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Screening the Effect of Impact Modifier Content to the Properties of Heterophasic Polypropylene

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Lisäainepitoisuuden vaikutus polypropeenituotteessa

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Opinnäytetyön aiheena on teräsputkien saumakohtien pinnoituksessa käytettävän polypropeenilaadun lisäainepitoisuuden tarkastelu uusissa raaka-aineissa sekä jo käytössä olevassa. Työn toimeksiantajan toimii muovin raaka-aineita valmistava Borealis Polymers Oy. Tutkimus sisältää luottamuksellista tietoa, joka on rajattu pois julkisesta versiosta. Tutkittava lisäaine on tärkeä tuotteessa, jonka kuuluu kestää pudotusta sekä kovaa painetta. Toimeksiantajalla on tulevaisuudessa tarkoitus ottaa käyttöön uusia lisäainelaatuja tuotannossa, minkä vuoksi reseptit oli tarpeen tarkistaa. Myös käytössä olevan reseptin toimivuutta kaupallisessa tuotteessa tarkasteltiin.

Tutkimuksen tavoitteena oli tutkia, kuinka uudet raaka-aineet vaikuttavat polymeerituotteen kemiallisiin ja fysikaalisiin ominaisuuksiin. Tarkastelun lähtökohtana oli siis selvittää optimaalinen sekoitussuhde polymeerin sekä lisäaineen välillä siten, että laatuvaatimukset täyttyvät, mutta raaka-ainetta käytettäisiin mahdollisimman vähän. Tavoitteena oli myös kartoittaa näytemateriaalien välisiä laatueroja ja ehdottaa saadun tiedon perusteella jatkotutkimusvaihtoehtoja toimeksiantajalle. Osasyy reseptien tarkastelulle on myös raaka-ainekustannuksissa, jotka ovat vuositasolla huomattavan korkeita nykyisellä sekoitussuhteella.

Tutkimuksissa näytteille tehtiin kemiallisiin omaisuuksiin liittyviä perustestejä sekä mekaanista kestävyyttä mittaavia kokeita. Kokonaisuudessaan tutkimuksen aikana tutkittiin kahta uutta lisäainelaatua sekä jo käytössä olevaa raaka-ainetta. Tarkoituksena oli valmistaa lisäaineista ja tutkittavasta polymeeristä viittä erilaista sekoitusta, joita tutkittiin muoville tyypillisin analyysimenetelmin. Saatuja tuloksia verrattiin referenssimateriaalina toimivaan kaupalliseen versioon kyseisestä polypropeenituotteesta.

Tavoitteet saavutettiin toivotulla tasolla, sillä lisäainelaatujen väliset laatuerot saatiin näkyviin ja analyyseista kerätyn datan perusteella voitiin tehdä johtopäätöksiä jatkoa varten. Yksi tutkittavista lisäaineista erottui laatuominaisuuksillaan ylitse muiden. Tulevaisuudessa raaka-ainetta testataan tuotantomittakaavassa koeajoin ja se tulee mahdollisesti korvaamaan tällä hetkellä käytössä olevan raaka-aineen. Näytteiden reologisia ominaisuuksia sekä morfologiaa tullaan tutkimaan tulevaisuudessa pohjautuen tässä opinnäytetyössä tehtyihin havaintoihin.

Asiasanat: polypropeeni, lisäaine, laatuparametrit, reseptinkehitys

ABSTRACT

Tampereen ammattikorkeakoulu
Tampere University of Applied Sciences
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UOSUKAINEN, ANNI:
Screening the Effect of Impact Modifier Content to the Properties of Heterophasic Polypropylene

Bachelor's thesis 52 pages, appendices 8 pages
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The objective of this thesis was to examine the effect of new impact modifier grades in polypropylene product, which is used as an adhesive material when coating joints of steel pipes. The impact modifier plays a major role in the PP product due to its physical requirements, and the coating material must cope with high pressure and stress during usage. When the impact modifier material changes, the product recipe must be examined and optimised again for each substance to make the quality parameters meet its specification.

The subject for this thesis arose from the need for Borealis Polymers Oy to find the optimal mixing ratio between the impact modifier and polypropylene material. This examination was necessary because the product must maintain its physical properties and quality parameters.

The purpose of this thesis was to inspect the effect of new impact modifiers when screening the product's properties. The examinations focused on the product's chemical and physical properties in different concentrations. This study also had a financial object as the impact modifier material is valuable, and with recipe modification, the annual costs of the material can be significantly decreased.

Furthermore, the objective of this thesis was to examine the effect of new impact modifier grades in polypropylene product and recognise differences between materials. The aim is to sort out material options, find the most potential ones for further examinations, and propose the most suitable impact modifier concentrations for the commissioner.

The samples were subjected to tests focusing on chemical and physical properties. Results were compared to the quality parameters of the product, the commercial reference material, and each other to perceive differences between substances. All in all, three impact modifier grades were investigated, including two new compounds and the in-use material.

The object of this study was mainly accomplished. One of the materials exceeded expectations with its physical properties. As a result of the examinations, Borealis Polymers Oy may proceed to the test run phase with the new material.

Key words: polypropylene, impact modifier, quality parameters, recipe modification

TABLE OF CONTENTS

ABBREVIATIONS

PP	polypropylene
PE	polyethene
PO	polyolefin
IM	impact modifier
MFI	melt flow index
MFR	melt flow rate
DSC	differential scanning calorimetry
FTIR	Fourier-transform infrared spectroscopy
SEM	scanning electron microscopy
PVC	polyvinyl chloride

1 INTRODUCTION

This study was executed in the Borealis Polymers Porvoo location during autumn 2022. All measurements were performed in the quality control laboratory using harmonised (ISO 9001) methods and equipment, which are approved and audited for commercial use. Borealis Polymers Oy is a part of a large-scale international company that is a global leader in chemical and polyolefin solutions. (Borealis Polymers 2022, NA.)

Polypropylene (PP) and polyethene (PE) raw materials are produced in three different plants in Porvoo Kilpilahti industrial area, which is a significant operator on a global scale. Polyolefins (PO) are mainly used for automotive, consumer products, energy, healthcare, pipes and fittings. In this study, the final product has particular usage as shockproof material covering steel pipes joints in high-performance use. (Borealis Polymers 2022, NA.)

The objective of this study is to provide an overview of the new materials and their suitability for functional usage of the PP product. The starting point of this study is to examine how the old recipe works with new impact modifiers because of changes in the raw materials in the future. The in-use impact modifier grade recipe is also observed if modification is needed.

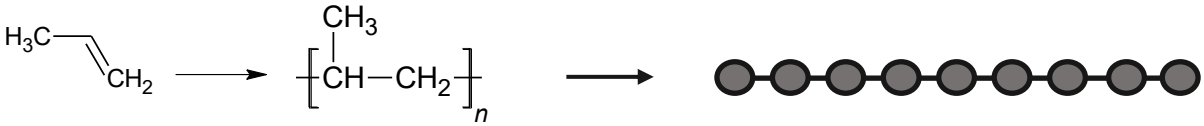
This thesis consists of a review of three impact modifiers and the conclusion of their appropriateness for polypropylene products. The results are presented in the form of charts, and the more precise analysis data is gathered in the appendices. The laboratory test results are stored in Borealis laboratory information management system (LIMS) under URSUS number 52717 and Nautilus number N58873.

2 THEORETICAL BACKGROUND

This chapter consists of the theoretical background of the polymerisation process and the general structure of a polymer. The analysis and pretreatment methods chosen for this study are presented in paragraphs 2.5 to 2.7.

2.1 Polymer structure

A polymer is created in a polymerisation process, where low molecular weight compounds called monomers (e g. ethylene or propylene) are attached to each other. In polymerisation, long chains of monomers are bound together with a single bond, creating the "necklace" structure as presented in figure 1 (Harper 2006, 2). When naming polymers in front of the monomer's name will be added a prefix poly, for example, polyethene or polypropylene.



propylene (monomer) — a single propylene unit in a polypropylene chain — a polypropylene chain ”necklace”

FIGURE 1. Polymerisation (Handbook of Plastic Technologies, Harper 2006. edited).

The synthesising process coincides even with hundreds to thousands of monomers at the same time. Polymer chains, which are generally linear, are formed due to this chemical chain reaction. There are other variations of polymer chains - for instance, branches, hyperbranched or cross-linked. The following figure 2. presents the most typical variations of polymer chains. (Braun 2004, 39.)

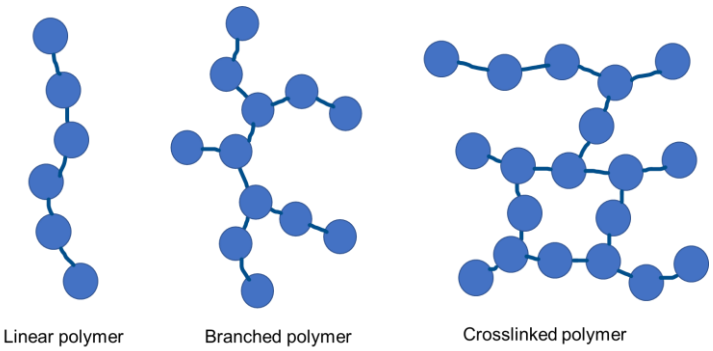


FIGURE 2. The most common alternatives of polymer chain structure.

The starting species of the polymer chain will determine the properties of the final polymer; in other words, the molecular weight of the chain's starting molecules will significantly affect the PP or PE product.

FIGURE 3. A copolymer simplified structure.

During the polymerisation between two different molecular weight monomers, the consequent compound is called a copolymer – for example, ethylene vinyl acetate. Figure 3 above presents the simplified structure of a copolymer. (Harper 2006, 2.)

2.2 Step reaction polymerisation

In step reaction, there is no need for a catalyst or any other specific active molecules, and the reaction does not occur as a cascade. The reaction requires at least two polyfunctional monomers before the step reaction can take place. The most typical chemical reaction types occurring between monomers are -ester, -ether, -amide or polyurethane formation. As the reaction continues, species with higher molecular weights are formed as more extended groups react. While this reaction goes on, typically, small molecules such as water are released. (Braun 2004, 41.)

2.3 Chain reaction polymerisation

Chain reaction polymerisation (also known as addition polymerisation) requires an initiation for polymerisation to maintain its state. An initiator (catalyst) could be a free radical, either anionic or cationic. The catalyst causes a monomer's double bond to open, and the reaction starts to proceed. Generally, chain reaction polymers chain contains only carbon molecules and polymers such as polystyrene, and polyvinyl chloride are incorporated. (Harper 2006. 2.)

2.4 Melt flow index

When defining different polyolefin (PO) grades, melt flow index (MFI) and melt flow rate (MFR) values are usually used as primary parameters. The MFI value of a material depends mainly on its molecular properties, generally the rate of compound distribution and the molecular weight of the polymer. In addition, the branching features, such as the branching type and the regularity of the distribution, also affect MFI and MFR values. The MFI result provides standard information on the average size of molecules in a resin (polymer mass) and their relations with each other. (Abbas-Abadi et al 2012. 1739-1740.) MFI and MFR are practically the same, and their measurement principles are identical, but the test conditions differ. Tests measure melt viscosity and resin flowability under a determined load. (Wagner et al. 2013, 195.)

The melt flow index is defined as the weight of polymer extruded in 10 min through a preheated capillary of specific diameter and length. The pressure directed to the polymer material depends on the test type, but the most common load is 2,16 kilograms, as figure 4 presents.

