

Tatu Sammalisto

**INFLUENCE OF ADDITIVATION ON  
CRYSTALLINITY, WATER VAPOR RE-  
SISTANCE, AND TENSILE PROPERTIES  
OF A POLYOLEFIN FILM**

Master's Thesis  
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October 2025

# ABSTRACT

Tatu Sammalisto: Influence of Additivation on Crystallinity, Water Vapor Resistance, and Tensile Properties of a Polyolefin Film

Master of Science Thesis

Tampere University

Materials Science

October 2025

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Polymers have outstanding properties in different applications in construction. Their versatility and ability to be subtly tailored is a property not many other types of materials possess. Crystallinity is commonly known well, but its effects on polymer films may certainly be understood deeper. Furthermore, the adjustment of crystallinity is an important topic in polymer science and there may never be too much information about it. Waterproofing needs to be taken into account whenever a new building is set to be constructed, and its importance may never be emphasized too highly. Proper waterproofing increases the lifespan of buildings and decreases the possible health-risks the users of a building may be exposed to. Even though waterproofing films nowadays are reliable when used properly, they certainly may be developed further to prevent water vapor damage in buildings even further. Higher degree of crystallinity of polymer films commonly leads to higher water vapor resistance, but may compromise the tensile properties of the film, leading to declines in usability.

This study was completed to gain information about how different properties of a polypropylene film may be influenced by means of adjusting its contents. The main focus of this study is to evaluate how the water vapor barrier of the film may be altered by two separate methods: replacing some of the polypropylene with high-density polyethylene, and decreasing the amount of calcium carbonate filler in the film. Additional factors that were evaluated, were degree of crystallinity, and tensile properties. The correlation between crystallinity and water vapor barrier was a very important topic in this study. Additionally, the changes in tensile properties along with additivation are evaluated, since the usability of this waterproofing film needs to remain at a suitable level.

The films used in this research were manufactured manually by mixing the components, calendering, and collecting the films from the calender. Degrees of crystallinity of the films were measured by differential scanning calorimetry. The tensile properties were determined by tensile testing. Finally, the water vapor barrier properties of the films were measured by a specific water vapor permeability test.

This study concluded that adjusting the contents of the polyolefin film greatly affects its water vapor barrier and tensile properties. The replacement of PP with PE as well as subtraction of the  $\text{CaCO}_3$  filler led to remarkable increases in water vapor resistance. Even though changes in ductilities and toughnesses of the films were detected in both adjustments, they remain insignificant. It was determined that the stiffness of the film increased notably along with added PE and decreased slightly with subtracted  $\text{CaCO}_3$ . However, these changes could not be detected when examined by hand. Based on the results obtained in this study, the addition of PE into the PP film greatly improves its water vapor resistance, maintains satisfactory usability, and is a more sustainable, effective and cost-effective method when compared to the subtraction of  $\text{CaCO}_3$ .

Keywords: Additivation, Crystallinity, Polymer, Polymer Film, Polyolefin, Polyolefin Film, Waterproofing

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# TIIVISTELMÄ

Tatu Sammalisto: Seostamisen vaikutus polyolefiinikalvon kiteisyyteen, vesihöyrynvastukseen ja lujuusominaisuuksiin

Diplomityö

Tampereen yliopisto

Teknisten tieteiden koulutus - Materiaalitekniikka

Lokakuu 2025

Polymeereillä on erinomaisia ominaisuuksia erilaisiin rakentamiseen liittyviin käyttökohteisiin. Monipuolisuutensa ja muokattavuutensa vuoksi ne erottuvat edukseen monien muiden materiaalien joukosta. Kiteisyys ominaisuutena on yleisesti tunnettu, mutta sen monista vaikutuksista polymeerikalvoihin voisi ehdottomasti saada laajempaa tutkimustietoa. Lisäksi, kiteisyyden räätälöinti on tärkeä seikka polymeeritieteessä, eikä siihen liittyvästä lisätiedosta voi koskaan olla haittaa. Vedeneristys on aina otettava huomioon uuden rakennuksen suunnitteluvaiheessa, eikä sen tärkeyttä voi alleviivata liikaa. Kunnollisesti tehty vedeneristys sekä kasvattaa rakennuksen pitkäikäisyyttä, että laskee rakennuksessa aikaa viettävien ihmisten altistumista mahdollisille terveysriskeille. Vaikka vedeneristyskalvot ovat nykyään luotettavia kunnollisesti käytettyinä, niitä todellakin voidaan jatkokehittää entisestään vastustamaan kosteusvaurioita rakennuksissa vieläkin paremmin. Polymeerikalvojen korkeampi kiteisyysaste johtaa usein korkeampaan vesihöyrynvastukseen, mutta saattaa samalla heikentää kalvojen lujuusominaisuuksia johtaen heikentyneeseen helppokäyttöisyyteen.

Tämä työ toteutettiin, jotta polypropeenikalvon erilaisten ominaisuuksien muuttamisesta lähtöainepitoisuuksiin vaikuttamalla saataisiin kattavaa tutkimustietoa. Työn pääasiallinen tavoite oli arvioida, kuinka polypropeenikalvon vesihöyrynvastukseen voidaan vaikuttaa kahdella eri tavalla: korvaamalla osa polypropeenista polyeteenillä, sekä vähentämällä täyteaineena toimivan kalsiumkarbonaatin määrää. Tutkittavia aiheita työn aikana olivat myös kiteisyysaste sekä lujuusominaisuudet. Kiteisyysasteen ja vesihöyrynvastuksen korrelaation arviointi oli myös erittäin tärkeä seikka tässä tutkimuksessa. Lisäksi lisäaineistuksen aiheuttamia muutoksia lujuusominaisuuksiin tarkasteltiin, sillä työhön liittyvän vedeneristyskalvon käytettävyyden tulee säilyä halutulla tasolla.

Kalvot, joita tässä työssä käytettiin, valmistettiin käsin sekoittamalla tarvittavat ainekset, sulattamalla ne kalanterissa ja keräämällä kalvot talteen kalanterista. Kalvojen kiteisyysasteet mitattiin differentiaalisella pyyhkäisykalorimetrialla. Lujuusominaisuudet määritettiin vetokokeilla. Vesihöyrynvastukset kalvoille mitattiin erityisellä vesihöyrynläpäisytestillä.

Tässä työssä saatiin selville, että tämän polypropeenikalvon sisällön muokkaaminen vaikuttaa voimakkaasti sen vesihöyrynvastukseen ja lujuuteen liittyviin ominaisuuksiin. Sekä polypropeenin korvaus polyeteenillä että täyteaineena toimivan kalsiumkarbonaatin vähentäminen johti valtaviin parannuksiin vesihöyrynvastuksessa. Vaikka molemmissa tapauksissa havaittiin muutoksia sitkeydessä ja venyvyydessä, ne jäivät merkityksettömälle tasolle. Kalvon jäykkyys kasvoi merkittävästi polyeteeniä lisätessä ja laski hieman kalsiumkarbonaattia poistaessa, mutta muutoksia ei voinut havaita käsin tutkimalla. Tämän tutkimuksen tulosten perusteella polyeteenin lisääminen polypropeenikalvoon parantaa sen vesihöyrynvastusta merkittäväällä tavalla, pitää kalvon käytettävyyden hyväksytyllä tasolla ja on kestävämpi, tehokkaampi ja taloudellisempi keino verrattuna kalsiumkarbonaatin eli täyteaineen määrän vähentämiseen.

Avainsanat: Kiteisyys, polymeeri, polymeerikalvo, polyolefiini, polyolefiinikalvo, seostus, vedeneristys

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## PREFACE

This Master's Thesis project was completed in cooperation with Kiilto Oy. Firstly, I want to collectively thank everyone at Kiilto for making the duration of my thesis project pleasant. My greatest gratitude belongs to my thesis supervisors, PhD Sarianna Palola, and Doctoral Student Iida Kangashaka for their continuous attention and support throughout the thesis work.

I want to thank everyone involved in the sampling and testing of materials during my thesis. I would not have succeeded without the aid of every single one of you. I want to thank Markku Vihanta for comprehensive guidance during sample preparation, Essi Korhonen for the great aid in water vapor permeability and tensile tests, Siru Rohola for helping me understand and analyze the results of water vapor permeability tests. Additionally, many thanks to everyone else even slightly involved in the thesis project.

This project concludes my studies in Tampere University, and I want to thank my family and friends for the support during my time at the University. I also owe a huge amount of gratitude towards Marissa, my dear girlfriend, who unconditionally has been there for me during my studies.

Tampere, 20 October 2025

Tatu Sammalisto

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## LIST OF SYMBOLS AND ABBREVIATIONS

$^{\circ}\text{C}$	temperature in Celsius
$\mu$	water vapor resistance factor
$A$	cross-sectional area
$\text{CaCO}_3$	calcium carbonate
$d$	thickness of water vapor permeability test samples
$DSC$	differential scanning calorimetry
$F$	force
$G$	water vapor flow rate
$HDPE$	high-density polyethylene
$\text{J/g}$	joule per gram
$\text{K/s}$	kelvin per second
$L$	final length
$L_0$	initial length
$m$	mass
$PE$	polyethylene
$PP$	polypropylene
$rHDPE$	recycled high-density polyethylene
$\text{rpm}$	revolutions per minute
$s_d$	water vapor diffusion-equivalent air layer thickness
$t$	time
$W$	water vapor permeance
$X_c$	degree of crystallinity
$Z$	water vapor resistance
$\delta$	water vapor permeability
$\Delta H(T_m)$	enthalpy of fusion of polymer at its melting temperature
$\Delta H(T_m)^0$	enthalpy of fusion of a fully crystalline polymer at its melting temperature
$\Delta m_i$	mass change rate
$\Delta p$	partial pressure difference
$\epsilon$	strain
$\sigma$	stress

# 1. INTRODUCTION

The history of polymers dates back to the first phases of life on earth. In other words, polymers have been present for a significantly longer time period than the mankind as we know it [1]. However, the earliest forms of polymers are restricted to natural polymers that were present in the form of e.g., cellulose, starch, and different proteins [2]. The use of polymers has been utilized by humans for several thousand years, since there's evidence of the use of natural fibers as early as in the Stone Age [3]. It may easily be figured out that polymers have played a crucial role when it comes to the development of life since every living organism contains proteins and they are one of the core components of human reproduction [4]. Based on the fact that nature uses polymers so diversely, it's very understandable that mankind has grown to use and modify polymers effectively and widely. Furthermore, polymers hold outstanding properties, such as versatility, cost-effectiveness, renewability and lightness, which enable the potential that may not necessarily be achieved with other materials [5,6]. Taking into account the previously mentioned factors, polymer research is a topic of very high importance when it comes to the further development of materials science and industry. While on the subject, this study concentrates on two synthetic, man-made polymers and their properties as well as their applications.

Polymers are complex materials that have several different factors which determine their properties and nature [7]. As mentioned above, people have had the ability to tailor polymers to their advantage and this craft continues to develop to this day. The first man-made, completely synthetic polymer dates all the way back to 1907 as a highly versatile polymer, Bakelite, was developed by means of polycondensation of phenol with formaldehyde [3,8].

According to several studies [7,9–11] discussing degree of crystallinity, it may be determined that it significantly affects both physical and chemical properties of polymers. Commonly the degree of crystallinity of a polymer lowers as its molecular weight increases. This happens because the structure of the polymer becomes more disoriented and thus crystalline structure becomes more difficult to be formed. As the degree of crystallinity of a polymer increases, its tensile strength and toughness increase and on the other hand, the brittleness within its system also becomes relevant. Amorphous polymers may also be initially brittle, particularly at low temperature levels [12]. Additionally, the

increased degree of crystallinity affects the glass transition temperature of a polymer by increasing it, which highly alters its usability in elevated temperatures. The most challenging factor of this study is the change in mechanical properties of the polymer film as the water vapor barrier increases. The compromising of mechanical properties regarding the application in this study includes increased stiffness and brittleness. Both of the mentioned properties commonly rise along with elevated degree of crystallinity [13,14]. The previously mentioned factors are not favorable considering the polymer film application in this study, since flexibility is essential to remain.

This study was conducted so that the degree of crystallinity as well as its practical effects on a polypropylene (PP) film and its properties could be understood better. The study consists of a theoretical literature review and a practical, experimental part. In the literature review, polymers, crystallinity and waterproofing applications are discussed. The practical research of this study concentrated on modifying the degree of crystallinity of a polypropylene film and evaluating its suitability for waterproofing applications. The modification of the film occurred via varying the relative amounts of polypropylene and two different additives: polyethylene (PE), and calcium carbonate ( $\text{CaCO}_3$ ). The polyethylene, which was used in this research, was post-consumer recycled high-density polyethylene (rHDPE), which was used as proportional replacement for the PP. Additional properties that were studied, were the following: stiffness, tensile strength and ductility. The most important topic of this study was the investigation of water vapor barrier and the influence of the degree of crystallinity on it. In conclusion, the desired outcome of this study was to determine the sample composition possessing the best combination of water vapor resistance and suitable mechanical properties.

Finally, the research questions are the following:

- 1) How do the relative amounts of PP and PE affect the degree of crystallinity of the polymer film?
- 2) How does additivation by means of  $\text{CaCO}_3$  affect the degree of crystallinity of the polyolefin film?
- 3) How does the degree of crystallinity affect the water vapor barrier and tensile properties of the film?

This thesis is commissioned by Kiilto Oy, and is a part of SUPER Healthy Buildings project, an ecosystem project which concentrates on sustainability and wellbeing in the building industry. SUPER stands for Sustainable, Productive, Ecological, and Reusable. The approach of the project, SUPER, is to decarbonize the built environment and to improve indoor living conditions. The project has received funding from Business Finland. The funding call from which the ecosystem has gained funding, is Low Carbon Built Environment: innovation funding call. The call is connected to the Sustainable Growth Programme for Finland, which is part of the EU's Recovery and Resilience Facility (RRF).



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## 2. POLYMERS

Polymers are macromolecules that are formed from smaller, repeating subunits, monomers, which determine the nature of the specific polymers. The atoms within a polymer molecule are most commonly connected by covalent bonds. A few of the most common types of synthetic polymers are rubbers, adhesives, and other types of plastics [15]. Polymers are commonly mistaken to only being plastics, but that presumption is very well wrong since many essential substances of life, e.g., proteins, cellulose, and carbohydrates, are polymers as well. Amorphous, semi-crystalline and crystalline are the three possible forms of polymers. A crystalline polymer follows a strictly orientated structure of constant components that are in perfect order [16]. Commonly, fully crystalline polymers are rarely used because their manufacturing is difficult due to the fact that commonly every material contains flaws of some sort which causes the presence of amorphous, disorientated regions that lead to the material being semi-crystalline [17]. The relevance of single crystals in polymer science is undisputed based on the understanding of pure materials and their behaviors [18]. However, the commercial use of fully crystalline polymers wouldn't actually be worthwhile for several applications due to the fact that with increasing crystallinity, the ductility of the polymer decreases and in other words, its brittleness increases [14]. This kind of behavior occurs due to the lack of amorphous regions within the polymer that would be able to prevent fracture by absorbing mechanical energy by means of deformation [19]. In addition, the degree of crystallinity of a polymer may be tuned via additives which usually are nanoparticles and act as crystallinity-modifying agents [20,21].

Several properties of polymers, including mechanical and barrier properties, may be tailored to match requirements of different applications. There are several different types of polymers that possess more or less varying properties and they are presented in the following subchapters.

### 2.1 Principal classes of polymers

There are three classes of polymers that may be separated due to their different structure and performance. Their characteristics and structures differ from each other and all of them possess different properties when compared with each other. These three classes are thermoplastics, thermosets, and elastomers.

### 2.1.1 Thermoplastics

Thermoplastics are a reusable class of plastics, which may be remolded several times by surpassing the glass transition temperature and melting point of the specific polymer [22]. The glass transition is a quality of amorphous or semi-crystalline materials, which causes the material to transform from a hard and glassy form into a rubbery and stretchable, possibly even soft state [23]. Understandably, the glass transition occurs as the elevated temperature reaches the glass transition temperature and the soft polymer hardens upon cooling, obtaining a new shape without the breakdown of the molecular structure [24]. The said atomic structure stands for a simple, linear composition which consists of chemically independent macromolecules [25]. Thermoplastics are marketed in, e.g., pellets, granules and sheets, and they may be processed using multiple ways, including injection molding, blow molding, extrusion and calendering [25]. Filler additives are vastly used in all polymer types, including thermoplastic applications, and their first purpose was to reduce costs [26]. Nowadays, as the price of plastics has decreased and cost-reduction is often less important, the use of fillers in thermoplastics is based on e.g., improved processability, flame retardancy and stiffness [26]. Examples of popular thermoplastics are polypropylene, polyethylene, polyvinylchloride, polycarbonate, polyamide and polystyrene [27].

