic liquid below magnet (Bakker et al., 2009).**

At the end of the channel, four splitters divide the feed into four products and a residue. The materials that float between the upper belt and the top splitter finally end up on the liquid surface in the tank. They can be considered as the residue of the process.

### 7.2.2 Pilot MDS

The pilot MDS (Fig. 7.3) was designed for high capacity production in an industrial environment. Compared to the lab-scale MDS, it should be able to work continuously, and the
entire sorting process needs to be easily operated. Therefore, several improvements were conducted to reach these purposes on the pilot MDS.

A major improvement is the wetting method. The wetting process in the pilot MDS is carried out in an inclined screw and a water tank. The water tank is filled up with water which is continuously being heated during operation by heating elements at the bottom of the tank to keep the temperature at around 100 °C. For a continuous process, the amount of water for wetting should be always sufficient, hence the water level in the screw is measured by two probes during the process and is automatically adjusted. The whole wetting process takes about one minute. After wetting, the polymer particles are dewatered and cooled on the vibrating feeder before they are mixed with the process liquid.

A second improvement is the particle feeding technique. Since the pilot MDS is designed for high throughput (~ 400 kg/h), the feeding method used in the lab-scale MDS is not very practical. In the pilot MDS, the polyolefin particles, after dewatering and cooling, are fed into a thin slit which is connected with the inclined injection tube under the middle injection (Fig. 7.3). By creating a downwards inner flow, the polymer flakes are driven against their buoyancy, and enter in the injection tube where they rise up and flow to the separation channel with the process liquid.

To reach a high throughput, the separation channel of the pilot MDS is 0.9 m, longer than the lab-scale setup. Such a long channel can process more materials than the short one, if the volume percentage of the materials in the channel is the same (typically 1 vol% in MDS). That is because, as introduced in Chapter 3, the residence time of the particles in the channel is essential to the separation. With a long channel, the flow speed can be increased and yet the residence time is sufficiently long to finish the sorting process. Another way of reaching a high throughput is to increase the volume percentage of the materials in the separation channel. However, this may result in a bad separation since particle interaction may reduce the sorting efficiency.

Another improvement is the membrane system connected with the pilot MDS. The membrane system is to remove extra water brought into the process liquid by the wetting process, and to avoid the reduction of the magnetization of the process liquid which is essential for an accurate separation (presented in Chapter 5). Considering the 10% moisture content of the wetted polymer particles and a throughput of 400 kg/h, the extra amount of water from wetting is about 40 l/h. That means the magnetization of the process liquid decreases 2% for every hour. The membrane system used in the pilot MDS is PERMAQ COMPACT 100 with one membrane unit which is able to extract a maximum of 50 l/h water from the system, which is suitable for a throughput of roughly 500 kg/h. The removed water can be reused for washing the products.

The magnet used in the pilot MDS is designed differently from the one in the lab-scale MDS. The global layout of the magnet was published in 2011 (Lahaye et al., 2011). Table 7.1 compares the parameters of the two magnets used in the lab and pilot MDS. Similar to that in the lab-scale MDS, the magnet of the pilot MDS produces a relatively flat magnetic field, and the pole size of it is 0.12m. However, the $B_0$ is lower, on average 0.21 T, compare
Fig. 7.3 Side views of the pilot MDS.
to the other one. As a result, a more concentrated magnetic liquid is needed in the pilot MDS in order to compensate for the weak field.

Table 7.1 Comparison between the magnets in the lab-scale and the pilot MDS.

<table>
<thead>
<tr>
<th></th>
<th>Lab-scale MDS</th>
<th>Pilot MDS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strength on the magnet surface, $B_0$ (T)</td>
<td>0.6</td>
<td>0.21</td>
</tr>
<tr>
<td>Pole size (m)</td>
<td>0.12</td>
<td>0.12</td>
</tr>
<tr>
<td>Entire length (m)</td>
<td>0.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Width (m)</td>
<td>0.54</td>
<td>0.61</td>
</tr>
<tr>
<td>Cost (€)</td>
<td>Not on the market</td>
<td>25,000*</td>
</tr>
</tbody>
</table>

* in 2013.

7.3 POLYOLEFIN RECYCLING

7.3.1 Sorting with lab-scale MDS

7.3.1.1 Materials and experiments

Materials

Two polyolefin wastes were used in the experiments: household packaging waste from Romania and from the Netherlands. A detailed study of these materials has been presented in Chapter 2. The shredded Romanian polyolefins were processed with MDS directly. However, the Dutch sample were divided into groups in order to: 1) obtain similar materials as the Romanian sample to study the stability of the MDS process in producing secondary polyolefins with a consistent quality, and 2) remove the less than 0.4 mm thick foils which contain only PP (see Chapter 2) and are not well separated by MDS (discussed in Chapter 6).

The blown and injected samples including bottles, buckets, caps and basins of NL HW are collected as Group 1; the rest, mainly boxes and lids of the injection moulds was named as Group 2. Group 1 which has similar characterizations as RO HW was processed by MDS directly. To remove the thin fractions, Group 2 was first treated by a ballistic separator before being processed by MDS.

The ballistic separator (Fig. 7.4) is able to sort mixtures into light, middle and heavy products. In this case, the input materials of Group 2, have a strong similarity in their density and flake size, thus the separator acts on particle flake thickness. The thin particles fell in the first chamber, the thick particles travelled further and finally ended up in the third chamber.
Table 7.2 introduces the mass distribution of Group 2 after being processed by the ballistic separator. The size distribution of the sorted polymer flakes in the three chambers is presented in Fig. 7.5. Group 2-1 and Group 2-2 successfully collected parts of the thin flakes (< 0.4 mm), but further analysis shows that only roughly 50% of the < 0.4 mm fraction were recovered in these two products, and the other half still remaining in Group 2-3.

**Table 7.2 Mass distribution of the Dutch polyolefin waste**

<table>
<thead>
<tr>
<th>Chamber</th>
<th>Mass (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>74</td>
</tr>
<tr>
<td>2</td>
<td>221</td>
</tr>
<tr>
<td>3</td>
<td>969</td>
</tr>
</tbody>
</table>

In total three tests were carried out by using the lab scale MDS. The input material of each test is listed in Table 7.3. The < 2 mm fractions of the inputs were removed to prevent the blockage of the filters in the MDS system. All the inputs were first wetted in boiling water for about one minute to eliminate air bubbles attached on the polymer surfaces and to remove the heavy fractions (> 1000 kg/m³). Then they were fed in the centre 20 cm of the separation channel to avoid turbulence created near the stagnant walls.
Table 7.3 Polyolefin flakes for each test.

<table>
<thead>
<tr>
<th>Test</th>
<th>Sample name</th>
<th>Sample source</th>
<th>Country</th>
<th>Type</th>
<th>Pre-sort</th>
<th>Mass (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>RO-HW</td>
<td>Romania</td>
<td>Household</td>
<td>No</td>
<td></td>
<td>2470</td>
</tr>
<tr>
<td>T2</td>
<td>NL-HW-Group 1</td>
<td>The Netherlands</td>
<td>Household</td>
<td>Non-Ballistic sorted</td>
<td></td>
<td>2400</td>
</tr>
<tr>
<td>T3</td>
<td>NL-HW-Group 2-3</td>
<td>The Netherlands</td>
<td>Household</td>
<td>Ballistic sorted: chamber 1</td>
<td></td>
<td>940</td>
</tr>
</tbody>
</table>

**MDS settings**

The process liquid, with a magnetization of 120 A/m, was made of magnetic liquid MSG 10 from Ferrotec. The liquid was moving from left to the right side of the separation channel with a speed of 0.18 m/s. Since the length of the channel was 0.6 m, the residence time for the shredded polyolefin samples was slightly more than 3s. The splitting positions in the MDS and their corresponding density are presented in Table 7.4. Four products and one residual fraction were created, on the basis of their floating height in the channel. Based on the density analysis in Chapter 2, most of the rigid PP and PE from household wastes have densities < 920 kg/m$^3$ and > 930 kg/m$^3$ respectively, thus the major component of P4 was expected to be PP, while P1, P2 and P3 should include mainly PE.

Table 7.4 Split position (distance from magnet surface) of product and theoretical corresponding density.

<table>
<thead>
<tr>
<th>Product</th>
<th>Position range [mm]</th>
<th>Corresponding density range [kg/m$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>81-103</td>
<td>980-1000</td>
</tr>
<tr>
<td>P2</td>
<td>59-81</td>
<td>960-980</td>
</tr>
<tr>
<td>P3</td>
<td>37-59</td>
<td>930-960</td>
</tr>
<tr>
<td>P4</td>
<td>10-37</td>
<td>860-930</td>
</tr>
<tr>
<td>Residue</td>
<td>0-10</td>
<td>-</td>
</tr>
</tbody>
</table>

7.3.1.2 Analysing methods

The sorted products from MDS were analysed with a series of liquid standards of different densities, in order to study their density distribution. Mixtures of demineralized water and 96% ethanol were used to make liquid standards with 10 kg/m$^3$ intervals covering the density range between 880 kg/m$^3$ and 1000 kg/m$^3$.

On the basis of the mass and density distribution of each product, the recovery rate ($R_\rho\%$) of polyolefins within a certain density interval ($\rho$) in a product can be calculated as:

$$R_\rho\% = \frac{\text{Mass}_\rho}{\sum_{i=1}^{n}\text{Mass}_{\rho_i}},$$
in which \( Mass_{\rho_i} \) is the mass of polyolefins in Product \( i \) within the density interval \( \rho \), and 
\[
\sum_{i=1}^{n} Mass_{\rho_i} = \text{the total mass of polyolefins within the same density interval } \rho \text{ in Products 1 to } n.
\]
According to the recovery rate of the particles with various densities, the separation inaccuracy around a splitter point, also known as probable error \( E_p \), can be determined as:
\[
E_p = \frac{\rho_{25} - \rho_{75}}{2},
\]
where \( \rho_{25} \) and \( \rho_{75} \) stand for the density with a recovery of 25% and 75% at the heavy side of the splitter, respectively.

### 7.3.1.3 Results and discussion

#### Mass Distribution

The mass distribution of products of each test is presented in Table 7.5. In addition to the heavy fraction (> 1000 kg/m\(^3\)), there were also small amounts of residues, mixtures of dust, dirt and few curved flakes, generated during the MDS process. The MDS processes recovered most of the polyolefins (< 1000 kg/m\(^3\)). In T1 and T2, the recovery of PP and PE reached around 99%. The product ratios of T1 and T2 are comparable, because of the similarity of the input materials.