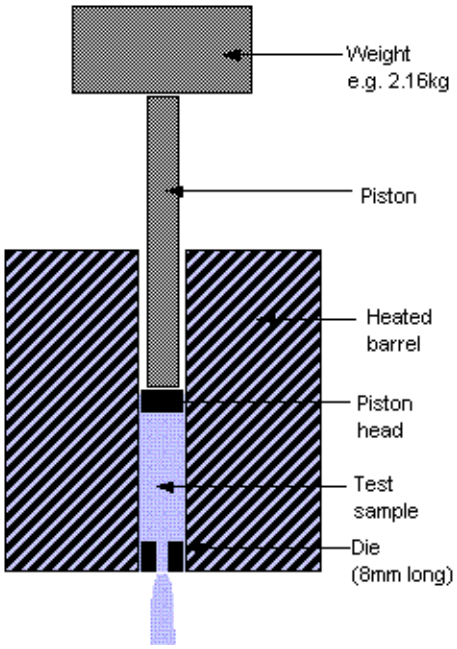


FIGURE 4. Melt Flow Index (MFI) instrument basic structure (Farhani 2014, Wikimedia Commons).

The cylinder temperature varies between 125 to 300 ℃ depending on the material to be tested. During the experiment, the material flow through the cylinder is monitored, and when the mass of the sample is known (g), the unit for the MFR can be derived. The unit resembles the amount of resin flow during the 10-minute measurement, g/10min. (Shenoy 1986, 2.)

2.5 Differential scanning calorimetry

Chemical reactions and physical transitions are either releasing or consuming heat. To examine such processes, calorimetry is a wide-known and reliable method. The basic principle of calorimetry is to measure changes in the specimen – more precisely, the amount of heat generated or lost in a physical or chemical change. (Newton 2021, 262-263.)

Differential scanning calorimetry (DSC) is a test method where an alteration in the heat flow is observed in a sample. The heat flow alteration is compared to the reference sample, measured with the same controlled temperature program. (Höhne et al. 2010, 1-2.)

The most common differential scanning calorimeters are heat flux and power compensation types. These instruments have differences between the design and measurement principles. Although generally, all DSC instruments share the same dual-type structure and a direct differential connection between two measurement systems of the same type. The system is relatively sensitive due to the difference signal monitor, which provides essential information about changes in the sample's heat quantity. The signal can be powerfully amplified because the high baseline signal (the signal from a single measurement system) is nearly compensated when the change appears. (Höhne et al. 2010, 9.)

2.5.1 Heat Flux DSC

The heat flux DSC is a heat-exchanging calorimeter whose sensitivity is based on a defined environmental change. It happens through a well-defined heat conduction path with a specific thermal resistance. As the basic principle of DSC technology, the temperature change is the main signal, which determines the exchange's intensity. The resulting heat flow rate (ϕ) depends on the temperature change. (Höhne et al. 2010, 10.)

A common analysis type uses a disk-type measurement system in which heat exchange occurs via a plate consisting of a holder for solid samples. It is a straightforward and reliable analysis with a high level of sensitivity, and the amount of sample required is small. However, there are some limitations in the rate of heating and cooling. (Höhne et al. 2010, 10.)

2.5.2 Crystalline and amorphous fractions of polymers

As the solid polymer melts, its amorphous fraction increases. Thoroughly melted polymer is amorphous, which means the substance is chemically unorganised. Most polymers are semicrystalline, and the material starts to cool down after reaching its melting point. During cooling, the polymer's internal structure begins to change, and crystalline structures are formed. A completely cooled semicrystalline polymer consists of both crystalline and amorphous

particles, and the fractions' ratio impacts the material's mechanical properties. The high crystallinity rate polymers are tough and durable compared to the more amorphous ones. (Manas et al. 2008, Chapter 1.6.1.)

2.6 Compounding and extrusion of thermoplastics

Compounding is a synonym for the term homogenising, which is a process of combining an impact modifier and a polymer product. As a result of compounding, homogenous pellets are formed. The additives usually enhance the material's properties and ensure that the homogenisation process is successful. The most common additives can include, for example, calcium carbonate, which is powdered limestone. If a soapy and slippery feel to the material is needed, talc is used. Carbon black is used as a black colourant and protector against UV radiation. Additives used to increase the material's mechanical strength are called reinforcements. (Davis et al. 2001, 16.)

For instance, extrusion processes are broadly used to produce a plastic film which is reproduced in plastic bags. (Harper 2006, 16.) To create such a mixture, the polymer material must be heated to an optimal temperature to make it flowable. A rotating screw inside a heated barrel combines materials as a homogenous paste.

Impact modifiers are added to the polymer resin to improve its physical properties, such as durability, stiffness or elasticity. They have a key role, especially in the products designed for physically demanding usage, for instance, coating purposes or the automotive industry. The impact modifier material depends on the polymer resin. For example, the impact modifiers added in the polyvinyl chloride (PVC) could be acetonitrile-containing copolymers to enhance the heat distortion resistance and improve tear strength. Some other known impact modifiers (acrylic-based impact modifiers) can affect the weather resistance of the material and rheological properties. (Wypych 2022, Chapter 2.2.)

2.6.1 Extruder structure and function

An instrument used for homogenising polymer products and additives is called an extruder. There are two types of extruders – single and twin screws, and the primary function of the screw is to push the material forward in the barrel while it homogenises the melted mixture. The temperature in the barrel varies between materials, but generally, PP requires higher temperatures than PE material. When materials are weighed and mixed precisely, the resin and impact modifier mixture will be poured into a hopper connected to the feed zone. The material falls into the feed section from the hopper, where a rotating screw pushes solid material gradually into the heated barrel. The solid material travels through the extruder's middle section, also called a transition zone, where the material is supposed to melt and mix. (Harper 2006, 16.) Figure five below portrays the basic structure of the extruder.

As materials have gone along the mid-section of the barrel, they will be pushed through a nozzle, resulting in a thin strip of homogenous polymer mass. The strip must be cooled rapidly using a water bath or airflow. The homogenised material is pelletised with a cutter into pellets, also known as granulates. (Harper 2006, 16.)

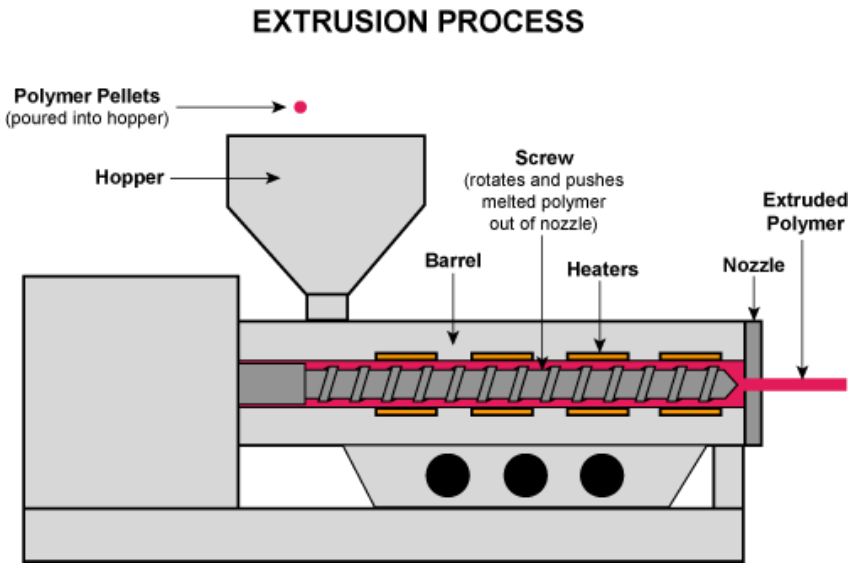


FIGURE 5. The basic structure of an extruder (Wikimedia Commons 2022, NA).

There are two types of twin extruder screws – counter- and co-rotating. The structure is practically the same, and the main difference is the rotation direction.

When compounding polyolefins, intermeshing twin screws are typically used due to the long flow path and increased elongational flow, which enhances mixing. (Harper 2006, 18.)

2.7 Injection moulding and mechanical tests

Injection moulding is a broadly used technique to manufacture different-sized plastic components. An injection moulding has four main components, which are presented in the following figure 6. (Harper 2006, 22.)

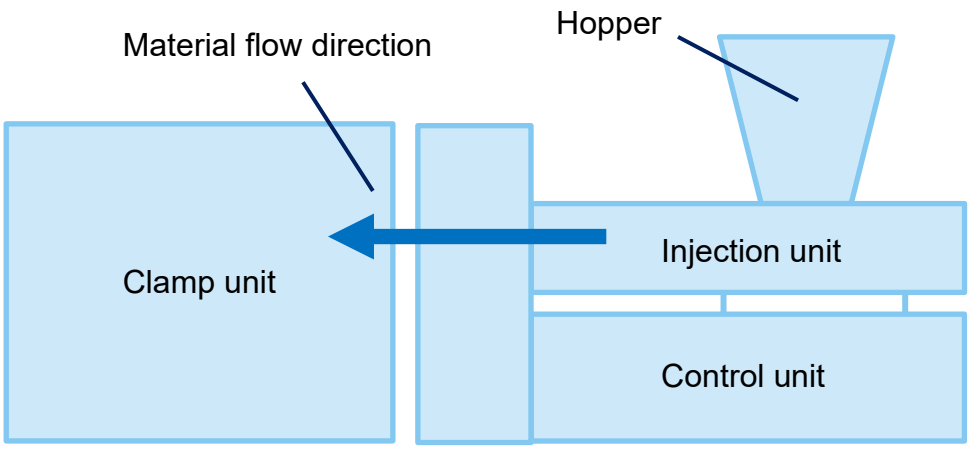


FIGURE 6. The basic structure of injection moulding machine (Olmsted et al. 2001, 2, edited).

The injection moulding process is a process which involves high temperatures and pressure, which results in some additives being essential for the material to endure these conditions. Like an extruder, the resin is poured into the hopper, which feeds material to the injection unit. The injection unit heats, melts, pumps and injects polymer material into the mould. The structure and function of the injection unit are similar to the extruder. The difference is that polymer mass steers into the mould instead of the nozzle. (Olmsted et al. 2001, 3.)

The mould has two postures – open and closed. When the material is injected into the mould, it is in a closed position. The metal mould is attached to the clamp unit, and its function is to open the mould when plastic parts need to be ejected

and keep it closed during the injection. The control unit monitors and controls the functioning of the injection and clamp unit. (Olmsted et al. 2001, 3.)

2.7.1 Vicat softening point

A Vicat test gives essential information about the material softening point during the heating process. In the Vicat softening point test, a polymer sample is heated gradually in an oven or a bath, and the temperature increases slowly. The rate can be either 50 or 120 °C degrees per hour. The softening point of the material is observed with a needle penetrating the sample while the temperature rises. The load from the needle is typically 10 or 50 Newtons, and measurement goes on until the needle has pierced one millimetre of the sample surface. (Brown 1999, 345.)

There are two types of Vicat tests – type A and B. The load is 10 Newtons in the Vicat A-test, and the temperature slope is 50 °C per hour. Type B has a steeper temperature rise, and the load is also higher (50 N) (Brown 1999, 345.) The softening point test is standardised with ISO 306, and the sample's thickness must be between 3 and 6,5 mm while the area must be at least 10 mm^3 (SFS 306 2014, 4).