Thermoplastics are nowadays very popular due to, for instance, their reusability, potential in sustainability, and simple processability [28]. They have been used and have meaningful functions in, for example, wind turbine blades, tissue engineering applications, and packaging materials [28–30]. The annual usage of food packaging materials is more than 200 million tons and the amount will keep on growing during following years [30]. There are several different eco-friendly thermoplastics that are used for packaging of food products, but their mechanical, antibacterial and barrier properties are more difficult to be optimally tailored for their specific uses when compared with their easily tunable fossil-based counterparts [30–32]. However, for instance, starches from different natural sources may be exploited in eco-friendly packaging applications, and they also may be modified by means of nature-derived additives, leading to biodegradable final products [30,31].

### 2.1.2 Thermosetting polymers

Thermosetting polymers, or more commonly thermosets, are specific kinds of polymers that are defined by their irreversible manufacturing process [33]. In other words, the processing of the heated and softened prepolymer by means of pressure, leads to perma-

nent crosslinking of the material and it cannot be processed again similarly [34]. In particular, the crosslinking occurs between individual chains of the material and thus a rigid 3D structure is obtained [25]. As their structure well suggests, the typical behavior of thermosets is stiff and brittle [26]. However, when it comes to advantageous properties, thermoset plastics possess more mechanical and physical strength and thus they provide a better solution for some applications when compared to thermoplastics, despite their renewability [35]. The production volume of thermoset is significantly lower than that of thermoplastics, and common thermoset materials include phenolic resins, melamines, polyurethanes, epoxy and silicones [25].

Thermosets have a broad variety of applications to which they may be used. They may, for example, be used as adhesives, electrical applications, construction, and vehicle industry [35]. Due to their rigidity, they are used in rather permanent applications, whereas thermoplastics are used in temporary, recyclable materials. Examples of these permanent solutions are automotive industry applications, flooring, boat hulls, refrigerator cooling systems etc. [33]. Currently, thermosets are particularly not considered environmentally friendly due to the fact that the difficulty of their recycling leads to minor end-of-life uses and applications [36]. At least until October 2022, the majority of thermoset waste was eventually shipped to landfills or incineration, but the recyclability of the waste is intensely studied and possibly a satisfactory solution for this major issue could be established [36,37].

### **2.1.3 Elastomers**

The most practical definition for elastomers is that they are materials that withstand significant amounts of elastic deformation at room temperature and restore their original form within a specified time limit [38]. In other words, the defining properties for elastomers are low Young's modulus and very high elongation at break [39]. The physical structure of elastomers contains long polymer chains that are connected by weak intramolecular forces, which allow the material to regain its shape after significant elongation [40]. Elastomers can typically be either thermoplastics or thermosets, but the main purpose, elastic behavior, of both types is the same [39].

Elastomers are considered a versatile group of materials due to their various uses in different applications. Moreover, their advantageous properties e.g., high barrier to gases, resistance to tear, and insulation capability promote their potential in industrial applications [41]. Elastomers are commonly used in adhesives, insulation and sealing

technologies, and they may either be derived synthetically or naturally [40]. The capability of elastomers to deform elastically under heavy loads is a superior quality when compared to most materials, and thus the use of elastomers in automotive and medical applications is common [42].

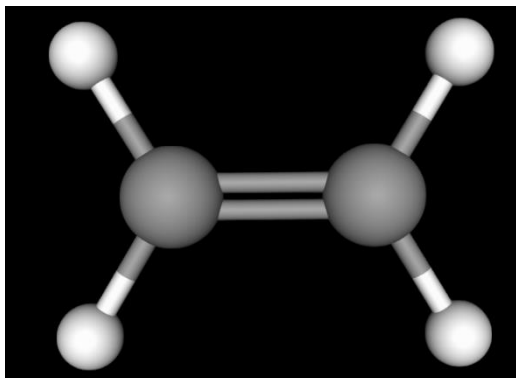
The application regarding this study is a thermoplastic elastomer film, which is used for waterproofing of wetrooms. The properties required by the functionality of the application include high water vapor barrier as well as elasticity and flexibility. The previously mentioned characteristics promote the efficiency and reliability of the material whilst used in waterproofing applications in construction.

## 2.2 Polymer types and structures

There are several different structures and types which polymers may appear in. This subchapter discusses the atomic-level investigation of polymers and the classification and properties of examples of the different structures and types.

### 2.2.1 Homopolymers

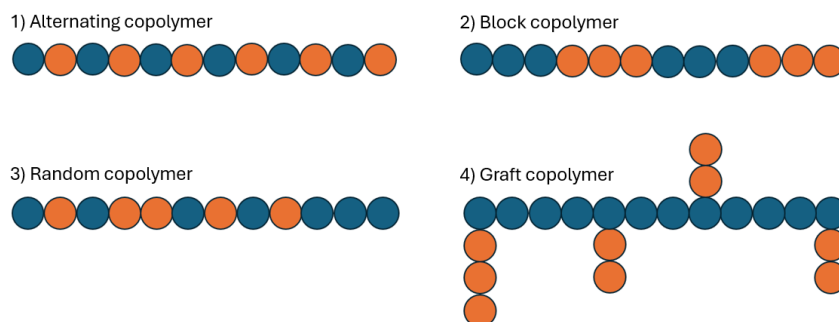
Homopolymers are defined to be polymers in which only one type of monomer constructs the repeating structure. In other words, homopolymers are the simplest forms of polymers due to the fact that they only contain one type of component which eventually gives the polymer its size and appearance. [43] A simple example of a homopolymer's structural component is illustrated in Figure 1. The illustration presents the monomer which forms a very elementary polymer, polyethylene. The black atoms are carbon atoms whereas the white ones are hydrogen atoms. In the actual polymer structure, the double bond is broken and the carbon atoms of the monomers may attach to each other, forming a long structure [44].



**Figure 1.** Ethylene, the repetitive unit of polyethylene [44].  
Image created in MolView.org

## 2.2.2 Copolymers

Copolymers are polymers that contain two or more different monomers that construct the macrostructure of the polymer [45]. There commonly are four different arrangements that may be present in copolymers and they are illustrated in Figure 2 [46]. The differently-colored circles indicate different types of monomers. The theoretical copolymers presented in Figure 2 contain only two types of monomers and it has to be considered that copolymers might include additional monomer types.

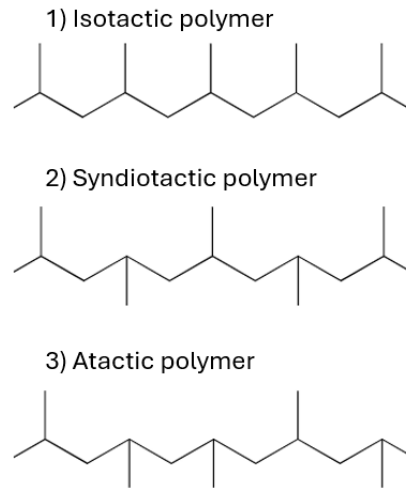


**Figure 2.** Different types of copolymers. Drawn according to [46].

An alternating copolymer has a structure in which every other component represents a specific monomer. A block copolymer contains sequences of the same monomers interrupted by sequences of other monomers. Despite the figure having even distribution, the sequences differ in length and size. A block copolymer is formed as a monomer tends to react with its copy rather than another type of monomer [43]. A random copolymer contains different types of monomers in a coincidental order. A graft copolymer's structure includes a long polymer chain backbone to which homopolymer chains with different lengths are connected [46]. Copolymers and their heterogeneous structure may be exploited in different applications regarding differences in hydrophobicity and crystallinity within the structure [47].

## 2.2.3 Polymer tacticity

Tacticity is a stereochemical topic which affects multiple properties of polymers. Tacticity of a material depends on how the functional groups of the monomer units have been distributed along the backbone of the polymer [48,49]. For instance, different tactic compositions affect e.g., the crystallinity, toughness, and impact strength of a polymer [48]. There are three different common types of tactic structures and they are presented in Figure 3. The continuous line of the skeletal formulas represent the backbone of polypropylene, and the individual branches indicate methyl groups that are present in every propylene molecule. Propene molecules act as the monomer units of polypropylene [50].



**Figure 3.** Different types of tacticities for polypropylene. Drawn according to [48].

Isotactic and syndiotactic polymers follow a regular, cyclic structure. In an isotactic polymer, the structure is arranged so that the functional groups are arranged on the same side of the backbone chain. On the other hand, in a syndiotactic polymer, the side of the functional groups alternate around to the backbone. Finally, atactic polymers follow no regular structure and thus the distribution of the methyl groups is entirely random.

According to a study [51] related to the investigation of tacticity of polymethyl methacrylate (PMMA), it was determined that it influences glass transition properties. The glass-transition temperature of syndiotactic PMMA is significantly higher than that of isotactic PMMA. Furthermore, the glass-transition temperature of isotactic PMMA is more prone to alteration as a result of pressure changes.

## 2.2.4 Polymer composites

Composite materials, or more commonly composites, are materials in which two or more individual components form a non-molecular level combination which has unique, enhanced properties when it comes to the behavior of the components alone [52]. Most commonly, composite materials contain a matrix material which gives the composite its shape and overall appearance, and a reinforcement material, which enhances the desired properties of the composite in the form of fibers or particles [53]. There are multiple natural composites that occur in the living world, including wood and bone [52,54]. The fact that nature has spontaneously constructed this kind of materials emphasizes the necessity of composite structures in the industrial world and their potential in sustainable construction materials. Polymer composites are materials in which a polymer matrix has been incorporated with reinforcing elements [55].

A very simple example of a polymer composite material is glass fiber reinforced polymer, in which a polymer acts as the matrix material, and the glass fibers are used as reinforcements [56]. Furthermore, glass fiber reinforced polymers are the most commonly manufactured polymer composites [56–58]. Due to their extraordinary structure, synthetic composites behave in a greatly different manner when compared to, e.g., metals, polymers and wood [54,59]. The modification of strength and stiffness of composites is relatively simple since it may be executed by varying the relative amounts of the matrix and reinforcement materials [56].

## 2.3 Polymer films

Polymer films are the most relevant form of polymers regarding this study, since the waterproofing application which is being concentrated on, contains a polymer film. In brief, a polymer film is a commonly used shape of polymer which has significantly greater length and width when compared to its thickness [60]. Polymer films also possess multiple unique and advantageous properties when it comes to material industry, and they may be manufactured from renewable and biodegradable raw materials using several different techniques [61,62]. Moreover, polymer films have multiple applications in which they may be used, including packaging and building construction as well as aerospace and automotive applications [63]. Processing of polymer films is relatively easy and their thickness, surface properties, and appearance may mostly be modified simply [64,65].

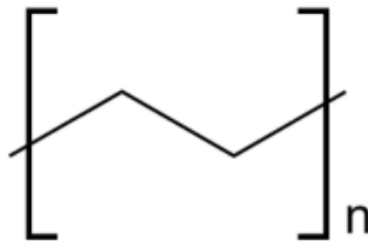
The simplicity of the processing of polymer films is highly advantageous when it comes to the use of them in different industrial applications. The modification of the thickness of the polymer films allows them to have several different uses, since the changes in thickness affect the flexibility, transparency etc. [66]. Furthermore, the surface of the polymer films may be modified regarding coatings and roughness and thus, different abilities related to, e.g., wettability and adhesion may be achieved [67–69]. Most polymer films are manufactured from thermoplastic raw materials and the multiple techniques that are used to construct the films include extrusion, calendaring and skiving [69]. Additivation may be used for improved processability, stability, orientation etc. [70–72].

There are several crucial topics that need to be addressed during the manufacturing process of polymer films. For instance for extrusion-based techniques, the processability of the raw materials depend highly on the amount of waste and the compatibility of the material for the processing machinery, i.e. the material should not attach to the machinery during the processing, and thus the production flow remains as efficient as possible

[73]. Furthermore, additives are used to improve the performance and processability of polymer films, which is discussed in chapter 2.3.3.

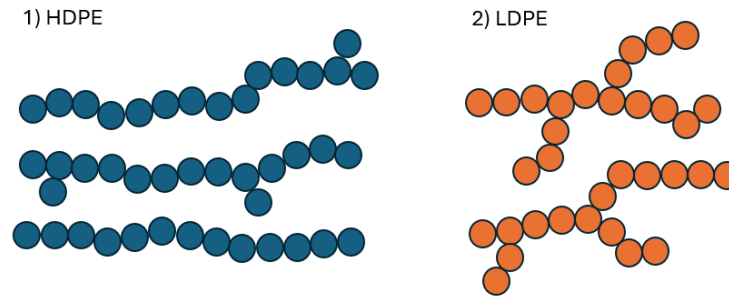
### 2.3.1 Polyolefins

Polyolefins are polymers which have, as a result of their simplicity, had a massive influence on the history as well as the present day of the polymer industry [74,75]. A polyolefin is defined to be a polymer, which consists of alkene monomers [76]. Alkenes are molecules which follow a formula of  $C_nH_{2n}$ . The relevance of the simplicity of polyolefins affecting their popularity was already mentioned, but additional reasons include abundance and versatility of raw materials, and cost-effectiveness [74,77]. The basic properties of polyolefins also comprise e.g., stability, lightness, recyclability, and processability, all of which emphasize their usefulness further [74]. The most common polyolefins are polypropylene, polyethylene and polybutylene [78]. The chemical structure of PP is shown in Figure 3 and that of PE is presented in Figure 4.



**Figure 4.** *The repetitive unit of polyethylene. [79]*

There are several common types of polyethylene, including the most popular ones, low-density polyethylene (LDPE) and high-density polyethylene (HDPE). Additional types include linear low-density polyethylene (LLDPE), medium-density polyethylene (MDPE) and ultra-high molecular weight polyethylene (UHMWPE) [80]. The most common way to classify different types of polyethylene is based on their molecular weight, which may range from thousands to millions g/mol [80]. The difference between LDPE and HDPE is based on the branching of the polymer chains of LDPE, which prevents the material to be packed compactly, reducing e.g., density, melting temperature, and tensile strength [13,81]. An illustration of the structures of LDPE and HDPE are presented in Figure 5. The structure of HDPE allows the material to possess higher crystallinity due to the fact that it contains less disorientation in the form of long side chains [13,79].



**Figure 5.** Examples of the basic structures of HDPE and LDPE. Drawn according to [81].

As discussed above, the relatively simple structure, which is fairly characteristic to polyolefins, affects several topics related to the use, processing, and cost-effectiveness of them. However, the simplicity of the material comes at the cost of compromised performance when compared to more complex, engineering plastics [75]. Engineering plastics stand for plastics which possess better mechanical properties and their performance often is more reliable [82]. Furthermore, the fact that polyolefins consist of nonpolar bonds, restricts their use because their interaction with other polymers is poor [83]. On the other hand, their mechanical properties, despite being lower than those of some other plastics, are suitable for multiple purposes, most of which being disposable products. Being the most manufactured plastics, polyolefins understandably are utilized in numerous applications in different industries, e.g., packaging, clothing and agriculture [84]. The previously mentioned lesser mechanical properties, however, may be enhanced by means of additivation.

### 2.3.2 Miscibility and compatibility of polypropylene and polyethylene

According to several publications [85–88] on the topic, the combining of PP and PE encounters multiple difficulties that affect not only the processability but also the final product consisting of the two polymers. Even though their structures are quite similar according to their hydrocarbon base, the miscibility of the two materials is poor. The immiscibility occurs due to the differences in the molecular structures of the two and is based on the Gibbs free energy of mixing being positive and thus the perfect blending of the materials is against the laws of thermodynamics [89]. However, the immiscibility is not a particularly relative issue due to the fact that water vapor barrier does not exclusively depend on that.

The compatibility of PE and PP in a film may be explained according to a study related to the miscibility of the two polymers [90]. The paper suggests that the two polymers tend

to form two different liquid phases. The polymer with higher concentration forms a continuous matrix and the one with lesser concentration forms smaller, separate units. The dominant matrix contributes most to the properties of the films, and the smaller units synergistically contribute to specific properties of the films. Polymer melts are connected together via intermolecular forces, as well as entanglement of the chains. These factors allow the materials to be connected to each other with no notable intramolecular forces [91].