<table>
<thead>
<tr>
<th>Product</th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.3%</td>
<td>1.2%</td>
<td>0.5%</td>
</tr>
<tr>
<td>2</td>
<td>19.4%</td>
<td>26.6%</td>
<td>3.2%</td>
</tr>
<tr>
<td>3</td>
<td>43.7%</td>
<td>40.3%</td>
<td>21.4%</td>
</tr>
<tr>
<td>4</td>
<td>35.1%</td>
<td>31.0%</td>
<td>63.1%</td>
</tr>
<tr>
<td>Residue</td>
<td>1.1%</td>
<td>0.9%</td>
<td>0.7%</td>
</tr>
<tr>
<td>&gt; 1000 kg/m(^3)</td>
<td>0.4%</td>
<td>-</td>
<td>11.1%</td>
</tr>
<tr>
<td>Total</td>
<td>100.0%</td>
<td>100.0%</td>
<td>100.0%</td>
</tr>
</tbody>
</table>

#### Density grade

Fig. 7.6 presents the density distribution of the products from all the tests.

The concentration of PP, LDPE and HDPE in the products of T1, T2 and T3 is inferred directly from the density distribution of the products, since the density analyses of the household polyolefins with blow and injection moulds in Chapter 2 concluded that PP, LDPE and HDPE are concentrated mainly in the density group of < 920 kg/m\(^3\), 920-930 kg/m\(^3\) and > 930 kg/m\(^3\), respectively. In both T1 and T2, the grade of PP (considered as the < 920 kg/m\(^3\) fraction) was more than 93% in Product 4 (Fig. 7.7). In Product 3, this
Fig. 7.6 Density distribution of products from T1 to T3 (continued).
Fig. 7.6 Density distribution of products from T1 to T3.

grade dropped to less than 4%. PE and in particular HDPE, was the major component of Product 1, 2 and 3, and the concentration of PE in these products combined was higher than 98%. T1 and T2 show that MDS is able to sort blown and injected polyolefin (in particular the thick walled injection moulds) mixtures into high grade PP and PE products. Similar to T1 and T2, P4 and P2 from T3 contain mostly PP and PE respectively, as indicated by the density grade. However, P3 is a mixture of PP and PE, instead of high purity PE like in T1 and T2. Considering that the cut densities between P3 and P4 should be similar in these tests because of the same settings of MDS, this is probably caused by the difference of the density distribution of the input materials (Fig. 2.11 and Fig. 7.8)
Fig. 7.7 Density grade of products from T1 (a, up left), T2 (b, up right) and T3 (c, below).
Fig. 7.8 Density distribution of the input materials of T3 (excluding > 1000 kg/m³).

Recovery rate
The recovery rate curves of the products from the three tests are presented in Fig. 7.9. The cut densities (ρ_cut) between the products and the corresponding E_p derived from these pictures are listed in Table 7.6. As shown, both the cut densities and E_p varied a bit, although the settings of the MDS system were not changed during the tests.

The actual cut densities are close to the predicted cut densities introduced in Table 7.4. The cut densities for separating PP and PE are around 920-930 kg/m³, as recommended according to the density analyses in Chapter 2. The other two cut densities (between P1 & P2, P2 & P3) at the high density range distinguished the PE with different densities.

The E_p varied with the cut densities (Table 7.9). In general, the average E_p found from the tests decreased with an increase of the cut density as expected from theory (see Chapter 6, Eq. (6.2)). For instance, the E_p at ρ_cut=980 kg/m³ is lower than that at ρ_cut=920 kg/m³. This is because the separation error in density E_p is not only determined by the separation error Δz_error, but also by the density difference between the process liquid and the polymer. Therefore, even though Δz_error caused by turbulence or other factors is the same, E_p at high density polyolefin separation will be lower than that for low density separation.

Table 7.6 Cut densities and corresponding separation inaccuracy of T1 to T3.

<table>
<thead>
<tr>
<th></th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
</tr>
</thead>
<tbody>
<tr>
<td>ρ_cut</td>
<td>E_p (kg/m³)</td>
<td>ρ_cut (kg/m³)</td>
<td>E_p (kg/m³)</td>
</tr>
<tr>
<td>P1-P2</td>
<td>-</td>
<td>980</td>
<td>4.5</td>
</tr>
<tr>
<td>P2-P3</td>
<td>961</td>
<td>5.5</td>
<td>958</td>
</tr>
<tr>
<td>P3-P4</td>
<td>932</td>
<td>5.5</td>
<td>926</td>
</tr>
</tbody>
</table>
Fig. 7.9 Recovery rate for polyolefins with varying density into products from T1 to T3 (continued).
7.3.2 Sorting with pilot MDS

7.3.2.1 Materials and experiments

Materials
The materials used to evaluate the performance of the pilot MDS were shredded PET bottle caps: a mixture of PP, PE and a small amount of PET flakes. The density distribution of the sample is introduced in Fig. 7.10. Most of the materials had a density between 900-910 kg/m³ and 940-960 kg/m³, which are the typical PP and PE density range respectively. There was less than 1% of > 1000 kg/m³ fraction, later identified as PET flakes. The particles floating in the density range between 960-1000 kg/m³ were PET flakes with air bubbles. Thus, in total, the amount of PET in the sample was about 1%.
Fig. 7.10 Density distribution of the materials processed by the pilot MDS.

**MDS settings**

The strength of the process liquid was roughly 380 A/m, stronger than that used in the lab-scale MDS, in order to compensate for the weaker magnetic field in the pilot MDS channel (introduced in 7.2.2). However, since the magnetic field and magnetization of the process liquid were different, the cut densities were slightly different. The liquid speed in the separation channel was 0.2 m/s. Correspondingly, the processing time of the particles in the pilot MDS was 4.5s. The split positions in the pilot MDS were similar to the lab-scale MDS (presented in Table 7.7).

**Table 7.7 Split position (distance from the magnet surface) of the pilot MDS.**

<table>
<thead>
<tr>
<th>Product</th>
<th>Split position (mm)</th>
<th>Corresponding density range (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>86-108</td>
<td>985-1000</td>
</tr>
<tr>
<td>P2</td>
<td>64-86</td>
<td>970-985</td>
</tr>
<tr>
<td>P3</td>
<td>42-64</td>
<td>940-970</td>
</tr>
<tr>
<td>P4</td>
<td>12-42</td>
<td>860-940</td>
</tr>
<tr>
<td>Residue</td>
<td>0-12</td>
<td>-</td>
</tr>
</tbody>
</table>

To evaluate the sorting performance of the MDS under different throughputs, tests were conducted with three different capacities: 125 kg/h (T4), 250 kg/h (T5) and 320 kg/h (T6). In these tests, the materials were fed into the middle 20 cm of the width of the separation channel, in order to avoid the turbulence effect caused by the stagnant walls on the sides of the separation channel. T7 was carried out with a throughput of 320 kg/h as well, same as T6, but the materials in this test were fed through the entire width of the
separation channel (40 cm) to understand whether the stagnant side walls or the inhomogeneous feeding is a dominant parameter influencing the separation process.

For each test, the separation process was carried out for 30 minutes. After each test, the products and the residues were collected for further analyses. As in T1 to T3, four products and one residual fraction were produced in each test.

### 7.3.2.2 Analysing methods

The products of T4-T7 were analysed with liquid standards of different densities, similarly as T1-T3. Instead of covering from 880-1000 kg/m³ in T1-T3, the analysed density range in T4-T7 was limited to 910-950 kg/m³, since more than 90% of the input materials was within this range.

### 7.3.2.3 Results and discussion

#### Mass distribution

More than 95% of the input materials were recovered in Product 3 and Product 4 for all the four tests, as shown in Table 7.8.

<table>
<thead>
<tr>
<th>Product</th>
<th>T4</th>
<th>T5</th>
<th>T6</th>
<th>T7</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.4%</td>
<td>0.5%</td>
<td>0.3%</td>
<td>0.4%</td>
</tr>
<tr>
<td>2</td>
<td>1.3%</td>
<td>1.2%</td>
<td>2.2%</td>
<td>1.0%</td>
</tr>
<tr>
<td>3</td>
<td>60.2%</td>
<td>40.5%</td>
<td>55.4%</td>
<td>43.5%</td>
</tr>
<tr>
<td>4</td>
<td>37.2%</td>
<td>56.2%</td>
<td>40.0%</td>
<td>54.3%</td>
</tr>
<tr>
<td>Residue</td>
<td>0.9%</td>
<td>1.6%</td>
<td>2.1%</td>
<td>0.8%</td>
</tr>
<tr>
<td>Total</td>
<td>100.0%</td>
<td>100.0%</td>
<td>100.0%</td>
<td>100.0%</td>
</tr>
</tbody>
</table>

#### Density grade

The density grade of the products of each test is presented in Fig. 7.11. Same as in T1-T3, Product 1 (P1) collected the heaviest materials (> 950 kg/m³), and Product 4 (P4) collected the lightest fraction. The fact that P4 also included a big amount of polymer particles with a density larger than 940 kg/m³ indicates that the cut density between the products 3 and 4 in these tests was too high to generate both PP and PE products with high purity.

#### Recovery rate

The recovery curves of the tests (Fig. 7.12) verify the conclusion derived from the density distributions. Table 7.9 summarizes the cut densities between Product 3 & 4 and the corresponding $E_p$ of each test. The cut density of T4-T7 is higher than those in T1-T3, although the weak magnetic field used in T4-T7 was compensated by the stronger magnetic liquid. The reason is that the splitter position between Product 3 and 4 in T4-T7 is 5 mm lower than that in T1-T3. This 5 mm difference results in an increase of 10 kg/m³ in density.
Fig. 7.11 Density distribution of the products from T4 to T7 (continued).
Fig. 7.11 Density distribution of the products from T4 to T7.
Chapter 7

The $E_p$ of T4-T6 is slightly higher than that of T1 to T3. Nevertheless, the tests demonstrated that polyolefins can be separated by the pilot MDS at a separation accuracy of less than 10 kg/m$^3$, and the separation accuracy was not significantly affected by the increased throughput. With such high separation accuracy, products containing 95% pure PP and PE can be achieved if the proper cut densities are applied. The fact that the $E_p$ of T6 is higher than that of T7 shows that it is best to feed the MDS over the full width of the channel, even though this means that flakes flowing near the side walls of the channel may be influenced by the turbulence created by these walls. Apparently, the turbulence created by inhomogeneous feeding of the channel is worse.

Table 7.9 Cut density and corresponding $E_p$ of T4-T7.

