Tampereen yliopisto

Mona Grann

POST-INDUSTRIAL PLASTICS SIDE STREAM RECYCLING

Master's Thesis
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August 2024

ABSTRACT

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Master’s Thesis
Tampere University
Materials Engineering
August 2024

Polyolefin pellet production generates a considerable amount of post-industrial materials, which are materials separated from waste streams during the manufacturing process. These materials represent a resource with great potential for promoting more efficient raw material use. This thesis investigates the feasibility of incorporating post-industrial waste from polypropylene compounding processes with prime grades, aiming to maintain the product performance while enhancing circular material use and creating economic value from the side streams. Conducted in collaboration with Borealis Polymers Oy, this study also explores the best practices, current policies, and industry trends in side stream recycling of polyolefins.

Pilot-scale compounding experiments were performed on two commercial polypropylene grades, incorporating up to 10 weight percent of post-industrial recycled content across four batches for each grade. The quality assessment compared the properties of the recycled compounds with those of the prime grades. Test methods were selected based on the end-use application and predetermined performance standards. The objective was to address whether the inclusion of post-industrial content could maintain the performance of the grade.

The experiments demonstrated that incorporation of post-industrial materials with prime resins did not significantly alter product properties. Therefore production-scale test runs are recommended for both examined products. The results are consistent with existing literature on polypropylene recyclate compounding. Moreover, it was found that due to regulatory aspects and trade secrets, several polyolefin pellet producers do not publicly disclose their methods relating to post-industrial material treatment. In general, more information is available about post-industrial content utilization in further conversion processes and by third parties.

Keywords: polyolefins, post-industrial

The originality of this thesis has been checked using the Turnitin Originality Check service.

TIIVISTELMÄ

Mona Grann: Teollisuudessa syntyvien muovisivuvirtojen kierrätys
Diplomityö
Tampereen yliopisto
Materiaalitekniikan tutkinto-ohjelma
Elokuu 2024

Polyolefiinien pelletointiprosessissa syntyy huomattava määrä jälkiteollisia materiaaleja, jotka ovat valmistusprosessin aikana jätevirrasta erotettuja materiaaleja. Näillä materiaaleilla on resurssina mahdollisuuksia edistää raaka-aineiden tehokkaampaa käyttöä. Tässä diplomityössä tutkitaan, onko mahdollista sisällyttää polypropeenin sekoitusprosesseissa syntyvää jälkiteollista materiaalia ensiömuoveihin, jotta tuotteen suorituskyky säilyisi ennallaan. Samalla tehostettaisiin materiaalien kiertokäyttöä ja luotaisiin sivuvirroista taloudellista arvoa. Tutkimuksessa, joka tehtiin yhteistyössä Borealis Polymers Oy:n kanssa, tutkitaan myös polyolefiinien sivuvirtakierrätyksen parhaita käytäntöjä, nykyisiä toimintatapoja ja alan suuntauksia.

Pilottimittakaavan sekoituskokeita suoritettiin kahdella kaupallisella polypropeenilaadulla, joihin sisältyi jopa 10 painoprosenttia teollisuuden jälkeistä kierrätyssisältöä neljässä erässä kummankin laadun osalta. Laadunarvioinnissa verrattiin kierrätettyjen yhdisteiden ominaisuuksia ensiömuovin ominaisuuksiin. Testimenetelmät valittiin loppukäytön ja ennalta määritettyjen suorituskykyvaatimusten perusteella. Tavoitteena oli selvittää, voidaanko jälkiteollisen materiaalin sisällyttämisellä säilyttää laatuluokan suorituskyky.

Tutkimus osoitti, että jälkiteollisten materiaalien lisääminen ensiömuovilaatuun ei muuttanut merkittävästi tuotteen ominaisuuksia. Tämän vuoksi suositellaan molemmille tutkituille tuotteille tuotantomittakaavan koeajoja. Tulokset ovat yhdenmukaisia polypropeenin kierrätysmateriaalin sekoittamista koskevan kirjallisuuden kanssa. Lisäksi havaittiin, että sääntelyn ja liikesalaisuuksien vuoksi useat polyolefiini pellettien tuottajat eivät julkisesti paljasta jälkiteollisten materiaalien käsittelyyn liittyviä menetelmiään. Yleisesti ottaen on saatavilla enemmän tietoa teollisuuden jälkeisen materiaalin hyödyntämisestä jatkokäsittelyprosesseissa ja kolmansien osapuolten toimesta.

Avainsanat: polyolefiinit, jälkiteollinen

PREFACE

This master's thesis was conducted in collaboration with Borealis Polymers Oy as part of their Sustainable Plastic Industry Transformation program, with a special focus on the Boremix compounding plant. This experience has been incredibly enriching, further fueling my passion for this field. I am profoundly grateful for the opportunity to contribute to such meaningful work.

I would like to express my sincere gratitude to my supervisor at Borealis, Andrei Ollikainen, for his invaluable mentorship and unwavering dedication. His expertise and guidance were crucial in maintaining the clarity of the project's goals and ensuring that its significance remained well-defined throughout the process. I am also deeply appreciative of my colleagues at Borealis, especially the Boremix team and the Porvoo laboratory services. Their collaborative spirit, insightful discussions, and collective efforts were instrumental to the success of this project and greatly expanded my understanding of the subject.

I am deeply thankful to my Tampere University supervisor, Ilari Jönkkäri, for his expert guidance and constructive feedback throughout this process. Special thanks are also due to Sanna Auvinen for her role in examining my thesis and, most importantly, for being such an inspiring teacher throughout my studies.

To my dear friends, whose support and encouragement have been a constant source of strength, I am truly grateful. Finally, I wish to express my heartfelt thanks to my family, especially my mother, for their unwavering support and encouragement throughout my academic journey and beyond.

Porvoo, 26th of August 2024

Mona Grann

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LIST OF SYMBOLS AND ABBREVIATIONS

AO	Antioxidant
aPP	Atactic polypropylene configuration
DSC	Differential scanning calorimetry
EPR	Ethylene-propylene rubber
ESCR	Environmental stress crack resistance
FCM	Food contact material
FTIR	Fourier-transform infrared spectroscopy
HPLC	High-performance liquid chromatography
ICT	Inter coating time
IEC	International Electrotechnical Commission
iPP	Isotactic polypropylene configuration
LVR	Linear viscoelastic region
MFR	Melt mass-flow rate
MWD	Molecular weight distribution
OIT	Oxygen induction time
OML	Overall migration limit
PCR	Post-consumer recycled
PDS	Product data sheet
PE	Polyethylene
PE-HD	High-density polyethylene
PE-LD	Low-density polyethylene
PE-LLD	Linear low-density polyethylene
PIR	Post-industrial recycled
PO	Polyolefin
PP	Polypropylene
PW	Powder
RSC	Reactive site content
SML	Specific migration limit
SPC	Steel pipe coating
sPP	Syndiotactic polypropylene
VST	Vicat softening temperature
XRF	X-ray fluorescence
YI	Yellowness index

G’	*Storage modulus*
G’’	*Loss modulus*
η^*	*Complex viscosity*
ETA0	*Zero shear viscosity*

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1. INTRODUCTION

The growing global concern over plastic waste has prompted significant efforts to develop sustainable recycling and repurposing strategies. In Europe, plastics generated 1.8 billion tons of greenhouse gas emissions in 2019, with 90% of these emissions resulting from production and conversion processes. [1] Within the spectrum of plastic waste, post-industrial plastic side streams, a byproduct of manufacturing processes, represent a substantial resource. These side streams, particularly those derived from polyolefin pellet production, hold the potential to contribute to circular economy initiatives by promoting more efficient use of raw materials, reducing waste, and minimizing the demand for prime materials. [2,3]

Closed loop recycling offers an effective means of reintegrating post-industrial materials into the production cycle. By recovering and reprocessing materials, manufacturers can produce products that closely resemble those made entirely from prime materials. [4] This process is of particular interest to polyolefin pellet producers, who can apply primary recycling methods directly to their processing lines by blending post-industrial materials with prime resins in specific proportions [5,6].

The aim of this thesis is to investigate the potential of reprocessing post-industrial materials, specifically from polyolefin pellet production. Thus, this research seeks to determine whether post-industrial materials can be compounded with prime resins without compromising the product performance, thereby offering a viable alternative to the exclusive use of prime materials in the production process. Conducted in collaboration with Borealis Polymers Oy, this study specifically focuses on their Boremix compounding plant, which produces polyolefin pellets via twin-screw extrusion. The involvement of Borealis Polymers Oy underscores their commitment to climate action through waste reduction and the promotion of effective circular material use.

The primary research question guiding this thesis is: *Can post-industrial materials from polyolefin pellet production be used as part of the corresponding prime product without compromising the required properties?* To address this question, pilot-scale compounding experiments will be conducted on two specific polypropylene products, exploring different mixing ratios of primary and post-industrial recycled resins. The results of these experiments will be evaluated to determine their potential for industrial-scale application.

In addition to the experimental investigation, the theoretical part of this thesis explores how other polyolefin producers manage their post-industrial side streams within a closed-loop recycling framework. A secondary research question is posed: *How other polyolefin pellet producers treat post-industrial side streams generated from their production operations?* By examining these practices, this research aims to discover best practices, current policies, and industry trends regarding side stream recycling. These insights will be used to guide improvements within the studied operations and contribute to broader advancements in sustainability and innovation across the polyolefin industry.

The thesis is organized into seven chapters. Chapter 2 covers the theoretical background, focusing on the compounding and mechanical recycling of polyolefins. Additionally, this chapter discusses practices for treating post-industrial side streams by polyolefin producers, end-users, and third parties. Chapter 3 details the processing techniques, including an evaluation of the functional equivalence between pilot- and industrial-scale pelletization processes. Chapter 4 specifies the characterization methods for quality control. Chapter 5 outlines the materials and methods, including recipe design, sample preparation, and test methods. The results are presented and discussed in Chapter 6, followed by the conclusions and recommendations for further research in Chapter 7.

2. POST-INDUSTRIAL SIDE STREAM TREATMENT OF POLYOLEFINS

This study focuses on evaluating the process residual materials of polyolefin pellet production, particularly through the lens of their potential for reuse. Specifically, this chapter examines the classification and qualification of post-industrial side streams, with an emphasis on the production residuals arising from polyolefin pellet production via extrusion compounding. This chapter begins by presenting the fundamentals of polyolefins, highlighting the significance of polyethylene and polypropylene, not only for the practical aspects of this research but also for their broader relevance within the industry. Following this, the role of twin-screw extrusion in compounding operations is discussed, along with principles of blending, compatibility, and compounding. In addition, this chapter discusses different approaches for plastic waste recycling, providing a foundation for understanding the subsequent discussions on the management of post-industrial recycled polyolefins. This includes an in-depth examination of how polyolefin pellet producers internally recycle their production side streams and the practices adopted by third parties and end-users.

Industrial side streams are valuable resources for circular economy initiatives. According to Leppänen et al., side streams can be classified into the following categories: process residual materials, process residual energy, energy generation waste and process residual structural components [5]. Process residue assessment seeks to uncover economic opportunities while simultaneously enhancing the sustainability of industrial manufacturing processes. [7] In this thesis, the primary focus is on process residual materials, also known as post-industrial (PI) materials, which are generated during manufacturing processes and separated from the waste streams. In contrast, prime grade materials are thermoplastics sourced from fossil fuels, that have not been subjected to any processing beyond what is necessary for their initial production. [8]

Polyolefin production has steadily increased, with polypropylene production performing accelerated growth [9]. In 2022, approximately 80% of the plastics manufactured in Europe were derived from fossil fuels, while the remaining 20% originated from recycled materials and biological sources. Figure 1 illustrates the shares of different plastics produced in Europe in 2022. This graph also takes into account the different methods of recycling the raw material used in polymer production. [9,10]

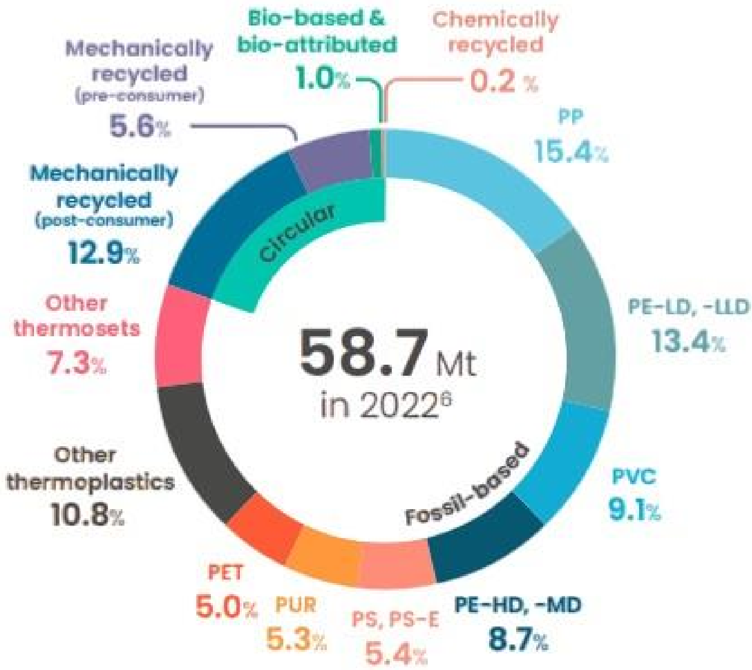

Figure 1. *European plastics production by polymer family in 2022 - Percentage share overview, adapted from [11]*

Taking circular plastics into account, polyolefins represent about one third of the plastics produced in Europe. In contrast, fossil-based polyolefins account for about 63% of total global plastic production. [9,10] According to Plastics Europe's report, in 2020 about one-third of post-consumer plastic waste end use was recycling. In contrast, more than 40% was incinerated for energy recovery, and approximately 23% was disposed to landfills. [12] To conclude, these statistics illustrate the current plastic production volumes, the share of circularity in it, and the methods of plastic waste disposal, while also highlighting opportunities for improvement.

2.1 Polyolefins

Polyolefins are a class of plastics classified by their monomer unit to ethylene-based polyolefins and propylene-based polyolefins. The most commercially applied thermoplastic polyolefins are polypropylene (PP) and polyethylene (PE). [13] Polyolefins have the largest production volumes and consumption among all polymers. The reason for their popularity lies in their versatile properties, which make them suitable for a wide range of applications. The fundamental properties typical for all PE grades include toughness, low stiffness, chemical resistance, lightness, and ease of processing. In addition, PEs maintain structural integrity better at low temperatures than PPs, which become brittle. Characteristic properties of PPs include stiffness and chemical resistance. PPs have greater resistance to elevated temperatures, and they are easier to process than

PEs, even though PPs are susceptible to oxidation during processing and use. [14] Blending, copolymerization, and additives are used to modify and tailor properties of polyolefins [15].

The molecules in thermoplastics are long polymer chains with no chemical bonds between them, only weak intermolecular forces. When plastics are heated, the intermolecular forces between the polymer chains decrease, leading to the softening of the material, which becomes moldable at specific temperatures. As the material cools, the intermolecular forces strengthen, causing the polymer to resolidify. This ability to repeatedly melt and solidify without substantial degradation is fundamental to the process of polyolefin pelletizing extrusion. [13]

The polyolefins to be studied in more detail, PE and PP, are semicrystalline materials, featuring a polymer matrix that contains both amorphous regions with random molecular structure, and crystalline regions with ordered structure. The degree of crystallinity varies based on their morphology, molecular structure, molecular weight, molecular weight distribution (MWD), tacticity in PP, processing conditions, copolymerization, and additives. [13] Figure 2 illustrates how amorphous and crystalline regions are arranged in semicrystalline polymer matrix [2].

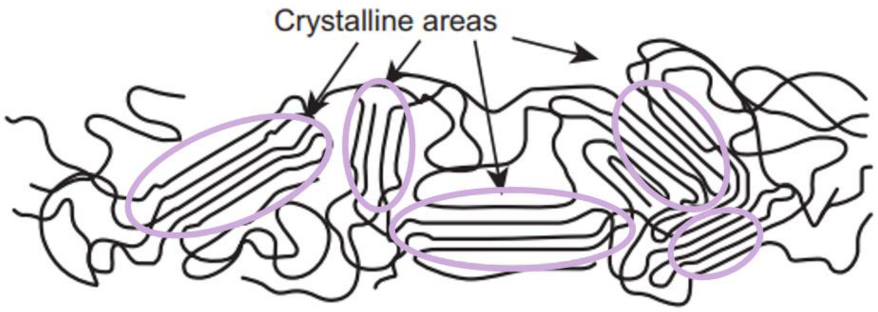

Figure 2. *Illustration of a semicrystalline polymer matrix, where crystalline regions are highlighted, adapted from [2]*

This structural arrangement significantly impacts thermal properties, including specific melting temperature (T_m) and the polymer softening point, called glass transition temperature (T_g). The operating temperature range for polyolefins is located between these temperatures. This is because below the melting temperature, polymers are soft and easily moldable, while below the glass transition they become brittle and rigid. In molten state both PP and PE are shear thinning materials, meaning they possess non-Newtonian behavior. Shear thinning nature arises from viscoelastic behavior, and it refers to the behavior of a fluid where viscosity decreases as a function of shear rate. This is the foundation of the rheological behavior of polyolefins, which describes polymers response to deformation. [16–18]

The size and structure of polymer molecules significantly influences their processability and mechanical characteristics. Polyolefins have a high average molecular weight which contributes to their high viscosity, melt elasticity and strength. [9] Varying chain lengths result from the polymerization process, making both molecular weight and MWD critical factors in assessing polyolefin compatibility, crucial for polymer compounding. Small polymer molecules facilitate easier molding due to reduced intermolecular forces, yet they compromise the mechanical strength of the final product. Furthermore, reduction in the number of intermolecular forces relates to chain entanglement, as shorter polymer molecules allow fewer entanglements to be formed. Conversely, very large molecules, while enhancing the strength of the product, pose challenges in mouldability due to stronger intermolecular forces. [16,18] However, Dealy et al. states that for highly branched polymers the decrease in viscosity initiates at low shear rate values, which facilitates mouldability [19]. When processing polymers at an industrial scale, polyolefins are characterized by their melt mass-flow rate (MFR), which provides information about the polymer's flow ability under pressure. In the context of extrusion processing, comparing branched and linear polymers reveals that branched polymers require higher energy to process due to their stiffness, higher viscosity, and lower MFR, yet they possess better melt strength and handling characteristics. [16,18]

Copolymerization offers a way to modify properties of existing homopolymers. During copolymerization at least two types of different monomers are bonded together by covalent bonds in a polymer structure. [9,13] Polyolefin copolymers can have two distinct kinds of arrangements which are random copolymer (a), and block copolymer (b), as presented in Figure 3. This figure illustrates the arrangement of a copolymer consisting of two different monomers, indicated in black and white. [20]

Figure 3. *Copolymer structural configurations, a) random copolymer, b) block copolymer [20]*

Randomly orientated copolymer is formed when both reacting homopolymers are evenly reactive, meaning the probability that a monomer joins the growing chain is proportional to its concentration in the reaction medium. Crystallinity of random copolymer is lower than the corresponding homopolymer, due to decreased structural regularity in the polymer structure. On the contrary, alternating block copolymer exhibits greater structural order than random copolymer, thus is able to form a higher degree of crystallinity. Moreover, block copolymers are formed when it is more probable that the joining monomer has a similar composition as the growing chain length. A combination of the two copolymer structures is also possible, with a block of one type of monomer followed by a random block containing both monomers. [13,16,21]

2.1.1 Polyethylene

PEs can be divided into three main types based on their density: low-density PE (PE-LD), linear low-density PE (PE-LLD), and high-density PE (PE-HD). The differences in densities are a result of the type and degree of side branching in molecular structure. These differences among PEs structures arise from the differentiating polymerization method. However, all PEs share the same chemical formula. [13,14] Figure 4 provides a simplified illustration of the molecular chain characteristics of these three main types [22].