### **2.3.3 Additives**

Additivation of polymers is a very common technique used to modify the properties of polymers and possibly turn them into composite materials [26]. There are several different forms of additives and they might be used to e.g., ease processability, crystallinity and mechanical as well as thermal properties [20,26,72]. Fillers usually are compounded to the polymers as plates, fibers or nanoparticles [20,26]. The most common additives in the polyolefin industry include for example, metal salts, metal oxides and halogen compounds [92].

The practical advantages of additivation of polymers include several different topics. The main reason of using additive materials is simply that the processability of the polymers may be improved significantly along with lowered costs [56]. Additives are vastly used in polyolefins and the proper utilizing of them leads to significant improvements in the mechanical properties of the application in question [93].

Despite the fact that polyolefins are usually defined as commodity resins, gaining less appreciation when compared to engineering plastics, they may be modified to possess improved mechanical and thermal properties. Additives may, in addition, be used to reduce the costs of a polymer by means of increasing the relative amount of the additive within the product [56]. The development of additivation of polyolefin applications has been significant and continues to this day. Furthermore, the use of nanofillers and plant-based fibers has been studied and their importance in the future could be remarkable [93].

The degree of crystallinity for PP is approximately 30–50 %, whereas that of HDPE reaches roughly 80–90 % [94]. From the values it may be determined that the more the blend of the two polyolefins contains HDPE, the higher its degree of crystallinity will be. Higher degree of crystallinity could lead to better water vapor barrier properties, and the influence of added PE will be discussed in chapter 6.

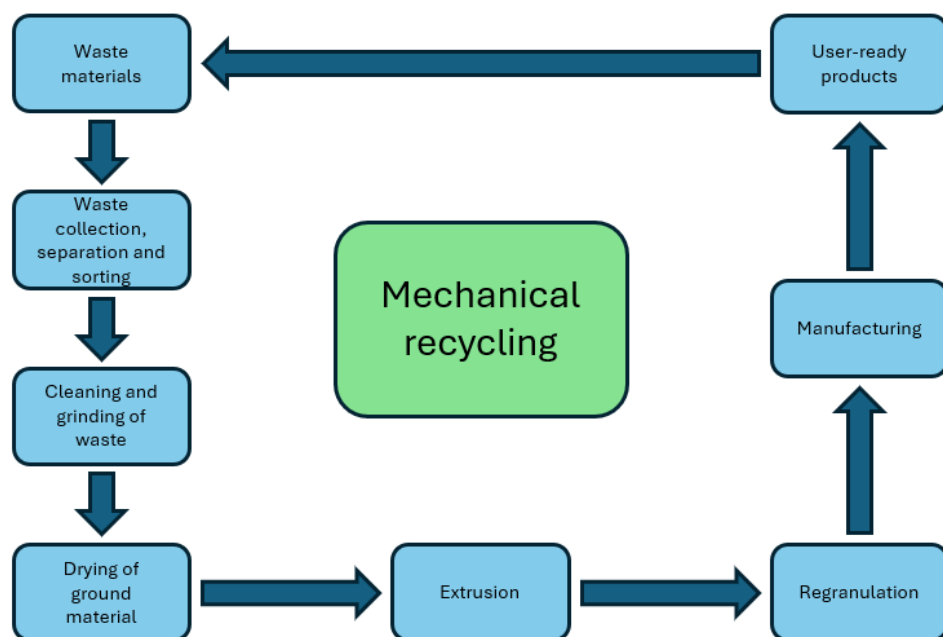
## 2.4 Environmental point of view and mechanical recycling

Polymers possess outstanding potential when it comes to environmental thinking due to their several advantageous properties. Due to the fact that the overall production of plastics has been aiming for disposability, durability and performance rather than evaluating the processes from an environmental point of view, the plastic manufacturing industry is predicted to become a significant consumer of petroleum [95]. However, by means of transition to renewable raw materials along with the biodegradability of final polymer products, the upcoming change of the polymer industry might become a revolutionary factor when it comes to the prevention of climate change and global pollution due to the fact that the greenhouse gas emissions and energy consumption would decrease massively [95]. Moreover, the current state at which the world is flooded with plastics would ease significantly by means of biodegradable plastic products. Nonetheless, the most effective way to improve plastic waste management would simply be decreasing the amount of it by manufacturing fewer temporary and disposable products [95].

There are several environmental topics that need to be addressed while discussing polyolefins. It's inevitable that there will always be unsuitable disposal of materials due to the fact that proper recycling of waste is always dependent on the consumer. As of 2017, polyolefin plastics covered the most of global waste, its amount being over 50 million tons [84]. Additionally, polyolefins are hydrophobic and their crystallinity, high molecular weight, and lack of nonpolar groups decrease their biodegradability and thus they remain stable in nature for long periods of time [84,96]. Apart from recycling, the most common end-of-life treatment types are landfilling, incineration, and, unfortunately, discarding to nature [77]. According to the European Parliament, in 2021, the proportion of plastic waste that was recycled in Europe, was ca. 40 %, with Ireland, Germany, Italy and Spain having the most contribution per capita [97].

Even though the carbon footprint of polyolefin production remains relatively low, there are multiple innovations which could be utilized to significantly decrease it [98]. For instance, exploiting currently best available technologies, including utilization of condensates and naphtha cracking could lower emissions by 20 %, whereas transitioning to green raw materials could reach a decrease of 45 % [99]. Additionally, despite the recycling of polyolefins doesn't enormously lower carbon emissions, it might significantly decrease the use of fossil raw materials and the role of the changes in the polyolefin industry might be significant for a sustainable future [99].

The main objective of mechanical recycling is to turn waste materials into suitable raw materials. Regarding environmental thinking, recycling is the most favorable option of handling polymer waste, and the usual steps include sorting, baling and washing, all of which influence the processability and handling of the waste material [100]. Mechanical recycling is a fairly simple technique which is based on utilizing force to restore the waste into reusable feedstock [101]. There are a few directive steps that are followed in mechanical recycling, including grinding the material into smaller units, and reprocessing the material into easily processable granules by means of heat and force [100]. The steps of mechanical recycling are presented in Figure 6. There are several guidelines that must be taken into account as successful recycling is to be achieved, including sustainable operation and proper sorting of the materials based on e.g., color, density and chemical composition [100]. Chemical recycling of polymers may be utilized in waste streams that contain e.g., mixed plastics or heavy contamination [102]. Chemical recycling breaks down plastics by means of heat and different chemical reactions, including pyrolysis and gasification [102].



**Figure 6.** Steps of mechanical recycling. Drawn according to [103].

The recycling process of plastics begins as the materials turn into waste after use. Waste materials from different sources are gathered, separated and sorted into groups containing, for instance, similar sizes, shapes, densities and chemical compositions [100]. The material is afterwards cleaned, ground and dried. The eventual outcomes after the grinding, flakes, often are reprocessed to obtain pellets that are easier to process by the user

of the recycled material [100]. Eventually, the recycled pellets are formed into new plastic products which will, after their lifespan, restart the life cycle.

According to a study in which HDPE material was recycled successively 100 times, the material retained 80 % of its initial hardness and stiffness, so it may be determined that rHDPE doesn't greatly lack mechanical properties when compared to its initial counterpart [104]. The crucial topics related to successful mechanical recycling of HDPE are: understanding the present conditions and equipment, as well as monitoring possible contamination, changes in chemical structure, and variations in molecular weight [105].

### 3. CRYSTALLINITY

Crystallinity is an essential property of all polymers which can be altered moderately easily. As mentioned earlier, practical polymers might exhibit a structure either semi-crystalline or amorphous [106]. By tuning the crystallinity of the polymer, the water barrier and mechanical properties of the polymer may be tailored to match the wanted applications and their requirements. Melt crystallization, in which amorphous regions within a polymer transform into crystalline regions, commonly occurs in three steps: primary nucleation, secondary nucleation, and secondary crystallization [107]. Primary nucleation stands for the initial forming of new nuclei, which may occur heterogeneously by means of foreign phases or rarely homogeneously by means of a spontaneous reaction [108]. The term secondary nucleation represents the growth of already existing crystals within a system, and it occurs mostly as a result of the collision of crystals with each other [109]. Secondary crystallization is defined as the further crystallization, which does not relate to the primary crystallization, which includes primary and secondary nucleation [110].

The degree of crystallinity stands for the relative amount of crystalline regions in a polymer and it commonly is calculated according to Equation 1,

$$\text{Degree of crystallinity [\%]} = X_c = \frac{\Delta H(T_m)}{\Delta H(T_m)^0} * 100 \%, \quad (1)$$

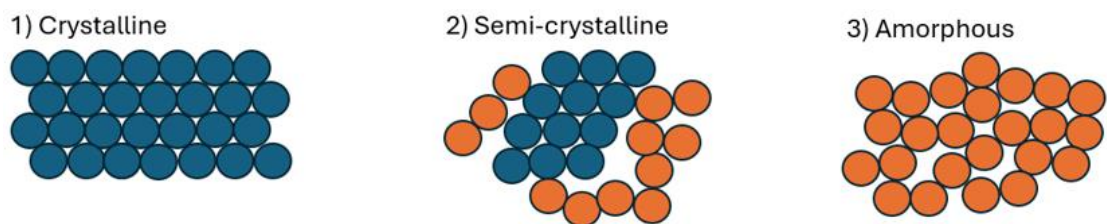
in which  $\Delta H(T_m)$  stands for the enthalpy of fusion of the measured polymer at its melting temperature, and  $\Delta H(T_m)^0$  indicates the enthalpy of fusion of the polymer at its melting temperature while in fully crystalline state [111]. There additionally are a few different techniques by which the degree of crystallinity can be investigated, and they will be presented below.

According to a study [112] related to the crystallinity of a polymer, different techniques that may be utilized for gaining information about the crystallinity of a material include evaluation of density, differential scanning calorimetry (DSC), X-ray diffraction, and Raman spectroscopy. The crystallinity of a material may be evaluated from its density, since the greater the density of a material is, the higher its degree of crystallinity will be. Differential scanning calorimetry exploits the heat flow during the melting of a material and thereby crystallinity may be determined by comparing the melting and crystallization enthalpies of a material with its single crystal counterpart. DSC is discussed further in the

next paragraph. X-ray diffraction is used to obtain Gaussian curves that may be studied to gain knowledge of the degree of crystallinity. This occurs by comparing the areas of crystalline and amorphous peaks from the obtained spectrum. Finally, Raman spectroscopy is based on measuring crystallinity from Raman spectra. According to Raman effect, when a material is exposed to light, different molecules reflect the light in a different wavelength and thus Raman spectrum may be obtained and evaluated since the peaks are characteristic to specific molecules [113].

As information about a material's crystallinity and thermal properties is wanted, DSC certainly is a very suitable method for a variety of materials and it has significant importance in polymer science [114]. DSC is based on the measurement of heat flow at sharply specific temperatures, and thus the above mentioned thermal properties, e.g., glass transition temperature, enthalpy of fusion, and melting temperature, of which enthalpy of fusion is the most important regarding this research due to its necessity in calculating crystallinities of materials [115]. Usually there are several heating and cooling phases in DSC measurements.

Different types of crystallinity are presented in Figure 7. Blue circles represent crystals, the organized parts of the molecule, whereas orange circles stand for disorientated parts. The most relevant insight related to different orientations is that in highly crystalline molecules, the amount of unoccupied space is minimal, while there is a remarkable amount of unfilled area within the amorphous structure. Due to the irregular orientation of amorphous materials, their properties also vary depending on the distribution and exact structure [116].



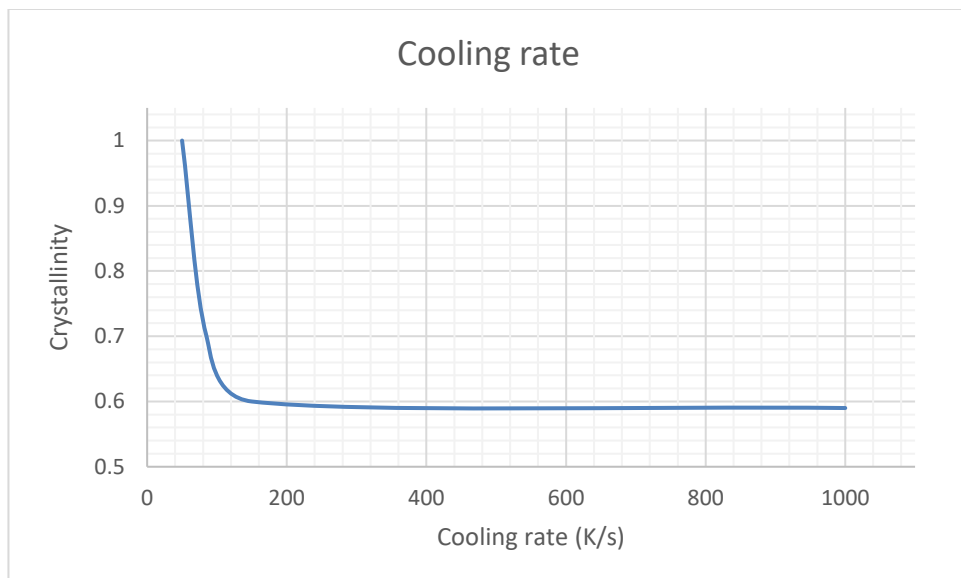
**Figure 7.** Different crystallinities. Drawn according to [117].

Highly crystalline materials possess advantageous properties for certain applications, e.g., polymer science and optical applications. However, they commonly also include flaws that need to be considered when pondering their uses and performance. Crystallographic defects affect mechanical properties of, for example, polymer composites by altering their strength, hardness, ductility and toughness, which may lead to imperfect products [118].

### 3.1 Controlling of the degree of crystallinity of polymers

The controlled modifying of the degree of crystallinity of polymers has a great importance in the polymer industry due to the fact that the final properties and performance of a polymer are greatly influenced by its crystallinity [20]. There are multiple ways by which degree of crystallinity may be modified, including cooling rate, and additivation [20,21,119]. Influencing the degree of crystallinity of polymer products is relatively easy, whether the aim is to increase or decrease it. The main methods of tailoring crystalline structures are presented further below.

The cooling rate of a polymer system greatly affects its crystallinity. This happens as a result of a shorter period of time during which the crystals within the material may be formed and grown [120,121]. According to a research [119] investigating the effect of cooling rate on crystallinity, the rapidity of the cooling of a polymer initially decreases the degree of crystallinity steeply, but the achieved crystallinity becomes constant above a specific point cooling rate. A rough example of the effect of cooling rate is illustrated in Figure 8, which emphasizes the rapid effect of the increased cooling rate as well as the fact that the degree of crystallinity becomes constant after a specific amount of cooling rate is surpassed.



**Figure 8.** Effect of cooling rate on the crystallinity of PP. Drawn according to [119].

The presence of additives within a polymer structure may influence differently based on the size and shape of the exploited nanoparticles [20]. Moreover, the relative amount of the additive within the material has an impact on the degree of crystallinity [21]. Additives may, for instance, act as nucleating agents and compatibilizers [122]. Nucleating agents

stand for fine particles which provide sites at which the nucleation and growth of spherulites are promoted [122]. Compatibilizers are very important in immiscible polymer blends because they may provide unitive interfaces between non-miscible polymers [123]. Filler additives may be used to affect the nucleation onset temperature of polypropylene significantly, which means that the procedure to gain different crystallinities in different circumstances may be optimized [26].

## **3.2 Effect of crystallinity on polymers**

Advanced understanding of the behavior of complex, crystalline polymer systems is a challenging topic, which may be reasoned further based on molecular theory and the understanding of intermolecular forces [11]. Increasing degree of crystallinity affects the properties of a polymer due to the presence and greater count of large crystals [121].

Briefly, based on the molecular theory of polymers, semi-crystalline and crystalline polymers exhibit difficulties when it comes to thermoforming due to their sharp melting points [124]. Furthermore, crystalline and semi-crystalline polymers contain tightly packed structures which affects the materials' rigidity and stiffness [11]. Polymers may also contain strong intermolecular forces which promote their ability to perform in demanding circumstances, e.g., structural and repetitive loads [11]. These topics emphasize the fact that polymers are a very versatile class of materials and their properties depend on several different aspects.