<table>
<thead>
<tr>
<th></th>
<th>T4</th>
<th>T5</th>
<th>T6</th>
<th>T7</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cut density (kg/m$^3$)</td>
<td>932</td>
<td>945</td>
<td>937</td>
<td>944</td>
</tr>
<tr>
<td>$E_p$ (kg/m$^3$)</td>
<td>12</td>
<td>10</td>
<td>9.5</td>
<td>7.5</td>
</tr>
</tbody>
</table>

Fig. 7.12 Recovery rate of materials with different densities into products from T4 to T7 (continued).
Fig. 7.12 Recovery rate of materials with different densities into products from T4 to T7 (continued).
CONCLUSIONS

This chapter introduced the lab-scale and pilot MDS and their sorting performance on polyolefins from household wastes. The Lab-scale MDS was designed to further understand the separation process as well as to determine the proper input materials for MDS by sorting small amounts of polyolefin wastes. The pilot MDS was designed for high capacity production in an industrial environment. With both plants, over 98% of polyolefins from household wastes with average thicknesses down to 0.5 mm were successfully recovered as products with sufficient separation accuracy: 10 kg/m³. The density distribution of the products from the lab-scale MDS indicates that the concentration of PP and PE in these products is high, thanks to the appropriate cut densities between PP & PE and high separation accuracy. The pilot MDS produced high concentration PE products, but the PP grade was not sufficient due to the improper cut density. The tests demonstrated that increasing throughput (up to 320 kg/h at least) does not significantly lower the accuracy of the MDS process, and it is best to feed the MDS over the full width of the channel at high capacities, even though this means that flakes flowing near the side walls of the channel are influenced by the turbulence created by these walls. Apparently, the turbulence created by inhomogeneous feeding of the channel is worse. Since the wall region in the pilot prototype is almost 50% of the width of the channel (0.4 m), and commercial plants will have width of 2-3 m, it is expected that their separation accuracy will be even better.
Chapter 8

Quality of Recycled Polyolefins
8.1 INTRODUCTION

Recycled polymers are not widely accepted by product manufacturers because of their uncertain quality (Toldy et al., 2009). Usually, manufacturers only reuse their own waste generated on production lines (post-industrial wastes), since its quality is close to that which recycled and it is predictable. The use of recycled post-consumer materials is possible if the quality of this material is tuned to the needs of the customer and it is constant. Specification systems or standards for recycled polymer are poorly developed, thus the quality of secondary polymers is difficult to be defined. Nevertheless, there are a few key aspects which are normally concerned when the quality of recycled secondary polymers is discussed: (a) the composition of the products: if the product contains more than one type of polymer; (b) the presence of non-polymer, e.g., fillers; (c) the melt flow behaviour of the material; (d) the degree of degradation, considering the mechanical properties of the recyclates; and (e) the colour of the products (Tall et al., 1998). It is also important that the supplied recycled polymers have consistent and stable quality, and they should be comparable with virgin materials to meet the functional requirements of an intended application (Rajendran et al., 2012). To produce high quality secondary polymers which can fulfil these requirements and to increase the market demands for recycled polymers, the sorting technologies from which secondary materials are derived still have to overcome some major technological and economic barriers.

The objective of this Chapter is to assess the quality of the sorted PP and PE products from T1 and T2 in Chapter 7. The quality of the products was evaluated from four perspectives: composition, filler contents, rheological property and mechanical properties.

8.2 ASSESSMENT METHODS

The product analyses covered the above-introduced first four key aspects concerning the quality of the secondary polymers presented in the introduction (Fig. 8.1): (a) the main composition and purity of the products were determined by spectroscopic and thermoanalytical analysis: FTIR and differential scanning calorimetry (DSC); (b) the filler content was measured by thermogravimetry (TG); (c) rheological property: melt flow rate (MFR) was used to characterize the melting properties of polymers which is in connection with the molecular weight and can be used in distinguishing between the polymer types and processing performance; (d) tests were carried out to define the mechanical properties of the products: tensile and impact strength, and flexural property. The colour of the product is not discussed here. After the composition and filler content measurements, the MDS products that contained the same type of polymer, e.g. PP or PE, were mixed as one product for further rheological and mechanical analyses.

In addition to the recycled PP and PE from MDS, virgin polyolefins were tested as well as references in order to evaluate the quality of MDS products. For the composition and filler contents analyses, Moplen HP 400 N type PP from LyondellBasell Industries and TIPELIN BA 550-13 type HDPE from TVK Plc were selected as the references. For the rheological and mechanical properties, besides these two references, three more virgin pol-
ymers from each type were selected, since the processing properties are often diverging. Because the rheological and mechanical properties are highly related to the production methods, the selection of the references took into account that PP and PE from packaging materials are popular in injection and blow moulds respectively. The properties of the references are listed in Table 8.1.

All the MDS product properties were measured by the Department of Organic Chemistry and Technology of Budapest University of Technology and Economics.

---

**Fig. 8.1 Flowchart of the product analyses.**
Table 8.1 Rheological and mechanical properties of the reference polymers.

<table>
<thead>
<tr>
<th>Polymers</th>
<th>Type</th>
<th>Supplier</th>
<th>Rheological</th>
<th>Mechanical</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>(g/10 min)</td>
<td>Tensile</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(MPa)</td>
<td>Strength</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(GPa)</td>
<td>(MPa)</td>
</tr>
<tr>
<td>PP</td>
<td>Moplen HP 400 N</td>
<td>LyondellBasell</td>
<td>11</td>
<td>32</td>
</tr>
<tr>
<td>PP</td>
<td>Moplen HP 400 R</td>
<td>LyondellBasell</td>
<td>25</td>
<td>-</td>
</tr>
<tr>
<td>PPR 9220</td>
<td>TOTAL</td>
<td>LyondellBasell</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>PP 2182*</td>
<td>MAB Polymers</td>
<td>MAB Polymers</td>
<td>35</td>
<td>-</td>
</tr>
<tr>
<td>HDPE</td>
<td>Tipeolin BA 550</td>
<td>TVK Plc</td>
<td>0.35</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>13</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>BB2581</td>
<td>Borealis Borstar</td>
<td>0.40</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>PE BS2541</td>
<td>Borealis</td>
<td>0.30</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>5502 HDPE</td>
<td>TOTAL</td>
<td>0.2</td>
<td>27</td>
</tr>
</tbody>
</table>

* recycled polymer
8.2.1 Composition analyses

All products with the exception of Product 1 of T1 (RO-P1) were ground in an analytical mill in the presence of liquid nitrogen to ensure the representativity of the samples used for spectroscopic and thermo-analytical analysis. Product 1 of T1 (RO-P1) was too little for these analyses. The ground samples were analysed with FTIR and DSC in order to characterize the main polymer components.

8.2.2 Determination of filler content

Thermogravimetry (TG) was used to determine the filler content of products. The analyses methods, the used equipment and the relevant parameters are listed in Table 8.2.

<table>
<thead>
<tr>
<th>Analysis method</th>
<th>Equipment</th>
<th>Relevant parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>FTIR</td>
<td>Bruker Tensor 37</td>
<td>· samples in KBr pastille, Ø10 mm and 1 mm thick;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· spectrometer equipped with NaCl windows, and deuterated triglycine sulfate (DTGS) detector;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· scanning range of wavenumber was 400-4000 cm(^{-1}) with a resolution of 4 cm(^{-1}).</td>
</tr>
<tr>
<td>DSC</td>
<td>Setaram DSC 92</td>
<td>· 10-15 mg of the ground sample;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· 10 µm Al crucible;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· N(_2) atmosphere;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· temperature program consisted of a 1 min isotherm part at room temperature and a subsequent linear part with a heating rate of 10°C/min from room temperature to 550°C.</td>
</tr>
<tr>
<td>TG</td>
<td>Setaram Labsys TG apparatus</td>
<td>· weight of each sample: 20 mg;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· in N(_2) atmosphere;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>· the temperature program consisted of a 1 min isotherm part at room temperature and a subsequent linear part with a heating rate of 10°C/min from room temperature to 700°C.</td>
</tr>
</tbody>
</table>

On the basis of the composition analyses, the products containing the same main polyolefin component were mixed and compounded homogenously using Brabender PlastiCorder PL 2000 machine equipped with a 50 cm\(^3\) kneader chamber. The samples were mixed in the chamber for ten minutes, at 190 °C in case of PP and 160 °C in case of PE, with a rotating speed of 30 RPM. Then the compounded blends were compressed into 4
mm thick sheets in the Collin P 200 E laboratory compression moulding machine at 190°C, with 90 bar. No melt screen was used in compounding.

### 8.2.3 Rheological property

MFR is used to characterize the flow properties of polymer melts in order to determine their optimal processing conditions, which also influences their fields of application. The MFR measurements were carried out with Ceast Modular Melt Flow 7027.000, at 190°C, applying with 21.6 N load.

### 8.2.4 Mechanical properties

Mechanical properties determine the application of a polymer, since they tell manufacturers whether a material is strong, flexible or elastic. These properties are essential to manufacturers to choose the right materials for their applications. Normally, recycled polymers have lower mechanical properties because of the impurities of other polymers or other non-polymer materials, and the manufacture processes may also cause degradation. Evaluating the mechanical properties by comparing to virgin materials tells the possibility of applying the materials in final products.

The compressed polymer sheets were cut into different sizes for the mechanical tests according to the requirements of the standards applied in this study (Table 8.3).

<table>
<thead>
<tr>
<th>Mechanical properties</th>
<th>Method (standard)</th>
<th>Equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>EN ISO 527-5:1997</td>
<td>Zwick Z020</td>
</tr>
<tr>
<td>Flexural strength</td>
<td>EN ISO 178</td>
<td>Zwick Z020</td>
</tr>
<tr>
<td>Impact strength</td>
<td>EN ISO 179 (Notched)</td>
<td>CEAST Resil Impactor Junior</td>
</tr>
</tbody>
</table>

For each composite type, at least five samples were tested under the same working conditions in case of each mechanical test. The reported results are the average of the single values determined.

### 8.3 RESULTS AND DISCUSSION

#### 8.3.1 Composition analyses

The FTIR analysis of the products from both sources (RO HW and NL HW) shows that P4 can be determined as polypropylene, and the main component in P3 and P2 is polyethylene (Fig. 8.2). However, a small amount of PP was observed in P3 from both the Romanian and Dutch samples, as expected from the density analyses. In NL-P1, besides PE, the peaks of PP were discovered as well. That is because of the existence of the high density PP (> 950 kg/m³) in the Dutch household waste (Fig. 2.12).
The DSC analyses verify the conclusions derived on the basis of the FTIR tests (Fig. 8.3). The measurements on the virgin polyolefins show that the melting temperature of PE
and PP was around 136 °C and 169 °C, respectively. The decomposition temperature of PE and PP was 493 °C and 470 °C. Compared with the melting and decomposition temperature of the reference PP and PE, P4 of both RO HW and NL HW contained high purity PP. The main component of both P3 were PE, but mixed with a small amount of PP. The grade of PE in P2 was higher than in P3, as expected from the density distributions. Similarly to the FTIR analysis results, the DSC measurement on NL-P1 indicates that this fraction contained some PP as well.

Fig. 8.3 DSC characterization of the MDS products from Romanian (above) and Dutch (below) household wastes, provided by Budapest University of Technology and Economics.
8.3.2 Filler content

The filler contents in the two PP products were similar: around 11% (Table 8.4). For the PE products from both samples, the filler content increases with the product density. For instance, the filler content in RO-P3 was lower than that in RO-P1.

Table 8.4 Results of TG analysis, provided by Budapest University of Technology and Economics.