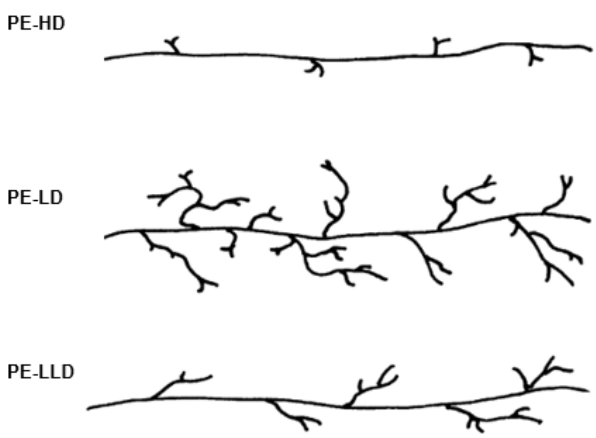

Figure 4. *Molecular chain characteristics of PE-HD, PE-LD, and PE-LLD, adapted from [22]*

PE-LD is polymerized via free radical initiated polymerization, whereas PE-HD is polymerized via Ziegler-Natta. As a result, PE-LD possesses a highly branched molecular structure with shorter non-uniform branch lengths, and density of 0,91-0,93 kg/dm^3 [14, p.21]. PE-HD has more linear molecular structure than PE-LD, because the branching is less frequent with longer chain lengths. The numerical density value of PE-HD is 0,95-0,98 kg/dm^3 [14, p.21]. As a results of lower degree of branching, and therefore increased regularity of molecular structure, PE-HD has a higher degree of crystallinity than PE-LD. Medium-density PE exhibits at a density range from 0,93-0,94 kg/dm^3, positioning it between the two densities previously defined [14, p.21]. To conclude, the density of PE is linked to its crystallinity, serving as a reliable measure for it. [13]

A higher degree of crystallinity contributes to the properties of the polymer, so that PE-HD is structurally stiffer, has a higher tensile strength, and the energy required to melt the material is higher due to increased intermolecular forces compared to low-density PEs. [14] In addition, crystallinity promotes chemical resistance of the material. On the other hand, highly crystalline materials experience embrittlement at low temperatures, and due to their increased stiffness, their elastic behavior is hindered. Moreover, transparency is a common characteristic of PE-HD. [9,23]

In addition to the three primary types outlined previously, PEs can be further classified into distinct sub-types. Linear low-density PE (PE-LLD) is a subtype of PE-LD. PE-LLD is produced by copolymerization, where ethylene monomers are combined with other types of monomers, such as butene, hexane or octene. Like PE-LD, PE-LLD has a highly branched molecular structure. However, the difference is that PE-LD has short-chain branching while PE-LD has long-chain branches. In the case of PE-LLD, the chain branching length is dependent on the comonomer used during the polymerization. Overall, PE-LLD has comparable properties to PE-LD but is slightly more ductile and stronger at both high and low operating temperatures. In addition, PE-LLD is less susceptible to stress cracking. [9,14]

PE-LD sub-types also include other copolymers such as E/VAC (EVA) and E/VAL (EVOH). Further metallocene catalyzed sub-types include metallocene PEs mPE-LD and mPE-LLD. Metallocene catalysts enable the polymerization of polymers with precisely predictable properties, giving manufacturers greater control over polymer properties. By choosing the proper polymerization reactor conditions, it is possible to control the following properties: molecular weight, comonomer content, molecular weight distribution, and tacticity. As a result, PEs prepared with these catalysts have a narrow molecular weight distribution, which improves their mechanical properties. [18,24] Metallocene catalysts also offer a significant improvement in the efficiency of polymerization, since its catalytic activity is approximately 100 times higher than the traditional Ziegler-Natta systems. However, mPE-LLD exhibits high surface roughness, which contributes negatively to optical transparency. [25]

PE-HD sub-types include high molecular weight PEs such as PE-HMW (high molecular weight) and PE-UHMV (ultra-high molecular weight). These high molecular weight grades of PE have excellent impact strength and abrasion resistance. In addition, they have a significantly lower tendency to stress cracking compared to other linear PEs. PE-HMW and PE-UHMW exhibit crystallinity-dependent strength properties that are equivalent to or lower than those of High- PE-HD. The limited ability of these polymers to form well-ordered crystal structures, as a direct consequence of their high molecular weight, adversely affects their strength properties. In addition, PE-HD has also metallocene subtype mPE-HD. [14,18]

Table 1 summarizes how the mechanical properties of PE depend on crystallinity and molecular weight. The table illustrates how variations in crystallinity or average molecular weight - either increasing or decreasing - affect the properties of PE and provides a straightforward guide for evaluating these changes. [18]

***Table 1**. Dependence of the mechanical properties of polyethylene on crystallinity and molecular weight, adapted from [18]*

Property	Crystallinity increases	Average molecular weight increases
Stiffness	Increase	Increases slightly
Impact strength	Decrease	Increase
Stress at yield point	Increase	Increases slightly
Tensile strength at break	No effect	Increase
Elongation at break	Decrease	Increase
Creep	Decrease	Increase
Hardness	Increase	Increases slightly
Embrittlement	Increase	Decrease
Softening temperature	Increase	No effect
Thermal conductivity	Increases slightly	No effect
Gas permeability	Decrease	Decreases slightly
Stress corrosion	Increase	Decrease
Relative permittivity	Increase	No effect

Most of the PE-HD produced, approximately 75%, is used to make films. Largest applications in Europe include packaging applications such as multilayer structures, stretch films, and pouches [14]. Medium density PE is specially used for the manufacture of strong films and vessels. PE-HD is used for more demanding applications where greater mechanical, thermal or chemical resistance is required. Examples of PE-HD applications include industrial pipes and tanks for chemical solutions and gasoline tanks. [18]

2.1.2 Polypropylene

PPs can be utilized as homopolymer (PP-H), which consists of polymer chains only constructed from the same monomer type, propylene. PP-H is the traditional form of PP. When PP forms a copolymer with ethylene, it can be either a randomly oriented PP-R copolymer or a block copolymer where the ethylene groups are located in the polymer in regular sequences, in this case PP-B. In a random copolymer, the ethylene is thus randomly located in both the main chain and the side branches of the polymer, resulting in a stable rubbery like copolymer called ethylene-propylene-rubber (EPR). Increasing the content of EPR enhances the impact strength of PP especially at low temperatures.

Thus, PP copolymers consisting of up to 40% EPR, are called impact copolymers or heterophasic copolymers. [14,15,26] The structure of the three main types of PPs are illustrated in Figure 5.

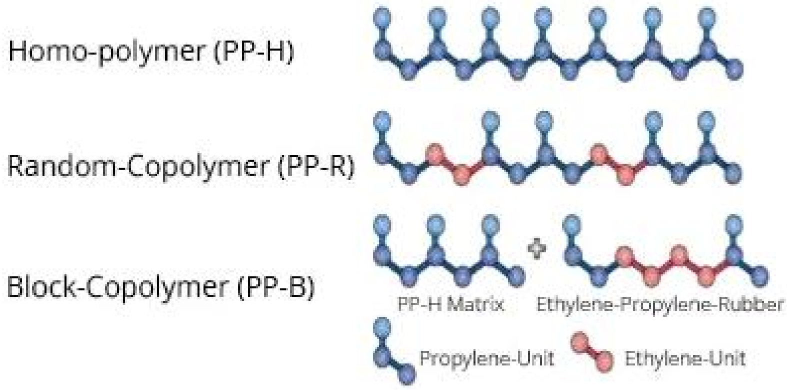

Figure 5. *Molecular structure of the three main types of polypropylenes [27]*

When ethylene monomers are distributed along the chain, the homogeneity of PP decreases due to disturbed regularity. PP-B structure further decreases homogeneity compared to PP-H and PP-R, since it creates regions within the polymer that have markedly different properties from each other. [26] Decreased homogeneity can reflect higher melt viscosity and a lower MFR value in case of PP-B compared to PP-H. On the other hand, when the composition of PP-R changes towards PP-B, the regularity of the polymer structure may increase, causing easier flow and increase in MFR value. Due to the structural regularity, PP-H is more resistant to heat when compared to its copolymers. Moreover, a distinctive feature of PP-H is its excellent hinge ability; it can endure repeated bending without compromising the polymer's structural integrity. On the contrary, PP-B is utilized in flexible products, and it exhibits better resistance to low temperatures compared to PP-H. PP-R, distinguished by its transparency, is tailored for specific uses that benefit from this characteristic. However, its resistance to frost does not match that of PP-B. [14,22] Conclusively, optimizing crystallinity and impact resistance by varying the ratio of the copolymer phase to the homopolymer phase allows PP to be used in a wide range of applications. [26]

Furthermore, depending on the polymerization conditions and utilized catalyst, PP-H can exist in three different structural configurations [13,18]. More specifically, PP is characterized by its stereoregularity, which is crucial for its structural and physical properties, such as mechanical properties, crystallization and melting behavior. This regularity is specifically attributed to the position of methyl groups attached to the polymer backbone. [9,28] The way these methyl groups are positioned relative to each other along the chain results in three main stereoisomeric configurations, illustrated in Figure 6 [29].

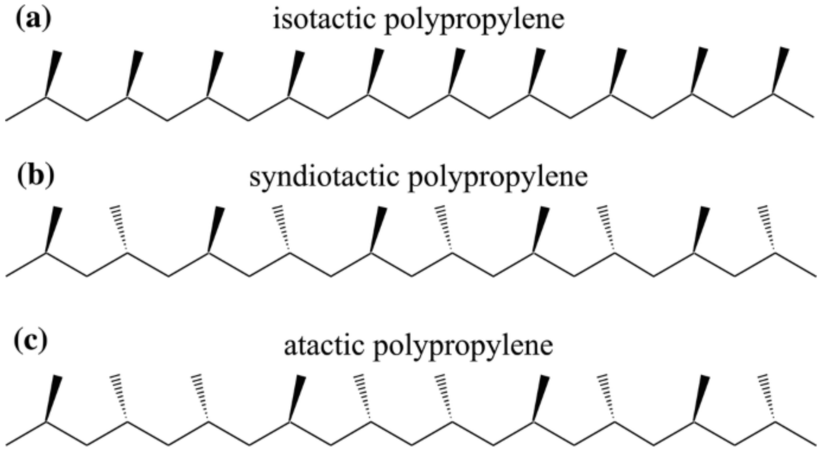

Figure 6. *Three main tacticity configurations for polypropylene [29]*

PP structure possess isotactic configuration (iPP), when all methyl groups are positioned on the same side of the polymer chain. Syndiotactic polypropylene (sPP) has a form where methyl groups are at alternating sides, and in atactic PP configuration (aPP) methyl groups are arranged randomly along the polymer chain. [9] The degree of crystallinity increases with configurations from atactic towards isotactic, due to the increased regularity in the molecular structure. In more detail, the methyl groups in sPP and aPP disturb the packing arrangement that is required for crystallization, and therefore a much lower degree of crystallization can be obtained compared to iPP [16]. While aPP is amorphous and mechanically weak, sPP produced commercially with metallocene catalysts can crystallize to high degrees. Moreover, high clarity is a common characteristic of sPP. The isotactic form is able to crystallize and thus gives PP the necessary strength properties. Due to the differences in degree of crystallization, almost all (96-100%) of PP used in commercial applications is in isotactic form. [13,18]

To summarize, similarly to PE, the mechanical properties of PP depend on both the degree of crystallinity and the molecular weight. Increasing the degree of crystallinity directly improves the hardness, stiffness and yield strength of the material. Similarly, an increase in average molecular weight contributes to greater ductility, elongation and melt stiffness of PP. Moreover, PP is an extremely versatile material in terms of its applications. Depending on the properties discussed previously, the primary applications of PP include rigid and flexible packaging, as well as fibers for the textile industry, among others. PP is the most widely used plastic in the automotive industry, specifically in the car interior. [14,18]

2.2 Twin-screw extrusion in compounding operations

Twin-screw extrusion is a cornerstone technology in polymer compounding and blending. Compounding is a process to create a new homogenous material by incorporating two or more components by melt mixing via extrusion [30]. Compounding is followed by conversion into plastic pellets, which are subsequently utilized in extrusion and other processes to form different products. Twin-screw extruder system involves two screws housed within a barrel which rotate and work together by mixing, shearing and conveying the material. The system can be categorized by three zones: [31,32]

1. **Feed zone**, material is fed from a hopper, preheated, and conveyed
2. **Compression zone**, material is melted by mechanical work and heat
3. **Metering zone**, final homogenization and conveying material to the die

Compared to single-screw extruder twin-screw extruder generally provide better mixing capability, shorter residence time, more uniform residence time distribution, and higher flexibility [33]. For these reasons twin-screw extruders are more popular choice for polymer compounding than single-screw extruders. The barrel structure of twin-screw extruder is modular, thus the element configurations can be modified to enable additional feed streams along the barrel length, liquid feed, or venting. The temperature profile of each segment is controlled to facilitate precise thermal management during processing. Both temperature and pressure are monitored during processing. Segment temperatures are typically described as temperature zones. The major equipment components of a twin-screw extruder are illustrated in Figure 7. [30]

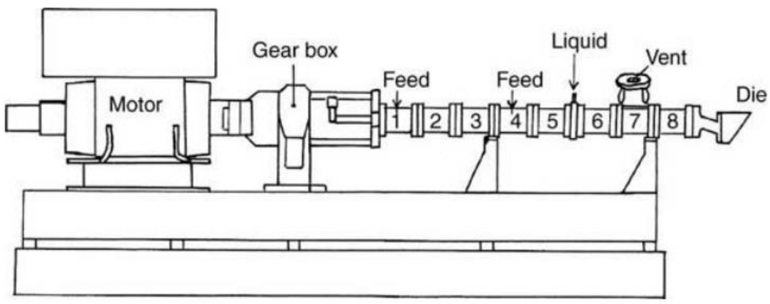

Figure 7. Schematic illustration of a parallel twin-screw extruder presenting the major equipment components [30]

Twin-screw extruders allow more complicated screw geometries compared to single-screw systems. The screw design can be parallel or conical. A conical design with decreasing diameter towards the die improves material conveying at the feed section and reduces the force exerted on the screw by back pressure and thus on the support bearings. Screws can be corotating or counterrotating, and the degree of contact between screws can be varied by choosing screw configuration between intermeshing or non-intermeshing. Both corotating and counterrotating screws offer high to medium mixing and conveying efficiency. Conveying of materials with corotating screw relies on frictional action, whereas counterrotating screw utilize forced mechanical conveyance [33]. Compounding most often utilizes a twin-screw extruder with closely intermeshing and co-rotating screws, which is presented in Figure 8 along with a counterrotating screw design for comparison [34]. Further, the degree of intermeshing between the screw has a major impact on material flow and the generation of shear. Likewise, to barrel sections, the screw sections are modular to serve a specific purpose over the three major zones and localize detailed material processing. To summarize, modular structure of a twin-screw extruder enables high flexibility along the system, making it an excellent equipment for diverse compounding needs. [30]

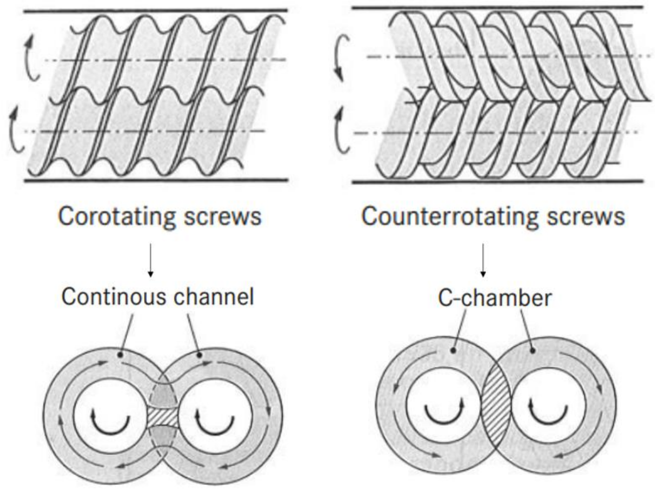

Figure 8. *Twin-screw extruder designs: corotating fully intermeshing and counterrotating fully intermeshing [34]*

Corotating screw transfers the material from one screw to another, creating an eight-shaped trajectory around the screws. Thus, this corotating design has a relatively short residence time making it suitable for sensitive material processing. [33] Residence time or dwell time indicates the time formulation is conveyed through the extruder. In contrast, counterrotating screw design squeezes the material between the screws creating higher shear and pressure compared to corotating screw design. [31,34]

Dispersive and distributive mixing are the two types of mixing utilized to form a homogenous compound. Dispersive mixing utilizes stresses to reduce the component size and uniformly disperse the melt. Distributive mixing refers to the uniform distribution of components during extrusion. [9,30]

2.3 Blending, compatibility, and compounding

Blending and compounding aims to optimize properties of polymer products, and to increase processing practicality. Blending is also used to reduce costs. [35] A polymer blend is a mixture in which two or more polymers are combined [36]. Polymer blends can be categorized into five main types based on the material types used in the blend. In the context of polymer compatibility of polyolefins, the emphasis is on thermoplastic-thermoplastic blends, and namely on *polyolefin only* blends. This emphasis aligns with the product portfolio of Borealis Boremix compounding plant, which concentrates mainly on PE-PE blends and PP-PP blends. On a commercial scale, polyolefin blends are fabricated during polyolefin polymerization (in-reactor), or alternatively after it (post-reactor). The subsequent involves mechanical blending in compounding extruders. [15,37]

For blending to take place as desired, the properties of the input materials and the output products must be characterized using a variety of testing methods, and only after the testing is completed the *compatibility* of the polyolefin blend can be confirmed. Essentially, compatibility in this context refers to all methods of creating stable blend microstructures that are essential to the performance of the part [13]. In other words, compatibility is considered as describing the degree to which polymers interact. [2,36]

Characterizing the properties of each polyolefin resin is crucial when blending them at a specific ratio. In practice, this process involves comparing the properties of the resultant polymer blend pellets with both the target specifications and the properties of the primary feedstock resin. By operating in this manner, the proportion at which the secondary feedstock can be added to meet the product specifications, can be determined. [35,37]

2.3.1 Miscibility

One way to determine polymer compatibility is to examine the *miscibility* of a polymer blend. Miscibility is dependent on these three factors: polymer blend composition, mixing temperature, and molecular structure. Polymer blends form a composition that is either miscible, partially immiscible, or immiscible. In practice, miscibility means that mixing conditions and polymer chemistry allows the components to mix completely and form a single-phase blend. Ullmann et al. state that for complete and stable mixing, the free energy of the mixture must be negative. However, negative free energy does not alone ensure miscibility of a blend. The free energy of mixing is expressed as a function of the enthalpy of mixing and the entropy of mixing. When the free energy of mixing is negative, the mixing reaction is spontaneous under constant temperature and pressure. As a result, polymer blend has single glass transition temperature, which value depends on the initial glass transition temperatures in proportion to the mixing ratio. Typically, two grades of the same polymer, such as two PE-LDs, exhibit compatibility and a miscible blend. [2,36,37]

On the contrary, immiscible polymer blends create two separate phases, thus no homogenous mixture is formed. In fact, polyolefin blends commonly form two separate phases due to the lack of polarity or hydrophilicity, which arises from the nature of polyolefins. As a result of phase separation, the blend exhibits poor mechanical properties. Furthermore, immiscibility negatively affects the optical clarity of the polyolefin blend causing for example haze in film products [36]. Immiscible polymer blends have two distinct glass transition and melting temperatures, which are formed by the characteristics of the blend components. One of these two methods can be applied to enhance the miscibility of a two-component system. The interaction energy between the two polymers can be increased or internal repulsion energy of random copolymers can be utilized. Increased interaction energy can be achieved for example with compatibilizers. [2,15]

2.3.2 Polyethylene blends

Despite a fundamental conflict regarding whether PE-HD is miscible with PE-LD or PE-LLD, there is consensus that the branching content of the branched component is the major molecular factor that determining the miscibility of both PE-HD/PE-LD and PE-HD/PE-LLD blends. A study conducted by Michler and Balta-Calleja explains that when strong mixing is applied, PE-HD/PE-LD blends form a lamellae structure consisting of linear or branched macromolecules, or co-crystallization occurs forming mixed lamellae [38]. Changes in mechanical properties will arise from these structural changes. [35,39].

A study conducted by Shebani et al. investigated the effects of varying ratio of PE-LD to PE-HD. Ratios that were tested for PE-HD/PE-LD blends respectively were 80/20, 60/40, 40/60, and 20/80. For greater clarity, the tested blend ratios are presented in Table 2. [35]

Table 2. *Tested blend ratios of PE-HD and PE-LD and name for each composition [35]*

Sample	PE-HD (%)	PE-LD (%)
PE-HD	100	0
Blend 1	80	20
Blend 2	60	40
Blend 3	40	60
Blend 4	20	80
PE-LD	0	100

These samples were tested for the following characteristics: hardness, elongation at break (%), tensile strength, and impact strength. In addition, stress-strain properties were evaluated. In Figure 9 the test results of hardness and elongation at break are presented for each blend. On the right-hand side in Figure 9 the impact strength test results are also presented. [35]

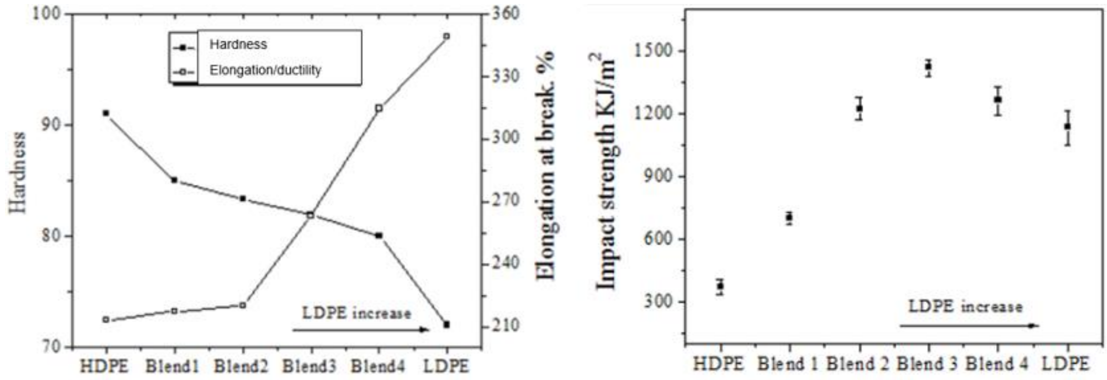

Figure 9. *Hardness and elongation at break (%) (left), and impact strength (right) as a function of PE-HD, PE-LD and their blends [35]*

The hardness and elongation at break results follow the crystallinity content ratio of the blend. PE-HD has a high degree of crystallinity which promotes greater hardness properties, on the contrary PE-LD promotes better elongation properties. From the right-hand side graph, it can be observed that increasing PE-LD content increases the impact strength to a certain point, and Blend 3 has the highest impact strength value. The study concluded that the blend with composition of PE-HD (40%) and PE-LD (60%) resulted in overall better mechanical properties compared to neat polymers and other blends. [35] Therefore PE-HD/PE-LD blends are frequently used [38].

Studies [36,38] claim that fabrication, optical properties, and solid-state properties of PE-LLD can be improved by the addition of PE-LD, and a miscible blend in molten state is formed within temperature range of 130-230°C. Moreover, improvement in optical properties indicates that the transparency of PE-LLD can be improved by blending. Hussein et al. has suggested that higher branching content is promoting better miscibility in this specific blend. [39] There is a strong possibility that the current observation is a result of the chaotic structure of PE-LD, corresponding to the highly branched mPE-LLD conformation. Furthermore, it has been pointed out that miscibility is more likely to develop in the PE-LLD rich blend range, and less probable in the PE-LD-rich blend range. [35,36,38]

On the contrary, for PE-HD/PE-LLD blends, the miscibility has been reported to decrease as branching content increases. Due to the molecular structure differences, typically this blend results in phase separation. [35,38,39] However, Morris highlights in his study [36] that blending PE-HD with PE-LLD is utilized for example in packaging applications to improve moisture barrier performance. Another study [24, p.2121] reports that blending less than 10% of PE-HD of the total composition, the haze of mPE-LLD blown films can be reduced. Hence, the compatibility and functionality of the blend is not entirely dependent on miscibility.

2.3.3 Polypropylene blends

Both chemical modification by copolymerization, and blending are utilized to improve impact resistance of PP. Although iPP is extensively utilized, the range of applications are limited by its brittleness at lower temperatures. Blending can alter brittleness by improving impact strength, among other mechanical properties. For instance, blending ethylene-propylene block copolymer with iPP has shown an improvement in toughness. Another advantage is improved tolerance for environmental stress-crack, which is achieved by addition of 10-20% of EPR [40, p.624]. Despite EPR and iPP forming a blend with limited compatibility, extensive research has been conducted to optimize blending and unlocking their potential. EPR/iPP blend can be produced either by in-reactor process or as a post reactor process, during which the random and block ratio can be altered. [15]

Molecular chain stiffness and the packing size of molecules affect the miscibility of polymer blends. Stereoregularity alters the stiffness of the chain, resulting in variation of the miscibility among PP tactic configurations. An immiscible blend is formed when iPP and sPP are blended in the molten phase. In more detail, when the blend is cooled down into solid state the PP grades crystallize separately and form a non-homogenous phase separated blend. Moreover, sPP also forms an immiscible blend with aPP. [15,41] According to a study conducted by Chen et al., a small amount of low molecular weight aPP can be blended with iPP to increase the crystallinity of iPP. In more detail, the study reports that a blend with 20% aPP content shows the highest crystallinity when the processing conditions such as temperature is optimized. Furthermore, the study states that aPP is locally miscible with iPP. [42]

Two incompatible polymers can be blended by utilizing functionalized PP, such as grafting with maleic anhydride. Grafting can be done either directly by adding functional groups onto the polyolefin structure during polymerization, or after polyolefin polymerization. Functionalized PP overcomes inherent interfacial tension differences by means of chemical bonding. Hence, they act as compatibilizers or impact modifiers and are typically characterized as additives rather than polymer matrix materials. [2,15,43]

2.3.4 Compatibility characterization

Mechanical testing can be utilized to determine whether two polymers have good compatibility. Good compatibility indicates good miscibility and interface actions, which should improve the overall mechanical properties of the blend compared to the neat polymers. For example, as stated for PE-HD/PE-LD blending. [44] While tensile testing is not a direct measurement of miscibility, it can reflect the overall compatibility. Therefore, improvement or change in tensile strength, elongation at break, or modulus can indicate polymer compatibility. [45]

Differential scanning calorimetry (DSC) is an analytical technique, which is used to analyze the degree of crystallinity, melting point and glass transition temperature of a given polymer resin or compound. The miscibility can be estimated from the number of observed glass transition temperatures. Similarly, more than one melting peak indicates that immiscibility causes phase separation in melt due to two groups of crystal structures. [15] DSC also provides information on reactions occurring in the sample during heating, such as enthalpies of phase transitions. Moreover, DSC can be used to study the oxidation, degradation, purity, additives, composition of copolymers and polymer blends. [2,18]

In polyolefin film products formation of gels, a defect or distortion, can be used to assess the compatibility between polymers. Gels can be generated from multiple sources including resin manufacturing, compounding, and converting operations. Blending of different polyolefin resin types typically results in gel formation, indicating incompatibility. On the contrary, materials with similar shear viscosities provide fewer defects indicating compatibility during extrusion operations. Another indication of compatibility that can be made from gel inspection is if the blended polymers have similar molecular weight. Even inside a polymer family, great differences in molecular weight can generate gels due to uneven melting. [36,46] Given the sensitivity of gel formation to contaminants, it represents a pivotal technique for assessing polymer compatibility.