### **3.2.1 Water vapor barrier**

The water vapor barrier of different polymers may be modified quite simply based on the fact that crystalline regions within a polymer offer higher barrier to gases and water, which leads to lower permeability along with higher crystallinity [13,125]. Amorphous regions within a polymer material are more permeable due to their random, disorientated structure, which contains less tortuous pathways along which gases are diffused through the material less efficiently [126,127].

Diffusion of water vapor through polymers is a very complex topic that relies on several different aspects, such as crystallinity and structure of the material, when comparing different polymers. The transmission of water vapor through a polymeric material occurs via diffusion, which is promoted by large enough empty spaces provided by a more amorphous structure [128].

### 3.2.2 Tensile strength

Tensile testing is a common measurement technique that may be used with all kinds of solid materials. Tensile test is a fairly simple method to gain knowledge of various mechanical properties including ductility, stiffness and elasticity. The test is one of the most popular measurements in metallurgy and materials science. The usual outcome of the test is a stress-strain curve in which stress is presented on the y-axis and strain is portrayed on the x-axis. The test is executed by applying vertical force to the specimen which afterwards exhibits some kind of deformation, depending on the test. Briefly, either one or both of the grips to which the ends of the specimen have been connected begin moving away from the sample and consequently it will be exposed to force.

According to several studies, the higher the degree of crystallinity of a polymer is, the higher its strength and stiffness will be [10,11,121,129]. The increasing degree of crystallinity additionally causes polymers to behave in a brittle manner due to the decreasing count of amorphous chains, which would enable ductility and elasticity [94]. The higher crystallinity of a material leads to higher water vapor barrier along with higher stiffness and brittleness.

### 3.3 Comparison of crystalline and amorphous polymers

Because fully crystalline polymers are impossible to be obtained by means of general manufacturing principles, a more descriptive term for most polymers would be “semi-crystalline” [17]. However, the headline of this subchapter is how it is, because of the importance of crystalline regions and the concentration on polymers containing significant amounts of them. According to the previously mentioned reasons, I concluded that the term “crystalline” would be more appropriate regarding this particular study application, since HDPE usually holds a crystallinity of roughly 80–90 % [94].

There is a significant amount of differences between crystalline and amorphous polymers that need to be taken into account while considering and comparing the two kinds. For instance, the processing into solid objects is easier for amorphous materials based on lower shrinkage and broad melting temperature range [130]. Other important topics that separate amorphous and crystalline polymers include transparency, impact resistance, and isotropy, all of which may be utilized for certain applications [131].

In conclusion, according to their performance and behavior, crystalline and amorphous polymers are completely different types of materials. Moreover, the degree of crystallinity

of a polymer material may easily be tailored to match the requirements of a certain application. The degree of crystallinity of a material highly influences a broad variety of its properties, and the understanding of its effect is crucial when it comes to the development of commercial products. The tunability and versatility of polymers as industrial materials emphasize the importance of them for tailored applications in which other materials might not reach similarly high levels of performance.

## 4. WATERPROOFING IN CONSTRUCTION

Waterproofing is a crucial topic that always needs to be considered while constructing a variety of structures. More or less harsh weather conditions often lead to the unwanted transition of water into the inner structures of the system in question. Moreover, the improper waterproofing of wetrooms may lead to leaks which enable and accelerate the condensation of water into the structures of a building. The presence of water usually leads to issues regarding the safety and comfort of dwelling as well as aesthetic deficits within a building.

According to [132–134], the presence of water accelerates the growth of mold spores within a building. Building materials being exposed to mold contamination remains a global issue and promotes the possible health risks of residents by means of corrupting the bases of buildings and enabling microbial emissions into indoor air [135]. In conclusion, proper waterproofing of buildings extends their longevity and prevents their contribution to health issues of the individuals spending time inside the building.

### 4.1 Waterproofing methods

Waterproofing may be completed using several different materials and methods [136]. For example, the materials and methods may be chosen to match a wanted application and the circumstances related to it. Some waterproofing materials provide better barrier against liquid water and on the other hand, some materials resist water vapor superiorly when compared to liquid water barriers [137]. Furthermore, proper drainage systems may also be utilized in preventing moisture damage [136].

As mentioned above, there are multiple, more or less different methods by which waterproofing of rooms may be executed efficiently. Two of them will be presented in this chapter. Both of the techniques are based on the attachment a seamless, uniform, impermeable layer between the innermost and outermost components of flooring, roofing or walls, depending on the specific application.

One of the most common and traditional ways of waterproofing wetrooms is the use of impermeable fluids that are evenly distributed onto the surface that requires prevention of exposure to water [136]. The methods that utilize fluids will in this chapter be referred to as membrane techniques. There are several ways to distribute the material onto the surface, including spraying and manual spreading by brushing or rolling [138]. There are

a couple of different materials that are commonly used in membrane waterproofing systems. There commonly are membranes that consist of one component but also ones that utilize two or more functional components. Common raw materials for membrane systems include styrene-butadiene rubber and styrene acrylate [139]. Two-component systems may, for instance, utilize cement or other powders [139]. Waterproofing membranes may harden by drying via water evaporation or by curing which involves a chemical reaction which turns the membrane from liquid into solid [140].

Films are nowadays used widely when it comes to the waterproofing of constructed systems. The using of them is based on placing the material directly with adhesive, onto the surface that requires waterproofing. Waterproofing films often are manufactured into long products and thus they may easily and rapidly be placed also onto large surfaces. Waterproofing film products commonly contain additional components along with the actual plastic waterproofing film. For example, the film may be coated with a fleece layer or something else which eases attaching and use [139]. Examples of waterproofing membranes and films are presented in Figure 9. The figure illustrates already placed waterproofing films and the distribution of a waterproofing membrane.



**Figure 9.** Use of waterproofing films and waterproofing membrane [139].

According to a manufacturer [139] of waterproofing materials, both of the presented techniques possess advantageous properties when compared to each other. For instance, the above mentioned membrane techniques include breathable solutions which may be efficiently used in underground systems. They may efficiently be used in the applications in which permeability of liquid water needs to be minimal, but the permeability of water vapor is required. Moreover, the membrane techniques are easily spreadable to uniform layers as well as according to delicate structures, such as penetrations. The advantageous aspects of waterproofing films include durability as well as installation rapidity and simplicity. They have also gained significant appreciation in modular buildings and the thickness of the film is constant. Finally, the elasticity of the films promotes their withstanding of structural movement within the constructed system.

However, along with the two above mentioned techniques, there are several alternative solutions for the installation of waterproofing materials in construction, and every one of them may be utilized for different applications and surfaces [136]. Furthermore, the used material itself doesn't define the suitability, since the characteristics of the surface material also need to be evaluated [138]. In addition, there are certifications related to waterproofing materials, according to which the selection of high-quality and sustainable materials may be done easily and trustworthily [139].

## **4.2 Additional applications**

There are several applications in different constructed systems which require prevention of water flow. Waterproofing in construction usually refers to wetrooms and residential buildings. However, also other types of built structures, including e.g., roofs, tunnels and swimming pools require proper protection from water. Waterproofing of roofs is a notably important topic when it comes to the functionality and longevity of a constructed system. According to the varying outside temperature and constant exposure to the sun and rain, the understanding of the importance of roofing is crucial when considering a waterproof building [141]. The waterproofing of swimming pools is done so that the final construct would resist leaks and contamination as efficiently as possible. The waterproofness of the used concrete possesses as high importance as correctly executed finishing and waterproofing work [142].

## 5. MATERIALS AND METHODS

This section of the study presents all the relevant steps that were followed in this study regarding raw materials, sample preparation, measurements, and investigation of the properties of the polymer films. Each subchapter contains detailed information of the material or method in question.

### 5.1 Raw materials

There are three main components that are used in the preparation of the polyolefin films in this study. The polyolefins that are used, polypropylene and polyethylene, act as the bulk polymer whereas calcium carbonate is used as the additive for tailoring the properties of the eventual film. Furthermore, there are a few components that are related to the processability and appearance of the films, but their further explanation won't be necessary since the relative amounts of main components and additives possess higher interest regarding this study. Briefly, they mostly act as dyeing agents and lubricants. The most relevant raw materials in this research are presented in the following subchapters.

This study is completed to gain understanding of the effects of the presence of an additional polyolefin in the polypropylene film. The added material is recycled high-density polyethylene (rHDPE), which is used by replacing specific amounts of the PP within the film. The most important factors that may occur along with the use of PE, include the changes in processability, performance, and uniformity of the material. Finally and most importantly, the fact that the use of rHDPE would be a more sustainable solution, promotes its potential in the application.

#### 5.1.1 Polypropylene and polyethylene

Polypropylene acts as the main component of the polyolefin film related to this study. Previously, it has been the only polymer within the application, but in this study, the presence of polyethylene will be studied. However, as the relative amounts of PE within the film in differently composed samples will be 0, 10, 20, 30 and 40 %, PP will remain the dominant polymer and component. According to the manufacturer of the used PP, it possesses several advantageous properties that may be utilized in this study. For instance, the material is suitable to be processed in several different ways and furthermore, it may be blended with other polyolefins including PE. Most importantly, it has high resistance for aqueous solutions and a variety of other fluids. The material is used as granules,

which are presented in Figure 10. The used PP is isotactic and its usability in the manufacturing of polymer films is brilliant.



**Figure 10.** *The used polypropylene granules.*

The polyethylene which is being used in this research project is post-consumer recycled HDPE. The material is entirely recycled and thus it would be a step towards a more sustainable product. Furthermore, the material is fully recyclable, which comports with the overall values of sustainable industries. The material itself does not particularly have superior properties when it comes to the use of it in this particular polymer film, but the possibility of exploiting it as a replacement or alternative for PP is studied. The PE that is used in this study is presented in Figure 11.



**Figure 11.** *The used polyethylene pellets.*

**Table 1.** The essential properties of the used polyolefins according to the manufacturers.

Property	Polypropylene	Polyethylene
Density (g/cm <sup>3</sup> )	0.9	1
Tensile strength (MPa)	8	25
Elongation (%)	30	15
Recycled content (%)	0	100
Processing temperature (°C)	160	170

Table 1 presents the essential properties of the polyolefins used in this research. It may be observed that the values somewhat differ from each other. The densities of the materials are highly similar, whereas the values of tensile strengths and elongations, which are derived from the point of yield, have notable differences. The tensile strength of PE is roughly three times greater than that of PP, whereas the elongation at yield of PE is half of the value of PP. The most radical difference between the materials is the relative amount of recycled content within the material. The used PE consists of entirely recycled material, whereas PP is fully manufactured from virgin raw materials. The amount of recycled content within the polyolefin film studied in this research is not the most relevant topic of this study, but the increased amount of the use of recycled materials is a very welcome addition.

Polypropylene and polyethylene are commonly not well miscible [85–88]. However, the problematic miscibility is not particularly essential in this research. This is because the compatibility of the two materials is evaluated based on the differences in water vapor barrier. Even if PP and PE would not form a greatly homogeneous compound, the presence of differently orientated crystalline structures may affect the barrier of the final film, either positively or negatively.

### 5.1.2 Calcium carbonate

Calcium carbonate is often included in polymer melts as it acts as a relatively low-cost filler material. The presence of CaCO<sub>3</sub> may also affect the mechanical properties of polymer melts, including tensile properties as well as impact strengths. The effect of CaCO<sub>3</sub> on the water vapor barrier properties of the polyolefin film possesses high importance and also the effect on tensile strength of the film is being studied. [143]

The calcium carbonate that is used in this research promotes the sustainability of the eventual product, given that it is entirely recycled. The material is recovered from side streams of the mining industry, which means that the material would be disposed as

waste if it wasn't utilized this way. The amounts of calcium carbonate in different samples are adjusted to match 40, 30, 20, 10 and 0 % of the amount of used polypropylene.

Regarding this research, the presence and use of additives is essential due to the fact that  $\text{CaCO}_3$  is exploited in order to tune the properties and reduce the cost of the polyolefin film. The influence of additivation on the degree of crystallinity of the film is one of the most relevant topics regarding this study.

## 5.2 Sample preparation

In this research, 10 different sample compositions are present. The parameters that are alternating between the samples are: the amount of polypropylene being replaced with polyethylene, and the amount of used calcium carbonate. These parameters are adjusted separately and they aren't combined in the same samples. In the samples into which PE is added, the amount of used  $\text{CaCO}_3$  remains constant. PE will neither be included in the samples in which the amount of  $\text{CaCO}_3$  is adjusted. The different sample types and compositions are presented in Table 2 and Table 3. Sample type 1 acts as the reference sample and all the other samples will be considered according to it. Table 2 represents the proportional amounts of the two polyolefins, and Table 3 exhibits the proportional amounts of PP and  $\text{CaCO}_3$ . Neither table takes any additional materials into account. The samples are presented in two separate sets to enhance the clarity of the results of the study.

**Table 2.** Sample set 1: compositions of the samples with adjusted polyolefin content.

Sample type	Amount of PP (%)	Amount of PE (%)
1	100	-
2	90	10
3	80	20
4	70	30
5	60	40

**Table 3.** Sample set 2: compositions of the samples with adjusted  $\text{CaCO}_3$  content.

Sample type	Amount of PP (%)	Amount of $\text{CaCO}_3$ (%)
1	71	29
2	77	23
3	84	16
4	91	9
5	100	0

Of every sample type, different amounts of samples are obtained for different purposes. For the cup test by means of which the water vapor permeability is measured, the required amount of samples is six. The tensile testing also requires six samples, from which a rough mean value from every sample composition is taken into account. DSC requires two samples of every type. In brief, the water permeability test and the tensile test both require 54 samples while DSC only needs 18. The steps of sample preparation are presented in Figure 12.



*Figure 12. Steps of sample preparation.*

### 5.2.1 Preparation of the polymer mix

The mixing of the raw materials was done by weighing all the components into a cup and stirring them together. The amount of used raw materials for different batches varied between 73 and 53 grams. Eventually, when the mixture was homogeneous enough, it was placed in between the rolls of the calender and thus the melting process was initiated and completed. The presence of calcium carbonate, practically, affects the processability of the polymer in different ways. For instance, the decreased amount of  $\text{CaCO}_3$  leads to easier mixing of the raw materials as well as more rapid melting of the compounds.

### 5.2.2 Calendering and obtaining of samples

Calendering is a very popular method that is used to manufacturing thin plastic sheets. The operation of a calender is based on the spinning motion of the heated rolls, which enables the material to become a uniformly thin film. There are different types of calenders which may contain a separate extruder or, perhaps, several rotating rolls [144]. However, the calender in this study, Schwabenthon Polymix 110 T. It is a fairly simple one, consisting of two heated rolls and one pressure roll, and it is presented in Figure 13 in which the order of the rolls from bottom to top is: front roll, back roll and pressure roll, respectively. Furthermore, as mentioned earlier, the material was manually dropped in between the rolls.



**Figure 13.** Schwabenthan Polymix 110 T.

The processing of the samples containing polyethylene was executed at a temperature of 170 °C, whereas the samples that only contained polypropylene as polymer raw material, were processed at 160 °C. The first step in the calendering was adjusting the desirable temperature and starting the machine.

As soon as the wanted temperature was reached, the polymer mixture was placed in between the rolls of the calender. Initially, the rotational speed of the front roll was set to roughly 15 revolutions per minute (rpm). The back roll also rotated, but very slowly. The heat and rotation of the rolls melted the polymer mixture and it began to horizontally spread along the front roll. Consequently, the polymer melt was distributed by means of a blunt knife to obtain a uniform, homogeneous material. As soon as the distribution of the raw materials within the melt was satisfactory, the rotational speed of the back roll was set to roughly 12 rpm, whereas the speed of the front roll was lowered to the minimum, 1–2 rpm. The material processed by means of a calender tends to attach onto the roll with greater speed, which allows the material to easily be transferred between the two rolls. As the material was successfully transferred onto the back roll, the adjustment of the gap between the two rolls was used to alter the thickness of the film until the desired area density, 200 g/m<sup>2</sup>, was reached.

The collection of the sample material occurred by means of the use of a fibrous sheet with the help of a rubbery pressure roll. Initially, the sheet was placed in between the pressure roll and the back roll, with the pressure roll yet being separated from the back roll. Consequently, the pressure roll was pressed against the back roll, and the sheet

was left in between of them. By manually pulling the end of the sheet and due to the spinning motion of the back roll, the sample material was transferred uniformly onto the sheet. After the wanted amount of the polymer was attached to the sheet, the pressure roll was released and subsequently, the polymer was cut by means of pulling the sheet and the remainder of the polymer melt was left on the calender to be collected later as additional samples.