2.7.2 Tensile stress-strain properties

The tensile stress-strain properties analysis is the most common mechanical measurement for polymers, and the parameters are generally considered a quality guide. The principle is quite simple – the sample will be stretched until it breaks, and force and elongation directed to the piece are measured at various stages. The results depend on test piece geometry, so the tensile properties are commonly regarded as erratic rather than absolute. (Brown 1999, 228.)

The basic parameters measured are strength, elongation at break, and modulus, but these can have different significance depending on the material. Plastics may yield before failure such that the strength at break is not the maximum stress

attained, and the elongation figure has little practical meaning. (Brown 1999, 228.)

Because polymers do not have linear stress-strain curves, several measurements are being adopted, and due to that, there are several definitions of modulus and yield points. It is also important to remember that the results will depend on the sample preparation method and that comparison can only be made between measurements where the procedures and the definitions of the parameters are the same. (Brown 1999, 228.) Figure 7 presents a typical stress-strain chart when measuring plastics or other solid and rigid materials.

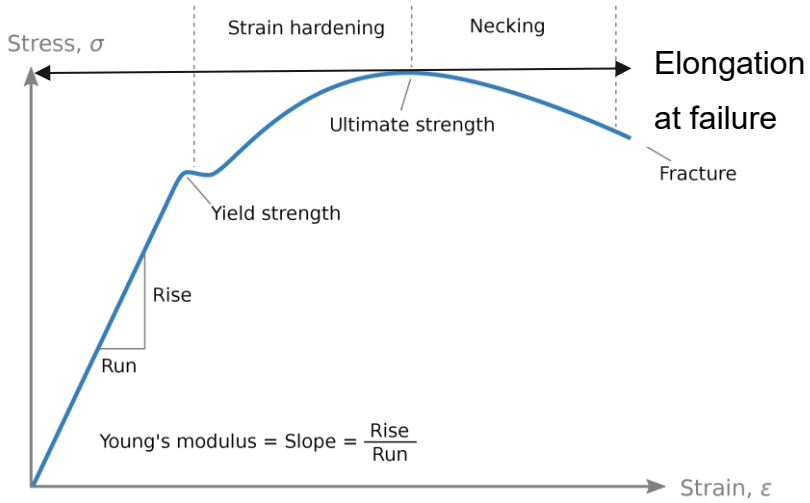


FIGURE 7. Typical stress-strain diagram (Wikimedia Commons, 2020. edited).

The stress-strain diagram resembles the relation between the tensile force affecting the material (stress) and the level of deformation caused by the load (strain). Ultimate strength, elongation, and elastic modulus (also known as Young's modulus or Tensile Modulus) can be discovered from the tensile experiment. (Manas et al. 2008, Chapter 2.2.2.) Specimens are the most typical shape of a two-dimensional dumbbell or flat strips, more familiar as "dog bones". Sample preparation requires injection moulding and stabilisation time before further examinations. During the measurement, the stress concentrates on the narrow part of the piece, and usually, the fracture occurs in that part instead of the grip section. The following figure 8 presents the general shape of a dog bone sample. There are dimensional differences between test pieces.

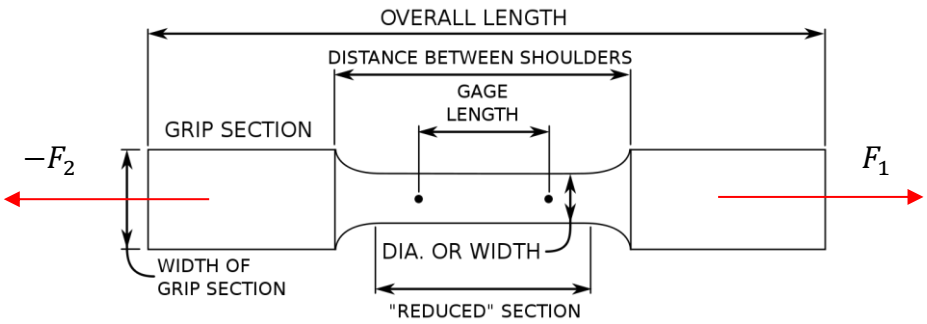


FIGURE 8. A common test piece is known as the “dog bone” (Davis 2014, Wikimedia Commons. edited).

The sample is stretched from its grip sections during the tensile-stress analysis. Forces F_1 and F_2 are equal but in the opposite direction.

2.7.3 Flexural properties

The flexural stress-strain properties of the material are primarily dependent on the stiffness. With rigid material, flexural modulus and strength are commonly measured. Flexural properties of the material (e.g. polymer or metal) give information about its stiffness and bending features. (Brown 1999, 237.)

When the specimen is bent, gradually increasing maximum tensile stress effects on one surface through a neutral axis and maximum compressive stress occurs on the other surface. For example, in figure 9, tensile stress occurs on side A, the indentor creates compressive stress to the material when force is aligned from above, and the maximum tensile stress occurs on side B when the material bends during measurement. (Brown 1999, 238.)

The test piece can be, for instance, an injection moulded rectangular or a dumbbell shape ("dog bone"). Three-point loading is the most popular, but four-point loading has the advantage of constant stress between the two inner supports. (Brown 1999, 238.)

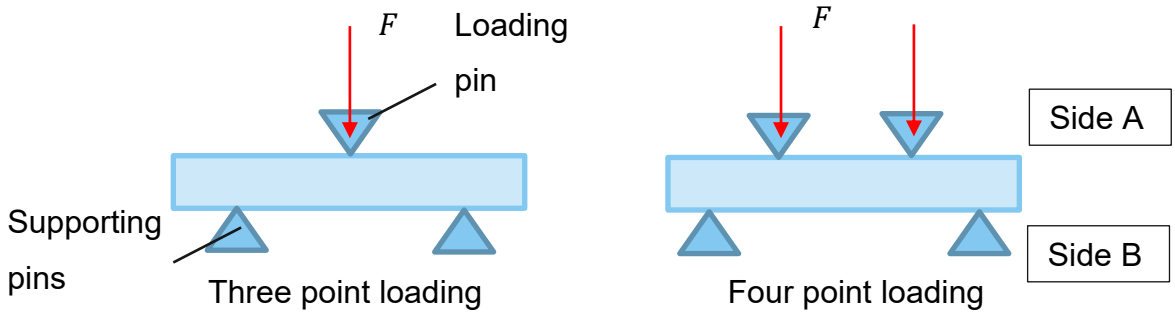


FIGURE 9. Flexural properties measurement simplified, loading types and the impact of the loaded force.

There is also a possibility to observe flexural properties with a simple loading experiment where mass is being hanged from the other end of the specimen. The other end is supported, and the sample does not lean on any surface. (Brown, 1999, 238.) For instance, the three-point loaded flexural stress (maximum fibre stress) can be determined with the following mathematical formula number one (Brown, 1999, 238).

$$\sigma_F = \frac{3FL}{2bh^2} \quad (1)$$

σ_F = Flexural stress, MPa

F = Force at the midpoint, N

L = Span (between supporting pins), $\mathrm{m, cm\ or\ mm}$

b = Width of the sample, $\mathrm{m, cm\ or\ mm}$

h = Thickness of the sample, $\mathrm{m, cm\ or\ mm}$

2.7.4 Charpy impact

The Charpy impact test is designed to determine the impact behaviour of materials when a loaded pendulum is hitting a notched bar. The main causes of failures in materials are high strain rates, low temperatures and triaxiality of stress (Manas et al. 2008, Chapter 2.2.19.) The triaxial stress means the multiaxial stress in the brittle specimen caused by the pendulum hit (Tiejun et al. 2002, Chapter 1.1).

A pendulum test is the most suitable way to subject material and its capability to cope with high stress in different environmental simulated conditions. The method gives essential information about the material's breaking type, predicting its suitability for its designed function. Charpy and Izod tests are standardised, and measurements are conducted per ISO 179-1 (SFS 179-1 2010, 2). The specimen has a standard-sized notch on the tension side whose radius is 0,25 millimetres (45°), or in the blunt version, 2 millimetres. (Manas et al. 2008, Chapter 2.2.19.)

In the Charpy test, the specimen is supported lightly in the sample holder, and the loaded pendulum is aligned at the midpoint. The unnotched side is directed toward the pendulum, as shown in figure 10.

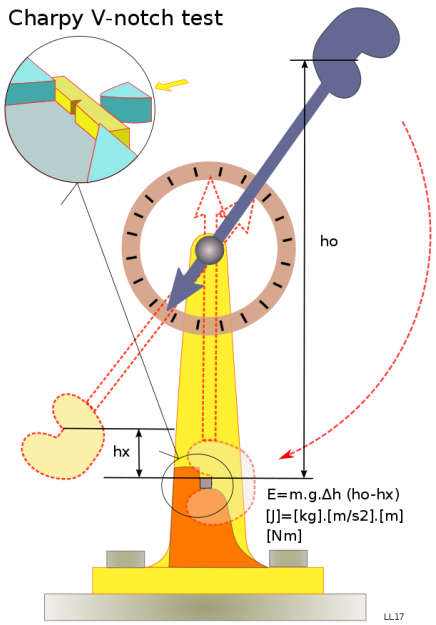


FIGURE 10. Charpy impact test basic principle (Van Lieshout 2017, Wikimedia Commons).

The energy absorbed by the specimen during breakage is documented. The result often has deviation, and the median is used rather than the average strength due to the irregularity; therefore, the median value is more presentable. Impact strengths are usually expressed as formula number 2, presents below. (Manas et al. 2008, Chapter 2.2.19)

$$Impact\ strength = \frac{Energy\ absorbed\ to\ break}{Area\ at\ notch\ section} = kJ/m^2 \quad (2)$$

2.7.5 Durometer hardness test

A durometer test is used to determine the degree of material hardness. The principle of the durometer hardness test is to press a needle-like indentor into the sample and examine the needle's creep in the material. The analysis' basic principle is to press the instrument against the material surface and force the indentor into the sample. A lever system converts the puncture depth into an indicator on a scale calibrated from 0 to 100. (Manas et al. 2008, Chapter 2.2.22.6.)

The most common types of durometers are Shore Type A and D. The difference between the types is in the spring force and the geometry of the indentor. In type, A indentor tip is blunter, and the force aimed at the specimen is lower. The type D tip is sharper, and the pressure used is higher. (Manas et al. 2008, Chapter 2.2.22.6.)

3 PRACTICAL PART

3.1 Polypropylene product

The polypropylene product used for this study is designed specifically for coating purposes, and its shockproof properties are essential. PP product examined is used as a coating material when steel pipes' joints are welded together in the construction site. As a result, long pipelines are formed, and the weakest part of the pipes are the welded seams. The shockproof coating material is added to the hem to ensure that the seam endures the high energy hit when it is dropped to the ground. This technique is called field joint coating, and the coating material's key role is to prevent seam breakage and any leakages. Field joint coating technique is commonly used, for instance, with gas pipes or sewage systems. (Smith, 2016, 105-115.)

3.2 Impact modifier grades

All in all, three different impact modifier (IM) types with different characteristics were studied. Information and names of the types have been modified due to confidential reasons.

TABLE 1. Impact modifier materials used for the study.

Impact modifier	Information
3.1	In-use
5.7	New material
3.2	New material

In this study, reference material was an impact modifier type already used in the commercial product. The previous table 1 includes IM materials used for this study. The IM types presented in this study are unrelated, and the commissioner names the materials.