Melt mass-flow rate (MFR) is a measuring technique to obtain information about viscosity and molecular weight of particular polymer resin. In other words, MFR describes the melt processability of a polymer [18]. The main principle of MFR measurement is quite simple: the polymer is heated up to a specific temperature, forced through a specified die with piston and additional weight, and the MFR value is determined according to the polymer that has flowed out in 10 minutes. A high MFR value indicates that the viscosity and molecular weight of the polymer resin being measured is low, and a correspondingly low MFR value indicates that the polymer resin possess a high viscosity and molecular weight. [13] Polymer compatibility cannot be directly detected from MFR value. However, variation and in MFR values can be monitored, and as Crawford and Martin emphasize it allows comparison between different polymer melts under equivalent conditions [31].

Determination of Vicat softening temperature (VST) allows evaluation of temperature at which thermoplastic material softens under a specific load and heating conditions. The testing apparatus measures the temperature at which a pointed needle penetrates 1 mm deep into the specimen. Another testing method to provide information about the physical properties of the blend is the yellowness Index (YI). With YI measurement, a color comparison between two specimens of the same material can be done. Increased yellowness of the sample is observed as a positive change in YI value, indicating that the object has become more yellow compared to its initial condition. YI is particularly useful when investigating the compatibility of recycled plastic with prime plastic. However, similarly to MFR, both VST and YI are more indicative of physical interaction and processing characteristics than direct measure of miscibility. [47–49]

Only after numerous testing practices can the compatibility between two polymer melts be verified. The implementation of the test methods used to characterize the compatibility is demonstrated in more detail in the following chapters.

2.3.5 Additives

Throughout compounding, processing, and end-use polyolefins are susceptible to conditions that can alter their properties by degrading the polymer structure. Additives are substances that are compounded with polyolefin resin in the extruder to improve or modify properties, processability, and resistance to environmental attacks. The resin polymer has inadequate properties for industrial scale plastic pellet production, so compounding is required. Specific pellet recipes are created based on the required property profile and existing technology. Additives are categorized into stabilizers, which are considered process additives, and modifiers, known as functional additives, based on their roles in enhancing the manufacturing processes and providing specific functionalities, respectively. To ease the compounding process of products, a masterbatch blend with high additive concentration and a carrier resin is typically utilized. [2,36,50]

During melt processing polyolefins are susceptible to degradation which proceeds in radical mechanism. Free-radicals are able to initiate chain-scission or chain combination by cross-linking. Eventually, a large-scale polymer degradation can be caused, unless the reaction chain is interfered and inhibited. Due to the molecular structure differences of polyolefins, one of the reactions is typically dominant over the other. PP has a greater tendency towards chain scission, resulting in a lower average molecular weight. PE is more likely to experience crosslinking. Chain scission can be observed from an increase in MFR value, while chain combination results in a decrease in MFR value due to increased viscosity. Tolinski highlights in his study, that the effect on MFR becomes more pronounced when multiple re-extrusion processes are applied. [50] Three common polyolefin product additives are presented in more detail below, with the emphasis on antioxidants.

Antioxidants (AO) are utilized upon exposure to oxygen or autoxidation. The process of autoxidation is initiated by free radicals and proceeds in a self-propagating circular manner producing hydroperoxides and other substances. AOs work by stabilizing free radicals. Primary AOs consume free radicals, and secondary AOs react with secondary autoxidation products of chain propagation – hydroperoxides. [50] A combination or a blend of multiple additives is utilized to achieve the desired property outcomes, as no single AO usually provides adequate effectiveness [2]. Moreover, it has been reported in the literature some AOs create a synergistic effect, meaning that greater protection is achieved as a combination of AOs than using a single additive. [51]

For long-term heat stability, hindered phenol AOs such as Irganox 1010 are the most used primary AOs for PE and PP compounds. Thioester-based secondary AOs such as DSTDP are utilized as a combination with phenol primary AOs to support the long-term heat stability and prevent heat aging. Irgafos 168 is a phosphite-type AO used to maintain short-term melt flow and color stability during PE and PP processing. Key advantages of utilizing synergistic effect of phosphite AO and hindered phenol AO as a dry blend include enhanced processability, material savings in the use of primary AOs, and ease of handling and feeding. According to a study by Drake et al., a 4:1 ratio of phosphite to phenol has shown the best performance when re-extruding PE-HD and PE-LLD [52]. Similarly, the most effective ratio for PP homopolymer under re-extrusion has been reported to be 2:1 ratio of phosphite to phenol. These measurements were done at temperature range of 220°C to 280°C. [2,50,51]

Ultraviolet (UV) degradation is mostly dependent on UV-absorbing species that are mixed with polymer resin, given that saturated polyolefin molecules inherently absorb minimal UV light. Also, if oxidation occurs during thermal processing, the oxidation products can further convert to other reaction products when exposed to UV. These wavelengths penetrate into the molecular structure of the polymer and create free reactive radicals, further inducing breakage of polymer molecule bonds. UV stabilizers are utilized for the purpose of preventing degradation reactions caused by light. [50,53] Wypych states that polypropylene deformation caused by any wavelengths of light, known as photodegradation, can result in yellowing, loss of mechanical properties and surface cracking [54]. Carbon black is typically used UV-blocker in pipe applications because it is inexpensive and stability. However, hindered amine light stabilizers are the most efficient class of UV stabilizers [55].

Both PE and PP are prone to accumulate statistic charges. For this reason, antistatic agents are essential additives for polyolefins used in applications including cable products and packaging. These additives reduce the material's dust attraction and the possibility of spark discharge, which can cause severe accidents in the form of vapor explosions. Antistatic agents can be compounded into the polymer resin for longer-term production, or they can be applied externally on the surface of the polymer. [30]

2.4 Approaches for plastic solid waste recycling

Waste refers to materials that have outlived their usefulness for their original or any alternative purposes and are incapable of being repurposed or recycled. Recycling refers to the process of recovering waste materials into new items, substances, or materials, which can serve the same function as they originally did or be applied in new ways. [56]

Plastic solid waste recycling operations can be divided into four main categories: primary, secondary, tertiary, and quaternary recycling. This classification is based on ASTM standards for the recycling and use of recycled plastics [48]. In Figure 10 these four approaches for recycling solid plastic waste are presented, and the processing possibilities associated with each of them. The main processing methods for circular plastics can be further divided into three: mechanical, chemical, and thermal [57]. It should be noted that the treatment methods presented in Figure 10 apply to different plastic polymer types and are not exclusively limited to polyolefins. [58]

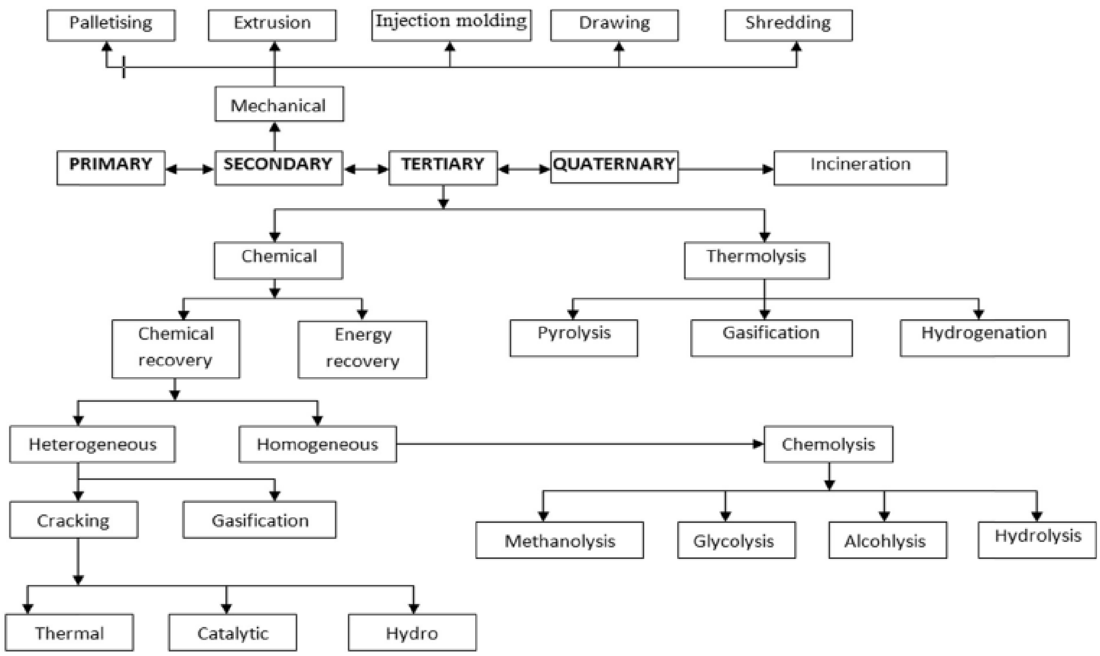

Figure 10. *Approaches for plastic solid waste recycling [58]*

Primary and secondary recycling utilizes mechanical operations to convert plastic solid waste into new feedstock. Therefore, they both can be classified as mechanical recycling. The basic principle of mechanical recycling is converting the polymer material back into a pellet form via melting. During this process prime polymers and additives are commonly added to recycled resin to form a blend. Considering this, additional mechanical operations such as grinding, sorting, washing, re-extrusion, blending, and compounding can be applied. More importantly, these two types of recycling are distinguished according to the purity and known composition of the recycled material, which is influenced by the source of the waste. The material sources can be categorized into pre-consumer materials and post-consumer recycled (PCR) materials. According to the above definition of recycling type, in general primary recycling utilizes pre-consumer materials. However, PCR materials can also be suitable for primary recycling if their composition is known in the required manner. Commonly PCR material is the source for secondary recycling. [36,57,59]

Primary recycling, also known as closed loop recycling or in-house processing, is the process of recycling non-contaminated polymers of a single type [58]. Primary recycled polymers may possess nearly prime polymer properties [60]. In thermomechanical processes, such as extrusion, the resulting side stream may have a low level of contamination, and high resemblance with the prime material’s structure, and therefore polyolefin pellet producers may apply primary recycling to their own processing lines [5]. This is a significant benefit for plastics recycling. According to Chu et al., closed loop recycling in the case of polyolefins takes advantage of the recovery of materials to produce new products for the same applications as the initial ones [4]. A popular method to utilize primary recycled polymers is to blend them in a specific proportion with prime material, to achieve desired properties. As a result, savings in materials can be obtained and weaknesses of recycled plastics minimized. [6,59]

Recycling procedures become more challenging when the material is collected from recyclers after consumer use (PCR). In this case, factors such as impurities and severe mechanical processing have a more pronounced negative impact on the properties of reprocessed polyolefins. Recycling becomes more difficult due to different product lifetimes, wide geographic distribution, and material inhomogeneity. [4,58]

Secondary recycling utilizes a series of mechanical operations where the physical form of plastic is changed to create new materials, for both similar and different applications than the original [57]. Secondary recycling of polyolefins begins with sorting the waste by physical characteristics, followed by removing contaminants and shredding into flakes. Further these flakes undergo compounding and pelletizing to meet specific standards before being extruded into final products. [58] This process is also called open-loop recycling [4]. Typically, secondary recycled materials are used in applications where the required performance is lower than the original, due to the quality inconsistencies [61]. A general recommendation suggests that post-consumer plastics should not be blended with prime plastics more than 20 wt.%, while pre-consumer plastics could be blended up to 80 wt.% [6, p.134]. Another study found [62, p.207], that properly preserved regrind could be incorporated into thermoplastic products in proportions of 10-30 wt.% with no significant change in their properties.

In tertiary recycling the polymer material structure is degraded by depolymerization. Chu, et al. describes that for polyolefins the tertiary recycling is also known as chemical upcycling [4]. As a result, monomers are recovered from the polyolefins. Tertiary recycling can be categorized based on the technique that is used for the monomer recovery. These recycling methods are either chemical or thermal (thermolysis). On the contrary, when plastic waste is incinerated to generate electricity, it is called quaternary recycling. This method of waste disposal is also called energy recovery from plastic waste. Plastics are valuable materials and therefore quaternary recycling should only be applied when other recycling methods are not feasible. [58]

2.5 Management of post-industrial recycled polyolefins

In the following chapters, the focus will be on examining the effects of mechanical recycling on polyolefins, and how polyolefin pellet producers utilize side streams from production in their own processes. In some processes the post-industrial material can be directly fed into the fabrication extrudes. This is a common approach of end-users utilizing plastic pellets, for example in flexible film manufacturing [36]. Furthermore, the extent to which polyolefin producers use external companies to manage side streams is also discussed.

Material which is generated during manufacturing process, is called post-industrial material [8]. If the material is re-grinded and induced back into the production process it is determined as post-industrial recycled (PIR) material [36]. In other words, post-industrial materials separated from the waste stream during manufacturing are classified as PIR. These materials can be obtained from the following stages of the plastic product manufacturing process: prime resin manufacturing and compounding including pelletization, plastic part manufacturing, assembly of plastic part, or packaging materials. [8,63]

The stage of production at which the PIR material is collected affects greatly its recovery possibility, contamination level, and quantity. Materials intended for re-granulation or re-use can be systematically classified into four types based on their characterization, potential for recovery and contamination level. These four types are presented in Table 3. [8,63]

Table 3. *Classification of post-industrial materials for re-granulation and recovery, adapted from [8]*

Classification	Description	Recovery possibility	Contamination level
Type 1	Incorrect products with visual defects such as wrong dimensions. Side streams generated from normal product operations such as screen pack changeover. Typically, fully recoverable through re-granulation.	High	Low
Type 2	Lightly contaminated products resulting from operational changes in processing, includes color and grade variations.	Moderate	Moderate
Type 3	Contaminated or difficult to handle products, resulting from product changeovers including, for example, purging. May contain contaminants such as machine leakage materials, materials from heaters or cross-contaminated resins.	Low	High
Type 4	Fallout products, easy to collect but challenging to clean and re-utilize.	Very low	Very high

During prime resin manufacturing and compounding processes, off-specification materials can be generated due to overprocessing, contaminations, or inconsistencies in the resin materials. Such distortions can be caused by production faults or technical flaws, such as incorrect dosing due to malfunctioning of the ingredient weighting instrument. Moderate distortions usually generate type 1 or type 2 materials. Post-industrial materials are also generated during normal processing operations. For example, during screen pack change, the extruded product is passed as second quality. Moreover, post-industrial materials are generated when new grades are tested with test runs to verify the processability. [2,3,8]

One of the largest and most challenging processing steps are the grade transitions, where side streams are generated during the start-up and shut-down phases. During these phases, the extrusion machinery must achieve or fall back from the desired operating parameters, resulting in type 2 or 3 production material that does not necessarily meet the quality requirements of the final product. Moreover, purging materials used to clean the inside parts of the extruder can be considered a non-desirable source of regrind material. This is because they have a high risk of contamination, degradation, and oxidation, therefore falling into type 3 classification category. Post-industrial materials also include fallout products (type 4), which typically cannot be utilized due to a highly increased risk of contamination. [2,3,64]

2.5.1 Effects of mechanical recycling on polyolefins

During re-extrusion, the polymer undergoes additional processing loops, which changes its performance and properties compared to prime polymer materials [57]. During reprocessing the material is exposed to additional stresses such as elevated temperature, oxygen, and shear. These stresses cause polyolefins to degrade, resulting in chain-scission and/or cross linking and chain branching. Strength, stiffness, and ductility are some mechanical properties that deteriorate from mechanical recycling, regardless of the type of polyolefin. Degradation causes a decrease in molecular weight and narrower MWD [64]. In more detail, material degradation reduces the number of intermolecular forces and entanglements in the molecular structure, thus reducing the viscosity of the polymer. These effects are observed as increased MFR value. Figure 11 demonstrates the correlation between re-extrusion temperature and MFR for PP. It illustrates how the temperature notably influences flow properties, particularly with an increasing number of extrusion cycles. [49–51,65]

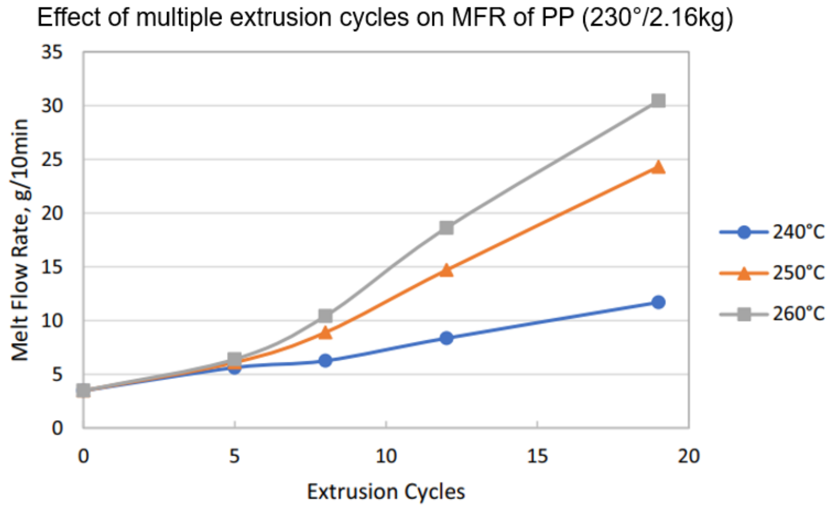

Figure 11. *MFR as a function of extrusion cycles for PP, highlighting the impact of temperature used for operation [65]*

Sin and Tueen highlight that blending recycled plastic with prime plastic compensates for the MFR remaining at an acceptable value [6]. Furthermore, structural degradation can cause discoloration, creating a yellowish tone or changes in haze values to recycled plastics. Magnitude of the change is compared to prime plastic and considered in the end product color setting. Moreover, reprocessing can negatively affect product clarity and transparency. [49–51]

When the number of re-extrusion cycles are increased it can be observed that the dominant reaction for PE-HD is chain scission and for PE-LD the dominant reaction is cross linking. Nevertheless, according to one research, chain scission has been reported to be the predominant reaction during the first two re-exclusion cycles of PE-LD using a single screw extruder operating at 30 rpm and 170 °C [66]. However, the presence or absence of oxygen may modify the mechanisms of PE thermo-mechanical degradation such that the absence of oxygen is more likely to favor branching and recombination. [51,67]

Similarly, as in polyolefin blending, AOs and heat stabilizers are utilized in mechanical recycling to control thermo-oxidative degradation during processing. In this context, re-stabilization is the term discussed. AOs and stabilizers might have been consumed during the first processing loop, so re-stabilization as an additional batch of these additives is often required for the second and further processing loops. Re-stabilization helps to achieve long-term stability for the product but does not recover any lost properties. To conclude, knowing the processing history of the resin provides essential information about the incorporation possibilities of rework. [62,68]

Other factors to consider when reprocessing polyolefins are increased risk of contamination and odor development. Increased risk of contamination may occur due to environmental factors. Moisture content in the regrind should be considered prior to reprocessing, as it exemplifies the impact of environmental conditions. Especially if the post-industrial material has been exposed to the atmosphere for an extended period of time, drying might be required. It has been reported in the literature that re-extruded polyolefins possess higher water absorption and swelling properties, due to poorer chain packing. All previously mentioned phenomena become more pronounced if the feedstock has time to age before re-extrusion. [62,64,68]

Mechanical recycling's effects on the chemical structure of polyolefins can be studied by Fourier-transform infrared spectroscopy (FTIR) -analysis. The evaluation of the effects is carried out by comparing the IR spectra of polyolefins before and after applying recycling technique. Thermo-oxidative degradation can be observed from FTIR spectrum as a presence of carbonyl groups. These carbonyl groups are formed as an intermediate in carbon oxidation reactions. However, there are uncertainties in the analysis as the identification and qualification of carbonyl groups can be challenging. Based on the FTIR results, one study strongly suggests that very little thermo-oxidative degradation occurs in the re-extrusion of PP [65]. Instead, the observed reactions are associated with thermomechanical processes, in the form of chain scission, as previously identified. However, for PE-LD and PE-HD the number of carbonyl groups has been reported to increase as a function of the number of extrusion cycles. This conclusion has been identified with FTIR analysis. [51,68,69]

In mechanical recycling, the oxidation resistance of polyolefins can be investigated with DSC by determining the oxygen induction time (OIT). OIT measurement is performed for a specimen under constant heating in an inert gas atmosphere. At a certain temperature, the sample is induced to oxygen. The apparatus measures the time at which the sample is oxidized. Thus, the analysis provides qualitative information on the activation level of additives such as AOs and is useful for assessing the processability of reprocessed polyolefins. OIT analysis is also utilized for comparison purposes, since it provides information whether structural changes have occurred during reprocessing. [70] Moreover, OIT is a useful analysis in reactive processing, as it can be utilized to determine the stability of a polymer compound [71].

Another method to analyze processability is to measure rheological behavior of reprocessed materials. Rheological measurements examine how materials respond to external forces by qualifying the correlation between stress and the rate of deformation. The two most commonly used rheometers are capillary rheometer and rotational rheometer. The capillary rheometer measures the flow of molten plastic in a thin capillary as a function of pressure drop. The viscosity of the melt can be determined with a capillary rheometer when the radius and length of the capillary, the pressure differential between the ends of the capillary and the volumetric flow rate of the melt are known. The pressure difference is a function of the shear rate. On the contrary, with rotational rheometer either the shear stress or the deformation rate can be determined, while the other is controlled. Therefore, this rheological measurement technique provides broader perspective on the determination of rheological properties. [18,70] The working principle of rotational rheometer in dynamic measurements will be described in more detail in the following chapters.