<table>
<thead>
<tr>
<th>Product</th>
<th>Residue (mass%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RO-P4</td>
<td>10.9</td>
</tr>
<tr>
<td>RO-P3</td>
<td>9.8</td>
</tr>
<tr>
<td>RO-P2</td>
<td>12.0</td>
</tr>
<tr>
<td>NL-P4</td>
<td>11.5</td>
</tr>
<tr>
<td>NL-P3</td>
<td>&lt;1.0</td>
</tr>
<tr>
<td>NL-P2</td>
<td>12.8</td>
</tr>
<tr>
<td>NL-P1</td>
<td>14.9</td>
</tr>
</tbody>
</table>

8.3.3 Rheological property

The melt flow rate (MFR) of the waste polymers was investigated at temperature of 190 ºC in case of all fractions. Fig. 8.4 shows the measured MFR of the PP and PE products of MDS and the MFR of the references obtained in online databases. After separation, the recycled PP and PE products from Romanian and Dutch household wastes had similar melt flow behaviour: MFR of PP and PE are around 11 g/10min and 0.5 g/10min, respectively. As expected, the MFR of PE is lower than that of PP due to the fact that most of the PE was blow moulded. Compared to the references, the recycled PE had higher MFR. However, the PP was much more viscous than the virgin materials. One of the main reasons can be that the MRF of the recycled PP were measured at 190 ºC instead of 230 ºC at which temperature the references were measured by their supplier.

8.3.4 Mechanical properties

8.3.4.1 Tensile properties

The tensile strength of both the references and the sorted PP and PE are presented in Fig. 8.5. The PP from household wastes have a tensile strength of around 19 MPa, while the sorted PE show higher results. Compared to the average tensile strength of the reference, both the recycled PP have lower values. Nevertheless, the recycled PP nearly reached the tensile strength of TOTAL PPR9220. The tensile moduli of the recycled PP and PE were very comparable with the references. (Fig. 8.6). The tensile modulus of the recycled PP did not decrease because of the manufacture processes. But the PE have lower tensile moduli compared to the virgin references, probably because of the compounding or the mix of a bit PP.
Fig. 8.4 Melt flow properties of the references and the separated waste fractions, provided by Budapest University of Technology and Economics.

Fig. 8.5 Tensile strength of the references and recycled PP and PE, provided by Budapest University of Technology and Economics.
8.3.4.2 Flexural properties
The flexural strength and the flexural modulus are shown in Fig. 8.7 and Fig. 8.8 respectively. It was difficult to determine the flexural strength degradation of the recycled PP and PE since no data were available from the references. The flexural moduli of the recycled PP presents that on average the recycled PP were stiffer than the references. However, the recycled PE showed less stiffness than the virgins, similar as the conclusion derived from the tensile property.

8.3.4.3 Impact strength
The measured Charpy impact strength of the recycled PP is approximately three times higher of the references (Fig. 8.9). The recycled PE showed higher impact strength than the recycled PP. But it was hard to tell whether the impact strength of the recycled PE satisfies the requirement of the applications that can be made with the references.
Fig. 8.7 Flexural strength of the references and recycled PP and PE, provided by Budapest University of Technology and Economics.

Fig. 8.8 Flexural moduli of the references and recycled PP and PE, provided by Budapest University of Technology and Economics.
8.4 CONCLUSIONS

The quality of the recycled PP and PE from both Romanian and Dutch packaging wastes was assessed by concerning four key aspects: (a) the composition of the products: if the product contains more than one type of polymer; (b) the presence of non-polymer, e.g. fillers; (c) the melt flow behaviour of the material; (d) the degree of degradation, considering the mechanical properties of the recyclates. Eight virgin PP and PE were selected as references in order to evaluate the purity and the degradation of the recycled products. According to the composition analyses measured by using FTIR and DSC, the derived products from MDS were high purity PP and PE. The TG analyses presented that there was about 11% filler content in PP, and the filler content of PE increased with the product density. The rheological and mechanical property measurements of the recycled PP and PE showed comparable qualities to the references.
Chapter 9

Modelling of MDS process
9.1 INTRODUCTION

Chapter 7 evaluates the MDS process on polyolefins from household wastes. The tests demonstrate that the $E_p$ of PP and PE separation can reach the 10 kg/m$^3$ accuracy required for high quality recycling. In general, $E_p$ is a function of the feed properties, and the product quality is strongly connected with the value of $E_p$. The smaller the $E_p$ is, the sharper the separation and as a result the purer the product. Therefore, it is necessary to investigate the factors that affect the separation process and contribute to the $E_p$. It is clear that the distribution of the polymers over the products is mainly determined by the feed characterization and the density and magnetization of the process liquid. While, next to these factors, the separation process is also affected by other factors in the MDS plant. Four main factors are discussed in this chapter: poor wettability of polymers, turbulence of the flow, magnetic field errors and the effect of the size of particles. A model was built on the basis of the combined effect of these four factors and validated with two sets of experiments.

9.2 FACTORS AFFECTING SEPARATION ACCURACY

9.2.1 Poor wettability

Poor wettability of polymers reduces the apparent density of polymers. This is because air bubbles are likely to attach to polymers. For the density-based MDS process, the decrease of apparent density of polymers due to air bubbles may result in a few centimetres separation error.

![Fig. 9.1 Floating position of 1-2 mm thick PP (square) and PE (circle) in MDS at different wetting conditions. Dashed lines are the theoretical equilibrium heights.](image)

Improvement of the polymer wettability is related to the wetting time, as introduced in Chapter 4. Fig. 9.1 presents the floating position of smaller than 5 mm PP (910-915...
kg/m³) and PE (950-955 kg/m³) flakes in an MDS system with a process liquid of 420 A/m for different wetting conditions. To show the floating height when there is absolutely no air on the polymers, one group of samples was wetted in boiling water for 10 minutes as a reference. When no wetting was applied, both PP and PE floated higher than their equilibrium height. With increasing boiling time, both polymers gradually moved to their equilibrium position. Note that every test was performed with a fresh sample of polymer flakes. This explains the outlier for PE at 120 s, where particles collect in a narrow range of positions but away from the predicted band.

The change of floating positions indicates that $\zeta_{\text{air}}$, the volume of air attached to the polymers as a fraction of the volume of polymer, decreases with boiling time. The amount of air can be calculated by inserting the actual floating position $z$ of polymers from Fig. 9.1 in Eq. (9.1). The results in Fig. 9.2 show that PE collected more than twice the volume of air compared with PP when no wetting was applied, i.e., an average 40 μm-layer of air for PE versus a 12 μm-layer of air for PP. After 60 seconds of boiling, the effect of air was no longer visible. Since PP and PE are among the most hydrophobic polymers (ACCU DYNE TEST, 2009), 60 seconds boiling is sufficient in order to eliminate the effect caused by air in MDS processes for most polymers to below 1 kg/m³ in terms of the density error.

$$
\zeta_{\text{air}} = 1 - \frac{\rho_i - C_1 e^{-C_2 z}}{\rho_p}
$$

(9.1)

$$C_2 = \frac{\pi}{\rho}
$$

Fig. 9.2 Calculated amount of air on flakes at different wetting conditions. The dash line at 0.1% corresponds to a separation error of approximately 1 kg/m³.
9.2.2 Turbulence

Three major sources of turbulence in MDS are discussed in Chapter 6. Turbulent flow is not completely avoidable, although the design of the MDS attempts to minimize turbulence. For instance the width of the injection tube cannot be too small in practice, consequently the perturbations caused by the shear instability in the injection tube transport into the separation channel, and further develop to turbulence. To study the effect of the turbulent flow on polymer flakes, the flow in the separation channel was studied both numerically and experimentally.

9.2.2.1 Numerical study

The numerical simulations were conducted for both $D = 120$ mm and $D = 50$ mm, and the detailed description of the CFD simulations has been introduced in 6.2.3.2. The simulation did not concern the laminators, consequently the detailed flow profile from the laminators was not included and the profile of $v_z$ coming from all the three injections was assumed perfectly square-shape. The floor and the ceiling of the middle injection of the MDS prototype were described realistically with a stick boundary condition. Therefore the simulated turbulence levels in the separation channel relate to the instability in the feeding tube and to the stick walls in the middle injection, but not to the stochastic flow profile of the laminator tubes. Fig. 9.3 shows the magnitude of the developed turbulence in the simulated separation channel. At $D = 120$ mm, the disturbance leads to a variation of $v_z$ from around -20 mm/s to 15 mm/s, with a root-mean-square speed ($v_{z_{\text{rms}}}$), defined with Eq.(9.2), of 5.6 mm/s. By narrowing the tube width to 50 mm, the magnitude of $v_z$ drops 25 times.

$$v_{z_{\text{rms}}} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} v_{z_{i}}^2}$$

9.2.2.2 Experimental study

The flow in the separation channel was studied experimentally for $D = 120$ mm and $D = 50$ mm as well. Since magnetic fluid is non-transparent, water, which has similar viscosity and density to magnetic liquid, was used instead. The experiments were carried out for a channel velocity of 0.18 m/s. In order to track the flow and to capture the streamlines, thin plastic foils were fed into the system as tracers. To minimize the influence of their own terminal velocity, the selected foils had a size of approximately $3 \times 3$ mm$^2$, with a thickness of 4.5 $\mu$m, and their terminal velocity in water was less than 1 mm/s. These polymer foils were injected through the feeding tube in MDS at seven positions along the horizontal $y$-direction of the 400 mm wide separation channel (Table 9.1). The whole process was recorded with a Canon 5DII camera, which had two million pixels and a recording speed of 30 frames/s. The motion of the flakes was recorded through a viewing window on one of the side walls of the MDS. The videos were analysed with Matlab to obtain the positions
and displacement of the foils. Velocities of the foils were calculated on the basis of their trajectory.

Fig. 9.3 Variation of $v_z$ for $D = 120$ mm (top) and $D = 50$ mm (bottom), simulation provided by BSC.
Table 9.1 Feeding locations along the width of the separation channel for flow measurements.

<table>
<thead>
<tr>
<th>Location y (mm)</th>
<th>P1</th>
<th>P2</th>
<th>P3</th>
<th>P4</th>
<th>P5</th>
<th>P6</th>
<th>P7</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>80</td>
<td>140</td>
<td>200</td>
<td>260</td>
<td>320</td>
<td>390</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 9.4 Snapshot of foils moving along the length of the separation channel.

A video snapshot of the moving foils in the separation channel is shown in Fig. 9.4. Fig. 9.5.a gives an example of the foil trajectories extracted from the video by means of Matlab. The trajectories are not continuous, since the foils often rotated in the flow due to the turbulence, and as a result Matlab failed to distinguish the particles from the background and furthermore to locate their position. Nevertheless, the displacements of the particles provide enough information to understand the vertical motion of the particles. To minimize the quantization noise, the particle positions were averaged for every three frames before calculating displacement and velocities. Fig. 9.5.b presents the $v_z$ values derived from Fig. 9.5.a.