3.3 Sample preparation

First, the polypropylene material and the impact modifier must be homogenised and pelletised before further examination. The preparation process starts with mixing a basic polymer material and an impact modifier following the planned recipes. Samples were made by weighing the desired amount of both materials together in a bucket, and the mixture was shaken thoroughly for ten minutes in a specific stirrer. Both raw materials were in a pellet form when making the sample mixtures. The impact modifier types were all pure, and dilutions were calculated with the expectation that the concentration of IM would be 100%. All recipes used are presented in the next chapter, 3.3.1.

3.3.1 Recipes

Impact modifier raw material and PP product were both in a pellet form before the homogenisation, and all compounds were made using the mass percentage principle. The concentration range was based on the original recipe tested and approved for the function of the designed product. Recipe D is the same as in the commercial product.

The exact amount of IM in the recipes is not presented in this thesis for confidential reasons, but recipe D is similar to the one used in the commercial product. The range for the IM content was linear, and data points were chosen below and beyond the expected optimal ratio. The impact modifier concentrations were selected to increase linearly from recipe A to E.

From the impact modifier grade 3.2, recipes B, C and D were examined. The lowest and the highest concentrations (recipes A and E) were left outside of this study because it was expected that results from the middle of the scale would be the most important ones. The same recipes were used with every impact modifier material. The following table 2 presents more precisely how the samples were named.

TABLE 2. Summary of samples and the naming policy.

Recipes	3.1	5.7	3.2
A	3.1-A	5.7-A	-
B	3.1-B	5.7-B	3.2-B
C	3.1-C	5.7-C	3.2-C
D	3.1-D	5.7-D	3.2-D
E	3.1-E	5.7-E	-

In the following chapters, samples will be presented using the naming policy introduced in table 2.

3.3.2 Compounding and pelletising

In this study a twin screw extruder Prism 16T, produced by Thermo Fisher Scientific was used. The capacity of the extruder was approximately 2 kilograms per hour, and the homogenised strip was cooled in a water bath right after it came out of the nozzle. The cooled strip was directed to a rotating cutter, which chopped the strip into small pellets. More precise information about the parameters is presented in appendix number 3.

3.3.3 Injection moulding

Injection moulding was conducted by laboratory personnel due to its complex usage and lack of time for additional education. In this study, two types of specimens were injection moulded. Type A bodies (dog bones) for tensile and flexural properties analyses. Type B bodies were prepared for Vicat A softening point, Shore D hardness studies, and Charpy impact tests. Before measurements were performed, samples were stabilised for seven days in a standard air-conditioned room to ensure that the environment did not affect the mechanical tests.

3.4 Measurements

Measurements were performed gradually as the sample material was prepared. The basic tests were made from pellets, and all mechanical studies required sample pieces injection moulded. Table 3 shows all examinations and devices used for this study.

TABLE 3. Examination summary and devices used.

Examination	Sample type	Characteristic observed	Instrument
MFR	Pellet	Formability, flowability	Göttfert MI-4
DSC	MFR Strip	Melting and crystallisation point, crystallisation rate → polymer hardness	Mettler Toledo DSC 3 and 2
Density	Pressed plastic discs from pellets	Compound density	Mettler Toledo XPE105 Scale
Vicat A	Injection moulded Type A	Material softness in high temperature	Coesfeld HDT/Vicat Tester
Tensile test	Injection moulded Type A	Material durability during a pull, maximum level of stress	Zwick/Roell Z010
Flexural test	Injection moulded Type A	Material stiffness during bending	Zwick/Roell Z010
Charpy	Injection moulded Type B	Material resistance to shock	Zwick/Roell HIT5P
Shore D	Injection moulded Type B	Degree of material hardness	Bareiss Test Stand BS61-II

All results presented in the next chapter are averages of each measurement's duplicate data points. The number of data points varied between measures, but overall, from the basic test results, MFR included eight data points, and density had three. Vicat A and flexural and tensile properties included six data points in one measurement. The Charpy impact test was conducted with eleven parallel data points. The DSC results were evaluated, and the result was derived using integration from the graph.

3.4.1 Basic tests

Basic tests (MFR, DSC and density) were conducted from the homogenised pellets. Before further examinations, IM concentration was checked using Fourier-transform infrared spectroscopy (FTIR) to confirm the intended quality of the sample. Results with FTIR are not presented in this thesis's public version because of the recipe's sensitivity.

A specific amount of pellets is weighed and packed into a preheated rheometer cylinder which a temperature is 230 °C. After the polymer was melted, a piston pushed the melt polymer mass through a standard-sized die with a determined load. The melt mass flowing through the die is then calculated in grams per ten minutes. The melt flow index is determined using a capillary rheometer.

The density of each sample was determined from compression moulded plaque in accordance with ISO 1183-1 (SFS 1183-1 2019, 2). The pellets were compression moulded into a rectangular plate using a press with controlled heating and cooling profile. The density was measured from a die-cut circular sample disc. Samples were pre-conditioned for 40 hours in the standard-conditioned room before measurement.

The melting and crystallisation point of compounds were determined using a differential scanning calorimeter (DSC). Samples were cut from an MFR string which comes out through the die during the experiment. After the string cools down, a thin slice is cut, weighed and placed into an aluminium crubicle which is sealed by using a press machine designed especially for the DSC. An automated

sample feeder robot inserts the crubicle into the DSC oven. The sample goes through an endo- and exothermic reaction, whereas data is recorded and gathered in a thermogram format. Information regarding the sample and method used is entered into DSC software.

3.4.2 Mechanical tests

Mechanical tests were conducted using injection moulded specimen types A and B, presented in the previous paragraph 3.3.3. Specimens were air-conditioned for seven days and then tested in accordance with ISO 291 (SFS 291 2008, 3).

The material's tensile properties were tested using a type B specimen (dog bones) and mounted to the instrument from its grip sections, as figure six presents. The device stretches the specimen gradually while sensors detect the material's deformation, elongation, and breakage.

The flexural properties analysis required only a precise alignment of the specimen to the sample holder, and measurement was ready to be started. Both experiments (tensile and flexural properties) were measured with six specimens.

The Charpy impact test sample parts (type B specimen) were notched 24 hours before the experiment. The notching instrument used for this study was Ceast AN, Type 6899.000. The exact dimensions of the notch according to the standard ISO 179-1:2010 are presented in chapter 2.7.4. (SFS 179:1 2010, 5).

The Charpy impact test was conducted in four temperatures to simulate different weather conditions. Samples were conditioned in an ethanol bath for two hours to ensure that the temperature was stable in every specimen before the actual experiment. Measuring temperatures for this study were +23, 0, -20 and -30 °C degrees. The -20 and -30 °C were chosen to demonstrate how suitable the material is in the Nordic conditions.

During the measurement, each specimen was separately lifted from the sample rack and placed immediately in the sample holder, as presented in figure ten. The

pendulum was released right after the specimen had been positioned. The pendulum size was altered depending on a sample's IM concentration to ensure that the energy absorbed by the material was not too high (optimal absorbed energy 15-35 %).

In a Vicat A softening point analysis, samples were cut in a rectangular shape using type A specimen's grip sections (see figure 8.) Samples were placed under each test station, which included a needle and a temperature sensor. The HDT Vicat Tester instrument sank stations in the silicone oil bath where the temperature rose according to the method's slope.

4 RESULTS

4.1 General information on the result data

The results are presented in charts in this chapter due to the high amount of result data. The X-axis indicates the recipes from A to E, and the Y-axis is the scale of each analysis result. A commercial reference batch was selected to demonstrate how the compounds produced for this study differ from a production-scale polypropylene. The commercial reference is shown in the charts as a yellow bar. The in-use IM material was observed in this study to assess if there is a possibility to modify the commercial recipe.

The raw data is presented in appendix number 2. In the legend, IM types are shown as they are in tables one and two in chapter three. Sample F presents the commercial reference result in the charts to distinguish it from laboratory-scale compounded samples. This report does not present information regarding the analysis target values or range of the commercial PP material due to its confidentiality.

4.2 Results of the basic tests

Basic tests included melt flow index, density, melting and crystallisation point analyses with DSC. Overall, the melt flow rate results were within the accepted range, and the samples were easy to handle. Some difference between the in-use IM and new types is observed in the results, but they are still acceptable.

The higher MFR results with IM types 5.7 and 3.2 were expected because it was known that the index is somewhat higher due to the initial properties of the IM types. When mirroring results to the commercial reference, data points are in line with each other. If observing this analysis only, every recipe and IM-grade are giving promising results, which is a good sign when looking at the bigger picture. The MFR results are presented in figure number 11 below.

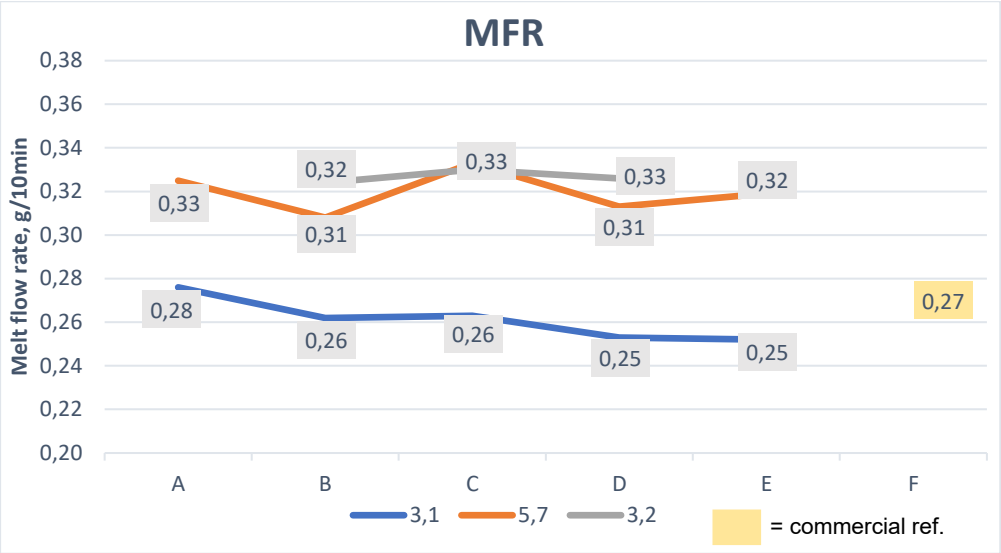


FIGURE 11. Melt flow rate results of the samples. The blue line represents the in-use IM, and the grey and orange lines are new IM types. The letters A to E are the recipe used with every IM type.

The results from the density analysis were also in the target when considering the quality parameters of the commercial product. It was expected that the density of the polymer would decrease when the IM concentration rose due to the IM material having a lower density than the basic PP product used for this study. The trend was overall linearly decreasing, and there was only a minor deviation between data points. To conclude, the lower IM concentration does not drastically affect the product's density. The results from the density analysis are presented in the following figure number 12.