Environmental stress crack resistance (ESCR) describes the materials' ability to resist cracking caused by environmental conditions. ESCR correlates positively with increasing polymer density, and therefore PE-HD has better ESCR than PE-LD [36]. In general, more rigid structure and long molecules with short chain branching shows good ESCR. PP typically resists environmental stress cracking well, but PP copolymers may exhibit increased susceptibility to certain chemicals due to decreased degree of crystallinity. ESCR deteriorates during re-extrusion due to changes in molecular weight, density and crystalline structure. Jubinville et al. states that PE-HD would exhibit a drop of 60% in ESCR value when re-extruded four times [68, p.13]. The reason given for this is that high molecular weight chains are consumed by chain scission, so that resistance to cleaving also declines [68]. However, the initial study by Zahavich et al. informs that the ESCR drop would only apply for prime copolymer, whereas in the case of re-extrusion of PE-HD homopolymers, the ESCR value only showed little or no reduction. The exact composition of the copolymer was not informed in the study. [72]

2.5.2 Treatment by polyolefin pellet producers

A majority of single resin plastics recycled from post-industrial sources are processed using standard equipment, including melting, extrusion, pelletization and final forming [26]. When post-industrial materials are circulated and reprocessed within the same process in which they were produced, a case-by-case assessment will be made to determine whether it is included in the recycling rate targets, as stated by the European Commission. However, often the recovery of pre-consumer materials is perceived as a reasonable use of raw material rather than recycling. [56] ISO 15343:2008 standard supports the previous claim by stating the material does not count towards recycled content if it is generated and recovered within the same manufacturing process [73]. It is likely that due to these regulations and concerns about potential trade secrets, many companies will refrain from providing comprehensive information about their PIR treatment methods.

LyondellBasell (LB) has a Circulen polymer brand which focuses on recycled and renewable-based polymers. CirculenRecover is one of the three sub-brands of the brand, specializing in mechanical recycling of both pre-consumer and post-consumer waste. The CirculenRecover products undergo mechanical processing to form pellets, which can be utilized either independently or by blending and compounding with prime fossil-based resin. [74] Thus, the approach of LB appears to differ from other PO producers, as they offer information regarding their products incorporating PIR materials.

In addition to LB, TotalEnergies have developed at least one grade of PP incorporating PIR material. Table 4 summarizes the resin grades by LD and TotalEnergies where PIR materials are utilized as a blend with prime resin. Grades produced by LD are available in Europe, but it is mentioned that their usage has been restricted for highly regulated applications such as food contact products. In contrast, the grade provided by TotalEnergies is claimed to be FDA compliant, making it suitable for food packaging and personal care applications [75]. Table 4 includes MFR value, density, and the percentage of post-industrial recycled resin used in the product. The MFR values are measured at 230°C under a load of 2.16 kg. Density is measured at 23°C. A more detailed description of the mechanical properties for some of these grades is given in Appendix A. [74,76]

Table 4. *Product information of PP grades containing post-industrial resin [75–77]*

Grade name	Producer	MFR [g/10min]	Density [g/cm^3]	Recyclate [%]	Source
PP340H Natural	LB	1.9	0.900	>30	PIR
PP522H Natural	LB	2.0	0.900	30-35	PIR
PPH804IF Natural	TotalEnergies	30.0	0.905	40	PIR
PP320S Pearl	LB	40.0	0.930	>98	PIR
PP520T Crystal	LB	45.0	0.910	>98	PIR
PP310U Graphite	LB	70.0	0.945	>98	PCR/PIR

The table demonstrates that the recycled content of colored products is significantly higher than that of natural transparent products. As described previously, mechanical recycling can affect the color of the resin due to structural degradation [50]. This correlation might explain why products containing high levels of PIR (>98%) are dyed. Commonly, polymers used in biaxial stretching applications require a much lower MFR value than injection molding [78]. The MFR value of PP340H therefore supports the processing techniques mentioned in the product data sheet (PDS), which are sheet extrusion and blow molding [79]. According to the PDS of PP522H, this pellet grade is utilized in biaxially oriented PP film production [77]. PPH804IF could be utilized in sheet extrusion or blow molding applications. PDS of PP520T and PP310U clearly informs that PIR is recovered from the waste stream created during manufacturing. In addition, injection molding is specified as the processing method for both grades. [80,81] Taking this into consideration alongside the product data presented in table 4, it is likely that PP320S grade would also be processes with injection molding.

A comparison is made between Moplen RP390H, a similar prime resin grade from LB, and PP340H, a grade containing PIR. Both resins are PP random copolymers and utilized for extrusion applications including sheet extrusion and blow molding. Furthermore, Moplen grade can also be processed with thermoforming. Both grades share the same characteristic of transparency. Table 5 presents typical properties informed for Moplen PP390H and CirculenRecover PP340H, illustrating their similarities and highlighting their differences in terms of performance. [76,79,82]

Table 5. *Property comparison between prime and recycled PP resins produced by LB [76,79,82]*

Material Properties	Moplen PP390H	CirculenRecover PP340H
MRF (230°C/2.16kg) [g/10min]	1.8	1.9
Density (23°C) [g/cm^3]	0.9	0.9
Tensile Modulus [MPa]	1050	1070
Tensile Stress at Yield [MPa]	29	30
Tensile Strain at Break [%]	**>50**	**>30**
Charpy impact, notched (23°C) [kJ/m^2]	**26**	**55**
Haze [%]	15	15

As indicated in Table 5, the properties of these materials have high resemblance. The most remarkable differences can be seen from two characteristics. First, the tensile strain at break is lower for recycled resin. This observation seems logical, because recycled resin has lower homogeneity, thus decreasing the tensile strain value at fracture. However, it should be noted that the given value of greater than does not reflect the real value, making it challenging to estimate the actual difference between the materials. Second, the Charpy impact strength shows a higher value for the PIR containing resin. This difference cannot be coherently explained without additional information from the material supplier, as typically recycling and reprocessing would deteriorate mechanical properties of PP as a result of chain scission. Chain scission of PP would be reflected as decreased impact strength and tensile elongation at break.

Moreover, the CirculenRecover PP522H product has a corresponding prime product called Moplen HP522H. These grades have corresponding properties according to the

PDS of the materials, and both are utilized in flexible packaging applications. However, natural silica is present in the CirculenRecover PP522H grade but is absent in the Moplen HP522H grade. Natural silica is utilized in the grade containing PIR content as an antiblocking agent, which reduces the adhesion between two adjacent film layers. The prime grade is used as a packaging material for food contact packaging, whereas the CirculenRecover grade is under development, thus cannot be used for food contact materials. [77,83]

To summarize, several PO producers appear to have a policy of not publishing their processing methods regarding PIR as publicly available information. Instead, companies producing POs have expanded their activities into processing PCR materials. There is extensive information available on the use of PCR materials. Companies processing PCR materials to manufacture recycled pellets include LyondellBasell, TotalEnergies, Dow, Hanwa, LG chem, and INEOS [74,84–88].

2.5.3 Practices by end-users

According to ISO 14021:2016 standard, pre-consumer material does not include materials that can be reprocessed within the same process which generated it. Therefore, when post-industrial material is reclaimed within the same manufacturing process it is called scrap or regrind. This topic especially concerns end-users' practices for PIR content treatment. [89]

Materials such as edge trim in sheet or film operations, and runners in injection molding (cold runner mold system) are scrap materials that have potential to be used as regrind products [13]. Post-industrial scrap materials can be collected and treated at the site of manufacture. For example, in a film-forming extrusion process, it is considered a standard practice to feed some part of produced scrap back into the processing apparatus. Before utilizing the scrap as a feedstock material, it must be pretreated to enhance its flow properties and reduce the size. Pretreatment is done, since scrap materials may have different physical forms. The actual reprocessing might be integrated into the film production line, which is the more economical way, or it can be placed at a remote location. [64,90]

Erema is one of numerous companies that has developed recycling systems to treat plastic waste for reprocessing purposes. This Austrian company provides diverse technologies for the treatment of post-industrial materials, including edge trim, sheet waste, and filaments, for a variety of polymeric materials. Such scrap grades typically have a remarkably similar material quality as the prime material [64]. As an example, a schematic picture of their recycling unit called Intarema K, is presented in Figure 12. This machinery is utilized for non-printed PE edge trim. [91,92]

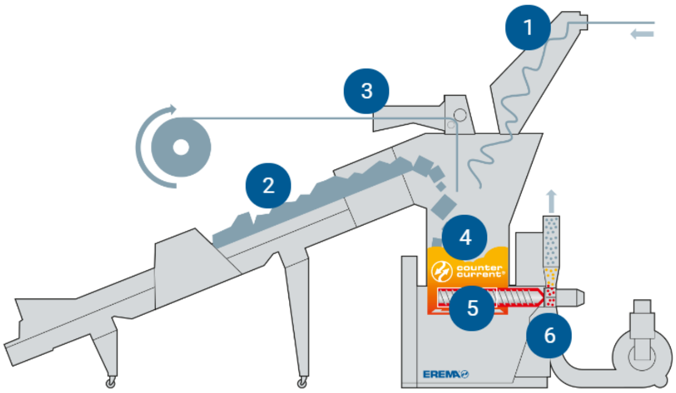

Figure 12. *Schematic representation of the Intarema K recycling unit [92]*

Intarema K contains six primary components. The method of feeding edge trim into the machinery varies based on its composition and quantity. It can either be automatically conveyed via pipelines and cyclone (1), transported by a conveyor belt (2), or fed in by feed rollers (3). Material is pretreated in the preconditioning unit (4), containing operations such as cutting, drying, mixing, compacting, and buffering. Next, material is fed into extruder, which operates in a continuous manner at low temperatures. Both the preconditioning unit (4) and extruder (5) are patented. Last, the melt is pelletized in an air-cooled pelletizing system (6). [92]

As another example, Lyondell Basell (LB) notes in their polyolefin film extrusion-guide that their coextrusion film production systems operate a recycling unit. A schematic representation of the recycling unit is presented in Figure 13. Waste material generated during film operations contains for example start-up waste, edge trim and slitting trim. This waste is automatically collected into pelletizing units, after which the material is combined with prime resin in specific proportion in the extruder hopper. [93]

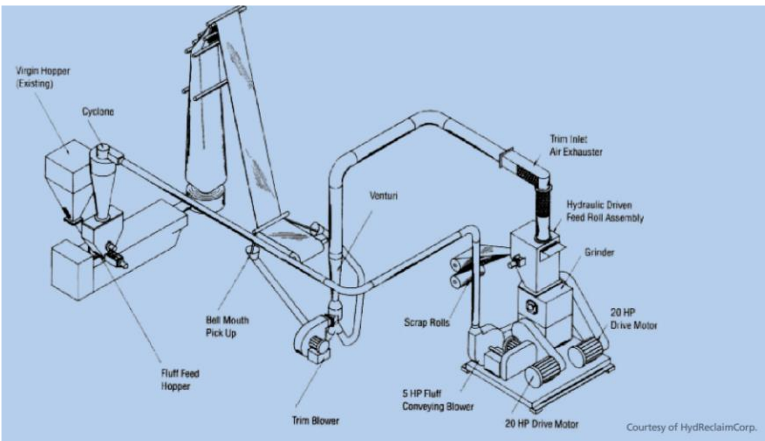

Figure 13. *LB recycling unit for processing scrap [93]*

On the contrary, another guide produced by Lyondell Basell which discusses extrusion coating, states that a substrate-free polyolefins could be sold as scrap to third parties. Meaning that, if the scrap only contains polyolefin film and no other film materials, it can be sold for other purposes or products, but it may not be used as feedstock within the process. The use is limited due to increased probability of defects such as gel formation and odor development. [94] It can therefore be concluded that the close loop recycling of materials within a process depends on the processing method and thus on the application of the product.

2.5.4 Treatment by third parties

By-products can be utilized on-site, as described in the previous chapters, or they can be exported to third parties for off-site treatment [58]. In particular, polyolefin producers have the option to sell post-industrial materials to third parties, serving as a feedstock for further processing. In this scenario the post-industrial material is either recycled in a crushed form or re-pelletized, making it suitable for the same production equipment used to process prime raw material. Alternatively, polyolefin producers may utilize external treatment facilities to recycle waste materials for their internal requirements. It's important to note that in this scenario, the external treatment facility does not assume ownership of the plastic at any stage of the processing. [26,95]

In general, recycled resin is often more affordable than prime plastics primarily due to the decreased homogeneity and uncertainties in its processing and additive history [95]. This perspective aligns with a study conducted by Kutz, suggesting that manufacturers often prioritize lower costs when selecting recycled resins over prime materials, taking into account the variations and uncertainties associated with the recycled material [26]. The economic feasibility of recycling PP and PE is also affected by the cost of prime resins, which can be a challenge to the competitiveness of recycled resins. However, this challenge is more pronounced for post-consumer plastics recycling due to a more complex and resource-intensive process compared to pre-consumer resin recycling. Moreover, additives are used to hinder degradation due to reprocessing, or to meet specific product requirements. However, their use is minimized to control the overall cost of recycled plastic in order to make recycling process profitable. [26]

A Finnish company Lassila & Tikanoja (L&T) specializes in the processing of industrial residues and rejects, encompassing various industrial process side streams. Its primary processing center is in Merikarvia, Finland. L&T informs us that the main suppliers of plastic waste are companies in the plastics industry. Collected materials are treated, granulated, and then recycled as raw material for the plastics industry. L&T produces over 100 distinct types of recycled plastic granulates. Alternatively, non-recyclable plastic materials are incinerated for energy production. [96]

RHSpolymere is a company which produces reprocessed PP and PE pellets made from post-industrial raw materials. These regranulates and rCompounds are compounded along with additives, such as stabilizers, to customize the product properties as desired. The company utilizes feedstocks in different forms, one of them being fallout products and residual resin materials. Furthermore, regranulates are available in various colors. RHSpolymere has classified the standard grades by market segments and processing methods. Market segments include for example packaging applications for transportation and products. [97]

M.Holland operates as a distributor and reseller of thermoplastic resins. In 2023, the company launched its Mfinity line, offering sustainable resins with up to 100% PIR content. M.Holland collaborates with manufacturers to produce these resins under the Mfinity brand. These grades are marketed to manufacturers as environmentally friendly alternatives to prime resin. Notably, the PP grade Mfinity SPP306-PPX-TR or WT, meets all U.S. Food & Drug Administration requirements for food packaging applications. [98] Similarly, Ter Plastics is another distributor company offering mechanically recycled commodity plastics. The company collaborates with polyolefin manufacturers to produce PIR resins under the TEREZ ECO brand. [99] Table 6 presents a selection of products offered by these companies. It should be noted that for both RHS Polymere and M.Holland, a number of other products containing PIR content are available. For certain products, the MFR value and the density value are given.

Table 6. *Polymer grades and properties comparison containing PIR [99–104]*

Producer	Polymer family and grade name	MFR [g/10min]	Density [g/cm^3]	PIR [%]
RHS Polymere	LDPE 210 F	0.5-1.0 (230°C/2.16kg)	0.920-0.940	100
RHS Polymere	HDPE 5009 E	0.5-0.9 (190°C/5kg)	0.955	100
RHS Polymere	PP 1500i	30-50 (230°C/2.16kg)	0.910-0.940	100
M.Holland	PP- Mfinity SPP306-PPX-TR or WT	6.0 (230°C/2.16kg)	Not specified	100
M.Holland	PP- Mfinity BK30-2-COPX-BK	32.0 (230°C/2.16kg)	Not specified	100
Ter Plastics	LLDPE film	Not specified	Not specified	100
Ter Plastics	HDPE BM	Not specified	Not specified	100

From Table 6, the diversity of PIR products can be observed, demonstrating a range of polymer family types and characteristics. This versality of products made of PIR content highlights its potential for addressing diverse needs across different sectors. In general, more information appeared to be available on PIR processing by external parties than on polyolefin pellet producers' own methods concerning PIR. This may be related to differences in business approaches, as external treaters base their business on the reutilization of waste. Therefore, it is reasonable that polymer grades containing PIR materials are marketed with more detailed specifications.

According to LyondellBasell's 2023 sustainability report, they successfully reduced the amount of material sent for disposal by 47% compared to 2022. This reduction was accomplished by pursuing reuse, recovery, and additional recycling options for non-hazardous waste streams. In particular, a key initiative was the implementation of an off-site project to convert waste material from the cleaning of production lines into reprocessed pellets suitable for commercial products. This project is estimated to result in a reduction of around 500 tons of waste, as it will be diverted to new uses. [105, p.46] This is a notable example of the quantities of savings that are achievable by investing in reprocessing material streams.

2.6 Legislation

This chapter briefly discusses the legislation relating to food contact materials and cable products. The European Union specifically regulates these materials to guarantee their safe usage.

2.6.1 Food contact materials

Materials and articles designed for direct interaction with food and beverages, such as packaging, are collectively known food contact materials (FCMs) [2]. Borealis Boremix compounding plant produces PP pellets that are further processed into packaging applications and FCMs. Materials that are intended to come into contact with food are applied with the Commission regulation No 10/2011. This Plastics regulation contains a Union list of authorized substances, containing for example monomers, additives, and production aids. Only substances included in this list can be used to manufacture FCMs. Plastics Regulation assign an overall migration limit (OML) of 10 mg/dm^2 for all FCMs. OML is measured with a representative food stimulant of the food that comes into contact with the material in the final product. Thus, the OML signifies the highest acceptable level of non-volatile substances that can transfer from a material or article into food simulants. [106]

Furthermore, to assure the safety of FCMs, specific migration limits (SMLs) are specified for some substances. The migration limits for these substances are stated as milligrams per kilogram of food (mg/kg). The Plastics regulation also takes into account the contribution of additives to SMLs. Meaning that if a substrate specified in the Union list contains additives, its effect on the SML of the substrate should be considered. [106]

There are two general regulations applied for all FCMs Framework Regulation EC 1935/2004 and Good Manufacturing Practices (GMP) in Regulation EC 2023/2006. These regulations apply to all materials and articles that come into direct or indirect contact with food, regardless of the material. The Framework regulations set restrictions so that FCM does not migrate to food in quantities which could be dangerous to human health, cause significant changes to the composition of the food, or cause distortion in the organoleptic characteristics. In addition, it includes regulations regarding aspects such as labelling and traceability. [107,108]

The Framework Regulation states that specific measures may be adopted for recycled FCMs [107]. Commission Regulation No 2022/1616 covers recycled FCMs in a more comprehensive manner. This Regulation specifies that it is essential to ensure that the plastic is thoroughly cleaned during the recycling process, to a point where residual contaminants have no risk to human health and no impact on food safety. Reusing materials from controlled and closed loops of productions is determined as suitable recycling method, including reprocessing scraps and off-cuts from plastic processing. [109] Following this legislation, the use of PIR content for FCMs can be perceived as a more feasible option than PCR material incorporation. Foremost, since the reprocessing of PIR content can be implemented as closed loop recycling, and the processing history is commonly better known than for PCR materials.

2.6.2 Cable products

Borealis produces various PE grades for cable protection applications. The applications include for example optic and data cables, where PE is utilized as a jacketing material. The purpose of cable jacketing is to form a protective outer layer to all other components of the cable product. This layer protects the interior from mechanical and environmental conditions such as corrosion causing substances, moisture, and impacts. [110,111]

Standardization of jacketing is integrated to power cable applications. Depending on the cable application, a specific standard can be applied. For instance, cables fall under distinct standards based on their voltage specifications, including the following: low voltage, medium voltage, and high voltage. The International Electrotechnical Commission (IEC) is a global organization providing framework standardization related to electronic devices and systems. The primary objective of IEC standardization is to recognize and address any safety concerns related to electrotechnical equipment. [112]

Power cable IEC standards describing jacketing requirements include for example IEC 60502 and IEC 60840. These standards define efficiency and safety criteria, including parameters such as the maximum conductor temperature, which varies according to the type of jacketing material. Moreover, common testing methods considering jacketing materials of electric and optical cables are specified in EN 60811 standard. [113]

3. DESCRIPTION OF PROCESSING TECHNIQUES

In order to determine whether post-industrial side streams could be utilized in the production of primary products, an understanding of the processing techniques is required. In this chapter the pellet production processes with twin-screw extrusion are explored. The experimental part will include batch production on a pilot-scale, and based on the results upscaling possibilities to industrial-scale extrusion are evaluated. It is therefore important to investigate how different processing settings between the pilot and industrial lines may affect the final product.

The purpose of polyolefin pellet production is to convert polymer formulations and additives into a more convenient form for further processing. The process includes compounding in twin-screw extruder, where the feedstock is melted, homogenized, and forced through a capillary shaped die. Pelletization occurs at the final stage of the extrusion process, when the continuous polymer strand is cut into pellets with a uniform cross-section. [31] The pelletization process utilized by these two lines differs, employing two of the most common methods: strand cut pelletization at pilot line, and underwater cut pelletization at industrial line.

3.1 Pilot-scale pelletization line

Borealis Polymers Oy operates a pilot-scale pelletizing line for research purposes within its laboratory facilities in Porvoo. The line utilizes a pilot-scale extruder called Werner & Fleiderer 30 (ZSK-30). The pilot-scale pelletization line is more commonly referred to as WP-30 line. A schematic picture of WP-30 pilot-scale extrusion pelletization process is presented in Figure 14. The figure represents a typical strand cut pelletization process. The production at WP-30 differs from the figure in that the pilot-line uses a two-hole extruder die to produce two polymer strands, and the haul-off is incorporated with a dryer unit.

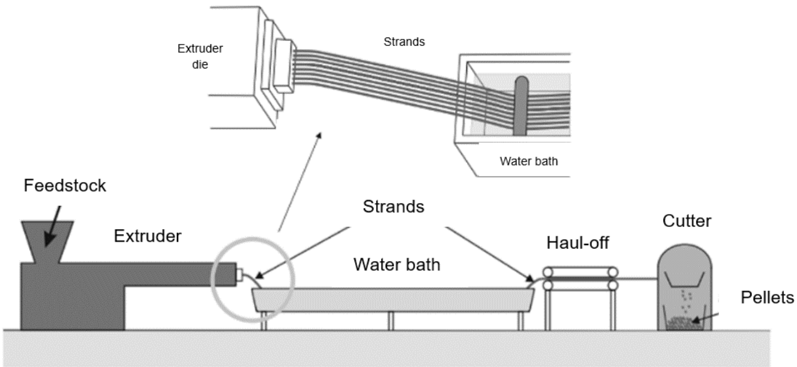

Figure 14. *Pilot-scale extrusion process to produce pellets, adapted from [31]*

For WP-30 processing the feedstock is applied manually into a hopper. The feedstock is applied as a premixed dry blend including base resins and additives. Vacuum and liquid feeds are possible due to the modular barrel structure. Moreover, the apparatus enables nitrogen feed to hopper. The material input rate is metered by a gravimetric scale, and during hopper filling the feeder is changed temporarily to a volumetric program.