The addition of PE into the raw material batch affects the processability and performance of the eventual polyolefin film in different ways. For example, the processability of the film during calendaring decreases due to the increased stickiness and decreased uniformity of the polymer melt. The stickiness of the melt leads to difficulties in collecting of the material due to increased risk of unwanted material attaching onto the sample surface. Furthermore, the differing melting temperatures of the two polymer precursors affect the initial processability negatively. However, with additional time consumption, the satisfactory uniformity of the polymer mix was achieved. In addition to the negative effects of added PE, the material to which the softened polyolefin film is collected, encounters difficulties as a result of the elevated temperature of the calender that's required to enable the melting of the used PE. The elevated temperature causes the fibrous sheet to stick more easily onto the calender rolls. Additionally, the processing needs to be done more rapidly due to the fact that the elevated temperature causes the melt to stick to the surface of the calender rolls. The collected polymer films were also determined to be less flat when compared to their counterparts with no added PE.

After the polymer film had cooled and solidified, the respective samples for the water permeability analysis, tensile testing, and differential scanning calorimetry, were obtained the following ways. The samples for the water vapor permeability tests were received by manually cutting the polymer film according to a correctly sized template. The samples for the tensile tests were obtained by means of a mold and pressure. The mold was placed on the sample, underneath the pressure machine, which applied uniform pressure to the mold. The samples required for the DSC measurements were obtained by using a tiny mold and hammer. The mold was placed vertically onto the sample surface and the pressure which was used to gain samples was applied by means of a couple strikes with the hammer. All of the methods for gaining ultimate samples are presented in Figure 14.



**Figure 14.** Obtaining of water vapor permeability test, tensile test and DSC samples, respectively.

The samples required for the DSC measurements were placed into a measurement cup, which is very typical for DSC measurements. Afterwards the cup was enveloped with a lid by means of a pressure machine presented in Figure 15. Consequently, a tiny puncture was made on top of the lid. The final form of a DSC sample is presented in Figure 16.



**Figure 15.** The machine used to close the DSC sample.



**Figure 16.** A ready-to-use DSC sample.

The samples that were used in the water vapor permeability test, were put into test-specific metallic measurement cups, presented in Figure 17. First, the inside of the cup was covered with a molten wax mixture to avoid the failing of the cup via corrosion during the testing period. Afterwards, a plastic petri dish was attached to the bottom of the cup by means of the liquid wax. 150 ml of saturated potassium nitrate ( $\text{KNO}_3$ ) solution was measured into the petri dish. Next, a circular mesh made of glass-fiber, was put onto the edges inside the cup. The circular sample was put onto the mesh ring and it was covered with a weight. Then, the visible remainder of the cup was filled with the wax mixture, and the test cup was left to solidify. After the wax had been solidified, the weight was taken out of the sample and thus the final test cup was obtained. An example of a ready-to-use cup is presented in Figure 18.



**Figure 17.** The test-specific measurement cup.



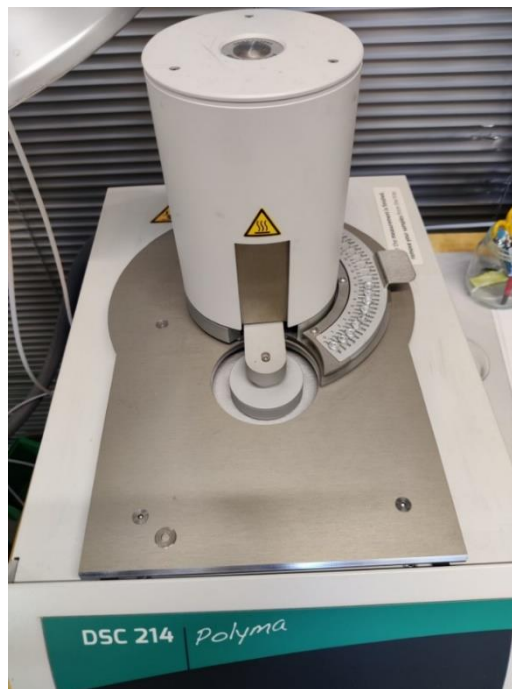
**Figure 18.** A ready-to-use cup for water vapor permeability tests.

## 5.3 Testing methods

Regarding this study, the essential testing methods are DSC, water vapor permeability test and tensile test. Briefly, DSC is used to determine the crystallinities of the samples, water vapor permeability test is utilized to evaluate the suitability of the samples for waterproofing applications, and finally, tensile testing is done so that the changes in the physical properties of the samples can be understood and evaluated.

### 5.3.1 Differential scanning calorimetry

DSC regarding this research was completed in a fairly usual way, according to the standard SFS-EN ISO 11357:6:2025. The machine used for the DSC measurements was Netzsch DSC 214 Polyma, which is presented in Figure 19. The samples may be seen on the right hand side of the machine. The test consisted of two heating phases, during which the temperature was elevated to 200 °C. Before the heating phases, the temperature was decreased to 0 °C by means of liquid nitrogen. The measurement cups were put into the DSC machine and it completed the initiated test itself. The provided data contained graphs from which the degree of crystallinity for each sample may be obtained using Formula 1. According to Netzsch, the manufacturer of the DSC machine, the  $\Delta H(T_m)^0$  is 207 J/g for PP and 293 J/g for PE [145]. These values will be used in the calculation of the degrees of crystallinity.



**Figure 19.** Netzsch DSC 214 Polyma.

The melting enthalpies of the samples in this research were obtained from the data of the DSC measurements, which are presented in Appendix A. The enthalpies are shown as the area of the peaks of the darker blue curve. The degree of crystallinity for each sample is calculated as average of two parallel samples.

### 5.3.2 Water vapor permeability test

The measuring of the water vapor barriers of the samples in this research was executed by means of a permeability test according to SFS-EN ISO 12572:2016. The most important property regarding the application in this study, indeed, is water vapor barrier, and thus the understanding of its tuning along with the changes in other properties is very much necessary in this particular research.

The cups were kept in a climate-controlled room, in which the temperature was set to be 23 °C and the relative humidity was set to be 50 %. The cups were weighed once a week for roughly 5 weeks. Every time the weighing was done, along with the weights of the cups, exact time, temperature, relative humidity, and air pressure were written down.

Essential parameters of the test were thicknesses  $d$  and areas  $A$  of the samples, and partial pressure difference across the samples  $\Delta p$ . The value of  $\Delta p$  across the samples of this study is roughly 1180 Pa. From the known parameters and the results of the weighings, different values related to water vapor permeability may be calculated.

Mass change rate  $\Delta m$ , for every weighing period, is calculated using Formula 2,

$$\Delta m_i = \frac{m_2 - m_1}{t_2 - t_1} \quad (2)$$

in which  $m_1$  and  $t_1$  are the mass at the previous weighing and the exact time at which the sample was weighed.  $m_2$  and  $t_2$  stand for the same values in the current weighing. The mean water vapor flow rate  $G$  is calculated using formula 3,

$$G = \frac{\sum_{i=1}^5 \Delta m_i}{5} \quad (3)$$

in which the mean of the mass change for the last five weighings is determined. Water vapor permeance  $W$  is calculated using Formula 4,

$$W = \frac{G}{\frac{A}{4p}} \quad (4)$$

in which  $A$  stands for the area of the sample. Water vapor permeability  $\delta$  is calculated using Formula 5,

$$\delta = W * d \quad (5)$$

in which  $d$  stands for the thickness of the sample. Water vapor resistance factor  $\mu$  is calculated using Formula 6,

$$\mu = \frac{\delta_{air}}{\delta} \quad (6)$$

in which  $\delta_{air}$  is water vapor permeability of air, which in the circumstances of this study is determined to be  $1.98 \cdot 10^{-10} \text{ kg}/(\text{m} \cdot \text{s} \cdot \text{Pa})$ . The most important values regarding water vapor resistance, are  $Z$  and  $s_d$ , units of which being  $\text{s}/\text{m}$  and  $\text{m}$ , respectively. Water vapor resistance  $Z$  is calculated using Formula 7,

$$Z = \frac{1}{W} \quad (7)$$

and water vapor diffusion-equivalent air layer thickness,  $s_d$  is calculated using Formula 8.

$$s_d = \mu * d \quad (8)$$

The values that are obtained by means of these formulas, may be used to understand and compare the differences of water vapor barrier of the materials that are studied.

### 5.3.3 Tensile testing

The measuring of the tensile properties of the samples in this research is completed utilizing a tensile test according to SFS-EN 12311-2:2013. The understanding of differences in the tensile strengths of the samples is of high importance because the usability of the polyolefin film application needs to remain at a suitable level. The test conditions of the measurements were the following: temperature was  $22,4 \text{ }^\circ\text{C}$  and relative humidity was  $50,0 \%$ . The most important values that are obtained from the tensile tests, are stress and strain, which may consequently be adjusted into a curve.

Stress may be calculated using Equation 9, and strain may be determined using Equation 10,

$$\text{Stress} = \sigma = \frac{F}{A} \quad (9)$$

$$\text{Strain} = \varepsilon = \frac{\Delta L}{L_0} = \frac{L-L_0}{L_0} \quad (10)$$

in which  $F$  indicates the force applied,  $A$  means the cross-sectional area of the specimen,  $L_0$  stands for initial length of the specimen and  $L$  denotes the final length of the specimen [146]. The used tensile testing setup in this study is presented in Figure 20 and stress-strain curves are presented in Appendix B.

Young's modulus is a value, which often is evaluated according to tensile tests. Young's modulus is related to the stiffness of the material. Young's modulus may be calculated as the slope at the beginning of the stress-strain curve in the linear region of the curve. In other words, the region in which the deformation of the material remains elastic.



**Figure 20.** The tensile testing machine used in this research.

The obtained data of the tensile tests contained values of load, expressed in Newtons, and displacement, expressed in mm. The values were converted into stress and strain using Equations 9 and 10.

## 6. RESULTS AND DISCUSSION

### 6.1 Degree of crystallinity

The degree of crystallinity was determined by means of DSC measurement, in which two parallel samples of each sample type were present. The results from the measurements are presented in Table 3 and Table 4. The average value of the two measurements for each sample is also disclosed.

**Table 3.** Results for DSC measurements for sample set 1.

Sample number	Crystallinity 1 (%)	Crystallinity 2 (%)	Average (%)	Standard deviation
1	8.1	8.1	8.1	0
2	34.0	32.1	33.1	0.95
3	34.0	35.4	34.7	0.7
4	39.2	38.7	39.0	0.25
5	38.5	39.7	39.1	0.6

The results for sample set 1 clearly indicate that even a slight replacement of PP with PE leads to dramatic increase in the degree of crystallinity of the film. The change may be considered presumable based on the degrees of crystallinity for PP and HDPE presented in chapter 2.3.3. The changes in crystallinities moderate after the first addition of the PE. However, the degree of crystallinity increases with every addition of PE even though the changes become notably smaller.

**Table 4.** Results for DSC measurements for sample set 2.

Sample number	Crystallinity 1 (%)	Crystallinity 2 (%)	Average (%)	Standard deviation
1	8.1	8.1	8.1	0
2	7.3	7.7	7.5	0.2
3	6.2	7.4	6.8	0.6
4	7.2	7.7	7.5	0.25
5	8.1	8.9	8.5	0.4

According to the results of DSC measurements in Table 4, the degree of crystallinity is lowest for sample 3, which contains 16 % of CaCO<sub>3</sub>. The degree of crystallinity increases

with both addition and subtraction of the amount of  $\text{CaCO}_3$ . The degree for pure PP film according to the results is 8.5 %, and the degree for sample 1, containing 29 % of  $\text{CaCO}_3$ , is determined to be 8.1 %. Even though the results follow the previously mentioned trend, the differences in results are relatively little. Furthermore, the deviations between the results of parallel samples are notably high. Based on the provided results, the consideration of the effect of reduction of  $\text{CaCO}_3$  on crystallinity would not be reliable. Additional parallel samples would be required to evaluate the actual contribution in a reliable manner.

The data obtained from the DSC measurements, presented in Appendix A, contains separate melting peaks for PP and PE at every sample composition. The presence of two peaks proves that the two polyolefins did not form a homogeneous compound. Based on the conclusion related to immiscibility in chapter 2.3.2, the observation is not surprising at all. However, the macroscopic appearance of the films was homogeneous and no separate phases could have been detected when examined with the bare eye. The homogeneity of the films may occur due to entanglement along with intermolecular forces, but the proving of that would require additional, microscopic investigation of the films.

## 6.2 Water vapor barrier properties

Water vapor barrier, regarding this study, was determined by means of water vapor permeability tests, in which five parallel samples along with a reference sample were present. Every sample which remained intact throughout the testing period, was taken into account. The results of the tests are presented in the tables below. The most important values in the tables are water vapor resistances  $Z$  and  $s_d$ , since the conclusion related to the performance of the films may be obtained directly from them.

The results obtained from the water vapor permeability tests for sample set 1, presented in Table 5, clearly indicate that the addition of PE to the PP film increases the water-proofing performance of the film, with increases in  $Z$  and  $s_d$  as a result from every addition. The steepest increase occurs between the films with 10 % and 20 % of added PE. Furthermore, a notable increase is observed between the films with 30 % and 40 % of added PE. According to the results, the proportional  $Z$  difference between the films with 0% and 40 % of added PE, is 46 %, which may be considered a remarkable performance increase.

**Table 5.** Results of the water vapor permeability tests for sample set 1.

Sam- ple	Thick- ness d (mm)	Water va- por flow G (kg/s)	Water va- por per- meance W ( $(\text{kg}/(\text{m}^2\text{s}\cdot\text{Pa}))$ )	Water vapor permeability $\delta = W\cdot d$ ( $\text{kg}/(\text{m}\cdot\text{s}\cdot\text{Pa})$ )	Water va- por re- sistance factor $\mu = \delta_{\text{air}}/\delta$	Water vapor re- sistance Z (s/m)	Water vapor re- sistance $s_d = \mu\cdot d$ (m)
1	0.358	$-4.78\cdot 10^{-11}$	$2.34\cdot 10^{-12}$	$8.37\cdot 10^{-16}$	$2.37\cdot 10^5$	$3.14\cdot 10^6$	84.9
2	0.341	$-4.43\cdot 10^{-11}$	$2.28\cdot 10^{-12}$	$7.76\cdot 10^{-16}$	$2.57\cdot 10^5$	$3.24\cdot 10^6$	87.6
3	0.337	$-3.59\cdot 10^{-11}$	$1.85\cdot 10^{-12}$	$6.22\cdot 10^{-16}$	$3.22\cdot 10^5$	$4.02\cdot 10^6$	108.5
4	0.332	$-3.45\cdot 10^{-11}$	$1.77\cdot 10^{-12}$	$5.89\cdot 10^{-16}$	$3.36\cdot 10^5$	$4.13\cdot 10^6$	112.4
5	0.330	$-3.14\cdot 10^{-11}$	$1.61\cdot 10^{-12}$	$5.32\cdot 10^{-16}$	$3.75\cdot 10^5$	$4.59\cdot 10^6$	123.9

The results obtained from the water vapor permeability tests for sample set 2, presented in Table 6, clearly indicate that the subtraction of  $\text{CaCO}_3$  filler in the film increases the waterproofing performance of the film, with increases in Z and  $s_d$  as a result from every subtraction. It may be concluded that the absence of filler leads to improved water vapor barrier when compared to the films to which filler has been included.