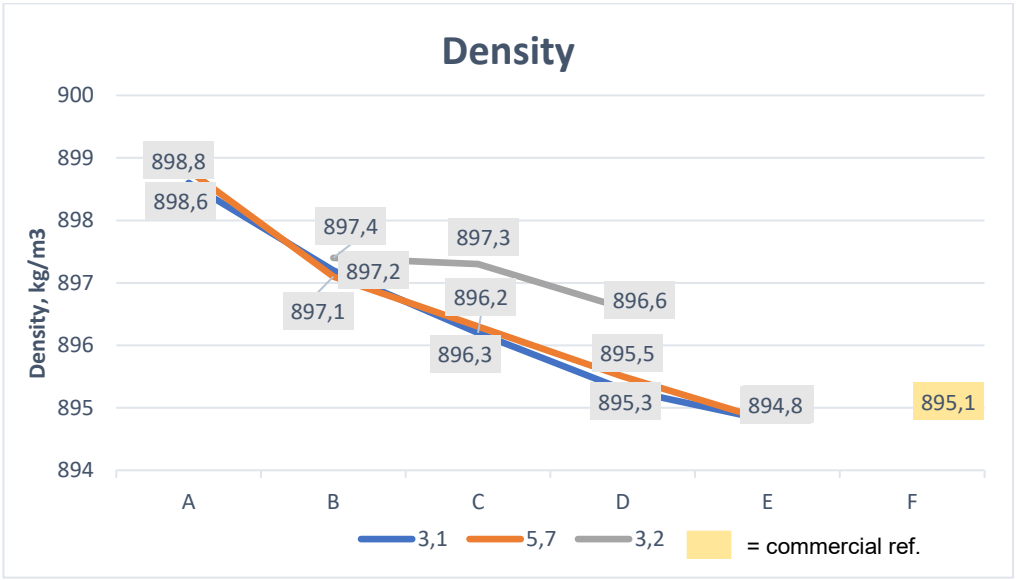


FIGURE 12. Density analysis results.

Melting and crystallisation points measured with DSC showed that samples performed as expected. Due to the sensitivity of the analysis method, a few degrees of deviation between the replicate results are typical and reflect that results were all in line with each other. (Höhne et al. 2010, 9.)

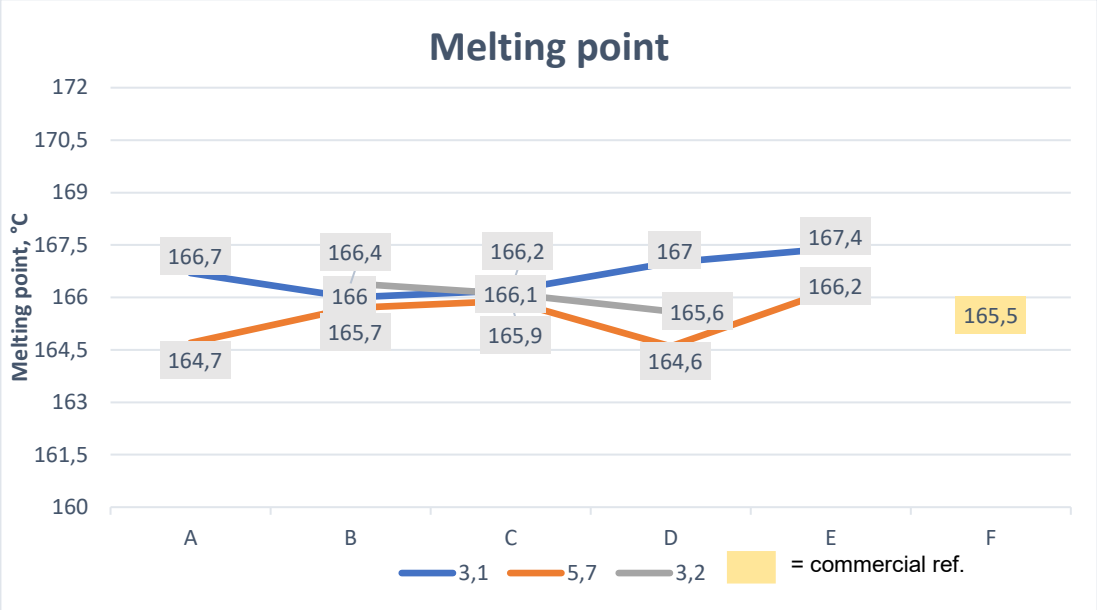


FIGURE 13. Melting point analysis results with DSC.

When comparing data points to the commercial reference, the results give promising signals with every recipe and IM type. To summarise, the changes in the IM concentration do not significantly affect the melting or crystallisation point. Figures 13 and 14 present melting and crystallisation point results with DSC.

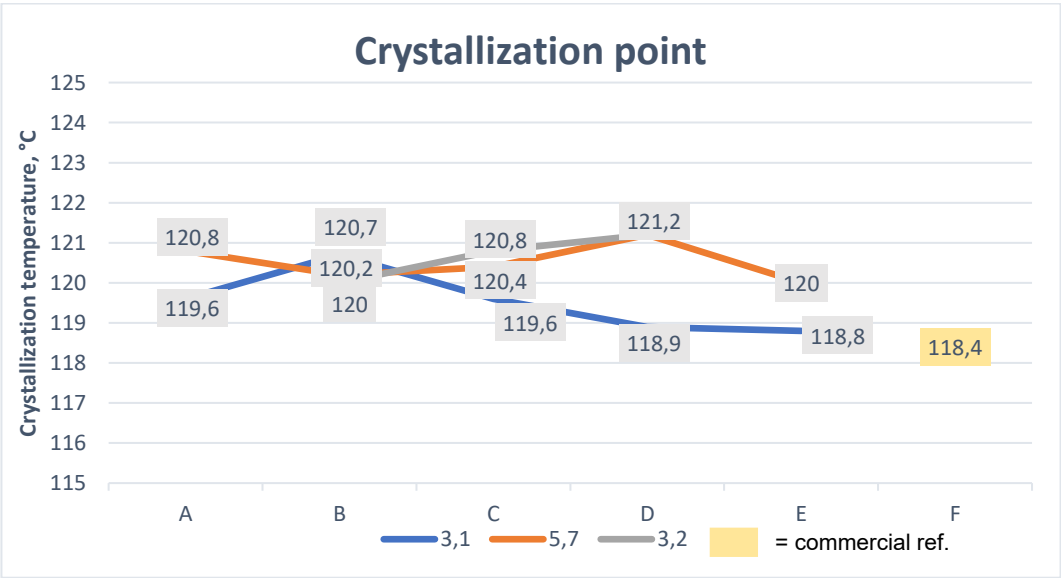


FIGURE 14. Crystallization point analysis results with DSC.

The crystal content of the polymer was observed in this study to ensure that the polymer's hardness decreases when the IM concentration changes, as presented in chapter 2.5.2.

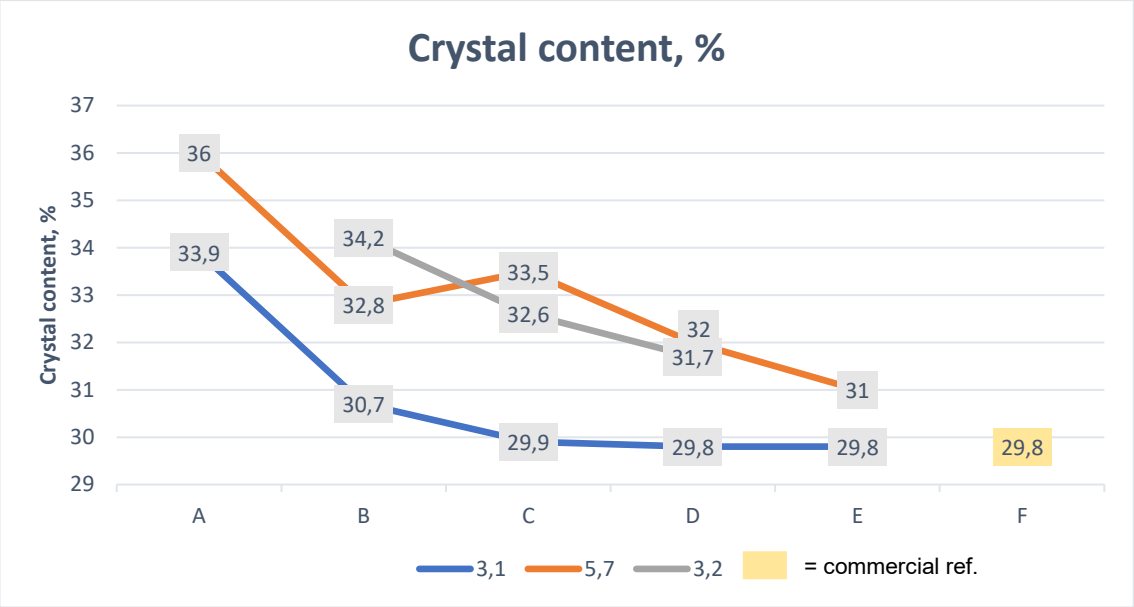


FIGURE 15. Crystallised fraction percentage in the samples.

Figure 15 indicates that the amorphous fraction in the samples correlates with the IM concentration. This result data provides essential information regarding the hardness of the sample and how the IM affects the properties of the polymer compounds.

Altogether when reviewing the measurement data from the basic tests, it can be stated that both 5.7 and 3.2 show potential results with every recipe. Nevertheless, it must be remembered that mechanical tests are the most critical when considering steel pipe coating's physical requirements and its function in general.

4.3 Results of the mechanical tests

The mechanical properties of the PP compounds were studied to find out how the compounds endure stress and pressure in different conditions. Mechanical analyses for this study are presented in table three in the previous chapter.

Tensile Modulus results are presented in the following figure, number 16. As chapter 2.7.2 presents, the tensile stress-strain analysis gives information on the material's stiffness. The higher the value is, the stiffer the material is. According to the quality requirements for this material, every recipe gives potential results because the minimum value is exceeded. This result generally means that even recipes A and B, consisting of the least IM, fulfil the quality requirements. Results are in line with each other, and the commercial reference is on the same scale. This graphical presentation indicates that changes in the IM concentrations of the samples significantly affect the properties of the polymer stiffness.

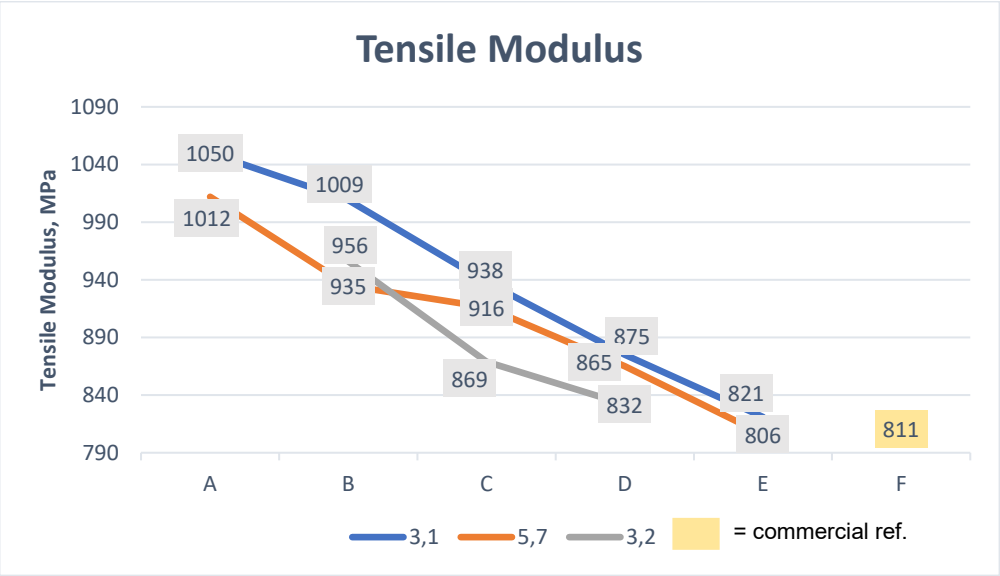


FIGURE 16. Tensile Modulus results.