The barrel consists of 12-barrel blocks that are divided into pairs to form 7 temperature zones, each of which has a separate temperature control. WP-30 line utilizes pelletization process, where polymer extrudate strands are first cooled then pelletized by rotating cutting blades, known as strand cut pelletization [114]. Material exiting the extrusion die is directed and immersed in a water bath for cooling. With the help of rolls and haul-off, polymer strands are fed into the dryer, which removes the excess water and further allows the material to solidify. After the dryer, the polymer strands are directed to cutter where pellets are formed and further collected.

3.2 Industrial pelletization line

The industrial pelletization line operating at Borealis Polymers Oy Porvoo facilities is called SL-200 line. The feedstock is applied automatically from raw material silos. Each ingredient has its own raw material silo, which enables addition of PIR content or additives along with prime base resins. The extruder consists of distinct barrel blocks with heating and cooling abilities. Additives in solid and liquid form can be applied to the compounding process. The working principle of SL-200 line reflects underwater cut pelletization process, as polyolefin strands exiting a die plate are immediately pelletized by a rotating cutting blade and immersed into circulating water [2]. The flowing water conveys the pellets into a centrifugal dryer which separates water from the pellets. The water is re-circulated within the process, while polyolefin pellets are conveyed to a classifier. The classifier ensures produced pellets are uniform in shape and size. The end product is conveyed pneumatically to packaging operations.

Overall, the industrial process is complex compared to the operations in the pilot-scale line. Some of the operational details are discussed. In the SL-200 line, a screen pack and breaker plate are positioned before the die plate to eliminate impurities and unhomogenized material. Moreover, the uniformity of pellets is ensured by the classifier. These features are not present in the pilot-scale line. In general, the WP-30 line operates with a higher degree of manual intervention compared to the highly automated processes of the industrial line.

At the industrial line, certain procedures have been developed to ensure the quality of the material. During operational stages where the quality of the product may deteriorate for some reason, the material collection is diverted to a separate container from the primary product. These side streams can be generated from multiple operational stages of the process. The largest side streams generated at SL-200, arise from the following production steps and can be divided into the quality categories (Chapter 2.5) described previously: product changeover purging (Type 3), stabilization to operating values, and screen pack changeover (Type 1 or Type 2). Given their levels of contamination, the latter two side-streams have a high potential for reuse. These materials constitute the primary focus of the experimental section and will be referred to as PIR content, emphasizing that the post-industrial waste is repurposed. However, it is possible that the PIR content composition is not fully known, and the generated material could be from other operational stages as well. These could include for example operational disturbances, which cause material to be collected separately, as they may have anomalies such as impurities.

The utilization of post-industrial materials on the SL-200 line is straightforward. During production, these side streams are generated and collected in octabines or containers. Each packaging method has its own dedicated pneumatical transfer line to the raw material silo. When post-industrial materials are used, they are fed from this separate silo into the process.

3.3 Functional equivalence of pilot- and industrial-scale

The pilot-scale extruder is structured to correspond with the industrial-scale production line. However, due to structural differences that arise from down-scaling of WP-30, the lines are not fully compatible. For example, equal temperature profiles between the lines can be used, but in practice they do not fully represent each other. Both pellet production lines utilize corotating fully intermeshing twin-screw design, but the modular screw sections known as the screw map is different due to the size differences of the extruders. Both lines utilize mainly gravimetric scales for raw material metering, but these scales can be converted into volumetric setting. Moreover, intermeshing corotating twin-screw extruders are always operated following a starve feeding principle [31]. This principle enables more efficient mixing as the material intake is smaller. Some operational parameters of the extruders are provided in Table 7, to highlight the structural differences.

Table 7. *Comparison of WP-30 pilot-scale extruder and SL-200 industrial-scale extruder*

Extruder information	WP-30 Pilot-scale	SL-200 Industrial-scale
Screw diameter [mm]	30	119
L/D ratio	38:1	48:1
Screw speed (rpm)	max 300	40-600
Expected throughput [kg/h]	5-10	up to 3000
Multi-hole mold	2	80-150
Typical residence time [s]	45-90	ND

WP-30 extruder has a smaller L/D-ratio compared to the SL-200 extruder. Screw and extrusion temperature profiles are set to match as much as feasible in both lines taking the scale difference into account. Therefore, there is a risk that even if the pilot-scale experiments provide good results, the results at industrial scale might be differ due to higher shearing and possibility of degradation, and vice versa.

SL-200 extruder screw has a higher screw length relative to its diameter. Thus, the material is more extensively mixed [62]. Moreover, the material is possibly subjected to greater shearing forces. Greater L/D ratio allows for higher pressure build up, which is linked to higher throughput rate at the industrial compounding line. [13] Moreover, the production-scale apparatus utilizes a multi-hole die for PP products, which enables significantly higher production rates compared to the pilot-scale machinery. At SL-200 line the material is expected to experience finer dispersion of additives and more uniform melting compared to WP-30. In summary, SL-200 extruder with a 48:1 L/D ratio offers thorough mixing, higher pressure capabilities, and better thermal control, making it suitable for complex and demanding extrusion processes. WP-30 extruder with a 38:1 L/D ratio provides a balanced approach with SL-200 extruder.

In practice, the SL-200 line operations primarily follow product specifications. Product properties such as the MFR value are measured during production, according to which operating parameters are modified if necessary. In other words, the control values of the operating parameters are modified to ensure that the final product meets the customer's requirements.

4. CHARACTERIZATION FOR QUALITY CONTROL

This chapter presents the main quality control characterization methods used in the experimental part of this study. The properties linked to the test methods are grouped into the following five groups: rheological, physical, mechanical, thermal, and chemical. Each method provides valuable information about properties, in the context of the modified material formulation. The chapters cover the following aspects of each testing method: its working principle, the type of information or results it provides, and their analysis, particularly in relation to studying the incorporation possibilities of PIR content.

4.1 Rheological properties

Rheological and flow properties of polymer melts are crucial aspects considered in the processing of polyolefins. Melt mass-flow rate (MFR) is a commonly utilized technique in quality control and for predicting the processing behavior of polymers. This method provides basic information about the flow characteristics of polyolefins. For more detailed and quantitative analysis of rheological properties, rheometry is often used in addition to the MFR determination. Rheometry encompasses various techniques, such as capillary and oscillatory, to determine material's response under different flow and deformation conditions. [115] Viscoelastic properties of molten polymers are typically studied with dynamic oscillatory testing, since it provides accurate information of material's response reflecting both viscous and elastic behavior. Such testing is particularly useful for gaining insights into structural properties such as average molecular weight and MWD [116]. When a material is modified, these are important characteristics to accurately predict the product performance and behavior in further converting processes [117].

4.1.1 Melt mass-flow rate

MFR value provides qualitative information about the melt processability of a polymer, and it directly correlates with viscosity and average molecular weight of the polymer. Primarily, MFR value is used for melt flow behavior comparison purposes and is not suitable for precise calculation of melt behavior. [118] The measurement result is a single value (g/10min) which is compared to the reference MFR value to analyze the impact PIR content on the product characteristics. Comparing MFR values between initial grade characteristics and compound with PIR content, the changes in molecular structure and processability can be qualified. However, it is important to notice since MFR is a single value it does not provide comprehensive information on material's rheological properties, such as differences in shear-thinning behavior or changes in MWD, thus other rheological measurements are often utilized [36]. The testing procedure is done according to ISO 1133-1:2022 standard. Method B will be utilized in the experimental section. PP is typically measured at 230°C with a load of 2.16kg [119]. A greater load of 5, 10, or 21.6 kg can be used for thermoplastics with higher viscosity [36].

4.1.2 Dynamic rheology measurements

Dynamic rheology measurements utilize rotational rheometer according to ISO 6721-10:2015 standard. The measurements are carried out with frequency sweeps at a constant temperature, and the physical response of the specimen is examined. [120] Oscillatory testing is a valuable test method to provide comprehensive understanding of the microstructure of the sample [117]. The measurement can be performed either under strain controlled mode or stress controlled mode. Under strain controlled mode, the functionality of the rheometer relies on sinusoidal oscillatory strain which is created by moving one of the measuring heads, while the other remains stationary. For measurements undertaken under strain controlled mode, the resultant torque and the phase shift between the displacement and torque are measured. [121]

The material is subjected to deformations in the linear viscoelastic region (LVR). The material response is also sinusoidal, and the viscoelastic nature of the material can be characterized by the phase difference. In LVR the induced changes are small and correlate with the molecular structure of the polymer. The study in the LVR allows the molecular weight, MWD, and molecular chain branching of the polymer to be investigated. [18] Results obtained from frequency sweep measurement include the following: complex modulus (G*), storage modulus (G'), loss modulus (G''), and complex viscosity (η*) [120]. Frequency sweep results are typically obtained as a diagram illustrating the relationship between moduli components (G' and G'') and complex modulus, as presented in Figure 15 [122]. These components are presented as a function of angular frequency at logarithmic scale, in order to obtain the viscoelastic frequency dependence of a material [117].

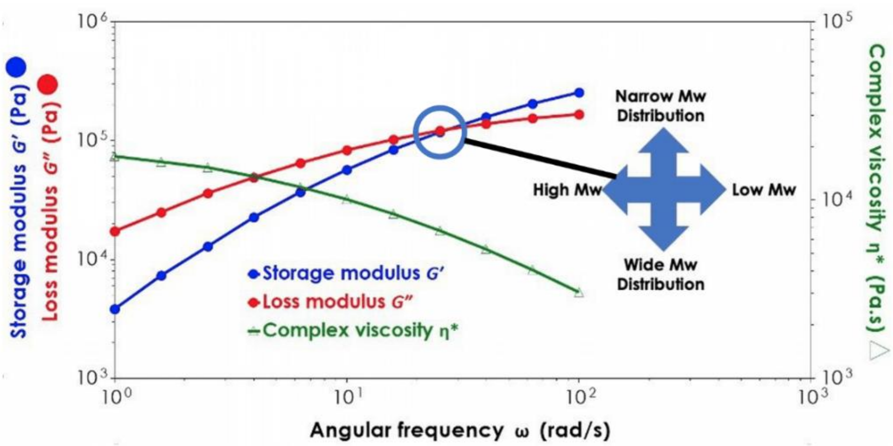

Figure 15. *Typical frequency sweep diagram, presenting the relationship of moduli components and complex viscosity as a function of angular frequency [123]*

As illustrated in Figure 15, at low frequencies the viscous response is dominant (G''), and at higher frequencies a change is observed, as the material exhibits more elastic response (G') [124]. The crossover point of storage modulus and loss modulus reveals information about the structural changes in average molecular weight and MWD and can be used to assess changes due to processing and composition [122]. Crossover frequency correlates with average molecular weight in an inverse way, and similarly crossover moduli is related to the MWD in inverse way [116]. Moreover, at low angular frequencies (low shear rate) most polymers exhibit Newtonian region where the complex viscosity approaches a finite value known as the zero shear viscosity, and this value correlates directly with the average molecular weight of the polymer [116,122]. The slope of complex viscosity curve reveals information about the MWD, as a polymer with narrow MWD exhibits non-Newtonian flow at a higher angular frequency than a polymer with the same zero shear viscosity but a broad MWD [123].

As previously stated, rotational rheometry provides a broader insight into the rheological properties of materials than capillary rheometry. Dynamic measurements are sensitive to structural changes, therefore relatively small deformations due to, for example, reprocessing, can be noted. [18] With dynamic frequency sweep it is possible to obtain information on both elastic and viscous behavior of the material, as previously stated [116]. Due to the versatility, the experimental section utilizes dynamic rheometry to study samples with PIR content.

4.2 Physical properties

In this study, the physical properties that are critical for the product quality are gel formation and yellowness index (YI). Gel formation can be used to assess PIR content and base resin compatibility and suitability of chosen operating parameters [36,46], from which both contribute to the overall performance of the product. While the YI can reveal changes in color that may arise from degradation associated with reprocessing of PIR content [49]. Given the complexity involved in the comprehensive measurement and perception of color, YI is a common parameter to assess the change in color [125]. Both of these physical characteristics impact the overall appearance of the product. The following subsections discuss the measurement of gels and the YI.

4.2.1 Gel formation

Gel inspection can be done for PP products according to ASTM D7310. The testing apparatus contains the following four systems: extruder, measurement software, pellet dryer, camera instrument and reel. The defects are inspected from an extruded cast film utilizing the camera instrument and presented as the number of detected findings per square meter (n/m^2). Gel index, a single numerical value, is the sum of all observed gel findings per size class, multiplied by their multiplication factor. [126]

Gels create visual distortion and structural defects, which decrease the processability, quality, and overall performance of the final product [46]. Three following types of findings, referring to physical anomalies in the polymer matrix, can be inspected with the measurement: gels, contaminants, and others. Gels appear as a lenticular non-blended structural change, matching the surrounding material color. Contaminations are identified based on color deviation and can be observed as less transparent in transmission compared to the rest of the polymer matrix. Lastly, other defects can be inspected. [126] The experimental part will primarily focus on gels, as they provide essential information on material behavior and compatibility of PIR content incorporation.

4.2.2 Yellowness Index

During pilot-scale experiments, the PIR material is reprocessed making it more susceptible to structural degradation. Based on the theory described earlier, it is known that structural degradation can cause color changes from an ideal white color, and therefore it is reasonable to measure the yellowness index (YI). The measurement is done according to ASTM E313 standard and carried out with a spectrophotometer. An increase in YI value indicates increased yellowness, whereas a decrease in YI value indicates a color change towards more bluish or greenish tone. [127] YI measurements provide information on whether PIR content changes the color profile of the product compared to the reference sample. In addition, correlation between PIR content and YI can be studied.

4.3 Mechanical properties

Mechanical properties are essential characteristics in quality assurance, and they provide a numerical indication of material performance. Peel strength is a mechanical test to evaluate the performance of 3-layer PP coating, which is utilized for steel pipes [128]. Mechanical properties of recycled plastics can be commonly characterized with tensile properties, material hardness, and ability to withstand impact or bending [18]. These characterization methods are described below.

4.3.1 Peel strength

The purpose of coating is to protect a steel pipe from mechanical damage and corrosion during end-use application. Procedure for determining the peel strength of a steel pipe coating (SPC) is an internally developed method by Borealis and is therefore not based on a specific standard. However, the method is related to ISO 21809-1:2018 and DIN 30678 standards [129,130]. Particularly, the PP coated steel pipes to be further examined are utilized in the petroleum and natural gas industries as both onshore and offshore pipeline transportation systems. For these applications, a 3-layer PP coating structure consists of the following: an epoxy primer, a grafted copolymer adhesive plastic (PP), and a PP plastic topcoat. The purpose of epoxy primer is to protect steel from corrosion and to improve the bonding of subsequent protective layers to the steel surface. PP adhesive is utilized to form a bond between the primer and the topcoat. The PP topcoat must withstand temperature changes and extreme mechanical stress related to application demands. [128] During SPC process inter coating time (ICT) is a critical parameter which refers to the time between applying epoxy coating followed by adhesive coating. ICT highly affects the adhesion formation, and it is chosen based on the reactivity of the epoxy grade to avoid excessive cross-linking of the epoxy prior to subsequent coating [131].

A peel strength test is used to assess the adhesion of the coating, informing whether the coating process has been successful or not. The test is conducted with a tensile testing apparatus, including a specific specimen grip which allows pull angle of 90° at all conditions. The traction program monitors the force as a function of strain. The peel strength test set up is shown in Figure 16.

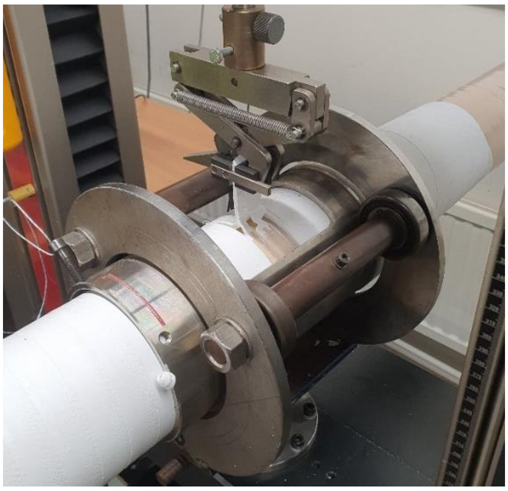

Figure 16. *SPC peel strength test set up*

Considering the application, peel strength is perceived as a critical quality characteristic. The experimental part evaluates the impact of PIR content on the 3-layer structure. During coating, the adhesive layer chemically bonds with the epoxy, and melt-fuses with the topcoat plastic [131]. For room temperature experiments peel strength results are characterized as *no peeling* (desired) or *peeling* to describe the ability of the 3-layer coating to remain intact. A coating resulting as no peeling elongates as an intact system without separation from the pipe surface. Figure 17 visualizes the difference between the results. Peel strength measurement is repeated at elevated temperature to study the strength of adhesive bond between the plastic and the epoxy, since poor adhesion is the weakest link of a 3-layer PP coating. Moreover, a temperature increase causes steel to experience thermal expansion, creating elongation of polymer coating layers. Thus, to ensure sufficient performance, the coating and formed adhesion must withstand elongation and shrinkage.

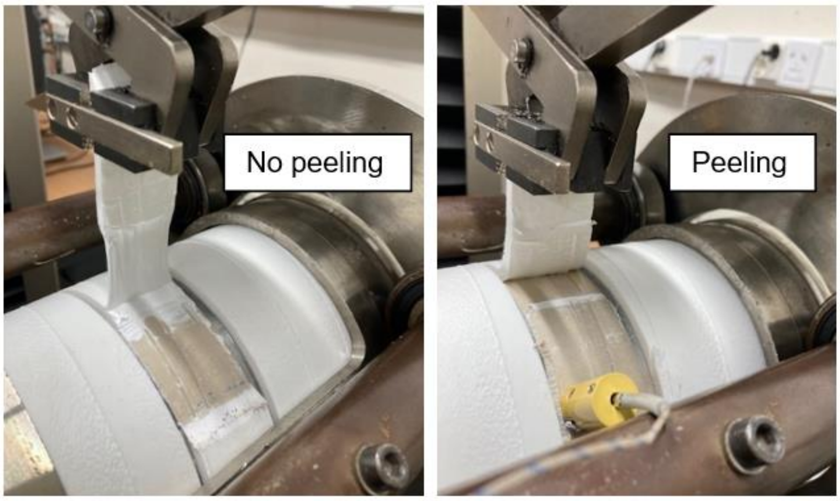

Figure 17. *Characterization of the peel strength result, no peeling 3-layer coating elongates as an intact system*

Moreover, melt strength of polyolefin in extrusion SPC can be evaluated visually. The purpose is to examine the behavior of the film as a change in width, thus the film's ability to remain intact during coating process. This qualification has a focus on polymer processability before the end-use of the product. Melt behavior and strength is qualitatively assessed by coating the steel pipe with polymer at specific extruder and line speed. Moreover, the strength is evaluated for increased line speeds as well. A uniform thickness of polymer layer over the primer is desired, and the coating should have a minimum overlap with itself. Especially at high production rates, the plastic elongates during coating procedure, making it more likely to tear than at low production rates. As specified in ISO 21809-1:2018, primer and topcoat should not be in direct contact, meaning that the adhesive plastic coating should be uniform to ensure adhesion between the layers [129]. Therefore, it is important to evaluate the melt behavior and the suitability of the polymer for certain operating parameters of further processing.

4.3.2 Tensile properties

Tensile property determination can be conducted in accordance with ISO 527-1:2019 and ISO 527-2:2012 [132,133]. Tensile properties are determined by applying a controlled force to elongate a standard test rod longitudinally at a constant rate, simultaneously measuring the resistance of the material to this elongation. The fundamental principle is to assess how material responds to applied forces. The results are presented as a stress-strain curve, from which the following parameters are determined: *tensile stress at break* (the maximum stress value at which the break of specimen occurs), *tensile strain at break* (the maximum strain when tensile stress at yield is reached), and *tensile stress at yield* (the maximum stress material can withstand without irreversible deformation) [134]. The properties reflect the mechanical behavior of the material and the limits of use in applications. [18]

When comparing compounds with varying amount of PIR content, the tensile property determination becomes particularly valuable. By subjecting these materials to identical testing conditions, a direct comparison of their mechanical performance can be made. Tensile testing allows analyzation on how the presence of PIR content influences tensile properties, aiding in optimization efforts.

4.3.3 Charpy impact strength

PP exhibits decreased flexibility and increased susceptibility to fracture at sub-zero temperatures [14]. The ability of PP to withstand impacts is an essential characteristic in pipeline transportation applications. As the structural composition of PIR content may vary, the impact of its incorporation needs to be assessed. To evaluate material's strength properties at sub-zero temperatures, Charpy impact strength measurement can be carried out according to ISO 179-1:2023 [135].

Charpy impact strength refers to the impact energy absorbed by a test specimen at the time of fracture, and it is determined per cross-sectional area of the test specimen. During the measurement a test specimen is supported from both ends by the testing apparatus and subjected to a single pendulum hammer impact midway between the supports. The pendulum hammer is released from known height, and the energy transferred to the specimen is measured. [135] The specimen may be either notched or unnotched, and this study utilizes notched samples. The calculation of impact strength is determined according to the specimen type. The primary result obtained from Charpy impact testing is a single value of the absorbed impact energy, usually reported in joules (J). This energy indicates the toughness or brittleness of the material, depending on the observed value, and it correlated with the material's ability to withstand impact loads. [18]

4.3.4 Shore hardness

Shore hardness determines the hardness of a material by utilizing spring-loaded pin. The principle of the method is based on the penetration depth of the pin into the sample at a constant force, which is determined as Shore hardness. [136] The test method is done according to ISO 868:2003 standard. For PP durometer type D is used which is intended for harder materials. The obtained Shore-D result is a single value determined as an average of 5 parallel determinations. [137] Variations in hardness typically reflect changes in other mechanical properties. Shore hardness results can be used to compare does incorporation of PIR content effect on strength properties of the product at room temperature.

4.3.5 Flexural Modulus

Flexural modulus describes material's resistance to bending which reflects the stiffness of the material. The bending behavior of a test specimen is measured under specific conditions according to ISO 178:2009 [138]. During the flexural test, the specimen is supported freely at both ends and the load is applied to the center of the specimen. This method is called three-point loading, as the load increases during experiment, the sample bends at the midpoint, and the resulting displacement is measured. Graphical flexural test results are obtained as a force-flexure curve from which the flexural modulus, breaking stress, and elongation at break can be calculated. If the specimen does not break during the test, its flexural strength is described by yield point. Flexural modulus is measured because non-ideal materials may exhibit different behaviors under bending compared to compression and tension (tensile properties). [53] The results can be used to compare how the PP batches with different composition respond to bending.