**Table 6.** Results of the water vapor permeability tests for sample set 2.

Sam- ple	Thick- ness d (mm)	Water va- por flow G (kg/s)	Water va- por per- meance W ( $(\text{kg}/(\text{m}^2\text{s}\cdot\text{Pa}))$ )	Water vapor permeability $\delta = W\cdot d$ ( $\text{kg}/(\text{m}\cdot\text{s}\cdot\text{Pa})$ )	Water va- por re- sistance factor $\mu = \delta_{\text{air}}/\delta$	Water vapor re- sistance Z (s/m)	Water vapor re- sistance $s_d = \mu\cdot d$ (m)
1	0.358	$-4.78\cdot 10^{-11}$	$2.34\cdot 10^{-12}$	$8.37\cdot 10^{-16}$	$2.37\cdot 10^5$	$3.14\cdot 10^6$	84.9
2	0.365	$-4.05\cdot 10^{-11}$	$2.08\cdot 10^{-12}$	$7.59\cdot 10^{-16}$	$2.61\cdot 10^5$	$3.53\cdot 10^6$	95.2
3	0.369	$-3.98\cdot 10^{-11}$	$2.04\cdot 10^{-12}$	$7.54\cdot 10^{-16}$	$2.64\cdot 10^5$	$3.60\cdot 10^6$	97.3
4	0.373	$-3.99\cdot 10^{-11}$	$1.95\cdot 10^{-12}$	$7.27\cdot 10^{-16}$	$2.75\cdot 10^5$	$3.80\cdot 10^6$	102.7
5	0.379	$-3.54\cdot 10^{-11}$	$1.73\cdot 10^{-12}$	$6.56\cdot 10^{-16}$	$3.02\cdot 10^5$	$4.24\cdot 10^6$	114.6

It may also be concluded that the thicknesses of the films alter due to additivation. Addition of PE to the films causes the film to be thinner when compared to films with no PE.

This leads to more abundant manufacturing patches due to the fact that thinner products provide more area in relation to the amount of used materials. Furthermore, the fact that the thinner films studied provide higher relative water vapor barriers, they might be manufactured to even thinner products and nevertheless possess satisfactory water vapor barriers. The manufacturing of thinner products leads to increased amounts of completed films per the used amount of raw materials.

### 6.3 Tensile properties

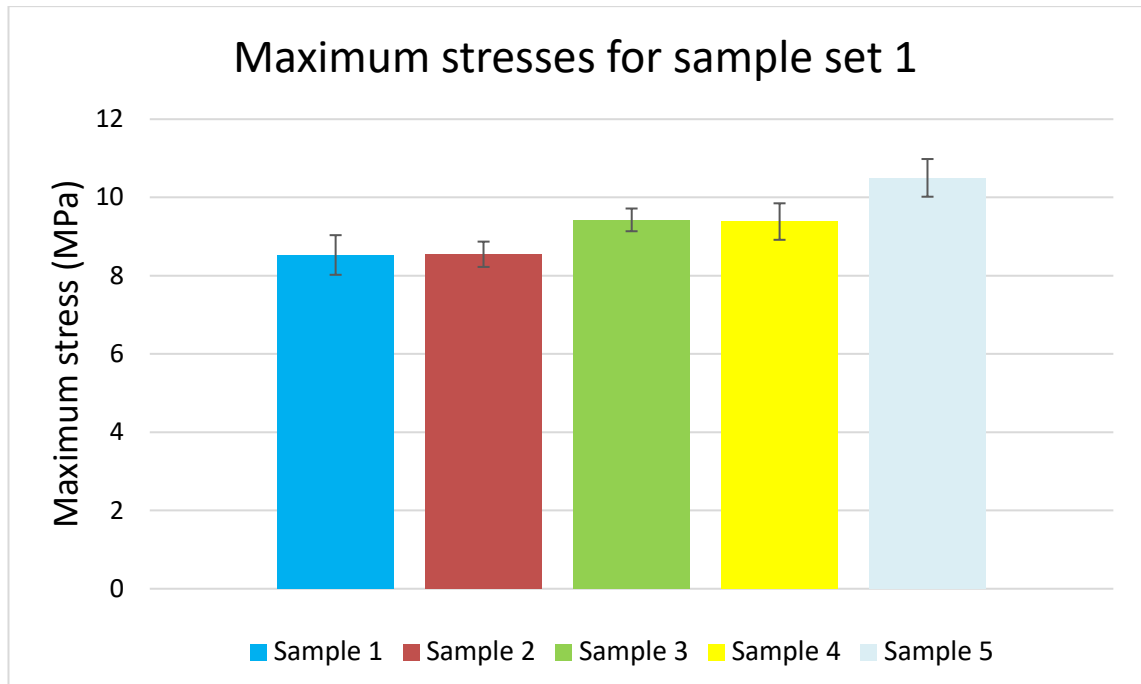
The evaluation of tensile properties of the samples in this study was executed by means of tensile tests. For every sample, there were 6 parallel specimens and the results are presented as their average. The following tables and figures represent the maximum stresses, strains at maximum stresses, and Young's moduli of the completed tests.

**Table 7.** Results of the tensile tests for sample set 1.

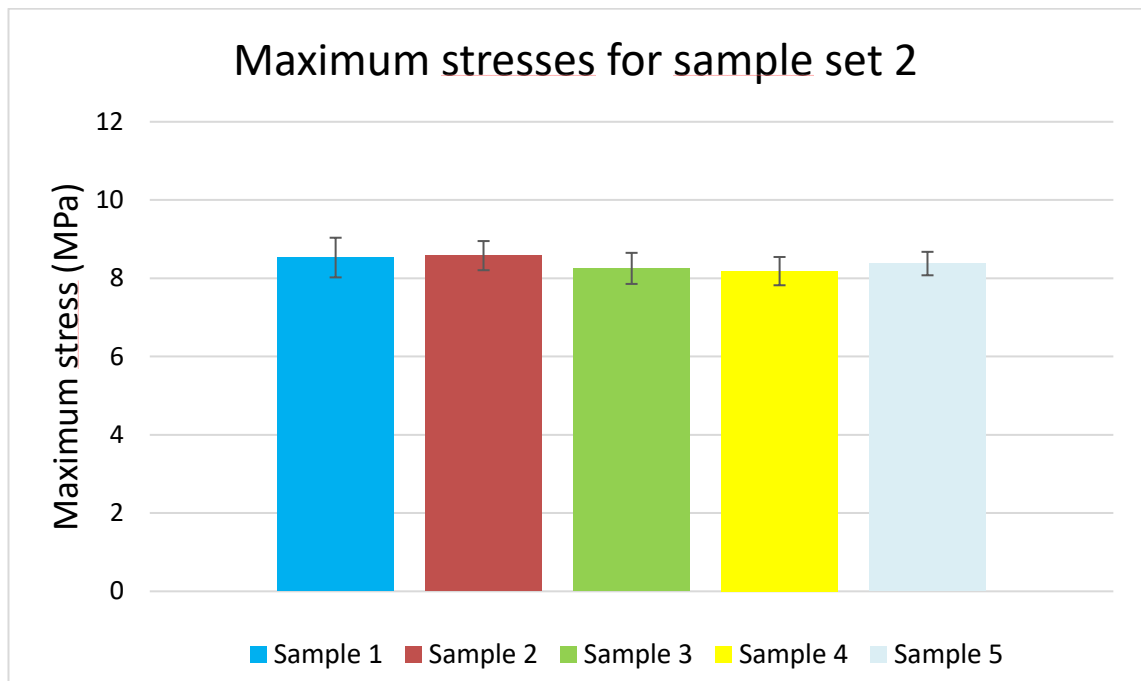
Sample	Maximum stress (MPa)	Strain at maximum stress (mm/mm)	Young's modulus (MPa)
1	8.53	0.81	168
2	8.55	0.82	191
3	9.43	0.89	235
4	9.38	0.90	238
5	10.50	0.89	294

**Table 8.** Results of the tensile tests for sample set 2.

Sample	Maximum stress (MPa)	Strain at maximum stress (mm/mm)	Young's modulus (MPa)
1	8.53	0.81	168
2	8.58	0.87	159
3	8.25	0.80	147
4	8.18	0.82	145
5	8.38	0.89	148



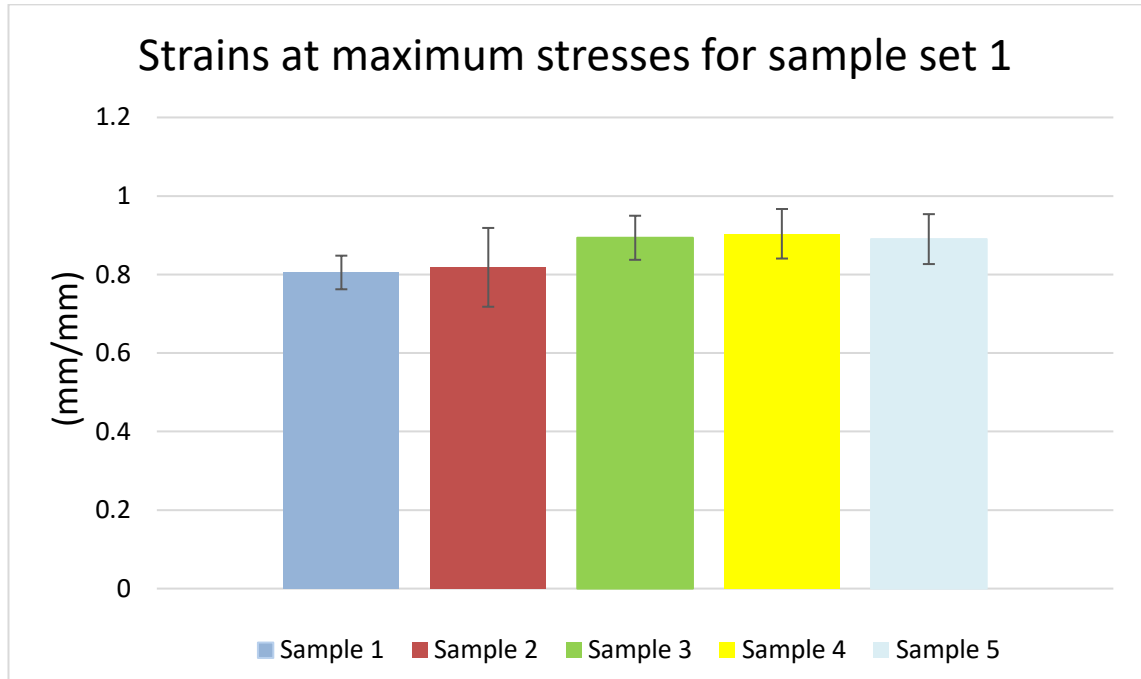
**Figure 21.** Maximum stresses for sample set 1.



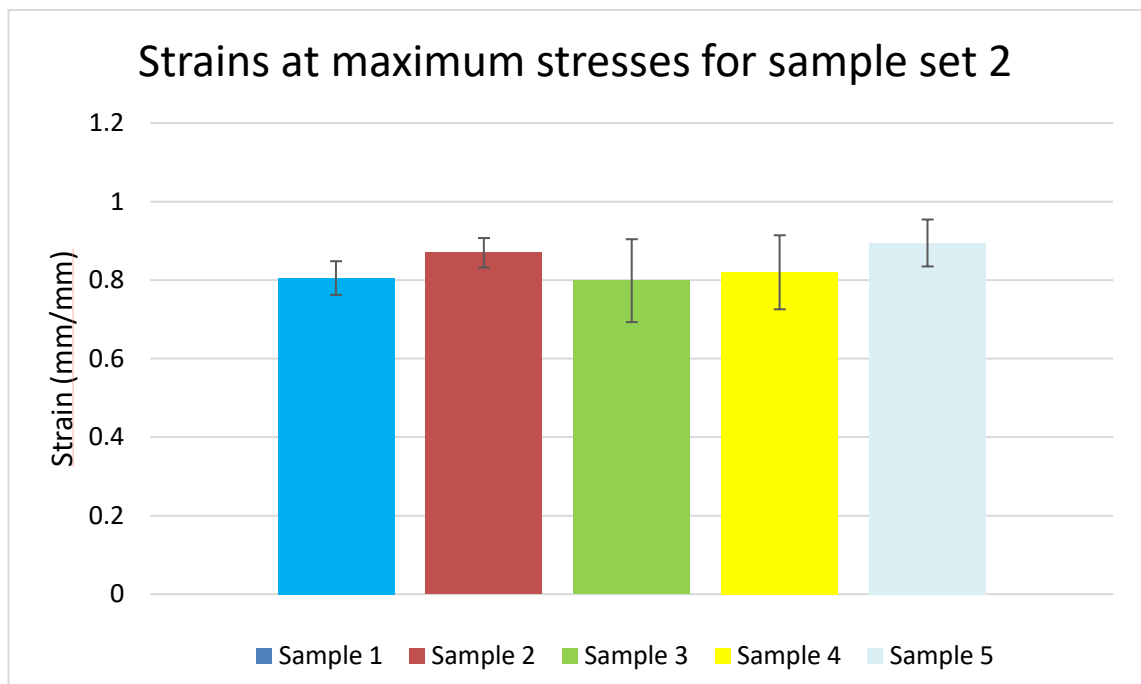
**Figure 22.** Maximum stresses for sample set 2.

According to Tables 7–8 and Figures 21–22, the increasing of maximum stresses for sample set 1 is present. However, the elevation is not significantly high. It may be concluded that the toughness of the material used in the waterproofing application does not decline. The results for sample set 2 show no significant differences between the samples, and it may be concluded based on the deviations, that the decreased amount of

CaCO<sub>3</sub> does not affect the toughness of the material. All of the sample types would be as suitable for using in the application as the reference sample.

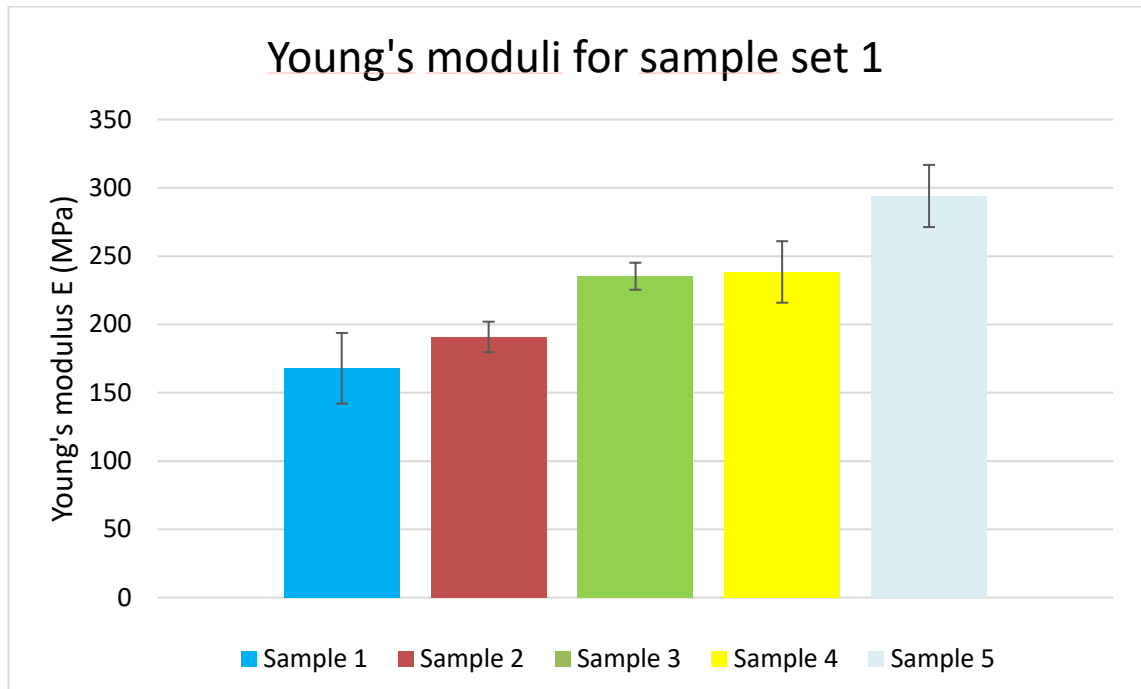


**Figure 23.** Strains at maximum stresses for sample set 1.

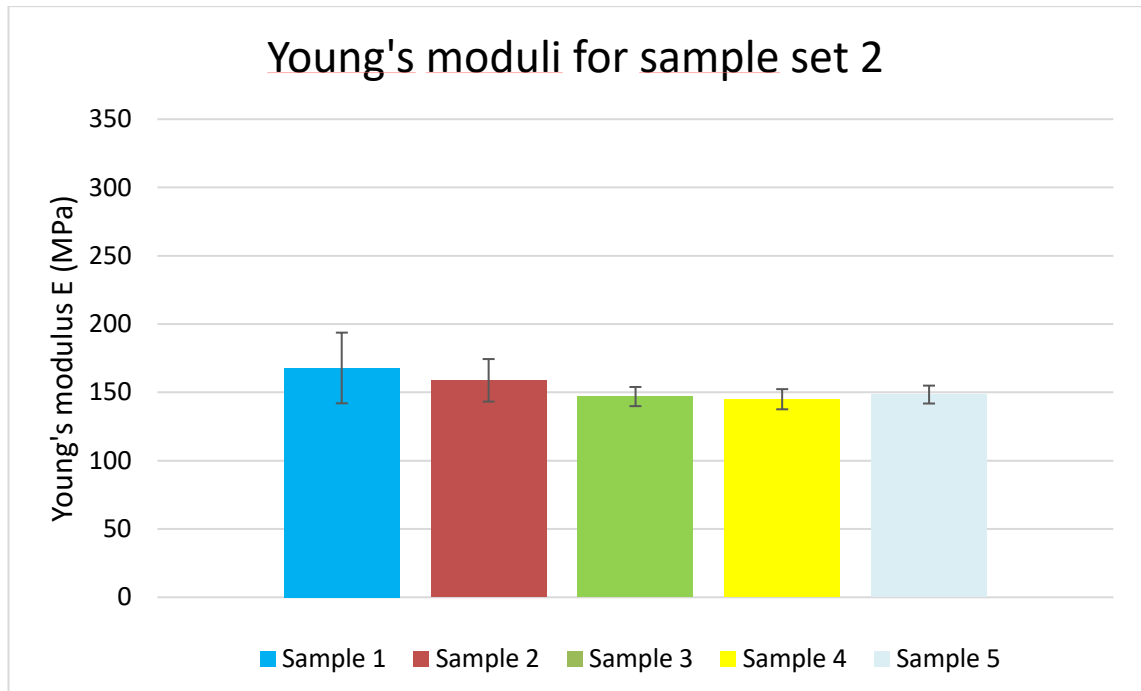


**Figure 24.** Strains at maximum stresses for sample set 2.

According to Tables 7–8 and Figures 23–24, the ductility of the samples, based on the completed tests, may not be said to be significantly higher or lower due to the fact that the graphs do not follow clear trends. Even though Figure 23 shows increased values in samples with higher PE content, the standard deviations are notably high, and thus no reliable conclusion into either direction can be made. In conclusion, all the sample types, based on ductility, would certainly be suitable for the waterproofing application to which the reference sample is used.