The tensile stress reveals the material's capability to endure deformation when tensile force affects the specimen. It is the maximum stress the material can tolerate before it deforms permanently. The tensile strain presents the change in elongation of the sample during tensile stress in the form of a percentage relation. Figures 17 and 18 below show the tensile strain and stress results at yield.

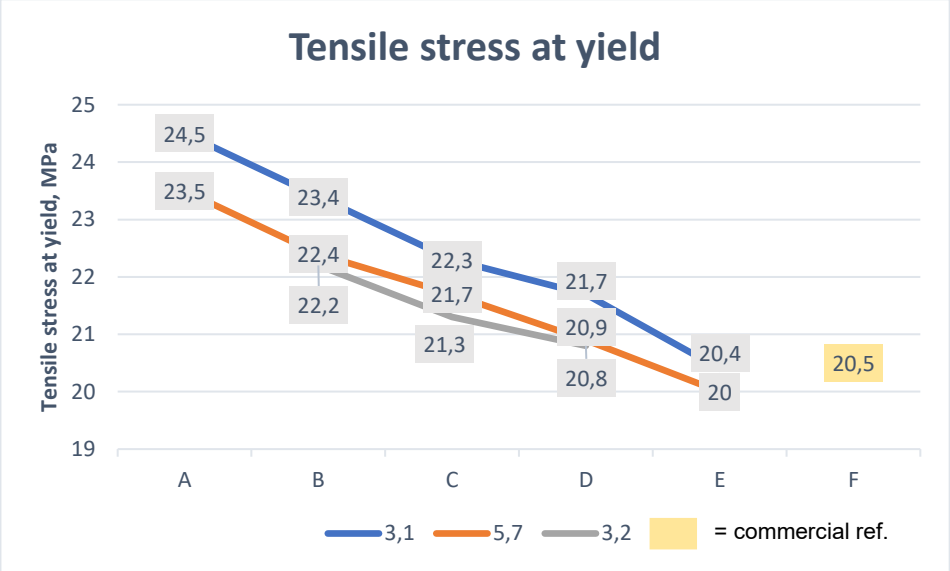


FIGURE 17. Tensile stress results at yield.

Tensile stress results show that the IM concentration directly affects the material's capability to withstand tensile force. As the graph shows, recipes A to C give too high results with every IM material, which means the material is too stiff. Types 5.7 and 3.2 are both closer to the target with recipes D or E. Results are also closer to the target than the in-use IM. This shows that the physical properties may be even better when using new raw materials with lower IM concentrations.

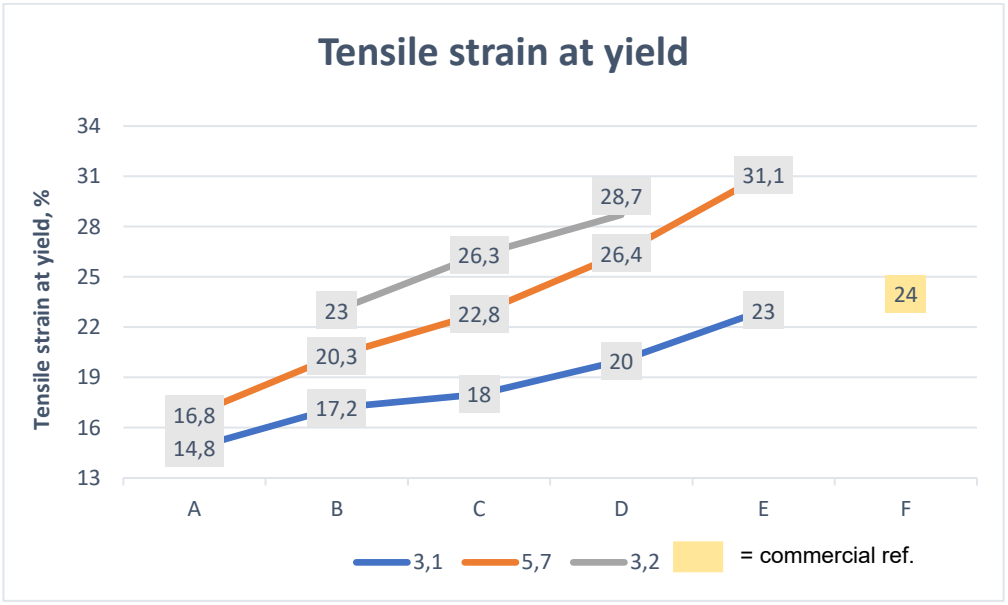


FIGURE 18. Tensile strain results at yield.

Tensile strain results are within an acceptable range, which is an optimistic sign. The compounds recover from deformation better than expected, and the lower

IM concentrations meet the quality requirements. When comparing the commercial reference and in-use IM to the new grades, results show that even the lower concentrations have potential because the result rate is systematically higher when comparing recipe C results with the IM types 5.7 and 3.2 to the in-use version.

The material's flexural properties provide information about material stiffness, as presented in chapter 2.8.3. The following figure 19 illustrates the results of the material flexural modulus. The data shows that the results are systematically too high with every IM material. Results are also decreasing as expected while the content of IM changes (the lower the result, the higher the flexibility). It means that the compounds are too flexible when comparing results to the commercial product. However, a high rate of flexibility is not that critical when observing the usage of this PP product.

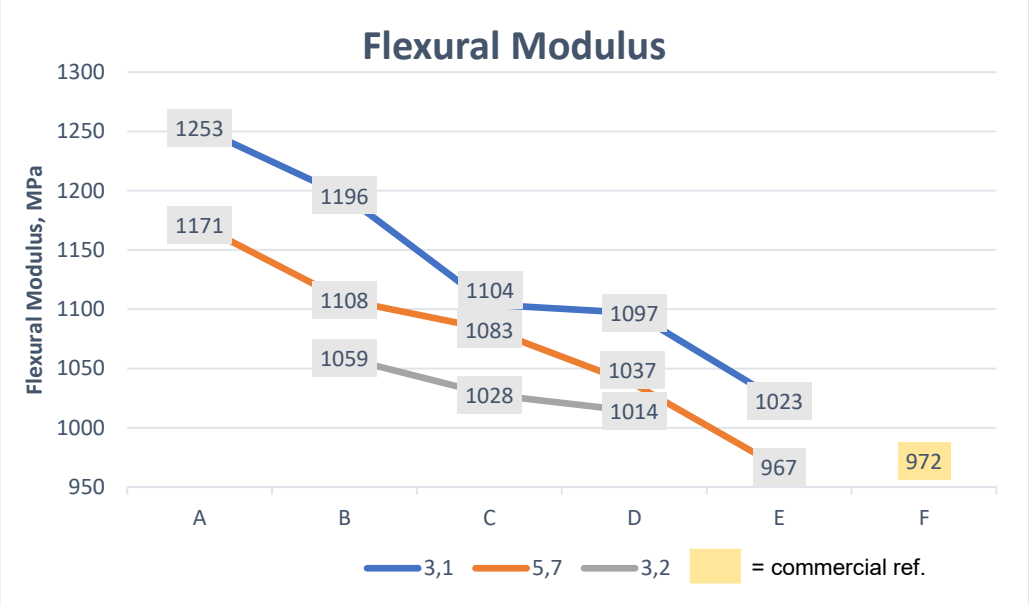


FIGURE 19. Flexural properties results.

Charpy impact results indicate the material's toughness when a loaded pendulum hits it. The following figures, 20, 21, 22, and 23, present the results of the experiment in temperatures 0 to -30 °C.

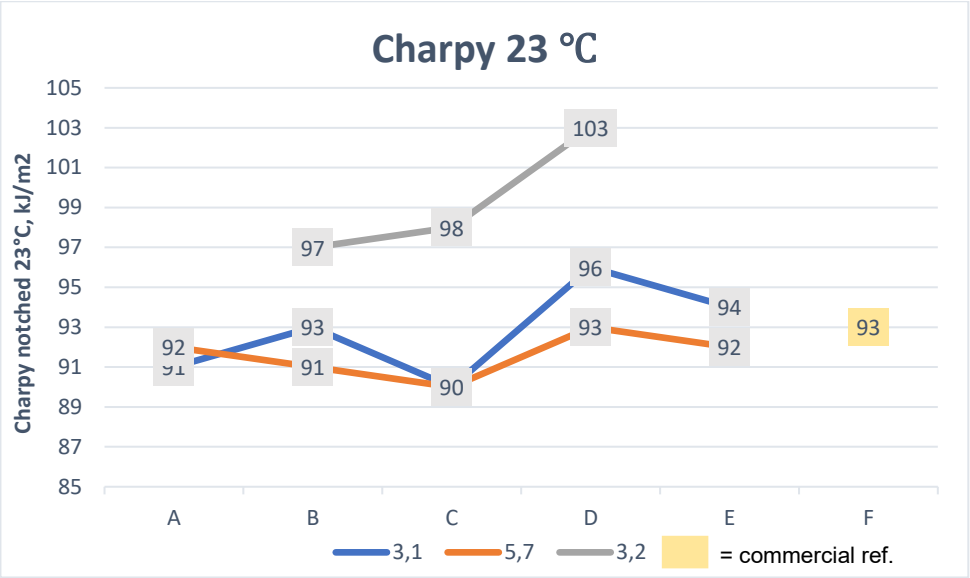


FIGURE 20. Charpy Impact test results, +23 °C.

The room temperature Charpy impact results were as expected, and the deviation scale was small. Somehow, IM type 3.2 gave higher results than any other raw material, indicating that the compounds were slightly softer than other IM types. The IM material 5.7 has the most potential compared to the commercial reference.

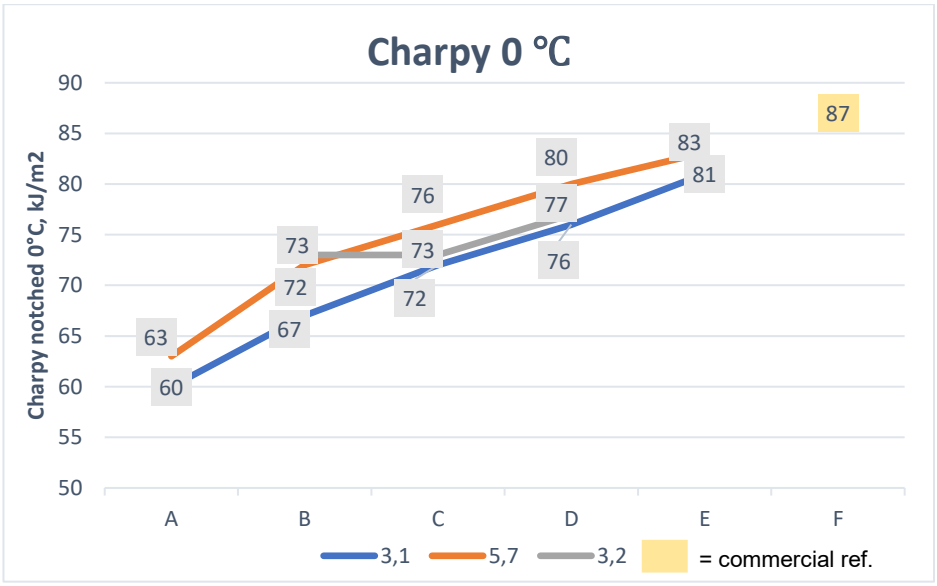


FIGURE 21. Charpy Impact test results, 0 °C.