4.4 Thermal properties

Thermal characterization methods are essential for assessing material properties, including physical or mechanical aspects, as a function of temperature or time at a constant temperature. Differential scanning calorimetry (DSC) is a widely used technique in polymer research due to its ease of use and ability to provide results rapidly. With DSC analyzation of physical properties of polyolefins can be conducted. [116] In addition, testing of thermal properties such as the Vicat softening temperature (VST) allows an assessment of how the combined effect of mechanical load and temperature affects the sample. Also, the dimensional changes in a polymer when exposed to heat can be studied. VST is beneficial for determining material characteristics relevant to specific conditions and applications. [18]

4.4.1 Differential scanning calorimetry

Differential scanning calorimetry (DSC) is a thermal analysis used to determine the thermal properties of a sample. The primary focus is on determining the melting and crystallization properties of examined samples, including crystallization temperature, degree of crystallinity, and melting temperature. DSC measurement can be done according to ISO 11357-3 standard [139]. The impact of PIR content incorporation on these properties is analyzed.

DSC analysis utilizes two samples: the specimen under examination and an inert reference material. Both samples are exposed to the same temperature condition, with the temperature difference between them kept near-zero. The temperature condition utilized is heating at a controlled rate. Most importantly, the thermal energy required to maintain this near-zero temperature difference is measured. This measurement provides insights into the thermal properties of the specimen, by determining the enthalpy changes associated with these thermal events. [139] A DSC scan is obtained as a result from the measurement. A schematic graph is presented in Figure 18, which illustrates how thermal changes are observed in the DSC scan of polymers. [18]

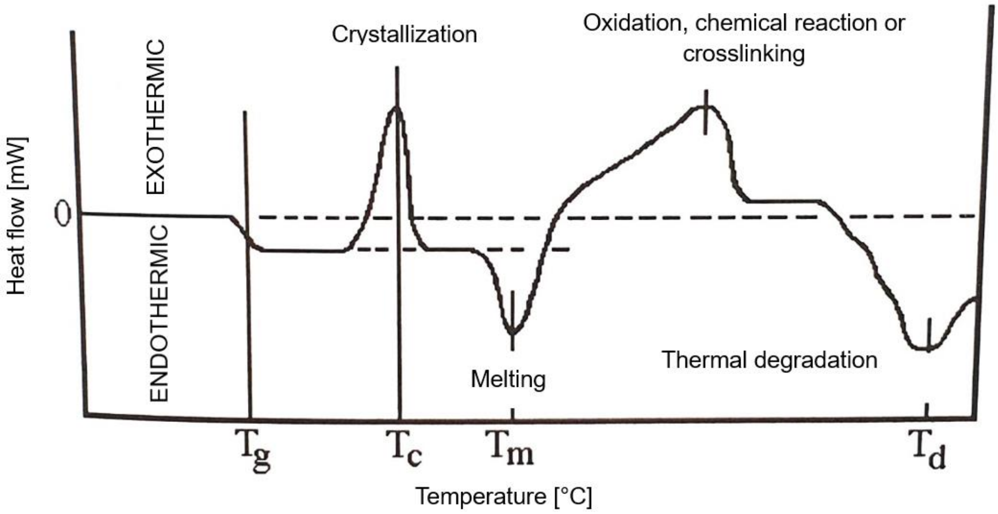

Figure 18. *Possible phenomena for polymers detected by DSC measurement, adapted from [18]*

The DSC scan provides a plot of heat flow (energy transferred per second) as a function of temperature, from which the transition enthalpies can be determined by analyzing the areas under the peaks. In more detail, thermal deviations are detected in the DSC scan as either exothermic (heat-releasing such as crystallization) or endothermic (heat-absorbing, such as melting) changes. [140]

4.4.2 Vicat softening temperature

VST measurement determines how elevated temperature affects the softening and strength properties of a polymer. Unlike DSC measurements, the values obtained from VST do not determine transformation temperatures such as T_g or T_m but depend on specific conditions of the measurement method. Therefore, VST does not fundamentally define material properties but serves as a physical test to verify the suitability of a material for a particular application. [18]

VST can be determined for thermoplastics according to ISO 306:2022 standard [141]. The VST of a semicrystalline polymer is typically located close to T_m [142]. The measured value is utilized as a guideline for processability, since above the VST the polymer structure experiences irreversible deformation observed as rapid softening [143]. Vicat measurements are used to study whether PIR content impacts the polymer structure and creates premature heat sensitivity, thus lowering the VST.

4.5 Chemical properties

The chemical properties of polymers have a major impact on material performance. The addition of PIR content to prime products may have unexpected impacts in terms of characteristics such as performance in the end-use application and further processing steps. Therefore, the following chemical properties are examined: additive content, and reactive site content (RSC). Both additive content characterization methods utilized in the experimental part are internally developed by Borealis. The concentrations of two specific additives are measured from one of the PP compounds in the experimental part. Particularly, the content of AO1 and AO2 are measured with high-performance liquid chromatography (HPLC) and X-ray fluorescence spectrometer (XRF), respectively. Furthermore, the determination of RSC from a polymer is an internally developed method by Borealis and is therefore not based on a specific standard. The method and measurement set-up will not be disclosed.

5. EXPERIMENTAL PROCEDURE

The experimental part is conducted for two distinct PP products Compound A and Compound B. The experimental procedure includes the following steps in order: preparation of the recipes, planning of the schedule and sequence of the pilot-scale trials, preparation of the batches with WP-30, and testing of the samples. The study contains three different variables, from which the most significant being the PIR content:

1. The impact of non-prime side-feed (PIR content)
2. The impact of prime side-feed
3. The impact of additive particle size

As defined in the research question of the thesis, the first variable aims to investigate how incorporation of PIR content affects the product characteristics and could the product performance be yielded despite PIR content. During the experimental part a specific proportion PIR content is compounded with prime feedstock. Beginning from a smaller amount of side-feed and increasing the amount for each test run. For both compounds a reference sample with no PIR content is prepared, to allow comparison on properties. The reference reflects the prime grade. Moreover, the experimental part aims to investigate whether there is a threshold at which an increase in the PIR content causes the product's properties to no longer fall within the control limits.

Secondly, the impact of prime side-feed to the product characteristics is evaluated. The prime side-feed is used to assess the nature of the compounding, and the results of this batch are compared to both the reference and the batches with PIR content. By comparing compounds with additional prime content and PIR content, it can be evaluated to which extent the attributes of recycled content correspond with the initial prime product. The third variable concerns Compound A and is used to compare whether the particle size of the additives affects the final product. Particle size in this context refers to additives in powder and granulate forms.

The experimental procedure is illustrated in Figure 19. The process begins with recipe design, followed by pilot-scale trial planning including scheduling and sequencing of trials. Batches are manufactured with WP-30 for further product characterization. The characterization methods for quality control are specified for both compounds based on the application and end use of the grade. Moreover, the sequencing and relationships between the test methods can also be observed.

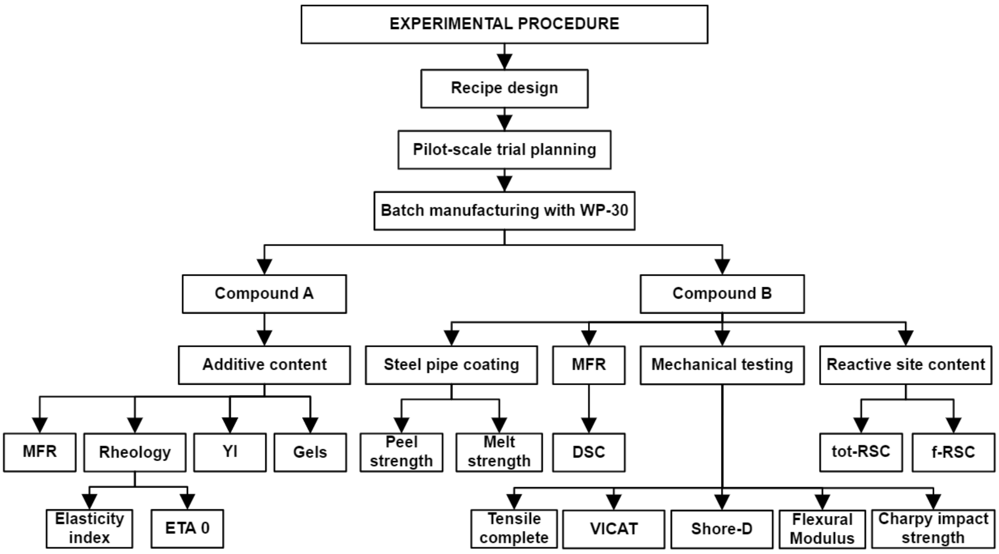

Figure 19. *Test methods and sequence of the experimental part*

For Compound A, additive content is measured prior to any other test method, which affects the further manufacturing procedure. MFR value is measured for both products. For Compound B, MFR testing should be performed prior to DSC, as the MFR strip is used as a test piece in DSC. This is a common procedure, as MFR string is considered as a homogenous product representation. Rheology measurement is used to assess the elasticity index and zero shear viscosity (ETA0). RSC content is measured to determine tot-RSC and f-RSC. For compound B, steel pipe coating is applied as part of sample preparation prior to peel strength measurements. Similarly, injection molding is used to prepare samples for mechanical testing.

5.1 Recipe design

Post-industrial material utilization is examined for two PP grades Compound A and Compound B. The material collected from the manufacturing process of prime resins is classified as PIR emphasizing that the waste is repurposed during the experiments. Thus, each PIR content corresponds to the prime product. A test plan was prepared for both grades, defining the recipes for each compounded batch. In the product recipes, the amount of ingredients was determined as weight percent (wt.%) based on the initial recipe of the grade. The percentage of PIR content in the total recipe was set as a variable, thus its amount was varied for each compound. The side-feed trials are started chronologically from smaller weight percentages (3 wt.%) to larger ones (10 wt.%). By working in this manner, it is possible to examine how the PIR content affects the product's characteristics compared to prime grade. And on the other hand, from an economic point of view, a higher PIR content within the allowed characterization limits would be desirable.

5.1.1 Product 1: Compound A

The first examined product is PP copolymer called Compound A. The product is utilized in packaging applications due to its excellent heat-sealing performance, high melt strength, and good resistance to elevated temperatures. This product consists of three ingredients A1, A2, and A3. A1 is one of the two PP homopolymer base resins of the product, and it is structurally isomeric modified. The other base resin of the product is A2, a reactor elastomer modified PP. Antioxidant utilized in Compound A is A3, which is a combination of AO1 and AO2. AOs are added to test batches to decrease the impact reprocessing may have on the MFR value.

Since the pilot-scale extruder does not fully reflect the production scale apparatus on addition of additives, the impact of additive particle size will be tested with two test runs. As presented in Table 8, the recipes remain same for both batches, but impact of the additive particle size is tested with granulate (G) and powder (PW) forms.

Table 8. *Test run reference recipes for product 1*

Formulation	SF_TR0_G [wt.%]	SF_TR0_PW [wt.%]
A1	X1	X1
A2	Y1	Y1
A3	Z1	Z1

Moreover, the proportion of the additive (A3) is low in the product, which poses the challenge of uniform mixing during pilot-scale compounding. In more detail, since additives are added from the hopper there is higher risk of some material remaining in the hopper causing varying concentration of AOs. The aim is to achieve homogenous additive distribution to match the prepared recipes, therefore the impact of additive particle size to the additive content is analyzed from the reference batches. The analysis determines which one of the additive forms, granulate or powder, will be used in the following batches. The rest of the test run recipes containing side-feed of prime Compound A and PIR content are presented in Table 9.

Table 9. *Test run recipes for product 1 containing side-feed materials*

Formulation	SF_TR_P [wt.%]	SF_TR1 [wt.%]	SF_TR2 [wt.%]	SF_TR3 [wt.%]	SF_TR4 [wt.%]
A1	X2	X3	X4	X5	X2
A2	Y2	Y3	Y4	Y5	Y2
A3	Z1	Z1	Z1	Z1	Z1
Prime Compound A	10.0	0.0	0.0	0.0	0.0
PIR Compound A	0.0	3.0	5.0	7.0	10.0

The weight percent of A1 and A2 decreases as the quantity of the PIR material increases. Therefore, the amount of A1 and A2 is the same for SF_TR_P and batch containing 10 wt.% of PIR content (SF_TR4). The recipes take into account that the exact composition of PIR content is not known. But for the recipe calculation an assumption is made, that the composition and relation of the base resins in the PIR content is near prime material. Moreover, the impact of prime side-feed is tested with the highest side-feed weight-percent. To conclude, there will be 7 distinct batches of Product 1 produced.

5.1.2 Product 2: Compound B

The second examined product is a PP grade called Compound B. This product is a RSC grafted PP which is utilized in applications such as steel pipe coating. In total, the product consists of two distinct PP base resins B1 and B2, and additives. The initial recipe utilized to produce prime Compound B contains the following two additive mixtures: B3 and B4. However, additives will not be added in the pilot-scale trials. With the pilot-scale apparatus arrangement applying additives could affect negatively on the polymer's performance in application. The test run reference recipe for product 2 compared to the initial product recipe is presented in Table 10.

Table 10. *Test run reference recipe for product 2, compared to initial product recipe*

Formulation	Initial recipe [wt.%]	BB_TR0 [wt.%]
B1	M1	M1
B2	N1	N1
Additives	O1	0.0

The weight percent of B1 and B2 in the total mass of the product decreases as the quantity of the PIR content increases. The reduction from the base resins is done so that the initial ratio between them remains constant. To summarize, 6 distinct batches of Product 2 will be produced. The recipes for Compound B test runs are presented in Table 11.

Table 11. *Test run recipes for product 2 containing side-feed materials*

Formulation	BB_TR_P [wt.%]	BB_TR1 [wt.%]	BB_TR2 [wt.%]	BB_TR3 [wt.%]	BB_TR4 [wt.%]
B1	M2	M3	M4	M5	M2
B2	N2	N3	N4	N5	N2
Prime Compound B	10.0	0.0	0.0	0.0	0.0
PIR Compound B	0.0	3.0	5.0	7.0	10.0

From Table 11, the first ingredient is a heterophasic high level impact copolymer PP called B1. The MFR value of this grade is low. The second ingredient B2 provides the functional part of the product. Compounding of this ingredient is a functional process, and during the production multiple extrusion steps are conducted. During the experiment, the impact of this material history is considered since it may increase the risk of discoloration and have negative impact on mechanical properties. B2 has a yellowish tone, and considerably higher MFR value than B1. The impact of significantly different MFR values between the resins is considered and evaluated during result analysis.

5.2 Sample preparation

This chapter focuses on the experimental part of the thesis including pilot-scale pellet production via compounding, and sample preparation with steel pipe coating and injection molding. All experimental procedures were carried out using the same processing parameters as would be utilized for prime product without PIR content. Primarily, to investigate the compatibility of PIR content with the product, and to ensure customer' qualifications and processability. To clarify, operating parameters are not considered as variables. More specifically, each batch was tested using the same methods and operational parameters to ensure comparability. Therefore, the methods describe the testing of a single sample.

5.2.1 Manufacturing batches with WP-30

Pilot-extrusion schedule and test sequence was determined to maximize production flow and optimize the number of purging the equipment to avoid cross-contamination. For both PP grades, the references were produced first. Second, the batch containing prime side-feed was compounded. Lastly the batches containing PIR content were produced, from lowest to highest PIR content.

The quantity of products to be compounded with the pilot-scale extruder was determined by calculating the amount of product required for each quality control characterization method. For Compound A the batch size was 13 kg, and for Compound B the batch size was 25 kg. To ensure sufficient product availability for testing, the quantity of produced product exceeded the minimum required quantity. Thus, quality control analysis could be repeated on the same production batch if necessary. Moreover, the WP-30 operation ability needed to be considered when determining the production volumes. An excess amount of product was prepared for each batch, so that the collected material for quality analysis did not include the first or the last produced pellets. The extra amount varied between the batches, but approximately 4-6 kg of additional product per batch was utilized for operational reasons. By operating in this manner, it was ensured that the collected material only contained the prepared product. Additionally, buffer material at the end of the production ensured that the last pellets collected were produced with a constant feed-rate.

The study was done in Borealis Polymers Porvoo laboratory facilities. Compounding of test batches with WP-30 were carried out in TS-hall. Each batch was produced in a comparable manner including the following processing steps: stabilization of the extruder to operating values, preparing production by purging with base resin, batch production, ending the production by purging with base resin, and shutdown. Test reports including the average values of the main operating parameters are presented in Appendix B. The pilot-scale extruder is shown in Figure 20.

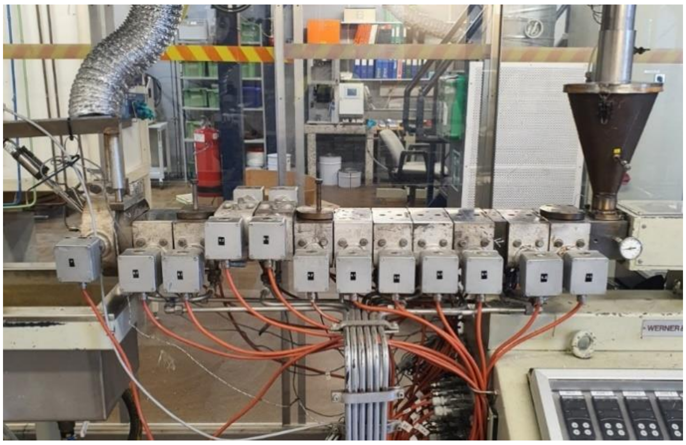

Figure 20. *Werner & Fleiderer 30 - extruder used for batch manufacturing*

Pilot-extruder experiments were started by preheating each of the 7 zones of the apparatus according to the values given in Appendix B. Once the heating was completed the barrel purging was performed with base resin. During purging about 5 kg of base resin was passed through the extruder, to ensure contamination removal and enable the extruder to achieve the desired operating parameters. The feed rate of the base resin was started with 2 kg/h and was gradually increased to 8 kg/h. Simultaneously, the screw rotation speed was slowly increased to 200 rpm. The melt exiting the die was visually inspected for particles and contaminants. This purging material was cut and removed from the water bath.

While the WP-30 preparations were running, the ingredients were weighted according to the recipes. The batches were prepared on the same day as they were operated. The weighted ingredients were combined in a plastic bag and placed in a barrel mixer for homogenization with a mixing time of 20 minutes. Before feeding the test material into the hopper, the excess base resin was vacuumed to avoid cross-contamination.

During the experiments pressure, torque, and screw speed were metered. Production capacity of 8 kg/h was utilized during the experiments. The pellet collection ended when the right amount had been collected, after this the pellets were collected to a separate container. During the shut-down phase operating parameters decreased gradually, including the feed rate. Finally, the apparatus was cooled rabidly with water circulation. Produced batches were weighted and labelled.

5.2.2 Steel pipe coating

Steel pipe coating was conducted as a 3-layer PP coating process in SPC pilot-line. SPC was carried out for batches of Compound B. All three layers were applied continuously as the pipe rotated throughout the process. Steel pipes utilized during the experiment were pretreated with shot-blasting off-site, to create a rough surface to allow epoxy to adhere, and to ensure the surface was contaminant-free. Epoxy was applied as a primer layer followed by adhesive layer and PP topcoat. A schematic illustration of the SPC process is presented in Figure 21, representing the main processing steps.

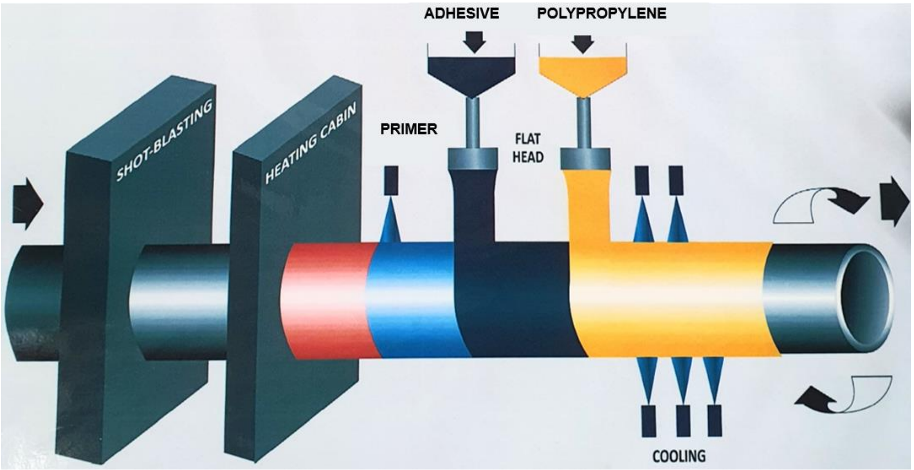

Figure 21. *General schematic illustration of 3-layer PP SPC process*

Twin-screw extruders Reiffenhauser and Extron were used for the coating application of topcoat and adhesive layer, respectively. Prior to coating both extruders were set to desired operating parameters. Reaching the temperature profile took 60 minutes in total, after which the barrel purging was conducted with the material to be coated. Simultaneously, an epoxy applier was placed at the correct distance to achieve the desired ICT. Other operational parameters such as extruder output rates were set to achieve the correct coating thickness. Cooling tank valves were adjusted correspondingly to the peripheral speed. Before the coating procedure, the temperature of the adhesive and topcoat were measured with IR thermometer. The film width and overlap were also measured. SPC pilot-line specimen dimensions and main operating parameter utilized during the experiments are presented in Table 12.

Table 12. *SPC pilot-line specimen dimensions and operating parameters*

Parameter	Value
Pipe diameter [mm]	114
Pipe length [mm]	930
Pitch set	3.3
ICT [s]	13
Die/pipe distance [cm]	38
Peripheral speed [m/min]	12

The coating process started by heating the steep pipe with an inductive heater. The steel temperature was tracked during the coating process since it highly affects the degree of crosslinking of epoxy. After heating, powder form epoxy was added pneumatically. The heated steel pipe enables epoxy to melt and form adhesion with the steel surface. Steel temperature was measured immediately after epoxy addition. Then adhesive was applied around the sample pipe using a flat head die extruder. Subsequently the top coating layer was applied similarly to the adhesive. After coating the specimen was cooled by a water-quenching process. After cooling the sample was allowed to equilibrate to room temperature. Resulted coating thicknesses are presented in Table 13.

Table 13. *SPC layer thicknesses*

Layer thickness	BB_TR 0	BB_TR_ P	BB_TR 1	BB_TR 2	BB_TR 3	BB_TR 4
Epoxy [µm]	170	170	200	200	200	200
Adhesive [µm]	450	450	400	400	400	400
Topcoat [mm]	4.5	4.5	4.5	4.5	4.5	4.5

The topcoat has the greatest thickness to ensure sufficient durability against environmental exposure. A slight variation between layer thicknesses can be observed from Table 13. Specifically, the epoxy and adhesive thickness of the reference and the prime side-feed batches differ from the rest. The difference is caused due to operational reasons related to parameter variation between batches. However, such small variations should not have a significant effect on the characterization results. The melt strength of adhesive layer was evaluated after the SPC process. With increased peripheral speeds, no significant neck-in or change in width was observed, thus the material remained intact.