In total, more than 400 sampling points of $v_z$ were obtained for all the seven locations together and for both values of the width of the injection tube. Fig. 9.6 presents the statistics of $|v_z|$. As expected from the numerical simulations, the fluctuation of $v_z$ with $D = 120$ mm was higher than that with $D = 50$ mm. The amount of sampling points with a vertical speed less than 5 mm/s was almost doubled by narrowing the width of the injection tube. For $D = 50$ mm, more than 70% of the $v_z$ had a vertical variation less than 10 mm/s. However, on average, $v_z$, after narrowing $D$, was not as low as expected on the basis of the numerical study. The root-mean-square values of $v_z$ for $D = 120$ mm and $D = 50$ mm were about 12.6 mm/s and 9.4 mm/s, respectively (versus 5.6 mm/s and 0.2 mm/s, respectively, for the simulations). According to the simulation, the shear instability caused turbulence is almost eliminated by reducing $D$ from 120 mm to 50 mm, hence the discrepancy observed during experiments was originated by the source which was not simulated but introduced in section 6.2.1 and 6.2.2: the stochastic flow through the laminators. An estimated $v_z$ of the turbulence from the shear instability is then $\sqrt{12.6^2 - 9.4^2} = 8.4$ mm/s, which is comparable to the simulated value 5.6 mm/s obtained by BSC.
Fig. 9.5 An example of trajectories of moving foils (a, up) and the corresponding variation of $v_z$ (b, below) ($D = 50$ mm).
The distribution of $|v_z|$ also shows that the flow was more turbulent near the sidewalls. For instance with the $D$ of 50 mm, less points reached the class of < 5 mm/s at P1 and P7 (Table 9.2). Especially at P7, some 30% of the points had a speed more than 20 mm/s, which was 3-30 times more than for the other positions.

**Table 9.2 Distribution of $|v_z|$ with $D = 50$ mm.**

<table>
<thead>
<tr>
<th></th>
<th>&lt; 5 mm/s</th>
<th>5-10 mm/s</th>
<th>10-20 mm/s</th>
<th>&gt; 20 mm/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>32.2%</td>
<td>37.4%</td>
<td>26.1%</td>
<td>4.3%</td>
</tr>
<tr>
<td>P2</td>
<td>38.2%</td>
<td>38.2%</td>
<td>22.1%</td>
<td>1.5%</td>
</tr>
<tr>
<td>P3</td>
<td>40.7%</td>
<td>23.7%</td>
<td>28.8%</td>
<td>6.8%</td>
</tr>
<tr>
<td>P4</td>
<td>47.1%</td>
<td>26.5%</td>
<td>26.5%</td>
<td>0.0%</td>
</tr>
<tr>
<td>P5</td>
<td>41.0%</td>
<td>20.5%</td>
<td>28.2%</td>
<td>10.3%</td>
</tr>
<tr>
<td>P6</td>
<td>48.0%</td>
<td>12.0%</td>
<td>36.0%</td>
<td>4.0%</td>
</tr>
<tr>
<td>P7</td>
<td>30.2%</td>
<td>11.6%</td>
<td>27.9%</td>
<td>30.2%</td>
</tr>
</tbody>
</table>

In order to model the effect of the turbulence on the separation for individual flakes, the vertical velocity component of the flow as experienced by a flake moving with the flow is characterised by an amplitude and a frequency. For the amplitude, the root-mean-square values mentioned before is taken. The characteristic frequency is derived from the time-autocorrelation function of $v_z$, introduced in Fig. 9.7, which indicates the “period” of the signal. The graph shows that the autocorrelation decayed to zero in $\tau = 0.5-2.5$ seconds, while half of the sampled fluctuations of $v_z$ de-correlated to zero between 1s and 1.5s. Since the turbulence movement has been simplified as a square wave in the section of 6.2.4 and
the relation between $\tau$ and the period ($T$) is $T = 4\tau$, the period of the turbulent flow in the MDS channel is on average 5s. On the basis of the experimental study of the turbulent flow, the maximum displacement of a polyolefin flake can be estimated with Eq.(6.15).

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig97.png}
\caption{Autocorrelation of $v_z$.}
\end{figure}

### 9.2.3 Magnetic field errors

One of the magnets used in experiments with the MDS system is 800 mm long and 600 mm wide, with a $B_0$ of 0.6 T and $\rho$ of 0.12 m. Overall, it produces a flat magnetic field, and its induction decreases exponentially with the distance to the magnet surface. Therefore, as indicated by Eq.(3.3) and Eq.(3.4), polymers with the same density float at the same height (Fig. 9.8). However, the magnetic field is not completely homogenous. The variation of the field can be indicated by the variation in height of the white and the red polymer balls in Fig. 9.8 at different horizontal locations in the field (Fig. 9.9). Near the borders, the dramatic decrease of the magnet induction results in a 5-20 mm drop of polymer ball height. In the inner area, more than 110 mm from the borders, induction varies much less, causing vertical variations less than 3 mm. Therefore, to avoid the inaccuracy due to the boundary effect, the MDS process should be conducted at least 110 mm from each magnet boundary. The standard deviation of the measurements within the accepted region is about 0.73 mm for the white balls and 1.35 mm for the red balls. That indicates that the separation error caused by uneven magnetic field decreases with the distance between the particle and the magnet.
9.2.4 Size of flakes

Polymer flake size is a final potential contribution to the positioning error. One effect of the flake size is due to the non-linear effective density profile of the magnetic fluid (Fig. 3.2). The other effect is due to the mechanical interaction of the flake with the splitters, making it go randomly above or below after hitting the edge. The former effect can be calculated, while the latter will be studied experimentally. For a circular polymer flake, oriented with its plane parallel to the vertical, with a density ($\rho_p$) and a radius $r$, its centre $(o)$ is slightly off from its equilibrium point $(z_{eq})$ (Fig. 9.10). The effective density of the magnetic liquid at $z$, according to Eq.(3.3) can be expressed with:
\[ \rho_{\text{eff}} = \rho_l + \Delta \rho_{p-l} \cdot e^{-\pi(z-z_o)/p}, \]

\[ \Delta \rho_{p-l} = \rho_p - \rho_l. \]

The area of the flake is

\[ A = \int_{z_o-r}^{z_o+r} 2 \cdot \sqrt{r^2 - (z - z_o)^2} \, dz, \]

therefore, the total force of the flake in the magnetic liquid with a density of \( \rho_l \) can be described as:

\[ F = d \cdot \int_{z_o-r}^{z_o+r} 2 \cdot \sqrt{r^2 - (z - z_o)^2} \cdot \left[ \rho_p - \left( \rho_l + \Delta \rho_{p-l} \cdot e^{-\pi(z-z_o)/p} \right) \right] \cdot dz = 0, \quad (9.3) \]

where \( d \) is the thickness of the flake.

**Fig. 9.10 A circular polymer flake in a magnetic field gradient.**

By solving Eq.(9.3), the correlation between \( z_o \) and \( z_{eq} \), for a “standing” flake, is:

\[ z_o = z_{eq} + \frac{\pi r^2}{8 p}. \quad (9.4) \]

Eq.(9.4) becomes Eq.(9.5), if \( \theta \) is the inclination angle of the flake with respect to the horizontal:

\[ z_o = z_{eq} + \frac{\pi r^2 \sin^2 \theta}{8 p}, \quad (9.5) \]

For small polymer flakes, e.g. \( r < 12 \)mm, this effect is too small (less than 0.5 mm) to contribute significantly to the separation error in practice.

A series of experiments were conducted to study the size effect due to interaction with the splitter. A simple setup was made to imitate the effect of a flake hitting the splitter (Fig. 9.11). In the MDS, the splitters are static, while polymer particles move with a speed of 0.2-0.3 m/s with the flow. In the setup shown in Fig. 9.11, the splitter moves from left to right with a speed of 0.3 m/s in a stagnant liquid. In this way, the mechanical interaction
between the splitter and the flakes is the same in these two setups. The setup was made of transparent Polycarbonate plates, and the process was recorded with a Nikon J1 camera, with filming speed of 400 frames/s. The polyolefin particles used in the tests were 0.7 mm thick, with varying diameters: 23 mm, 20 mm, 16 mm and 10 mm. Circular flakes were used, since it is relatively easy to calculate the orientation angles by image processing. All particles had the same density: 960-970 kg/m³, and the setup was filled with a water-ethanol mixture with a density of 960 kg/m³ in order to minimize the proper motion of the flakes.

**Fig. 9.11** Setup to measure the size effect due to mechanical interaction with the splitter.

In the tests, three parameters of the polyolefin flake were studied to understand their effect on the particle movement (see Fig. 9.12): the distance ($\Delta z$) between the centre point of the flake ($o$) and the splitter at the moment of hitting, the angle ($\Theta$) of the axis $ab$ to the $x$-$y$ plane, and the rotation angle ($\beta$) of the axis $cd$ along $ab$. $\beta$ varied from 0° (if the flake surface was parallel to the $y$-axis) to 90° (if the flake surface was parallel to the $x$-axis). The second picture of Fig. 9.12 illustrates a snapshot of a flake just hit by the splitter.

**Fig. 9.12** A polymer flake hit by a splitter (left: a schema, right: a snapshot).
Fig. 9.13 Correlation among $\theta$, $\Delta z'$ and the final position of the flake (triangle: 23 mm, rhombus: 20 mm, square: 16 mm, circle: 10 mm; red: below the splitter, blue: above the splitter).

Fig. 9.14 Correlation among $\beta$, $\Delta z'$ and the final position of the flake (triangle: 23 mm, rhombus: 20 mm, square: 16 mm, circle: 10 mm; red: below the splitter, blue: above the splitter).

Fig. 9.13 and Fig. 9.14 indicate respectively the effect of $\theta \& \Delta z'$ and $\beta \& \Delta z'$ on the final position of flakes with different sizes after being hit by the splitter. According to the tests, the flakes almost invariably moved above the splitter if $\Delta z' > 0$ and below the splitter if $\Delta z' < 0$, regardless of the flake size, and the angle $\theta$ or $\beta$. In four cases, 20 mm flakes with $\Delta z' > 0$, instead of moving above the splitter, ended up below the splitter after the hit. Coincidentally, this behaviour was only observed for 20 mm flakes. Therefore, the tests conclude that the effect because of the sizes of the input flakes is negligible for MDS separation.
9.2.5 Combined effect of all factors

All the above-mentioned factors influence the separation process independently of each other, thus the combined effect of these four factors ($\sigma$) can be estimated with:

$$\sigma = \sqrt{\sigma_{air}^2 + \sigma_{turbulence}^2 + \sigma_{magnet}^2 + \sigma_{size}^2}$$

(9.6)

in which $\sigma_{air}$, $\sigma_{turbulence}$, $\sigma_{magnet}$ and $\sigma_{size}$ are the errors caused by poor wettability, turbulence, uneven magnetic field and polymer sizes, respectively.