The experiment in zero degrees proceeded as expected, and the results were overall acceptable. Figure 21 above presents the results of the measurement. The in-use IM is giving the lowest results, which means that the durability and

toughness of the 3.1 compounds are not at the same level with new IM types 5.7 and 3.2. The difference is marginal, but still, 5.7 and 3.2 are both having potential compared to the in-use version. This means that recipes B or C might work with new raw materials, especially type 5.7.

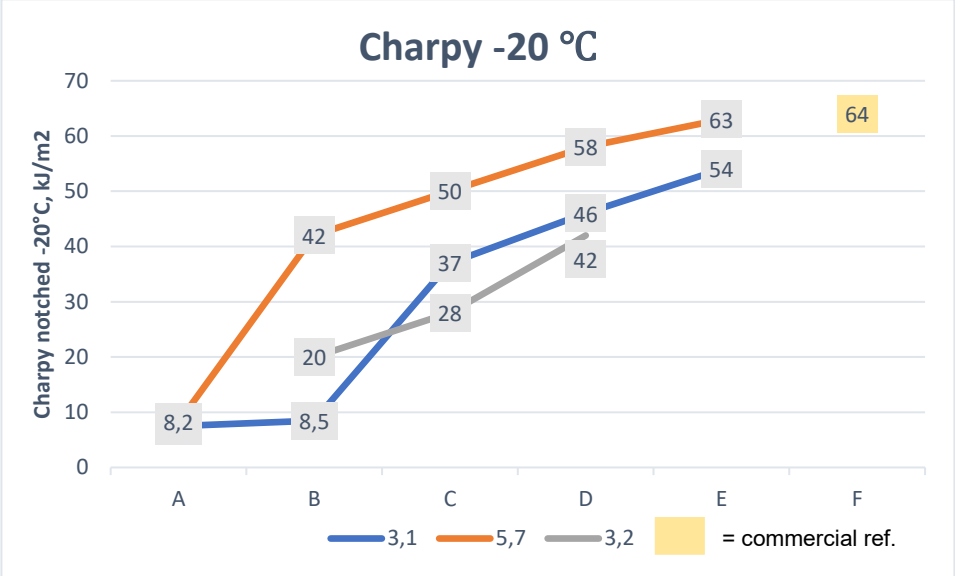


FIGURE 22. Charpy Impact test results, -20 °C.

The same trend continues in the below-zero measurements, as shown in figures 22 and 23. The IM material 5.7 stands out in these graphs with the best properties due to its higher result rate (the higher the result, the more likely the material endures the hit). These low-temperature measurements expose the differences between raw materials, and as we can see, 5.7 gives the most potential results overall.

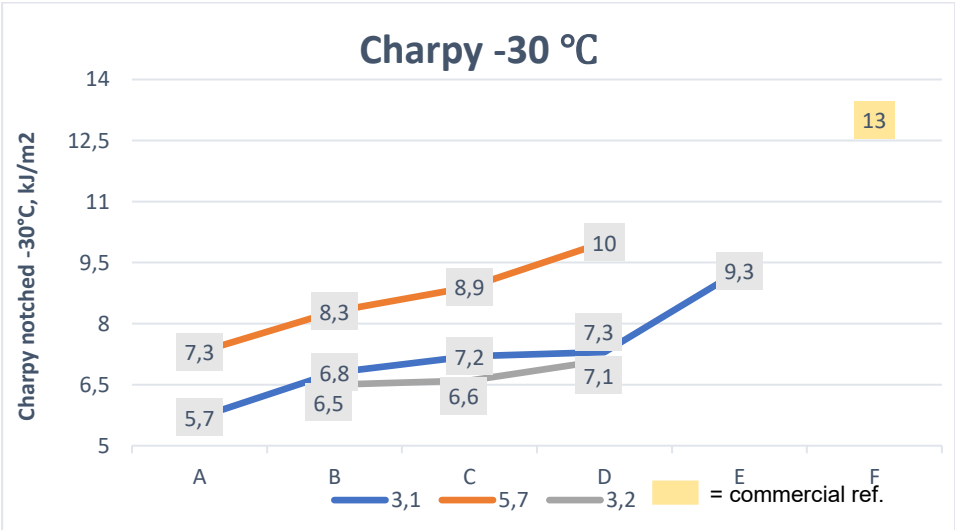


FIGURE 23. Charpy Impact test results, -30 °C.

The Vicat A softening point analysis showed a higher deviation between the replicate results, resulting in some data points being discarded. Nevertheless, all the results are still within the acceptable range. The results below 140 °C are a bit too low, but when considering the PP product function, this value is not too critical. Figure number 24 below presents the results of the softening point analysis.

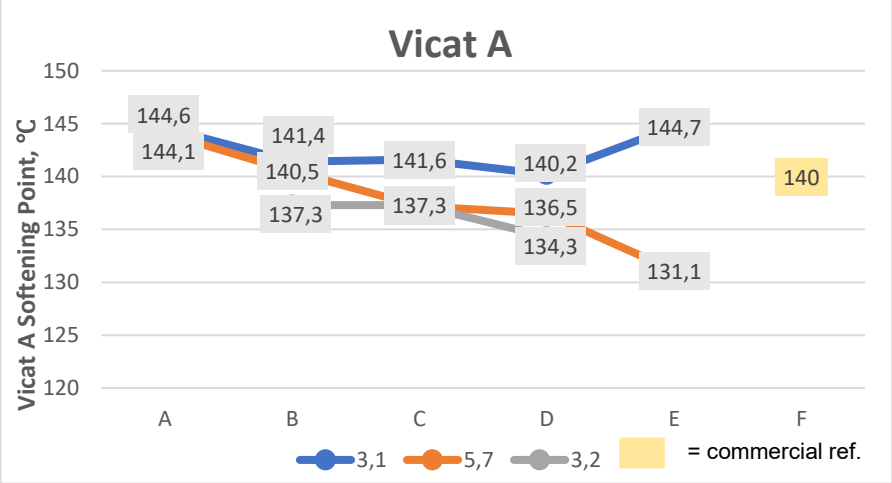


FIGURE 24. Vicat A Softening point analysis results.

The Shore D hardness analysis results are generally approvable. Figure 25 shows that the IM type 5.7 is resulting the compound being slightly harder than other materials. This discovery may have a relation with the higher Charpy results as well. The results with recipes A to C are systematically higher than expected, which means they are too rigid when considering the quality parameters. Recipes D and E are giving promising results with both 5.7 and 3.2.

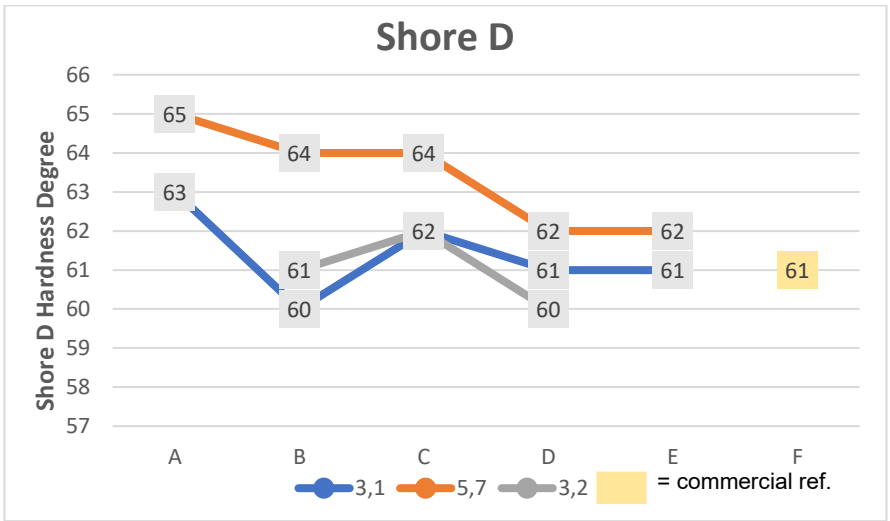


FIGURE 25. Shore D hardness degree results.

5 DISCUSSION AND CONCLUSIONS

To conclude, this study shows that results with basic tests are generally promising. They revealed that the impact modifier recipe could be modified because the results are mainly in the same range as the reference, even though the IM concentration scale is relatively high. When observing the basic analysis in a bigger picture, there are no "limiting factors" whose properties did not meet the quality parameters. Altogether, more tests will need to be made to ensure that there are no restrictions with any IM material.

Initially, a sweep rheology analysis was part of the study plan. Unfortunately, it had to be dropped from this report due to instrument failure, which caused a delay to receive the result data. The rheological analysis provides more information about the polymer's flowability, molecular weight distribution and chain branching type than other physical or chemical methods. Measurements are used to differentiate polymer grades and are essential when discussing computer-simulated injection moulding or extrusion in modern processes. (Polychronopoulos et al. 2018, 5.) Due to the amount of crucial information from the rheological analyses, the analysis is recommended to be incorporated into the future research plan.

Scanning electron microscopy (SEM) provides essential information about the dispersion of IM particles and basic polymer in the specimen surface. SEM is primarily used to observe the fracture and failure of mechanics as well as the particle's size and shape. With the SEM analysis, the differences between specimens A to E can be studied to understand morphological differences between compounds. From high-quality images, the aberration in the morphology and homogeneity of the material can be ensured if unusual data is noticed. (Guise et. al. 2011, 1278-1285.) For example, this observation could be advantageous between IM types 5.7 and 3.1.

When considering the mechanical properties analysis data, the most critical parameters are obtained from the tensile, flexural and Charpy impact tests. The product must withstand high pressure and endure dropping from an elevated

surface. Primarily the Charpy impact analysis simulates the tendency of the material to withstand such forces in different weather conditions. Especially this parameter is crucial when considering the usage of the PP product.

The Charpy measurements below zero degrees differentiate the characteristics of the materials examined most efficiently. As the Charpy results present, the rate of the results with both IM 3.2 and 5.7 are relatively higher than the in-use IM 3.1, indicating better shockproof properties. Especially, differences are remarkable between the 5.7 and 3.1 in the -20 °C measurement where the sample 3.1-B result was 8,5 kJ/m^2 and 5.7 with the same IM concentration was 42. This result indicates that the 5.7 has nearly five times better capability to absorb energy from a directed hit, which is an important discovery when investigating new potential material alternatives.

The same trend also occurs in the Charpy test -30˚C, but the IM 3.2 does not meet the quality expectations anymore, and a steep decrease in the results is observed. As can be concluded from the results, the 3.2 mechanical properties are acceptable and promising at room temperature, but the issues appear when circumstances change. For this reason, IM 3.2 is not the best option for the PP product when looking at the bigger picture, even though the basic tests and some mechanical properties are in line with the quality parameters.

Even with recipes B or C, the Charpy results are mainly in the target, indicating a possibility for recipe modification. At the same time, the product's properties are maintained at the same level or even better. Impact modifier 5.7 mechanical properties are systematically standing out from the result data, and the Charpy impact test confirmed that the material has the best properties of the samples overall.

To perceive the differences between materials and their properties, Figure 26 below presents the Charpy impact (-20˚C) and tensile properties gathered in the same chart. It is known that as the Charpy impact result increases, the sample's tensile properties (tensile modulus) decrease (Sugimoto et al. 2018, Chapter 2.3). The phenomenon can be explained as the softness of the material increases, the shock absorbing features improves but the value of the stiffness reduces.