**Figure 25.** Young's moduli for sample set 1.



**Figure 26.** Young's moduli for sample set 2.

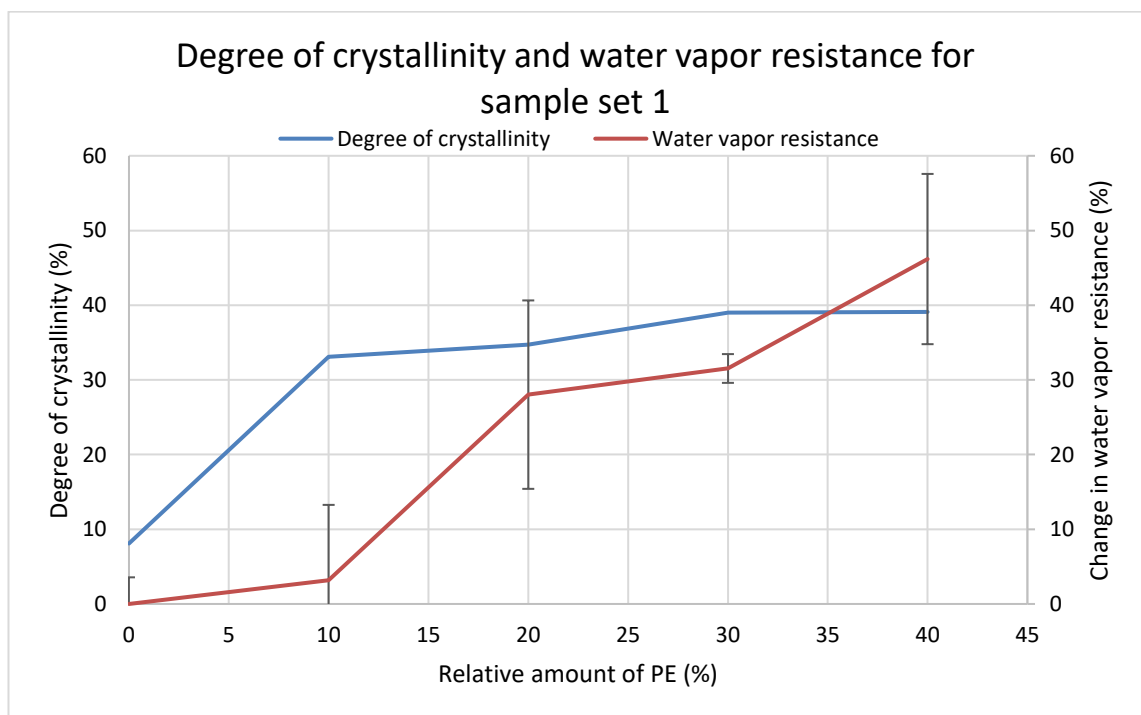
According to Tables 7–8 and Figures 25–26, the changes in Young's modulus between the samples may be evaluated. Based on Figure 25, it may be concluded that Young's modulus increases significantly with added PE. Sample set 2, on the other hand, shows no clear changes between samples due to high standard deviations of the first two samples. However, the final three sample types contain highly similar results, which underlines the fact that the reduction of  $\text{CaCO}_3$  does not dramatically decrease stiffness.

Even though sample set 1 shows steep increases in Young's modulus with added PE, the materials do not feel different when felt with hand. If the change in stiffness was critical, the materials would feel brittle or stiff when held and studied. However, the suitability for the waterproofing application will be necessary to be tested in practical circumstances. In conclusion, based on the test results, all the sample types should be suitable for the application.

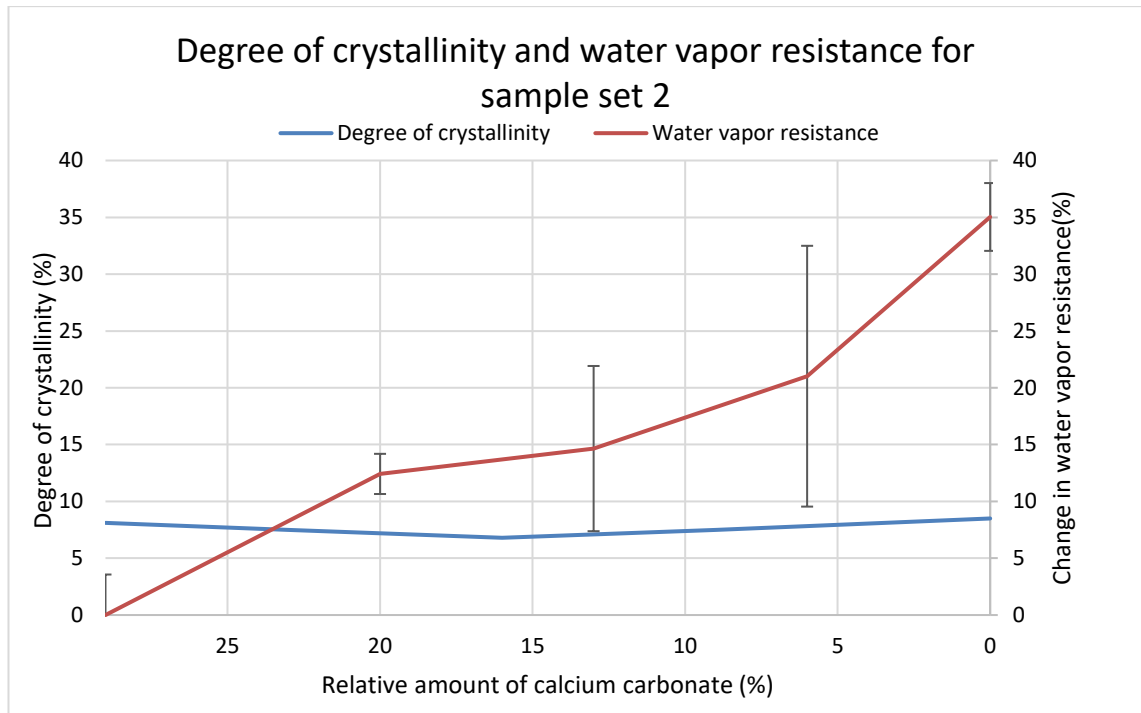
## 6.4 Summary of different results

According to the results obtained from the DSC measurements, the degree of crystallinity of the PP film increases continuously with the addition of PE, at least until the proportion of PE reaches 40 wt-%. The subtraction of the  $\text{CaCO}_3$  did not follow as clear a trend, but based on the results of DSC, the degree of crystallinity reaches its lowest point at 16 wt-% of  $\text{CaCO}_3$ . Both with addition and subtraction of  $\text{CaCO}_3$ , a slight increase in the degree of crystallinity is observed.

Figures 27 and 28 represent the degrees of crystallinity and proportional changes in water vapor resistance according to the reference film, for both sample sets. As concluded earlier, the addition of PE to the PP film notably increases the water vapor barrier of the film. According to Figure 27, the highest water vapor barrier is reached at 40 % of PE, which also is the highest amount studied in this research. It may additionally be observed that the addition of PE increases the water vapor barrier without exception at every measured amount, when compared to the previous sample with less PE. No clear correlation between degree of crystallinity and water vapor resistance may be concluded apart from the fact that both of them increase remarkably with added PE. Based on figure 28, the water vapor barrier also increases with every subtraction of  $\text{CaCO}_3$ , with a 35 % increase between amounts 29 % and 0 % of  $\text{CaCO}_3$  filler. However, the film with 40 % of added PE reaches a 46 % increase in water vapor barrier with respect to the film with no added PE, so it may be concluded that the subtraction of  $\text{CaCO}_3$  would not be worthwhile due to the fact that it would lead to a more expensive product due to the absence of the filler. Among the studied films in this study, it may be, based on the reasons above, concluded that the film with 40 % of added PE performs superiorly when compared to the other films.

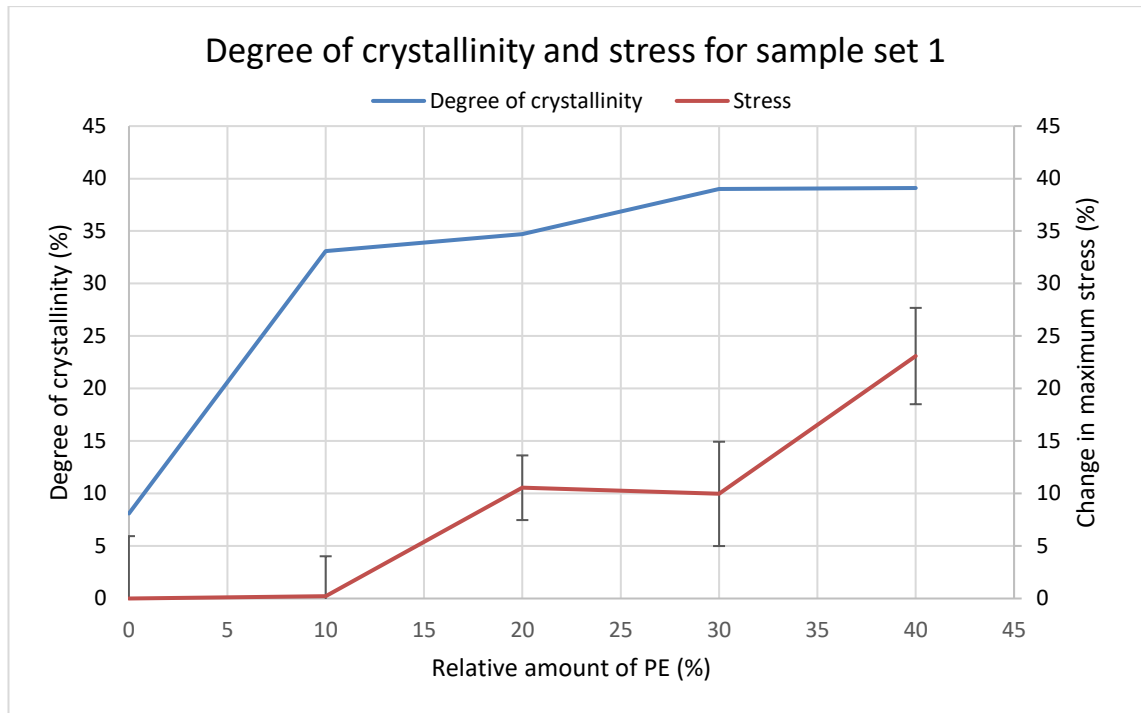


**Figure 27.** Degree of crystallinity and change in water vapor resistance for sample set 1.

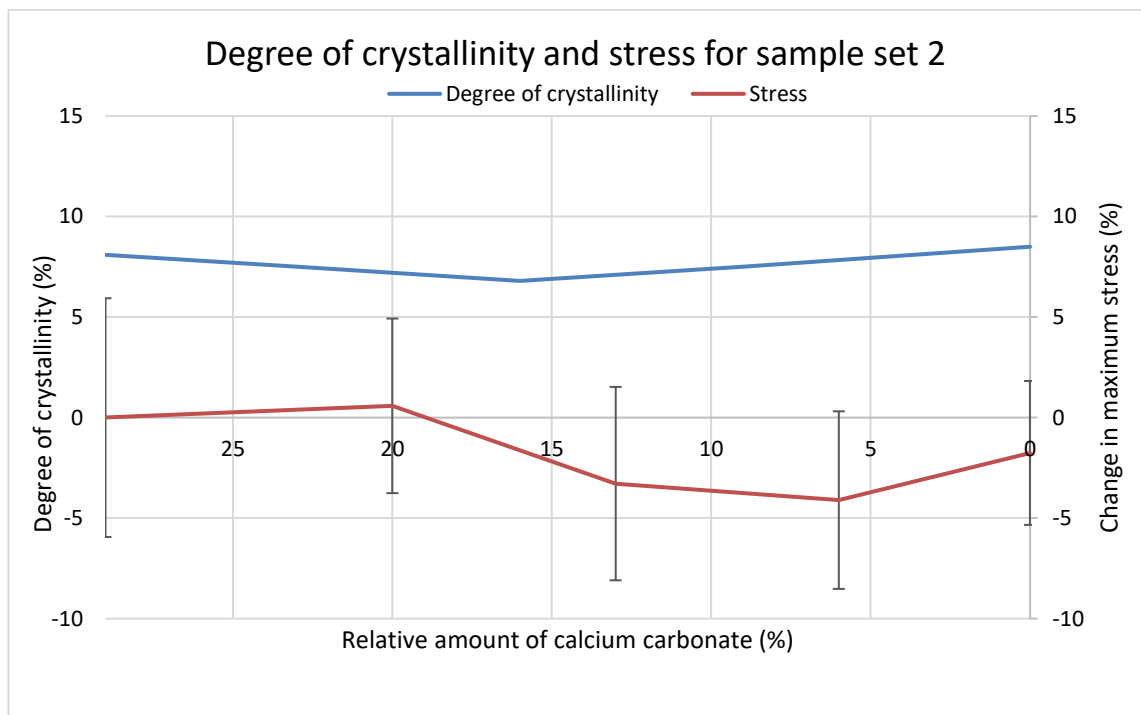


**Figure 28.** Degree of crystallinity and change in water vapor resistance for sample set 2.

Figures 29 and 30 illustrate the degrees of crystallinity, and changes of maximum stresses for both sample sets based on tensile testing. The change in maximum stress is presented in relative change when compared to the reference sample that contains 0 % of PE, or 29 % of CaCO<sub>3</sub>. Based on Figure 29, It may be observed that the degree of crystallinity does not exclusively affect the maximum stress of the material, since the most dramatic increase in degree of crystallinity occurs between 0 % and 10 % of PE content, and no notable changes in maximum stress are observed between the values. However, the addition of PE increases the maximum stress of the material along with the increase in degree of crystallinity. Based on Figure 30, no clear conclusions regarding the degrees of crystallinity may be made due to the fact that the measured values for crystallinity are so close to each other, and the changes in maximum stresses are not particularly high. However, it may be concluded that the subtraction of CaCO<sub>3</sub> does not significantly deteriorate, or on the other hand, increase the ultimate strength of the material.