As shown in Section 9.2.1, the effect of air on the MDS process can be entirely eliminated, and also the error due to the size of the polymer flakes is negligible. Therefore Eq.(9.6) becomes:

$$\sigma = \sqrt{\sigma_{turbulence}^2 + \sigma_{magnet}^2}.$$  

(9.7)

$\sigma$ is the expected vertical displacement of a polymer particle from its equilibrium position, and from it, the corresponding density error can be calculated by using Eq. (6.2). Fig. 9.15 gives an example of the variation of $\sigma_{turbulence}$, $\sigma_{magnet}$ and $\sigma$, in kg/m$^3$, as a function of the differential density between the process liquid and polymers. Since the process liquid densities used in polymer separation are close to the density of water, each polymer type has a typical differential density: e.g. the differential density between PP and the process liquid is about 80 kg/m$^3$, for PE 50 kg/m$^3$ and for PVC 400 kg/m$^3$. As shown, the effect of the uneven magnetic field increases linearly with the differential density and gradually becomes the dominant error, whereas the effect of turbulence becomes less important for particles with larger differences in density with respect to the process liquid, and finally the turbulence error remains at a constant value. Therefore, for polymers having a density close to the process liquid, in particular PP, PE, PS and ABS, turbulence is the major cause of an inaccurate separation. For other polymer types, the MDS process can be improved by having a more evenly distributed magnetic field, or let them float far from the magnet surface. As presented in Fig. 9.9, the magnetic field becomes less smooth when reaching the magnet surface. The two dashed lines in Fig. 9.15 indicate that the particles floating close to the magnet surface feel the uneven magnetic field more than those particles suspending far from the magnet.
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Fig. 9.15 Simulated dependence of $\sigma$ with differential densities between polymers and process liquid (polymers are 1 mm thick with $\rho = 1008 \text{ kg/m}^3$, $v_{z,\text{rms}} = 9.5 \text{ mm/s}$, $T = 5\text{s}$ and $C_d = 2$). The purple dash line has a $\sigma_{\text{magnet}}$ of 1.35 mm, and the black dash line has a $\sigma_{\text{magnet}}$ of 0.73 mm.

9.3 EXPERIMENTS

To evaluate the model described by Eq.(9.7), in addition to the tests with Romanian household waste introduced in Chapter 7 (T1), one test was conducted with the residues from shredded window frames (T2).

9.3.1 Materials

PVC window frames consist of a mix of PVC, glass, metals, rubber and other plastics. The glass and metals are usually manually removed, the other materials, mainly polymers, are shredded and sent to different recycling processes. Electrostatic separation is utilized to recover part of the PVC, yet the residue (Fig. 9.16) contains a large amount of PVC mixed with rubbers (Luciania et al., 2013). An example flow of this material, named 1403 quality, described by Luciani et al., included 55% PVC and three types of rubbers representing 45%.

Fig. 9.16 PVC residue stream from window frame waste sample (“1403 quality”) (Luciania et al., 2013).
The density distributions of the “1403 quality” are presented in Fig. 9.17. As shown, the density difference between the rubbers and the PVC is clear: at 1400 kg/m$^3$. But to obtain a high recovery rate, the separation process should be carefully controlled and the separation inaccuracy by using MDS should be no more than 20 kg/m$^3$. The densities of the two main types of rubbers, black and grey rubbers, are different as well. The black rubbers, the majority in the rubber fraction, have mainly two density ranges: < 1160 kg/m$^3$ and 1280-1420 kg/m$^3$. The grey ones are in the density range of 1160-1280 kg/m$^3$. The other difference between PVC and rubbers is their thickness. In general, the PVC is thicker than the rubbers. On average, the PVC and the rubbers are 2mm and 1mm thick respectively.

In both samples, more than 99% of the particles were smaller than 8 mm. The amounts of polymers processed in the tests are listed in Table 9.3. In T2, the samples were wetted with boiling water for 1 minute in order to eliminate the effect of air on the separation. At the same time, the polymers lighter than 1000 kg/m$^3$ in 1403 quality were removed from the input materials.

Table 9.3 Materials for MDS processes.

<table>
<thead>
<tr>
<th>Test no.</th>
<th>Materials</th>
<th>Mass (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>RO HW</td>
<td>2364</td>
</tr>
<tr>
<td>T2</td>
<td>1403 quality</td>
<td>155</td>
</tr>
</tbody>
</table>

9.3.2 Setup

The Lab-scale MDS introduced in Chapter 7 was also utilized respectively for T2. Four products were made in each test, and the splitting positions for T2 are presented in Table 9.4.

Table 9.4 Splitting positions from the magnet surface in T2.

<table>
<thead>
<tr>
<th>Product</th>
<th>Splitting position (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>10-37</td>
</tr>
<tr>
<td>II</td>
<td>37-59</td>
</tr>
<tr>
<td>III</td>
<td>59-81</td>
</tr>
<tr>
<td>IV</td>
<td>81-103</td>
</tr>
</tbody>
</table>

Concentrated magnetic liquid MSG10 (FerroTec, 2011) was mixed with water to make the process liquids that can create a density range covering the entire density distribution of the input materials in the magnetic field (Table 9.5). For T2, the liquid was moving in the separation channel with a speed of 0.18m/s.

Table 9.5 Properties of process liquids.

<table>
<thead>
<tr>
<th>Test</th>
<th>Magnetization (A/m)</th>
<th>Liquid density (kg/m$^3$)</th>
<th>Effective density range in MDS (kg/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T2</td>
<td>650</td>
<td>1008</td>
<td>1000-2000</td>
</tr>
</tbody>
</table>
Fig. 9.17 Density distribution of sample “1403 quality”.

**Fig. 9.17 Density distribution of sample “1403 quality”**.
9.3.3 Product analyses

After the test, the products were analysed with a series of liquid standards of different densities, in order to study the density distribution of each product and furthermore to obtain the inaccuracy of separation. Two types of solutes were used to make the liquid standards for T2: NaCl and sodium polytungstate (SPT). The density of solutions of water and salt ranged from 1000 kg/m³ to 1200 kg/m³, and the mixtures of water and SPT had a density from 1200 kg/m³ to 1500 kg/m³. The density intervals for both were 20 kg/m³.

9.3.4 Separation results and discussion

Three cut densities were produced in T2. The cut density between Product I and Product II was nearly 1370 kg/m³ (Fig. 9.18). Although it is lower than 1400 kg/m³, the point in between the density distributions of rubber and PVC, a high PVC grade of 97.9% was achieved in Product I with a recovery of 99.7%. This is because only approximately 1% of the input was between 1370 kg/m³ and 1400 kg/m³, and the material in this density range accounted for 1.2% of Product I. In addition to the PVC product, T2 generated two clean rubber products as well (Table 9.6), with the two cut densities: 1240 kg/m³ between Product II and Product III, and 1120 kg/m³ between Product III and Product IV.

The cut densities between every two products were intentionally selected at those densities where two types of polymers were distinguished and also the least materials were discovered. Consequently, the amount of the polymers near the cut densities in each product is often too small to obtain an accurate density analysis. For instance, the recovery rates of the polymers from Product II and III at 1250 kg/m³ and 1270 kg/m³ did not follow the shape of the curves (also known as Partition curve), due to the tiny amounts of the polymers with such densities recovered in these two products (less than 1.2 g). To eliminate the inaccuracy caused by this fact, additional fitting curves are added into Fig. 9.18. These fitting curves concern the original points derived from sufficient amount of samples as well as the shape of the Partition curve. The separation efficiencies, calculated according to these recovery curves, at the three cut densities are presented in Table 9.7.

<table>
<thead>
<tr>
<th>Product</th>
<th>Black rubber</th>
<th>Grey rubber</th>
<th>PVC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade</td>
<td>Recovery</td>
<td>Grade</td>
<td>Recovery</td>
</tr>
<tr>
<td>I</td>
<td>2.3%</td>
<td>0.0%</td>
<td>97.7%</td>
</tr>
<tr>
<td>II</td>
<td>97.7%</td>
<td>1.9%</td>
<td>0.4%</td>
</tr>
<tr>
<td>III</td>
<td>19.1%</td>
<td>80.9%</td>
<td>0.0%</td>
</tr>
<tr>
<td>IV</td>
<td>88.3%</td>
<td>11.7%</td>
<td>0.0%</td>
</tr>
</tbody>
</table>

Table 9.7 $E_p$ at different cut densities in T2.

<table>
<thead>
<tr>
<th>Cut density (kg/m³)</th>
<th>$E_p$ [kg/m³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1120</td>
<td>7.5</td>
</tr>
<tr>
<td>1240</td>
<td>11.8</td>
</tr>
<tr>
<td>1370</td>
<td>12.4</td>
</tr>
</tbody>
</table>

*derived from the fitted recovery curves in Fig. 9.18.
Fig. 9.18 Recovery rate of the polyolefins with different densities in each product for T2 (the dash lines are the fitted recovery curves).

Fig. 9.19 compares the experimental and simulated $E_p$ for the polymers in tests T1 and T2. It shows that the model described in Eq.(9.7) successfully predicts the trend and level of the separation inaccuracy with differential densities.

From both the experiments and the simulations, we can conclude that MDS can accurately separate polymer mixtures based on their density. With such high accuracies in density, polymer mixtures can be collected into different polymer types by MDS in a single step provided that there is a minimum gap of 20–40 kg/m$^3$ between the density ranges of the target polymers.
9.4 CONCLUSIONS

Four main factors that may cause separation errors were discussed: poor wettability of polymers, turbulence of the flow, magnetic field errors and the effect of the size of particles. Experiments showed that improvement of the polymer wettability is related to the wetting time. 60 seconds boiling is sufficient in order to eliminate the effect caused by air in MDS processes. A major effect that influences the MDS separation is turbulence. Numerical simulations and experiments were carried out to measure the turbulence in the separation channel. The error caused by the uneven magnetic field was obtained by measuring the floating height of polymer balls in a glass of magnetic liquid. The measurements showed that the inaccuracy of industrial permanent magnets can be around 1 mm, if the MDS process is conducted at least 110 mm from each boundary of the magnet. Polymer flake size is a final potential cause of the position error. One effect of the flake size is due to the non-linear effective density profile of the magnetic fluid, the other effect is due to the mechanical interaction of the flake with the splitters, making it go randomly above or below. For small polymer flakes, e.g. < 23 mm, the size effect is negligible for MDS separation.