5.2.3 Injection molding for mechanical testing

ISO 527-1:2019 specifies the injection molding principles of thermoplastics, and the experimental part was conducted according to the standard [133]. Injection molding was carried out for batches of Compound B. Two types of test specimens with distinct dimensions were produced via injection molding called type 1A and type 1B. The test specimen dimensions and requirements are specified in ISO 527-2:2012 [132]. Injection molding operating conditions for PP are determined in ISO 19069-2:2016 [144], presenting some of the main parameters in Table 14.

Table 14. *Injection molding operating conditions [144]*

Engel Operating conditions	Value
Mold temperature [°C]	40
Melt velocity in the mold cavity [mm/s]	200 ± 20
Holding pressure time [s]	40
Cycle time [s]	60
Cooling time [s]	14
Injection time 1A / 1B [s]	1.73 / 1.68

Multipurpose 1A test specimens were utilized for the following tests: tensile properties, flexural modulus, VST, and shore hardness. The Charpy impact strength test was made of 1B sample. The injection molding was performed with ENGEL VC 200/60. Prior to injection molding, the equipment was purged with the material to be tested to ensure the equipment was clean and contained no residues from the previous product.

5.3 Test methods

This chapter focuses on quality control of the prepared samples. Each test specimen from a distinct batch was tested using the same methods and parameters to ensure comparability. Thus, the methods describe the testing of a single sample.

5.3.1 Melt mass-flow rate

MFR value was determined for all examined batches from pellet form samples. The testing was carried out in accordance with ISO 1133-1:2022 standard utilizing method B [119]. This method utilizes instrument software to calculate the MFR value based on PP melt density at 230°C specified in ISO 19069-2:2016 [144]. The testing apparatus COCO QAC015 was heated to 230°C and the correct program was selected. The load utilized during the experiment was 2.16 kg. The instrument software determined the sample size based on assumed range of MFR values. The assumed range of MFR values was selected based on the specifications of the prime product. The sample was weighed and placed in the apparatus once the temperature had stabilized. Measurement was performed.

5.3.2 Dynamic rheology measurements

Determination of dynamic mechanical properties was done according to ISO 6721-10:2015 standard [120]. The testing was performed using an oscillatory rheometer Anton Paar MCR301 with cone-plate geometry. Measurements were performed under strain-controlled mode, and the main operating conditions are given in Table 15.

Table 15. *Anton Paar MCR301 rheometer operating conditions*

Operating conditions	Value
Diameter [mm]	25
Gap [mm]	0.1
Strain [%]	10
Temperature [°C]	200
Frequency sweep range [rad/s]	0.01-500

For sample preparation the pellets were compression molded with Fortune apparatus. The linear viscoelastic region was determined for the sample with a strain sweep before starting the measurement. The actual frequency sweep measurement was done at linear viscoelastic plateau from highest to lowest frequency values. Nitrogen atmosphere was utilized during the measurements to prevent structural degradation of the polymer at elevated temperatures. Calculation of the results was conducted by the rheometer software Rheoplus utilizing the Carreau-Yasuda model. The resultant torque and the phase shift between the displacement and torque were measured

5.3.3 Gel formation

Inspection of gels was performed according to ASTM D7310 [126]. As part of the preparations, the extruder equipment OCS ME25 was set to operating conditions and purged with the tested product. Product changeover was tracked from operational trend-curve to ensure no anomalies occurred. Moreover, prior to measurement the extruded film thickness was measured to ensure it fulfilled the test specifications. Operating conditions were set according to the standard, and the examined specimen was extruded to a film with a constant thickness and speed. Camera device OCS CR9/MFA was utilized to distinguish the findings from the polymer matrix background. Results were obtained as a finding per square meter by the measuring program OCS FSA100.

5.3.4 Yellowness Index

Yellowness index measurement was done according to ASTM E313 standard [127]. Utilized apparatus was Datacolor 650/800/850 spectrophotometer. The operating parameters were set according to the standard and measurement was performed. The YI result was calculated by the spectrophotometer apparatus as an average of five parallel measurements, describing the color change of the test sample from white to yellow.

5.3.5 Peel strength

Peel strength was measured from the coated steel pipe sample. For peel strength measurement, a 3 cm wide strip was detached from the steel pipe surface. The peel strength measurement was conducted with Zwick/Roell tensile testing apparatus. The specimen was attached to the apparatus and the strip was placed between the grips. An elongation rate of 10 ± 1 mm/min was used. The instrument recorded measuring data after reaching certain preload values. Preload values were product and temperature specific. For each measurement, the sample was elongated for 180 s. The test was repeated at elevated temperatures of 80 °C, 110 °C, and 140 °C. The coated pipe specimen was heated from the inside with a heating coil, and the temperature was monitored throughout the test. At elevated temperatures, the force required to peel off the coating decreases as a function of temperature.

5.3.6 Tensile properties

Determination of tensile properties was done with Zwick/Roell tensile testing apparatus using the 1A specimen. Prior to testing, injection molded samples were stabilized for 168 h under controlled conditions (T=23°C ± 2 °C, rH=50%± 10%). The test was carried out under the same conditions. The width and thickness of the test specimens were measured prior testing. The test specimen was placed between the grips along with the extensometers. Test speed of 50 mm/min was used to determine the tensile properties. Other operating parameters were set according to ISO 527-2:2019 [132], and the measurement was performed.

5.3.7 Charpy impact strength

Charpy notched impact strength was performed at -20 °C to 1B-type specimen according to ISO 179-1:2023 [135]. Prior testing, A-type notch was prepared according to ISO 2818:2018 [145] to ensure full fracture and comparable results. The sample was stabilized as mentioned in the tensile properties paragraph. The sample dimensions were measured before exposure to testing conditions. The edgewise impact test was performed with Zwick/Roell HIT5P Charpy -apparatus, using pendulum hammer with energy capacity of 1.00 J.

5.3.8 Shore hardness

Injection molded 1A sample was utilized to measure the shore hardness according to ISO 868:2003 [137]. Sample was stabilized as mentioned in the tensile properties paragraph. The shore hardness measurement was performed with a 5 kg weight (method D). The dial gauge result was read after 1 ± 0,1 s according to ASTM D2240 standard. The Shore-D (1s) result was calculated as an average of the five determinations.

5.3.9 Flexural modulus

The flexural modulus was measured using Zwick apparatus, according to ISO 178:2019 [138]. Prior to testing, 1A test specimen was stabilized as mentioned in the tensile properties paragraph. The dimensions of the test specimen were measured, and the values were utilized to set the correct distance between the test apparatus support. The measurement was performed with a testing speed of 2 mm/min. Force and corresponding bending of the test specimen were recorded.

5.3.10 Differential scanning calorimetry

DSC measurement was done with heat flux Mettler DSC -apparatus according to ISO 11357-3:2018 [139]. The test sample was cut from the beginning of the MFR string for a better sample homogeneity and preparation purposes. The measurement was performed over a range of 30 °C to 225 °C at 10 °C/min, with inert nitrogen atmosphere to avoid oxidation. The thermal history of the sample was removed within the first heating round. The data collected during the cooling run and the second melting run were used for property evaluation.

5.3.11 Vicat softening temperature

VST measurement was done with Ceast HDT VICAT -apparatus. Prior to testing, 1A-type test specimen was stabilized as mentioned in the tensile properties paragraph. Testing was performed according to ISO 306:2022 [141], with a load of 10 N (method A50) and heating rate of 50 °C/h. The VST was determined by the computer program of the test apparatus as an average of two parallel measurements.

5.3.12 Additive content

Additive content determination was made for Compound A sample. AO1 content was determined from the filtered solution with HPLC Agilent Technologies Series 1100- apparatus. A standard solution with a known AO1 concentration was run in parallel with the examined sample. AO2 content was determined using XRF apparatus called PANalytial

Zetium SuperQ. For both analysis results were obtained and calculated by the apparatus software.

The purpose of additive content measurements at the early stage of experimental part was ensure the quality of the reference batches. Meaning that the additive content was measured to evaluate the impact of particle size to the additive concentration. The results of additive content determinations are given in Table 16.

Table 16. *Additive content results for batches with varying particle sizes: Granulate vs. Powder form*

Sample	SF_TR_G	SF_TR_PW
AO1 content [ppm]	1320	1390
AO2 content [ppm]	1359	1464

The batch containing additives in powder-form generated a higher additive content for both AO1 and AO2. Thus, the experiments for remaining batches were continued by applying additives in powder form. Differences between the additive contents may arise from operational reasons, since additives may have remained in the processing apparatus such as hopper. Moreover, non-uniform mixing may have caused deviation between the batches. Furthermore, additives including AO1 and AO2 are added to the base resins already during polymerization. Therefore, changes in the quality of the base resins may be reflected in further processing steps such as compounding.

6. RESULTS AND DISCUSSION

In this chapter, the results of pilot-scale experiments are presented. The evaluation of batch quality is based on comparing the results to the reference batch indicating prime product performance. Product specification limits for characteristics will not be disclosed. From this perspective, the main research question is assessed whether PIR materials generated from polyolefin pellet production could be utilized as part of the corresponding primary product, ensuring that the required properties are maintained. Based on the evaluation of product quality characteristics, possibilities for upscaling to industrial scale are assessed.

The incorporation of PIR content with prime feedstock in proportions up to 10 wt.% was examined. Prior to discussing the results, it is important to note that, according to existing literature, the inclusion of PIR up to 10 wt.% is not expected to significantly impact material properties compared to initial prime product [6,62]. However, the following analysis focuses on evaluating any deviations in properties compared to the reference, as these deviations may impact further conversion processes and end-use application. Potential factors influencing the deviations include, the unknown composition of PIR (relation of base resins), the varying amount of PIR, properties of prime base resins, effect of reprocessing, and compatibility of PIR with other ingredients. Notably, the compounded product reflects the feedstock properties, as supported by several studies on PP recyclate compounding with base resins [146,147].

6.1 Product 1: Compound A

Product 1 is utilized in packaging applications. Characterization results are compared to the reference batch containing only prime resins and additives in powder form (SF_TR_PW). MFR value provides information about the polymer processability, and it is a common quality control test among resin producers [36]. MFR value is inversely related to molecular weight and viscosity. Obtained results are presented in Table 17.

Table 17. *MFR results for Product 1*

Sample	SF_TR_PW	SF_TR1	SF_TR2	SF_TR3	SF_TR4	SF_TR_P
MFR [g/10min]	13.31	13.08	13.05	13.14	13.27	13.18

MFR results are consistent with one another and reflect the target value of the product well. Surprisingly, the batch containing the highest amount of PIR (SF_TR_4) resulted with the most equivalent MFR value compared to the reference. For other test run batches the MFR value showed a slightly more pronounced decrease, indicating higher viscosity and thus lower flowability. However, the differences are small, as the overall standard deviation across the six batches is 0.1, with an average value of 13.17 g/10min. Structural degradation is not observed from the results since it would have caused a significant increase in MFR value. All results obtained were found to be well within the tolerance limits. Thus, incorporating PIR content to Compound A does not seem to change the processability of the product, which is a particularly promising finding.

6.1.1 Critical performance indicator – Gel formation

Gel inspection is a critical analyzation method for Compound A since gels are the most common defect in film products [46]. Characterization of gel formation was carried out through film extrusion to simulate the converting operations utilized by customers for this grade. Formation of gels in Compound A may result from factors such as non-compatibility between the ingredients or poor homogenization during compounding process. Gels are known to make polymer film during extrusion less durable and more prone to tearing. Further, grades containing a considerable number of gels decrease the functionality of the material to provide barrier properties. Therefore, a reduced quantity of total gels, and consequently a lower gel index, signifies enhanced product performance. Gel inspection results are presented in Table 18 including total gel findings, and calculated gel index value.

Table 18. *Gel inspection results including total amount, and calculated gel index*

Sample	SF_TR_PW	SF_TR1	SF_TR2	SF_TR3	SF_TR4	SF_TR_P
Total [pcs/m^2]	6.4	7.4	4.9	5.7	5.4	7.1
Gel Index [-]	0.8	1.9	0.8	0.8	0.8	1.2

According to the data presented in Table 18, all batches correlate with the reference batch, with a standard deviation of 0.4. The highest gel index can be observed for batches SF_TR1 and SF_TR_P representing a greater total number of gels compared to the other batches. Remaining batches exhibit homogenous behavior compared to the reference with a consistent gel index of 0.8. Based on the results, no correlation between PIR content and the number of gels is observed.

Given the sensitivity of gel formation, it represents a pivotal measurement technique for assessing the influence of PIR content on product properties. As indicated in the literature, non-compatibilities arising from significant differences in molecular weight, chemical composition, or shear viscosities can lead to gel formation [36,46]. Thus, moderate gel formation may be taken as an indication of the compatibility of post-industrial material and prime material. This level of gel formation suggests that PIR content have sufficient resemblance with prime feedstock and they can blend effectively. As a result, no significant incompatibility issues are formed at PIR to prime material ratio up to 10 / 90, respectively.

Moreover, operating conditions of compounding such as temperature, shear rate, and residence time can have major impact on gel formation [36,46]. Since gel formation was subtle, it may be concluded that during compounding the operating conditions were proper for the product even though it contained PIR content.

6.1.2 Rheological properties

MFR values provide a general guidance on polymer processability, but are not alone sufficient to assess polymer performance, hence other rheological measurements are required [36]. To provide more comprehensive understanding of the rheological performance of the test runs, dynamic frequency sweep measurements were performed. Dynamic frequency sweep measurements provide information on the rheological and microstructural properties of polymers. Thus, the measurements are used to evaluate whether the feedstock composition including PIR effects on structural properties. In other words, with dynamic rheology results it is possible to qualify how different the recycled polymer composition is compared to prime polymer composition. [122] Obtained results at 200 °C are presented in Figure 22 at logarithmic scale as a function of angular frequency, illustrating the relation of storage modulus (G'), loss modulus (G''), and complex viscosity (η*). Figure 22 includes measured data points from all examined batches.

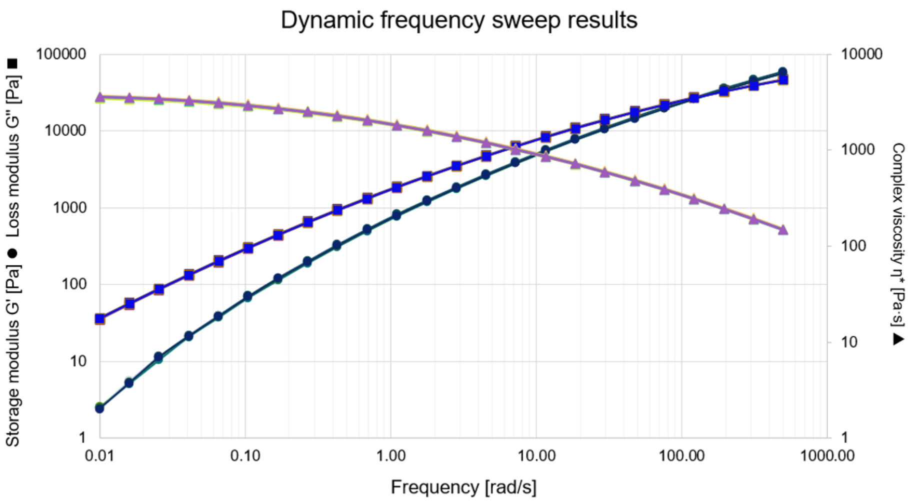

Figure 22. *Dynamic frequency sweep results for Product 1 at 200℃*

Figure 22 illustrates that the rheological performance of the batches correlates extremely well, as single measurement points overlap each other. Thus, no differences in rheological behavior between the batches are observed. Figure 22 illustrates typical rate dependency of G' and G'', as at higher frequency values the magnitude of G' increases at a faster rate, and the crossover point between relative viscous and elastic responses is observed [124]. A slope of G'' curve at high frequency indicates an overall broad MWD [123,148], which is desired. The results align with obtained MRF values, as a decrease in molecular weight or narrower MWD due to thermal degradation is not observed. This result is expected, since the PIR content is low, and the effects of PP reprocessing would only become apparent after further extrusion loops [65]. Moreover, the batches seem to exhibit good thermal stability, since zero shear viscosity plateau is observed at low angular frequency values [123].

In packaging applications, the rigidity of a material must be adequate. Product specific target and tolerance limits for the elasticity index (G') and zero shear viscosity (ETA0) values are determined. Elasticity index is defined as the corresponding storage modulus value, when loss modulus equals to 0.5 kPa. ETA0 values are calculated with Carreau-Yasuda model. The results are presented in Table 19.

Table 19. *Results obtained from dynamic frequency sweep measurements*

Sample	SF_TR_PW	SF_TR1	SF_TR2	SF_TR3	SF_TR4	SF_TR_P
Elasticity Index G' [Pa]	139	143	139	141	141	139
ETA0 [Pa·s]	4330	4420	4250	4450	4470	4560

Obtained elasticity index values presented in Table 19 indicate homogenous behavior among the samples, with highest deviation of 2.9 % (SF_TR1) compared to the reference. ETA0 values indicate slight changes between the batches, but overall the variation is small. These results vary from 4250 Pa·s (SF_TR2) to 4560 Pa·s (SF_TR_P), with deviation of 1.8% and 5.3% compared to the reference, respectively. From batches including PIR content the largest deviation compared to the reference is an increase of 3.2% (SF_TR4). Increase in average molecular weight typically results in an increase zero shear viscosity [123], but in this case the deviations are small. From a quality assurance perspective, slight fluctuations (<5%) are accepted in the measured values, due to the measurement procedure, including manual working steps such as removal of flash.

Inspection of crossover point between storage and loss modulus provides information on molecular weight and MWD change between prime product and product containing PIR content. The frequency of modulus crossover point also correlates with relaxation time of the polymer. [123,148] Frequency and corresponding moduli value at crossover points for each batch are presented in Table 20.

Table 20. *Frequency and moduli of crossover points at 200°*

Sample	Frequency [rad/s]	Moduli [MPa]
SF_TR_PW	140	0.0281
SF_TR1	139	0.0280
SF_TR2	139	0.0281
SF_TR3	138	0.0281
SF_TR4	137	0.0279
SF_TR_P	138	0.0290

The results presented in Table 20, express homogenous behavior between the samples, indicating similar average molecular weight and MWD. A slight increase in the moduli value of SF_TR_P can be noted, which could indicate a narrower MWD [148]. The change is extremely small, therefore degradation is not a probable cause for the result. To summarize, based on the results, there is no evidence to suggest that the addition of PIR content up to 10 wt.% would affect negatively on the rheological properties of Product 1.

6.1.3 Color assessment

Yellowness index measurement is a comparative tool used for color assessment. In general, minimizing yellowness is desirable for natural color polymers. The results obtained are presented in Table 21.

Table 21. *Yellowness index results*

Sample	SF_TR_PW	SF_TR1	SF_TR2	SF_TR3	SF_TR4	SF_TR_P
Yellowness index [-]	-0.03	-0.08	-0.12	-0.59	-0.47	-0.39

Yellowness index results show no indication of yellowish tones, that could indicate polymer degradation [125]. On the contrary, batches with the highest PIR content appear more bluish or greenish (SF_TR_3 & SF_TR4) with a lower YI value compared to the reference. Further, the batch containing 10 wt.% of prime Compound A (SF_TR_P) also has a slightly lower YI value than the reference. Batches with lower PIR content (SF_TR1 & SF_TR2) appeared whiter to neutral YI closer to the reference sample. The overall standard deviation across the batches is 0.2. Uneven distribution of additives during processing may cause deviations in yellowness. Additionally, the temperature of the pellets may influence the results. If the pellets are not fully stabilized at room temperature, the yellowness index tends to increase. To conclude, all obtained results were found to be well within the acceptable limits of yellowness. Surprisingly, the addition of small quantities of post-industrial materials to base resins may even yield a beneficial effect on the YI.

6.2 Product 2: Compound B

Product 2 is utilized in multi-layer PP coatings. Characterization results are compared to the reference batch containing only prime feedstock (BB_TR0). MFR value is a characteristic that can be affected by compounding and especially recipe changes. To evaluate these results correctly, the significant MFR value difference of the base resins is considered. The obtained MFR values of Compound B test runs are presented in Table 22.

Table 22. *MFR results for Product 2*

Sample	BB_TR0	BB_TR1	BB_TR2	BB_TR3	BB_TR4	BB_TR_P
MFR [g/10min]	6.93	6.77	6.80	6.12	6.38	6.36

In general, batches compounded with prime and non-prime content demonstrated a slight decrease in MFR value, with SF_TR3 exhibiting the lowest MFR value. The overall standard deviation across the six measured MFR values is 0.3, with an average value of 6.56 g/10min. The decrease in MFR value is partly expected since the MFR of a compound is approximately the weighted sum of the feedstock characteristics. In the recipe design chapter, it was previously mentioned that ingredient B2 has a higher MFR value than B1. As the base resins are replaced with PIR content which MFR value is probable to represent the properties of the prime product, it is reasonable that the MFR value is reduced. From the perspective of product functionality, however, it is important that the reduction is made from both base resins. Moreover, batches BB_TR4 (10 wt.% PIR) and BB_TR_P (10 wt.% prime Compound B) show corresponding MFR results. This result suggests that PIR material composition and prime grade composition regarding flow properties are similar. No observation on structural degradation due to reprocessing can be observed, since the MFR value is lower compared to the reference.

More importantly, another possible explanation for the results might be the composition of PIR, arising from the production operations before conducting the pilot-scale compounding. Since a large part of post-industrial material is generated from start-up phase during industrial production, the composition and relation of base resins may vary, causing range of MFR values. This could explain why the reduction in MFR value is not linear as a function of PIR content. For example, a possible decrease in B2 content in the PIR content could result in a pronounced shift in MFR towards lower values.

Controlling MFR values is a tricky aspect when incorporating prime materials with PIR content with not coherently known composition. However, MFR values are monitored throughout industrial manufacturing to eliminate the effect of varying compositions, whether it is arising from resin manufacturing or compounding operations.

6.2.1 Critical performance indicator – Peel strength

Peel strength is an essential characteristic of a 3-layer SPC system. The results are presented as the average force per centimeter (N/cm), expressing the quantity of force per unit width required to separate two bonded materials. The peel strength results and overall standard deviation of the samples at different measuring conditions are given in Table 23. A clear increase or decrease in peel strength compared to the reference value is highlighted.

Table 23. *Peel strength results at examined temperatures*

Sample	BB_TR0	BB_TR1	BB_TR2	BB_TR3	BB_TR4	BB_TR_P	SD
Peel strength 23 °C [N/cm]	576	**620**	578	578	610	624	22.7
Peel strength 80 °C [N/cm]	276	265	295	290	**319**	**311**	20.4
Peel strength 110 °C [N/cm]	179	186	181	191	**128**	**220**	29.9
Peel strength 140 °C [N/cm]	82	99	88	**117**	77	63	18.7

Without exceptions, the peel strength results at room temperature were characterized as *no peeling*. The results show no linear correlation between the amount of PIR and peel strength. Further, no specific batch showed consistently lower or higher peel strength at every testing condition. For instance, BB_TR_P exhibits the highest peel strength value at room temperature and at 110°C, but the lowest peel strength value at 140°C compared to other batches.