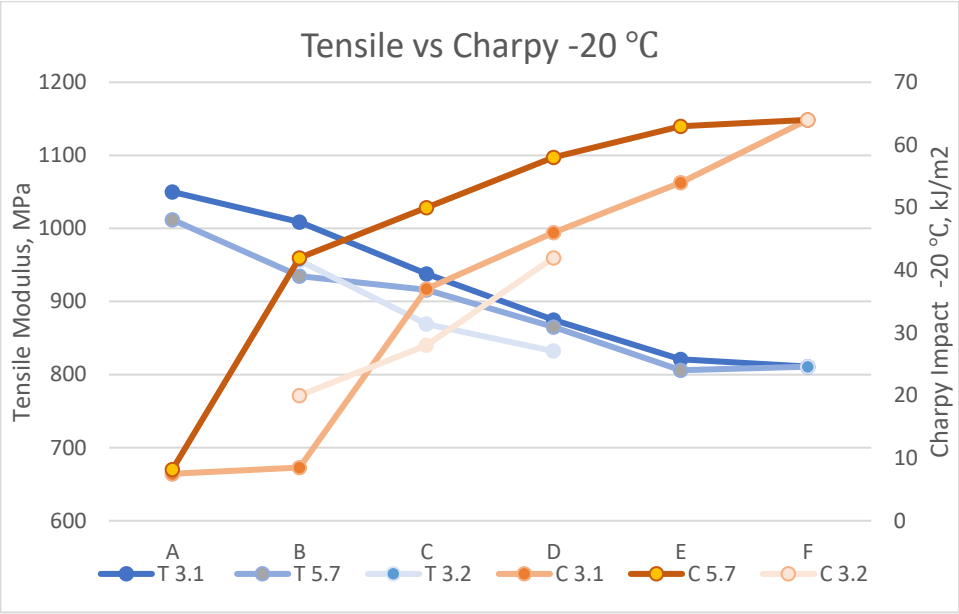


FIGURE 26. Tensile Modulus vs Charpy Impact (-20˚C) results comparison.

In the figure above, the blue lines represent tensile modulus results and the red lines correspondingly the Charpy impact results. When considering the optimal ratio of the IM 5.7, the Charpy result is already in the target with recipe A, and the minimum value for the tensile modulus is exceeded with every recipe. It must be remembered that the IM recipes A and B concentration is relatively low, and the material is slightly too stiff. To conclude, recipe C would be the most suitable and cost-efficient option with the 5.7 when considering the whole picture. Table 4 below sums up the conclusions of this study for all three impact modifiers focusing on the mechanical properties results.

TABLE 4. The result summary for the impact modifiers

Impact Modifier	Conclusion
3.1	Recipes D (in-use) and E gave the best results when considering the whole test data. → Unnecessary to alter the IM concentration → **The recipe D maintains to be the most optimal version**
5.7	Mechanical properties were promising to start from recipes B and C. → Further examinations are required, and there is the possibility of proceeding to the test run phase → **The best overall potential and cost-efficiency**
3.2	Mechanical properties were mainly in target starting from recipe C. → Weak results with Charpy -30˚C → The material does not perform as expected in the extreme environment → The results are near to the in-use IM (3.1) → **Is it necessary to replace the in-use IM with material 3.2?**

As presented in the previous table, it can be concluded that the target set for this study was achieved, and material differences were discovered. The aim was to observe how new materials affect polypropylene properties, and straightforward deductions were conducted from the analysis data.

Regarding the impact modifier 5.7, which turned out to be technically the most suitable and it might be the most cost-efficient option compared to other IM types. In the future, there will be further examinations to confirm this discovery. Borealis Polymers Oy will utilise the result data collected in this study as a basis when planning a production-scale test run.

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APPENDICES

Appendix 1. Result data basic tests

TABLE 5. Melt flow rate result data.

Melt Flow Rate, g/10min			
Recipe	**3.1**	**5.7**	**3.2**
A	0,28	0,33	-
B	0,26	0,31	0,32
C	0,26	0,33	0,33
D	0,25	0,31	0,33
F	0,25	0,32	-
Commercial Ref. (F)	0,27	0,27	0,27

TABLE 6. Density result data.

Density, kg/m3			
Recipe	**3.1**	**5.7**	**3.2**
A	898,6	898,8	-
B	897,2	897,1	897,4
C	896,2	896,3	897,3
D	895,3	895,5	896,6
E	894,8	894,8	-
Commercial Ref. (F)	895,1	895,1	895,1

TABLE 6. Melting point result data.

Melting Point, °C			
Recipe	**3.1**	**5.7**	**3.2**
A	166,7	164,7	-
B	166	165,7	166,4
C	166,2	165,9	166,1
D	167	164,6	165,6
E	167,4	166,2	-
Commercial Ref. (F)	165,5	165,5	165,5

TABLE 8. Crystallisation point result data.

Crystallisation Point, °C			
Recipe	**3.1**	**5.7**	**3.2**
A	119,6	120,8	-
B	120,7	120,2	120
C	119,6	120,4	120,8
D	118,9	121,2	121,2
E	118,8	120	-
Commercial Ref. (F)	118,4	118,4	118,4

TABLE 9. Crystalline content in polymer result data.

Crystal content, %			
Recipe	**3.1**	**5.7**	**3.2**
A	33,9	36	-
B	30,7	32,8	34,2
C	29,9	33,5	32,6
D	29,8	32	31,7
E	29,8	31	-
Commercial Ref. (F)	29,8	29,8	29,8

Appendix 2. Result data mechanical tests

TABLE 10. Tensile test results for the IM 3.1

Impact Modifier 3.1			
Recipe	**Tensile Modulus, MPa**	**Tensile stress at yield, MPa**	**Tensile strain at yield, %**
A	1050	24,5	14,8
B	1009	23,4	17,2
C	938	22,3	18
D	875	21,7	20
E	821	20,4	23
Commercial Ref. (F)	811	20,5	24

TABLE 11. Tensile test results for the IM 5.7

Impact Modifier 5.7			
Recipe	**Tensile Modulus, MPa**	**Tensile stress at yield, MPa**	**Tensile strain at yield, %**
A	1012	23,5	16,8
B	935	22,4	20,3
C	916	21,7	22,8
D	865	20,9	26,4
E	806	20	31,1
Commercial Ref. (F)	811	20,5	24

TABLE 12. Tensile test results for the IM 3.2

Impact Modifier 3.2			
Recipe	**Tensile Modulus, MPa**	**Tensile stress at yield, MPa**	**Tensile strain at yield, %**
B	956	22,2	23
C	869	21,3	26,3
D	832	20,8	28,7
Commercial Ref. (F)	811	20,5	24

TABLE 13. Flexural modulus result data.

Flexural Modulus, MPa			
Recipe	**3.1**	**5.7**	**3.2**
A	1253	1171	-
B	1196	1108	1059
C	1104	1083	1028
D	1097	1037	1014
E	1023	967	-
Commercial Ref. (F)	972	972	972

P = Partial Break

C = Complete Break

TABLE 14. Charpy Impact result data for the IM 3.1

Impact Modifier 3.1								
Recipe	**Charpy kJ/m2, 23 ℃**	**Break type, 23℃**	**Charpy 0℃**	**Break type 0℃**	**Charpy -20 ℃**	**Break type, -20 ℃**	**Charpy -30 ℃**	**Break type, -30 ℃**
A	91	P	60	P	7,5	C	5,7	C
B	93	P	67	P	8,5	P	6,8	C
C	90	P	72	P	37	P	7,2	C
D	96	P	76	P	46	P	7,3	C
E	94	P	81	P	54	C	9,3	C
(F)	93	P	87	P	64	P	13	C/P

TABLE 15. Charpy Impact result data for the IM 5.7

Impact Modifier 5.7								
Recipe	**Charpy kJ/m2, 23 ℃**	**Break type, 23℃**	**Charpy 0℃**	**Break type 0℃**	**Charpy -20 ℃**	**Break type, -20 ℃**	**Charpy -30 ℃**	**Break type, -30 ℃**
A	92	P	63	P	8,2	C	7,3	C
B	91	P	72	P	42	P	8,3	C
C	90	P	76	P	50	P	8,9	C
D	93	P	80	P	58	P	10	C
E	92	P	83	P	63	P		C
(F)	93	P	87	P	64	P	13	C/P

TABLE 16. Charpy Impact result data for the IM 3.2

Impact Modifier 3.2								
Recipe	**Charpy kJ/m2, 23 ℃**	**Break type, 23℃**	**Charpy 0℃**	**Break type 0℃**	**Charpy -20 ℃**	**Break type, -20 ℃**	**Charpy -30 ℃**	**Break type, -30 ℃**
B	97	P	73	P	20	P	6,5	C
C	98	P	73	P	28	P	6,6	C
D	103	P	77	P	42	P	7,1	C
(F)	93	P	87	P	64	P	13	C/P

TABLE 17. Vicat A Softening Point result data.

Vicat A Softening Point, ℃			
Recipe	**3.1**	**5.7**	**3.2**
A	144,6	144,1	-
B	141,4	140,5	137,3
C	141,6	137,2	137,3
D	140,2	136,5	134,3
E	144,7	131,1	-
Commercial Ref. (F)	140,0	140	140

TABLE 18. Shore D Hardness result data.

Shore D Hardness			
Recipe	**3.1**	**5.7**	**3.2**
A	63	65	-
B	60	64	61
C	62	64	62
D	61	62	60
E	61	62	-
Commercial Ref. (F)	61	61	61

Appendix 3. Prism extruder, parameters

BOREALIS POLYMERS OY — **TEST RUN REPORT**
COMPOUNDING **PRISM TSE-16-TC** **L/D 25:1**

Project **Originator** **Title**
Done by VPS/ **Week** 36 **Year** 2022

Sample		3.2-B	3.2-C	3.2-D	3.2-D/2	3.2-B/2	3.2-C/2
Date		6.9.22	7.9.22	7.9.22	7.9.22	9.9.22	9.9.22
Screw							
Feeder set		1.0	1.0	1.0	0,95	0,95	0,95
Feeder kg/h		2.0	2.04	1,95	1,95	2.0	2.0
Screw / rpm		250	250	250	250	250	250
Cutter / rmp							
Melt Pressure / bar		40	42	41	40	40	40
Melt Temp C		248	250	248	248	249	248
Torque Nm		~ 11	~11	~11	~ 11	~ 11	~ 11
Torque %		50	50	50	50	50	50
SEI = kwh / kg		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

Zone	Temp C	actual	actual	actual	actual	actual	actual
1	210	210	210	210	210	210	210
2	220	220	220	220	220	220	220
3	230	230	230	230	230	230	230
4	230	231	231	231	231	231	231
5	230	230	230	230	230	230	230
N2 flush		YES	YES	Yes	Yes	Yes	Yes
Water / air cooling		W	W	W	W	W	W

Comments

3.2-B :
3.2-C :
3.2-D :

The more precise information regarding the ratio between IM and the polypropylene product has been removed from this version.

3.2-D/2 : 2. homogenisation 3.2-D
3.2-B/2 : 2. homogenisation 3.2-B
3.2-C/2 : 2. homogenisation 3.2-C

Prism max rpm = 500
max motor power = 1.25 kw

SEI = rpm / max rpm x torguet % x motor max power kw / outcome kg/h

Appendix 5. DSC evaluated graph