A model was built on the basis of the combined effect of these four factors, and it was validated with two experiments. The evaluation shows that the model can successfully predict the level and trend of the separation inaccuracy with differential densities. The effect of the uneven magnetic field increases linearly with the differential density, whereas the effect of turbulence becomes less important for polymers with larger differences to the process liquid in density. Therefore, for polymers having a density close to the process liquid, e.g. PP, PE, PS and ABS, turbulence is the major cause of an inaccurate separation.
For other polymer types, the MDS process can be improved by having a more evenly distributed magnetic field or let them float far from the magnet surface. From both the experiments and the simulations, it is concluded that MDS can accurately separate polymer mixtures based on their density if the intended polymer products have a gap in their density range of 20-40 kg/m³. With such high accuracies in density, polymer mixtures can be collected into different polymers according to their type by MDS in a single step.
Chapter 10

Conclusions and Recommendations
Polymer recycling has significantly positive impacts on the environment, economics and society. Using recycled polymer to replace virgins reduces the growing amount of post-consumer polymer wastes, decreases the demand for fossil fuel and creates local jobs. As the most used polymer types, polyolefins certainly have attracted attention of recycling. Mechanical sorting has been pointed out as the preferred route for the recovery of relatively clean and homogeneous plastic waste streams. Whereas washing and compounding technologies for polymer scrap have dramatically improved over the last decade, there is as yet no sorting technology commercially available that is both cheap and effective. Consequently, the amount of recycled post-consumer mixed plastics remains low and the recycled polymer value is even worse. Innovative recycling technologies for post-consumer wastes should be extremely powerful, simple, cost-effective and accurate enough to create high quality products.

Magnetic density separation is a density-based sorting technology that is similar to the traditional sink-float method. Its innovation is to use a medium not with a single density but one that has a density gradient. By applying artificial gravity in the form of a magnetic force that varies exponentially in the vertical direction, the effective density of the liquid varies in this direction as well. Particles with the same density will collect at a level in the liquid where the effective density is equal to their density. This process takes a few seconds and multiple products can be achieved in one single step. No polluted liquid needs to be discharged, even though it is a wet process, since the separation medium circulates during the process. Such advantages allow MDS to be more cost-effective than other recycling techniques. Nevertheless, early results demonstrated that substantial research on MDS process with a target of improving the polyolefin separation should be undertaken to obtain high quality secondary polyolefins from complex waste mixtures.

To effectively recycle polyolefins, the first important step is to determine and characterize the proper input waste streams. Characterization of the waste streams should reflect the parameters that are vital for predicting the result of recycling processes and the quality of the recycled products. Concerning the lifetimes and the total consumption of polyolefin applications, household packaging and car components are two interesting streams for MDS. The characterization of packaging wastes from both Romania and the Netherlands show that PP and HDPE are in general found to be injection and blow moulded respectively. The density difference between PP and HDPE indicates that density separation can sort these two types of polyolefins and the appropriate cut density should be between 920 kg/m³ and 930 kg/m³. The separation inaccuracy for such technologies is recommended to be lower than 10 kg/m³ to obtain products with both high grade and high recovery. In addition to the type of polymers, packaging polyolefins can be identified by their melt flow behaviour. Melting temperature and rheological properties distinguish the moulding type used in production. Therefore, it is necessary to sort not only on polymer types but also on mould types for the purpose of reaching secondary polyolefin products with good mechanical properties.

The investigation on over 100 kg of larger car components shows that most of the analysed components are PP. The density distribution of the PP components covered from
880 kg/m³ to even > 1000 kg/m³, because some of the PP are reinforced for certain applications. The PE, from the two blow moulded water tanks, have a typical PE density: 930-950 kg/m³. According to the density distribution, it is possible to remove PE from the PP mixtures less than 1000 kg/m³ by extracting the particles with a density between 930 kg/m³ and 950 kg/m³ as one product, and the rests as other products. This is very useful especially for shredded car polymer components. However, for the hand-sorted car components (for instance this method is carried out in Eastern European countries since the labour cost is low), the applicability of MDS is low.

A deep study on magnetic density separation is obviously an essential step to apply this technology into polyolefin recycling. To reach the necessary separation accuracy for gaining high quality secondary PP and PE from packaging wastes in one single step, the separation technology needs to be sensitive to very small differences in the physical properties of the input materials. Moreover, in order to be economical and ecological, processes should recover most of the polyolefins in a useful product and minimize process residues. On the basis of these aspects, a scheme for an MDS system is provided in this thesis. Considering the introduced MDS procedure, three main aspects have received attention: improvement of polymer wettability, magnetization control of the process liquid and minimization of turbulence.

Good wettability of polymers is essential for high-accuracy sink-float separation in water-based media such as MDS, because the grade and recovery of the products are critically influenced by even a small percentage of air bubbles. The study shows that the wettability of most polymers improve with immersion in boiling tap water, and the success of wetting in boiling water can be explained by the deposition of a thin layer of calcium carbonate on the surface of the flakes. Shredded plastics are easier to be wetted than virgin polymers, probably because a relatively rougher surface is caused by the shredder process (as predicted by the Wenzel equation). A new wetting process to eliminate air bubbles was developed on the basis of these results. An important feature is that the wetted flakes carry a small amount of heat and water with the feed to the MDS process liquid.

The cut-density of sink-float separations in magnetic liquids depends on the magnetization of the process liquid. The control of the magnetization of the liquid is therefore a critical part of the MDS technology. However, a potential problem is that the process liquid may become inhomogeneous and will segregate in the magnetic field region. There are two parameters governing the mixing of concentrated magnetic liquid in the process liquid with different magnetization: the size of the inhomogeneity and the value of the magnetic field. For a magnetic field amplitude of 0.6 T and pole size of 120 mm, the critical size of droplets of concentrated magnetic liquid (12000 A/m) to dissolve into the main flow and to avoid segregation into layers of different magnetization is 60 μm. Segregation of the process liquid due to incomplete mixing can be avoided by utilizing a static mixer which is able to reduce the size of structure of concentrated magnetic fluid down to 60 μm. Based on a magneto-gravimetric principle, a magnetization measurement tool was designed to detect and control fluctuations of in an industrial environment.
Turbulence is a final major factor that affects MDS processes for polyolefin recycling. To obtain valuable secondary polyolefins using MDS, the separation accuracy should reach 4-5 mm in the vertical coordinate. It is thus critical to prevent turbulent flow. Three types of sources of turbulence in the MDS setup were studied: turbulence produced near static boundaries, turbulence generated by the laminator tubes, and the shear instability created at the interface between the flow from the middle laminator and the static liquid in the injection tube. The laminator tubes in the MDS system can effectively prevent big eddies entering the separation channel, but they may also produce eddies due to the fact that flow through individual tubes is itself stochastic in nature. Both the analytical and numerical studies reveal that shear instability in the injection tube is growing exponentially along the flow direction. By narrowing the width of the injection tube, the frequency of the developed turbulence increases, resulting in a faster decay of the eddies transported to the separation channel.

Two MDS setups designed on the basis of the above-mentioned concerns were constructed for laboratorial and industrial environment. Their performance on post-consumer polyolefins from household wastes demonstrates that over 98% of the polyolefins waste can be successfully recycled with sufficient separation accuracy: 10 kg/m³. The concentration of PP and PE in the products of lab-scale MDS as indicated by their density distribution is of the order of 95%. The separation results of using pilot MDS conclude that increasing throughput does not lower the accuracy of the MDS process, and it is best to feed the MDS over the full width of the channel, even though this means that flakes flowing near the side walls of the channel are influenced by the turbulence created by these walls. Apparently, the turbulence created by inhomogeneous feeding of the channel is worse than the turbulence created the side walls.

The evaluation of the quality of the recycled PP and PE from the lab-scale MDS concerns four key aspects of the polymers: a) the main composition and purity of the products; b) the presence of non-polymer, e.g. fillers; c) the melt flow behaviour of the material and d) the degree of degradation, considering the mechanical properties: tensile and impact strength, and flexural property. According to the analyses, the derived products from MDS are high purity PP and PE. Based on the mechanical analyses, the separated household wastes show good and consistent properties independently from their origin, which are comparable with neat polymers.

Further improvement on MDS process may be necessary especially if thinner materials in the future become interesting. Therefore, attention has been paid to the factors that influence the MDS separation process. Next to the distribution of the polymers over the products, there are four main factors: poor wettability of polymers, turbulence of the flow, magnetic field errors and the effect of the size of particles. The investigations of these four factors display that for polymer flakes less than 23 mm, if the wetting time is sufficient (more than one minute), the separation accuracy is only determined by the turbulence error and the magnet error. A model built on the basis of the combined effect of these factors shows that the effect of the uneven magnetic field increases linearly with the differential density, whereas the effect of turbulence becomes stable for polymers with larger
differences to the process liquid in density. The combined effect shows that for most of the polymers (density < 1400 kg/m³) turbulence is the major cause of an inaccurate separation. With both the experiments and the simulations, it is concluded that MDS can accurately separate polyolefin mixtures based on their density. With such high accuracies in density, polyolefin mixtures can be collected into different polymers according to their type by MDS in a single step.