However, variations in peel strength values of the batches compared to the reference are observed. Notably, peel strength results of SF_TR4 and BB_TR_P exhibit the highest variance compared to the reference. For both batches at temperatures of 23 °C and 80 °C, the average force required to elongate the 3-layer PP coating is considerably higher than the reference, suggesting homogeneous behavior between non-prime and prime side-feed. On the other hand, at 110 °C, the peel strength value of BB_TR4 shows clearly lower values compared to the reference batch and BB_TR_P. At this temperature, BB_TR_P exhibits significantly higher values in peel strength compared to the reference sample. The overall standard deviation across the samples is also the highest at 110 °C.

A graphical representation of peel strength as a function of temperature is provided in Figure 23. In general, as can be seen from the graph, the batches containing PIR content correspond well with the reference. The most notable deviations are observed at 110 °C, where the peel strength of SF_TR4 and SF_TR_P differs from that of the other samples.

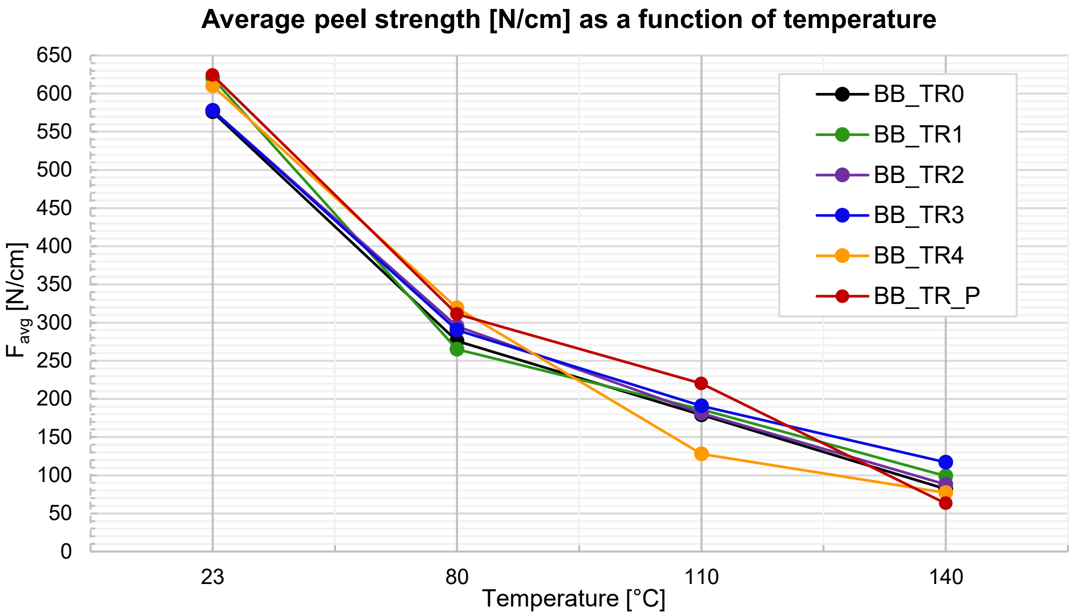

Figure 23. *Average peel strength values represented as a function of temperature for each examined batch*

Differences between batches may arise due to matters related to the measurement procedure. For instance, depending on where the elongation occurs, some error may be caused whether all coating layers are involved in the exact same manner or not. Similarly, at elevated temperatures the testing conditions may vary slightly between the batches, causing a minor deviation. Another possible explanation for the results is PIR content composition, which may have anomalies that cause differences in peel strength values. However, no apparent differences were observed in the behavior of the samples at elevated temperatures.

In summary, PIR content does not cause a linear dependence on peel strength properties, and all batches tested met the product requirements successfully. However, the results suggest that a higher (10 wt.%) side-feed content of non-prime or prime may cause more variation in peel strength values compared to prime product. The impact was mainly positive, as the peel strength value increased. Further analysis would be required to coherently confirm the conclusion that increasing the PIR content would introduce additional risk to peel strength fluctuations.

6.2.2 Thermal properties

DSC measurement was done to inspect if incorporation of post-industrial material effects on crystallization temperature, melting point, or degree of crystallinity. Thus, these results reflect the structural properties of the compounds. Results of DSC analysis is presented in Table 24.

Table 24. *DSC results*

Sample	BB_TR0	BB_TR1	BB_TR2	BB_TR3	BB_TR4	BB_TR_P
Melting point [°C]	165.3	164.9	164.5	164.9	164.4	163.9
Crystallization [°C]	120.3	120.4	120.6	120.4	120.6	120.9
Crystallinity [%]	41.0	42.4	42.1	35.2	41.4	40.6

The obtained results indicate that the reference batch has the highest melting point. On the other hand, batch containing 10 wt.% of prime side-feed has the lowest melting point. Melting temperatures of batches containing non-prime are slightly lower compared to the reference, but the difference is smaller than one degree Celsius, with overall standard deviation of 0.2 across the batches. Thus, the tested batches show homogenous behavior when compared to the reference, and no noticeable correlation between the melting point and PIR content is observed.

No significant shift in crystallization temperatures is observed when comparing the examined batches with the reference. Surprisingly, the crystallization temperature is higher for those batches with lower melting point. This finding is contrary to previous studies which have shown that a higher melting point of would correspond to higher crystallization temperatures [18]. This anomaly may arise from measurement procedures.

The degree of crystallinity is consistent, with the only exception being BB_TR3 which has a considerably lower degree of crystallinity than the reference. Anomalies were not inspected in main processing conditions during compounding, as presented in Appendix B. Moreover, the batch containing higher PIR content does not show reduced crystallinity, which suggest that the fluctuation could be related to factors other than PIR content, however minor variations are possible within the PIR content composition. Factors such as subtle impurities, inconsistencies in material handling or sampling procedures may have affected the crystallization behavior. Typically, a higher degree of crystallinity increases the melting temperature of polyolefins, and vice versa [18]. This correlation is not directly observed from the obtained results. However, since the percentage crystallinity is measured indirectly via enthalpy of fusion, it does not necessarily correspond to the peak temperature indicating the melting point. Overall, the results appear favorable; however, there are inconsistencies.

To evaluate material performance at elevated temperatures VST was inspected. This measurement provides information whether PIR content impacts the polymer structure by creating premature heat sensitivity. Thus, VST reflects the temperature value at which material composition is expected to soften, evaluating its performance at elevated temperatures. The results obtained are presented in Table 25.

Table 25. *Vicat softening temperature results*

Sample	BB_TR0	BB_TR1	BB_TR2	BB_TR3	BB_TR4	BB_TR_P
VST [°C]	145.7	146.4	146.6	146.1	145.1	145.8

The results presented in Table 25 show that there is no notable effect of side-feeding, regardless of whether it is a non-prime or prime material. BB_TR2 resulted with the highest VST and BB_TR4 the lowest. The overall standard deviation of the samples is 0.53, with the average value of 145.95°C. Considering the application in steel pipe coating the results are favorable and consistent. The VST value exceeds temperature of 140°C, demonstrating that the material retains its quality. Thus, these findings align with the peel strength results.

6.2.3 Mechanical properties

Tensile tests were conducted to evaluate the impact of incorporating PIR content on the mechanical performance of the product. Mechanical properties are important characteristics considering the grade's application in steel pipe coating. Complete tensile test results are presented as numerical data in Table 26, along with the standard deviations within the parallel determinations.

Table 26. *Complete tensile test results*

Sample	Tensile stress at break [MPa]	Tensile strain at break [%]	Tensile stress at yield [MPa]
BB_TR0	18.5 ± 0.6	505 ± 203	23.2 ± 0.1
BB_TR1	17.2 ± 1.2	457 ± 33	23.2 ± 0.0
BB_TR2	18.3 ± 0.7	506 ± 115	23.2 ± 0.1
BB_TR3	16.2 ± 3.0	344 ± 171	23.3 ± 0.0
BB_TR4	18.9 ± 0.4	619 ± 19	23.2 ± 0.1
BB_TR_P	17.4 ± 1.5	328 ± 196	23.3 ± 0.1

The results of tensile stress at break from Table 26, suggest a fairly consistent behavior across samples and the reference. The stress at break is the lowest for BB_TR3 with the highest standard deviation. BB_TR3 also showed clearly lower values on the degree of crystallinity (35.2 %), which typically results in lower strength properties [18].

The batches seem to vary strongly in tensile strain at break in the range of 328% (BB_TR_P) to 619% (BB_TR4). Sample preparation is known to have a major impact on tensile testing results, and compounds with a lower MFR value tend to show lower tensile strain at break values due to orientation [149]. Thus, a low strain at break of BB_TR3 may be perceived as an indication of its low MFR value (6.12 g/10min), and a low strain at break of BB_TR_P may be a result of sample preparation. According to Table 27, no significant difference in the tensile stress at yield values is observed among the samples. Thus, BB_TR3 correlates well with other batches, which may suggest that earlier deviations are more probably a cause of sample preparation, than degree of crystallinity. Moreover, tensile stress at yield is perceived as a more relevant indication of mechanical performance considering the application in multilayer corrosion protection.

Furthermore, tensile strain at break and stress at break are illustrated in Figure 24, along with the standard deviations within the parallel determinations. From the visual graph, tensile stress at break values exhibits a narrow deviation within the batches, but for BB_TR3 the standard deviation is the highest. On the contrary the standard deviation values in tensile strain measurements are quite large, especially for some specific batches (BB_TR0, BB_TR4, and BB_TR_P).

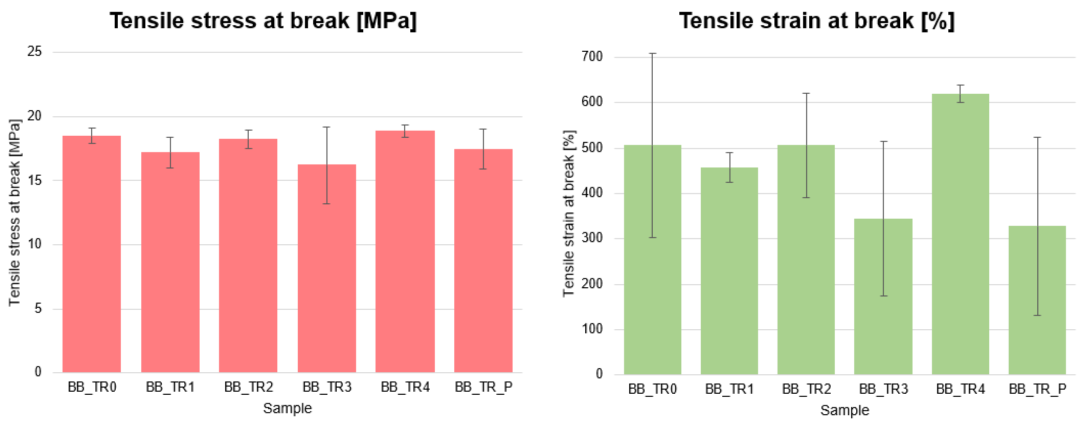

Figure 24. *Tensile stress at break and strain at break of Product 2 samples*

According to Figure 24, batches BB_TR1 and BB_TR4 represent a high degree of uniformity for both characteristics. Research of the use of PCR materials in PP compounds concluded that fundamentally the feedstock composition determines the MFR value which is highly reflected on tensile properties [146]. Thus, some indication of correlation between the flowability and results can be observed. However, since a difference in the tensile properties of BB_TR4 and PP_TR_P occurs despite the equal MFR value, it supports the suggestion of sample preparation affecting the results. In addition, another study found that incorporating up to 70 wt.% reprocessed PP into prime product does not significantly affect the mechanical performance [147], which again supports the claim of sample preparation.

In addition to the tensile tests, the following mechanical properties were measured: Charpy impact strength, shore hardness, and flexural modulus. Investigating these characteristics will provide additional information and a more comprehensive assessment of how the addition of PIR content to product 2 affects its mechanical properties. The results are presented in Table 27, along with the standard deviations within the parallel determinations.

Table 27. *Other mechanical properties of Product 2 samples*

Sample	Charpy notched (-20°C) [kJ/m^2]	Shore hardness (1s) [-]	Flexural modulus [MPa]
BB_TR0	6.96 ± 0.26	65.6 ± 1.3	1234 ± 4.7
BB_TR1	7.12 ± 0.18	65.4 ± 0.6	1236 ± 7.5
BB_TR2	7.05 ± 0.17	65.7 ± 1.7	1229 ± 2.8
BB_TR3	7.11 ± 0.15	66.3 ± 0.4	1249 ± 3.0
BB_TR4	7.13 ± 0.20	66.4 ± 1.1	1242 ± 9.4
BB_TR_P	6.95 ± 0.21	65.3 ± 0.9	1237 ± 8.9

PP exhibits decreased flexibility and increased susceptibility to fracture at sub-zero temperatures [14]. Therefore, Compound B samples were subjected to Charpy impact testing at -20°C to assess their resistance to fracture under low-temperature conditions. The results of Charpy impact strength, as presented in Table 27, show only a slight variation between the reference and the examined batches. Highest percentage difference is observed for BB_TR4, with increase of 2.4% in impact strength compared to the reference. This indicates that PIR content does not have a significant effect on impact strength, and no linear correlation between the amount of PIR material and impact strength is observed. Moreover, batches containing PIR content have a slightly higher impact strength compared to the reference and samples with prime Compound B, indicating that the material could be able to withstand impacts better at sub-zero temperatures. Moreover, the standard deviations within each batch are small, indicating consistency over impact strength.

Shore hardness expresses material's resistance to indentation, and higher value indicates harder material [136]. As shown in Table 26, test run batches with PIR content correlate with the reference, since no great variation is observed. Highest percentage difference compared to the reference is 1.2% for BB_TR3. The obtained data in Table 31 does not reveal any identifiable trend concerning the amount of PIR material and shore hardness. Moreover, the results are consistent with previous findings, as both Charpy impact strength and shore hardness values for all tested samples, including the reference, are within specification. Thus, the specimens behaved in a comparable manner, and the results are quite homogenous with small standard deviations within each batch.

Flexural modulus results are consistent with one another. The deviations compared to the reference are relatively small, with the highest percentage difference of 1.2% between BB_TR0 and BB_TR3. This kind of variation can be perceived as acceptable in daily operations. Moreover, increasing the amount of PIR does not lead to a predictable linear change in the flexural modulus values, since no consistent increase or decrease is observed. Therefore, it can be concluded that incorporating up to 10 wt.% of PIR within prime product results in an acceptable flexural modulus value.

To conclude, the findings regarding mechanical properties suggest that incorporation of PIR content up to 10 wt.% does not negatively effect on the material properties. These are promising results, and support utilization of PIR content as part of prime products.

7. CONCLUSIONS

Recovery of post-industrial materials can be perceived as a reasonable and sustainable use of raw materials. A popular method to utilize primary recycled polymers is to blend them in a specific proportion with prime resins to achieve desired properties. The aim of this thesis was to investigate the potential of incorporating post-industrial recycled (PIR) materials, specifically from polyolefin pellet production, with the corresponding prime grades, to reduce the dependency on prime materials and create a more sustainable and effective use of raw materials. The economic analysis considering the use of PIR content was carried out separately and is outside the scope of this study. However, it showed significant economic benefits which support the arguments of this study. The second research question considered how other polyolefin pellet producers treat post-industrial side-streams generated from their production operations.

Due to regulatory aspects and to protect trade secrets, most polyolefin pellet producers do not publicly disclose their methods relating to PIR material treatment. The approach of only a few companies differs from the rest. These companies typically have a separate polymer brand under which products containing PIR content are marketed. The PIR content of the products ranges from 30-98 wt.%, and the material properties are comparable to prime grades. In general, more information is available about PIR content utilization in further conversion processes and by third parties.

Pilot-scale compounding experiments were conducted on two polypropylene grades, and the PIR content was varied across four batches for both grades. The evaluation of the batch quality was made by comparing the results to a compounded reference batch indicating primary product performance. Moreover, a batch with incorporation of primary grade was compounded, to assess the extent to which the properties of the PIR material match those of the primary grade. Comparative analysis of material properties with varying amount of PIR content relative to the reference is presented for Product 1 and Product 2 in Table 28 and Table 29, respectively. Changes relative to the reference are characterized as follows: major increase/decrease (↑↑/↓↓), minor increase/decrease (↑/↓), and no effect (±0).

Table 28. *Product 1: Comparative analysis of material properties with varying amount of PIR relative to the reference*

Sample **PIR content [wt.%]**	**SF_TR1** **3**	**SF_TR2** **5**	**SF_TR3** **7**	**SF_TR4** **10**
Gel index	± 0	± 0	± 0	± 0
MFR (230°C/2.16kg)	± 0	± 0	± 0	± 0
Elasticity index	± 0	± 0	± 0	± 0
Zero shear viscosity	± 0	± 0	± 0	± 0
Yellowness index	± 0	± 0	↓↓	↓↓

The examined batches of Product 1 exhibit homogenous behavior compared to the reference, as observed in Table 28. Gel formation described with gel index is perceived as a critical performance indicator, as Product 1 is utilized in packaging applications. The PIR content shows compatibility and high resemblance with the prime feedstock, as gel formation remained moderate. Moreover, the incorporation of PIR content does not have an effect on MFR value, which corresponds to the results on rheological properties. The most notable change is observed in the yellowness index, which surprisingly shows a positive impact with the addition of PIR content, as the yellowness is reduced.

Table 29. *Product 2: Comparative analysis of material properties with varying amount of PIR relative to the reference*

Sample **PIR content [wt.%]**	**BB_TR1** **3**	**BB_TR2** **5**	**BB_TR3** **7**	**BB_TR4** **10**
MFR (230°C/2.16kg)	± 0	± 0	↓↓	↓
Peel strength (23°C)	↑↑	± 0	± 0	↑↑
Peel strength (80°C)	± 0	↑	± 0	↑↑
Peel strength (110°C)	± 0	± 0	± 0	↓↓
Peel strength (140°C)	± 0	± 0	↑↑	± 0
Reactive site content	± 0	± 0	↓	↓
Melting temperature	± 0	± 0	± 0	± 0
Crystallization temperature	± 0	± 0	± 0	± 0
Degree of crystallinity	± 0	± 0	↓↓	± 0
Vicat softening temperature	± 0	± 0	± 0	± 0
Tensile stress at break	± 0	± 0	↓	± 0
Tensile strain at break	± 0	± 0	↓	↑
Tensile stress at yield	± 0	± 0	± 0	± 0
Charpy notched (-20°C)	± 0	± 0	± 0	± 0
Shore hardness (1s)	± 0	± 0	± 0	± 0
Flexural modulus	± 0	± 0	± 0	± 0

Each compounded batch of Product 2 fulfilled the product requirements successfully. Some batches showed a decrease in MFR value, partially due to the reduction of the base resins as PIR content increased. However, the lack of a linear correlation suggests that the composition of PIR content may also be a factor. These fluctuations arise from the production operations before conducting the pilot-scale compounding. Peel strength is perceived as a critical performance indicator, as Product 2 is utilized in 3-layer polypropylene steel pipe coating systems. All examined batches fulfill the product requirements, but greater fluctuations are observed with PIR content of 10 wt.%. Further analysis would be required to coherently confirm the conclusion that increasing the PIR content could introduce additional risk to peel strength fluctuations. The examined batches generally displayed homogenous and favorable thermal properties compared to the reference. However, one batch exhibited considerably lower degree of crystallinity, likely due to factors other than PIR content such as subtle impurities or handling inconsistencies. The lower degree of crystallinity affected the mechanical properties of BB_TR3. Given the context of functional application performance, tensile stress at yield is more perceived as more essential characteristic than the other two tensile properties presented in Table 29. Therefore, slight variations observed in tensile stress and tensile strain at break are considered as less relevant.

A common factor is noticed that is most likely to cause variation; the composition of PIR content. No changes linked to reprocessing or material degradation were observed and PIR content exhibited compatibility with the prime resins. Further studies should focus on developing robust methodologies for the classification of generated PIR content. By creating tools for quality analyzation the risk of variation related to PIR content composition could be minimized. This could be done by, for example, examining the extreme cases, if the PIR content would only consist of one base resin, and assessing its impact on the properties. Then the quality and composition of generated PIR material could be linked with the product operations, as well as noted in the recipe design. Another valuable area of study could be conducting a life cycle assessment to evaluate the environmental benefits of utilizing PIR content. This assessment could provide critical insights into the environmental impact reduction compared to current practices, offering a more comprehensive understanding of the sustainability advantages linked to reprocessing of PIR content.

To conclude, the experiments demonstrated that incorporation of PIR content with prime feedstock does not cause significant changes in product properties, yielding results comparable to prime grades despite PIR content. The results align with existing literature concerning polypropylene recyclate compounding with base resins [6,62]. Based on the results, production-scale test runs are recommended for both examined products. After completing the production-scale test runs, the next steps in advancing this research would be to conduct customer sampling and initiate trial manufacturing of the final product. Reutilization of PIR materials present significant advantages by reducing raw material costs and enhancing the valorization of waste products, thereby aligning with sustainable manufacturing practices and advancing circular economy objectives.

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APPENDIX A:

Mechanical properties of grades containing PIR content.

Table 30. *Product Selection Guide – CirculenRecover PP by LyondellBasell [69,70]*

Grade name	Tensile modulus [MPa]	Tensile stress at yield [MPa]	Tensile strain at break [%]	Charpy impact strength, notched 23°C [kJ/m^2]
PP (PP340H Natural)	1070	30	>30	55
PP (PP522H Natural)	-	34	-	-
PP (PP320S Pearl)	1150	26	40	3.0
PP (PP520T Crystal)	1500	32	40	2.5
PP (PP310U Graphite)	1200	25	8	3.0

APPENDIX B:

Manufacturing batches with WP-30.

Table 31. *WP-30: Test run report Compound A*

Sample	SF_TR0_G	SF_TR0_PW	SF_TR_P	SF_TR1	SF_TR2	SF_TR3	SF_TR4
Screw speed [rpm]	200	200	200	200	200	200	200
Melt temperature [°C]	209	209	209	209	209	208	208
Melt pressure [bar]	10.2	10.2	10.3	10.3	10.1	10.3	10.3
Torque [%]	55.9	56.0	56.0	56.8	56.4	56.7	57.0

Table 32. *WP-30: Test run report Compound B*

Sample	BB_TR0	BB_TR_P	BB_TR1	BB_TR2	BB_TR3	BB_TR4
Screw speed [rpm]	200	200	200	200	200	200
Melt temperature [°C]	230	230	229	229	229	229
Melt pressure [bar]	12.7	12.8	12.7	12.7	12.7	12.8
Torque [%]	58.9	59.9	58.7	59.7	59.0	59.9

Table 33. *WP-30: Zone temperatures for Compound A and B*

Zone number	Compound A [°C]	Compound B [°C]
1	160	180
2	190	200
3	200	200
4	200	200
5	200	210
6	200	220
7	200	230