To further improve the MDS technology for industry purposes and for other potential applications, the following aspects should deserve more attention. 1) Developing new wetting technologies with low energy consumption. The current pilot MDS contains three heating elements with a total energy consumption of 26 KW. Such consumption is nearly double of the total consumption of the other MDS components (15KW). During the boiling, steam is continuously generated and it can be dangerous. New wetting technologies with low energy consumption are encouraged. In addition, considering that most of the polymer recycling lines include a washing step, hence it is a good idea to connect MDS directly after the washing so that the wetting procedure can be skipped when the wettability of the polymers are proper for the MDS process. 2) Further turbulence control. With current flow situation in the separation channel, the MDS can accurately separate polymers with a thickness larger than 0.3-0.5 mm. For polymers thinner than 0.3 mm, in particular polymer foils (thickness less than 0.1 mm), MDS is not able to sort them precisely. According to the above study, turbulence control is the key of solving this problem. Based on the numerical simulation, the shear instability is no longer an issue when the injection tube is narrowed to 50 mm, but instead, turbulence caused by the laminator tubes becomes visible. In order to process thinner materials, the stochastic of these tubes needs to be reduced or even eliminated and the size of the tubes should be smaller. 3) Particle interaction in MDS. The capacity of the MDS is often calculated based on 1-2% polymer volume percentage in the separation channel. Such low volume percentage is to ensure no particle interaction occurs during separation. However, the upper limit of the polymer volume percentage without particle interaction is unknown. More investigations are desired to address this problem so the capacity of MDS can be increased. 4) One MDS setup to sort both light and heavy polymers. The current MDS can process either light or heavy polymers, depending on the configuration. With a polymer mixture for instance containing PP, PE, PS and PET, the existing MDS is able to produce three products: a mixture of PP & PE, PS and PET; or PP, PE and a mixture of PS & PET. However, it is interesting to achieve four products in one step by applying two magnets both on the top and on the bottom of the separation channel.
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<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>ABS</td>
<td>Acrylonitrile butadiene styrene</td>
</tr>
<tr>
<td>ASR</td>
<td>Automotive shredder residue</td>
</tr>
<tr>
<td>B&amp;C</td>
<td>Building and construction</td>
</tr>
<tr>
<td>BM</td>
<td>Blow moulding</td>
</tr>
<tr>
<td>BSC</td>
<td>Barcelona Supercomputing Centre</td>
</tr>
<tr>
<td>CFD</td>
<td>Computational fluid dynamics</td>
</tr>
<tr>
<td>DSC</td>
<td>Differential scanning calorimetry</td>
</tr>
<tr>
<td>DTGS</td>
<td>Deuterated triglycine sulfate</td>
</tr>
<tr>
<td>FTIR</td>
<td>Fourier transform infrared spectroscopy</td>
</tr>
<tr>
<td>HDPE</td>
<td>High density polyethylene</td>
</tr>
<tr>
<td>HW</td>
<td>Household waste</td>
</tr>
<tr>
<td>IDIS</td>
<td>International dismantling information system</td>
</tr>
<tr>
<td>IM</td>
<td>Injection moulding</td>
</tr>
<tr>
<td>LDPE</td>
<td>Low density polyethylene</td>
</tr>
<tr>
<td>LLDPE</td>
<td>Linear low-density polyethylene</td>
</tr>
<tr>
<td>MDS</td>
<td>Magnetic density separation</td>
</tr>
<tr>
<td>MFR</td>
<td>Melt flow rate</td>
</tr>
<tr>
<td>MIP</td>
<td>Maximum intensity projection</td>
</tr>
<tr>
<td>MSW</td>
<td>Municipal solid waste</td>
</tr>
<tr>
<td>NIR</td>
<td>Near infrared sorting</td>
</tr>
<tr>
<td>NL HW</td>
<td>Dutch household waste</td>
</tr>
<tr>
<td>PBT</td>
<td>Polybutylene terephthalate</td>
</tr>
<tr>
<td>PC</td>
<td>Polycarbonate</td>
</tr>
<tr>
<td>PE</td>
<td>Polyethylene</td>
</tr>
<tr>
<td>PEF</td>
<td>Polyethylene furanoate</td>
</tr>
<tr>
<td>PET</td>
<td>Polyethylene terephthalate</td>
</tr>
<tr>
<td>PHB</td>
<td>Polyhydroxybutyrate</td>
</tr>
<tr>
<td>PLA</td>
<td>Polylactic acid</td>
</tr>
<tr>
<td>PLC</td>
<td>Programmable logic controller</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
</tr>
<tr>
<td>--------------</td>
<td>--------------------------------</td>
</tr>
<tr>
<td>PO</td>
<td>Polyolefins</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>PS</td>
<td>Polystyrene</td>
</tr>
<tr>
<td>PS-E</td>
<td>Expanded polystyrene</td>
</tr>
<tr>
<td>PUR</td>
<td>Polyurethane</td>
</tr>
<tr>
<td>PVC</td>
<td>Polyvinyl chloride</td>
</tr>
<tr>
<td>RO HW</td>
<td>Romanian household waste</td>
</tr>
<tr>
<td>RPM</td>
<td>Rotation per minute</td>
</tr>
<tr>
<td>SPT</td>
<td>Sodium polytungstate</td>
</tr>
<tr>
<td>TG</td>
<td>Thermogravimetry</td>
</tr>
<tr>
<td>TPS</td>
<td>Thermoplastic starch</td>
</tr>
<tr>
<td>WEEE</td>
<td>Waste electrical and electronic equipment</td>
</tr>
</tbody>
</table>
### Notations

**Greek Lower Case**

\( \alpha \)  =  decay factor of eddies  
or a geometrical factor

\( \beta \)  =  rotation angle of a circular polymer flake

\( \delta \)  =  thickness of flow boundary layer

\( \delta_{\text{air}} \)  =  thickness of the laminar air layer

\( \delta_{\text{water}} \)  =  thickness of water vapour

\( \eta \)  =  dynamic viscosity

\( \phi \)  =  contact angle

\( \lambda \)  =  wavelength

\( \lambda_p \)  =  polymer thermal conductivity

\( \nu \)  =  kinematic viscosity

\( \theta \)  =  inclination angle of steaming screw  
or rotation angle of a circular polymer flake

\( \rho \)  =  density  
or density interval

\( \rho_{25} \)  =  density with a recovery in the heavy product of 25%

\( \rho_{75} \)  =  density with a recovery in the heavy product of 75%

\( \rho_{\text{cut}} \)  =  cut density

\( \rho_d \)  =  material density of a small droplet of water or magnetic liquid

\( \rho_d' \)  =  effective density of magnetic droplets

\( \rho_{\text{eff}} \)  =  effective density of magnetic liquid

\( \rho_m \)  =  material density of magnetic liquid

\( \rho_p \)  =  non-magnetic particle density

\( \rho_{p,\text{eff}} \)  =  effective density of a polymer flake

\( \rho_w \)  =  water density

\( \sigma \)  =  standard deviation of a set of data  
or combined separation error

\( \sigma_{\text{air}} \)  =  separation error caused by air bubble

\( \sigma_{\text{magnet}} \)  =  separation error caused by uneven magnetic field

\( \sigma_{\text{size}} \)  =  separation error caused by flake size
\( \sigma_{\text{turbulence}} \) = separation error caused by turbulence
\( \tau \) = time-lag of autocorrelation function or elapsed time
\( \omega \) = frequency of perturbation
\( \zeta \) = displacement in \( z \)-direction of points on the surface of discontinuity due to perturbation
\( \zeta_{\text{air}} \) = volume percentage of air attached to a polymer particle

**Greek Upper Case**

\( \Delta \rho_{\text{error}} \) = separation error in density
\( \Delta \rho_{p-l} \) = \( \rho_p - \rho_l \)
\( \Delta u \) = decay of fluctuations caused by the stochastic behaviour of the laminator tubes
\( \Delta z \) = distance between a polymer particle to its equilibrium height
\( \Delta z' \) = distance between the centre of a polymer flake and a splitter
\( \Delta z_{\text{error}} \) = separation error in distance
\( \Delta z_{\text{max}} \) = maximum displacement of a particle from its \( z_{eq} \) caused by turbulence
\( \Phi \) = flux of heat

**Roman Lower Case**

\( d \) = particle thickness
\( d_a \) = diameter of a small droplet of water or magnetic liquid
\( d_{\text{nano}} \) = diameter of the nano-size particle in magnetic liquid
\( g \) = gravity acceleration
\( k \) = wavenumber of perturbation
\( k_0 \) = \( 1.3/\delta \)
\( p \) = pole size of a magnet
\( p \) = pressure perturbation
\( p_i \) = pressure perturbation at Layer \( i \)
\( r \) = radius of a circular polymer particle or roughness of polymer surface
\( t \) = time in second
\( u \) = \( u' + v \)
**Notations**

- \( u', u' \) = small perturbation
- \( u_x \) = profile of periodic flow
- \( u'_x \) = component of \( u' \) in \( x \)-direction
- \( u_z \) = component of \( u \) in \( z \)-direction
- \( u'_z \) = component of \( u' \) in \( z \)-direction
- \( v \) = flow velocity
- \( v_{av} \) = average velocity
- \( v_b \) = velocity profile in the boundary layer in shear instability analysis
- \( v_p \) = particle velocity in \( z \)-direction
- \( v_t \) = terminal velocity in \( z \)-direction
- \( v_x \) = flow velocity in \( x \)-direction
- \( v_z \) = flow velocity in \( z \)-direction
- \( v_{z_{rms}} \) = root-mean-square average \( v_z \)
- \( x \) = horizontal Cartesian axis or distance \( x \)-direction
- \( x_0 \) = length of the region of flow establishment in \( x \)-direction
- \( y \) = distance from the plane defined by the \( x \) and \( z \) axes
- \( z \) = vertical Cartesian axis or particle position in \( z \)-direction
- \( z_{eq} \) = equilibrium height of a polymer particle in \( z \)-direction
- \( z_0 \) = height of the centre of a circular polymer flake

**Roman Upper Case**

- \( A \) = area of a flake or a constant
- \( A \) = profile of the amplitude of \( u' \) in \( z \)-direction
- \( A_{projected} \) = projected area of surface
- \( A_{true} \) = true area of surface
- \( B \) = magnetic induction or a constant
- \( B_0 \) = magnetic induction on a magnet surface
- \( C_1 \) = \( \pi MB_0/gp \)
- \( C_2 \) = \( \pi/p \)
- \( C_d \) = drag coefficient
\( C_p \) = polymer heat capacity
\( D \) = diameter of injection tube of MDS
\( D' \) = diffusion coefficient
\( D_{air,vapour} \) = air vapour diffusion coefficient \((10^{-5}-10^{-6} \text{ m}^2\text{s}^{-1})\)
\( E_p \) = probable error of a separation
\( F \) = total weight on magnetic liquid
\( F_b \) = buoyancy
\( F_{buoyancy+gravity} \) = total force of buoyancy and gravity
\( F_{drag} \) = drag
\( F_{mag} \) = magnetic force
\( G \) = gravity
\( H \) = depth of the solid boundary in shear instability analysis or the height of the middle injection of MDS
\( H_0 \) = initial heat content of a flake
\( H_t \) = heat content of a flake at time \( t \)
\( H_{vapour} \) = water enthalpy
\( L \) = Avogadro’s number
\( M \) = magnetization of magnetic liquid
\( Mасс_{pi} \) = mass of polyolefins in product \( i \)
\( P_0 \) = pressure at 1 atm
\( P_{T,air} \) = air pressure at \( T \) (K)
\( P_{T,sat} \) = water vapour pressure
\( Q_w \) = heat of evaporation
\( R \) = gas constant
\( Re \) = Reynolds number
\( R_p \) = recovery rate within a certain density interval \( p \)
\( S \) = diffusion length scale
\( T \) = temperature or period of turbulent flow
\( V_{293,air} \) = air volume at temperature 293 K
\( V_d \) = volume of a small droplet of water or magnetic liquid
\( V_l \) = volume of magnetic liquid
\( V_p \) = volume of a polymer particle
\( V_{T, \text{air}} \) = air volume at temperature \( T \) (K)
\( V_{T, \text{bubble}} \) = bubble volume at \( T \) (K)
\( V_{T, \text{vapour}} \) = vapour volume at temperature \( T \) (K)
The journey of purchasing the Dr. title was full of contradictions: happy & sad, exciting & depressing, confused & confident, lonely & sociable, sacrifice & reward, failure & success…… Our life is unique because of the memories we have stored, the things we have been through and the people we have met. Certainly, my PhD life will be unforgotten because of all the people with whom I have spent wonderful time here.

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In January of 2009, she began as a PhD candidate at TU Delft under the supervision of Prof.dr. Peter Rem. The research topic was “Magnetic density separation of polyolefin wastes”, funded by the EU FP7 project “W2Plastics”.

From September of 2013, she continued her career at Fondel Urban Mining Ventures for the further development of the Magnetic Density Separation technology.
Polymer recycling reduces the growing amount of post-consumer polymer wastes, decreases the demand for fossil fuel and creates local jobs. However, the amount of recycled post-consumer mixed plastics remains low and the recycled polymer value is even worse. This fact calls for innovative recycling technologies that are extremely powerful, simple, cost-effective and accurate enough to create high quality products. Magnetic Density Separation (MDS) is such a technology. This thesis focuses on both the basic principle and the practical issues of MDS processes in polyolefin recycling. The study leads to a new solution for polyolefin wastes.