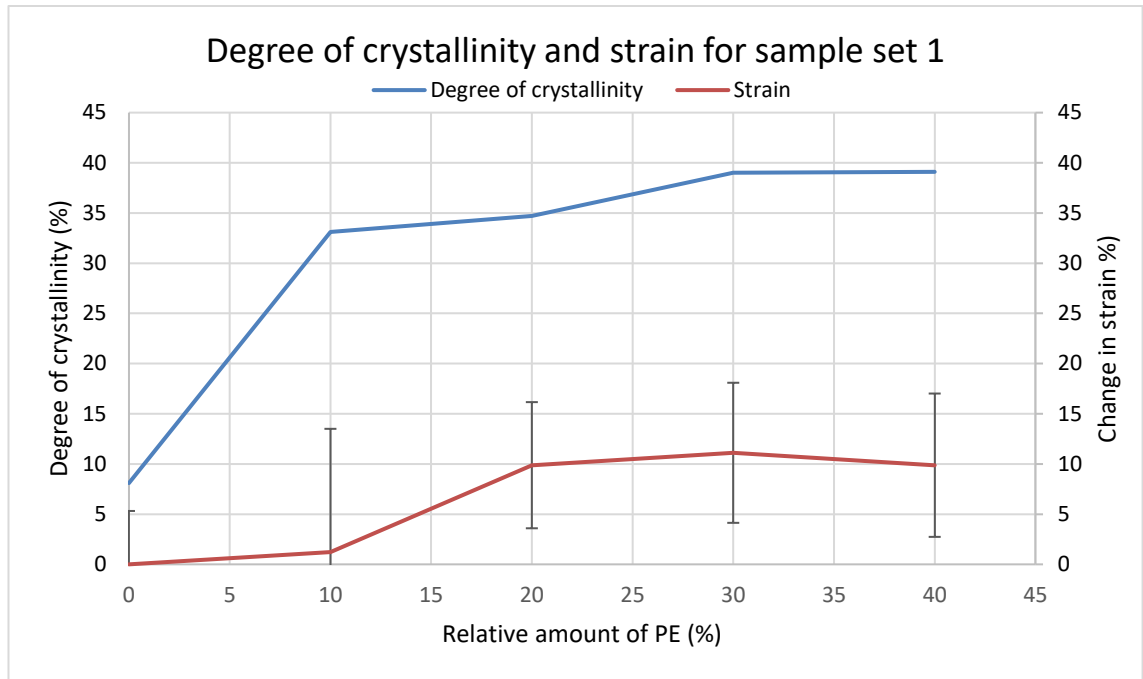
**Figure 29.** Degree of crystallinity and change in maximum stress for sample set 1.



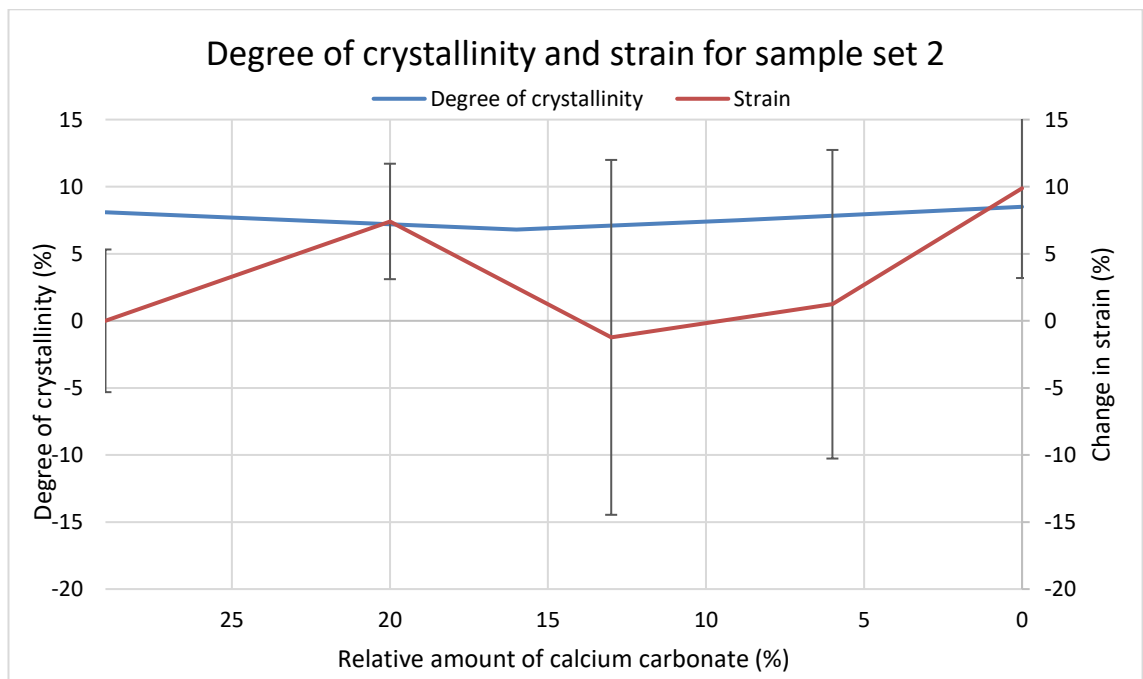
**Figure 30.** Degree of crystallinity and change in maximum stress for sample set 2.

Figures 31 and 32 illustrate the degrees of crystallinity, and changes of strains at maximum stresses for both sample sets based on tensile testing. According to Figure 31, it may be observed that the addition of PE increases the strain at maximum stress of the material up to 11 %. Based on Figure 32, no reliable conclusions may be made due

to similar reasons explained based on Figure 2. However, based on the results, it may be concluded that the addition of PE and subtraction of  $\text{CaCO}_3$  increase the ductility of the polyolefin film in most of the tested samples when compared to the reference film.

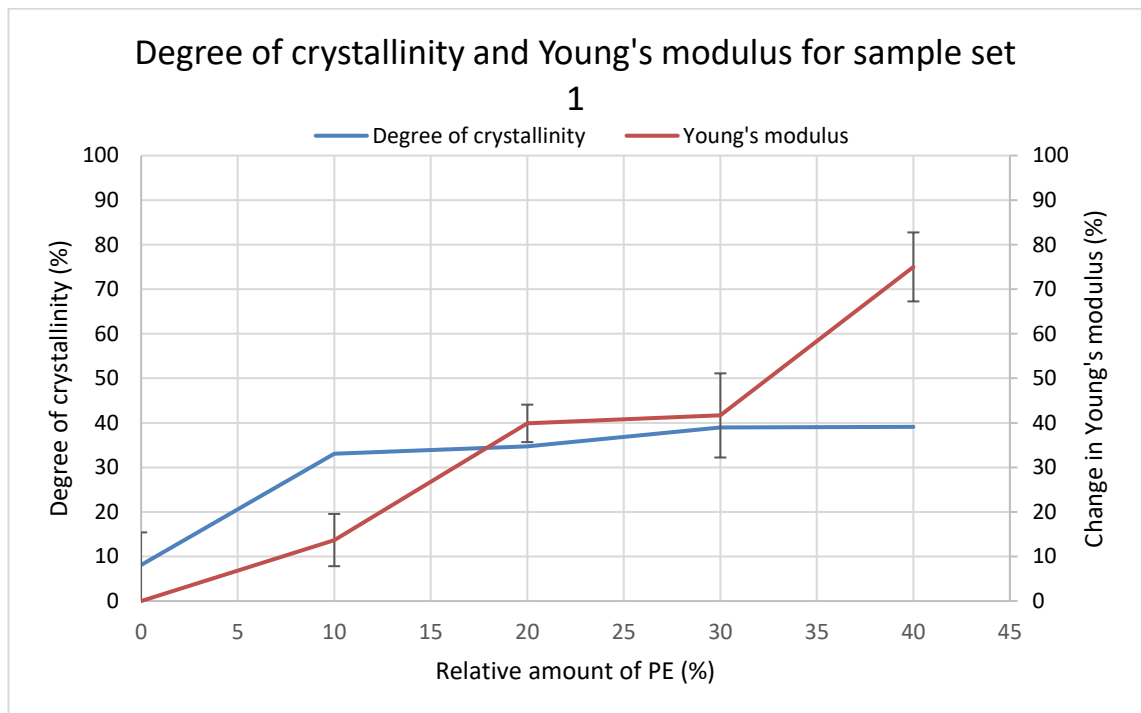


**Figure 31.** Degree of crystallinity and change in strain at maximum stress for sample set 1.

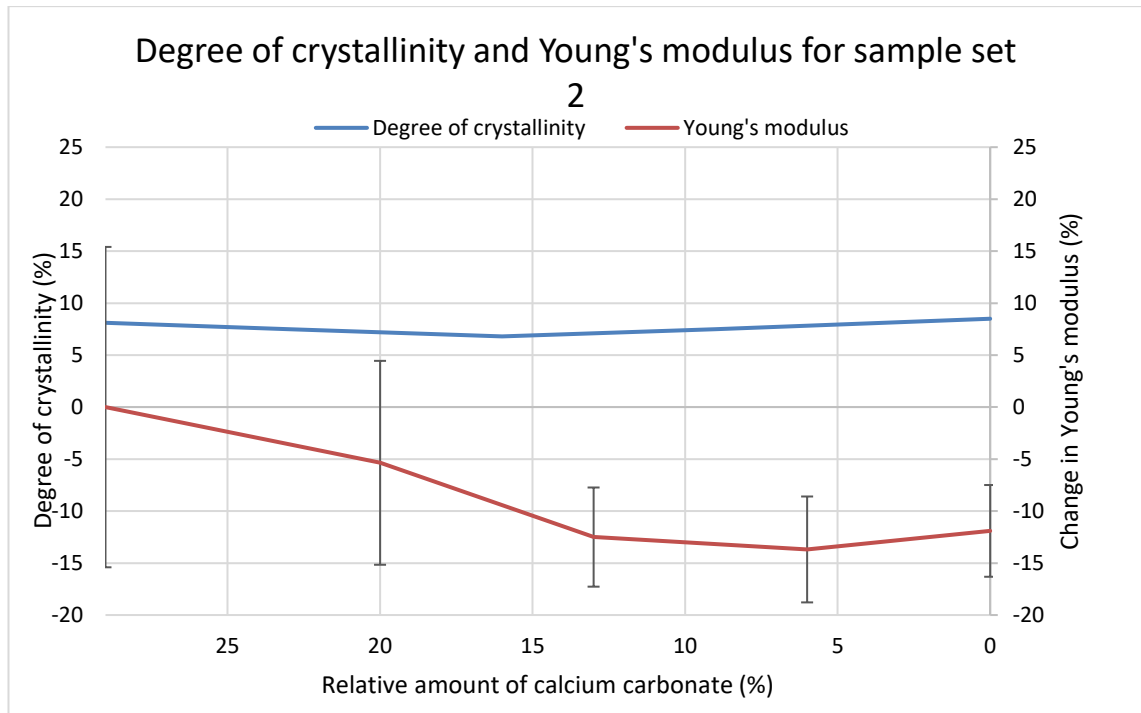


**Figure 32.** Degree of crystallinity and change in strain at maximum stress for sample set 2.

Figures 33 and 34 illustrate the degrees of crystallinity, and changes in Young's moduli for both sample sets based on tensile testing. When compared with the two other values being evaluated, stress and strain, the change in Young's moduli is the most distinct. According to Figure 33, it may be concluded that the addition of PE notably increases the Young's modulus of the samples. The highest increase is reached at 40 % of PE, being 75 %. On the other hand, based on Figure 34, the subtraction of  $\text{CaCO}_3$  reduces the Young's modulus of the samples notably. Based on these observations, it may be concluded that the addition of PE increases the stiffness of the material, whereas subtraction of  $\text{CaCO}_3$  decreases the stiffness of the material.



**Figure 33.** Degree of crystallinity and change in Young's modulus for sample set 1.



**Figure 34.** Degree of crystallinity and change in Young's modulus for sample set 2.

## 7. CONCLUSIONS

In this study, polyolefin films with different compositions were manufactured and their suitability for waterproofing applications was evaluated. The adjustment of the compositions included two separate trends: replacing proportions of the reference polypropylene film's plastic content with post-consumer recycled high-density polyethylene, and subtracting the amount of calcium carbonate filler in the reference film. Crystallinities, tensile properties, and water vapor barriers of the films were evaluated by means of different tests. Crystallinities were measured using differential scanning calorimetry. Tensile properties were determined via tensile testing. Water vapor barriers of the films were measured with specific water vapor permeability tests.

The samples were successfully prepared, and the measurement tests were conducted properly. The results that were obtained during the measurements in this study revealed that the addition of polypropylene films, indeed, notably alters their water vapor barrier and tensile properties. The replacement of polypropylene with polyethylene had a significant effect on the degree of crystallinity and water vapor barrier. The highest value for water vapor resistance was reached with a film that contained 40 % of PE and 60 % of PP, as the proportional increase was 46 % compared to the reference film. Additionally, the subtraction of the amount of calcium carbonate filler in the polypropylene film led to increased water vapor barrier but no significant changes in degrees of crystallinity were observed. The proportional increase of water vapor barrier in the film with 0 % of  $\text{CaCO}_3$  reached 35 % in comparison with the reference film with 29 % of  $\text{CaCO}_3$ . The completed tensile tests for every film revealed that the tensile properties of the films change along with addition. The presence of PE led to increases in maximum stress, strain at maximum stress, and Young's modulus. The subtraction of  $\text{CaCO}_3$ , according to the results, led to decreased maximum stress and Young's modulus, and increased strain at maximum stress. The amounts of the alterations in stresses and strains were determined to be minimal and insignificant. Even though the stiffness of the films increased up to 75 % with added PE, the changes couldn't be noticed when evaluating by hand.

This study concludes that the use of polyethylene is a promising method to enhance the water vapor resistance of polypropylene waterproofing films. Subtracting the amount of calcium carbonate filler also promotes the water vapor resistance of the polypropylene film. However, the use of PE is a more cost-effective method due to the fact that the use

of fillers is economical. The films did not exhibit remarkable changes in tensile properties and so far, the performance of the films may be concluded to remain suitable for the specific waterproofing application.

The film with 40 % of PE and 60 % of PP was determined to perform in a superior way when compared to other films evaluated in this study. Furthermore, the presence of polyethylene led to thinner films with satisfactory water vapor resistance and thus more films are possible to be manufactured with the same amount of raw materials when compared to the reference film. Finally, the fact that the used polyethylene was post-consumer recycled, leads to a more sustainable product and matches the objectives pursued in the SUPER healthy buildings ecosystem.

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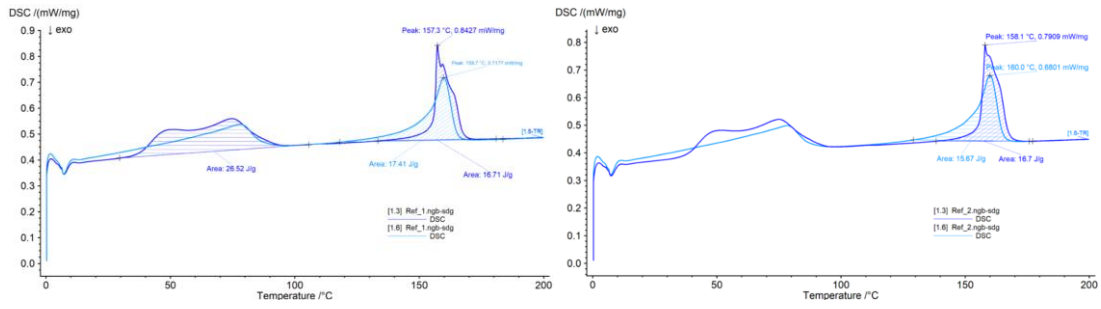
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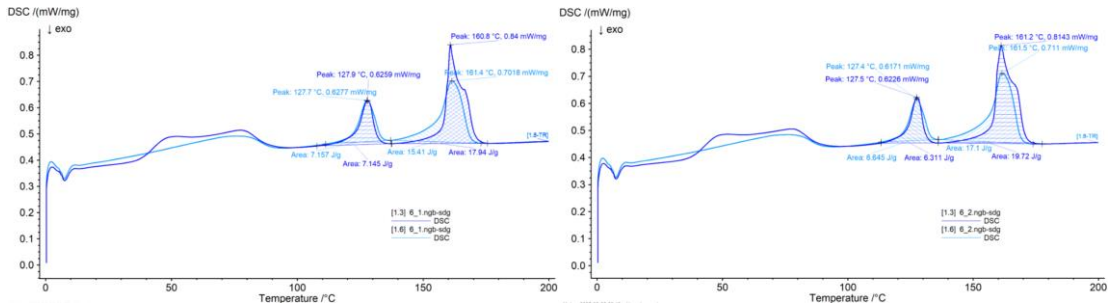
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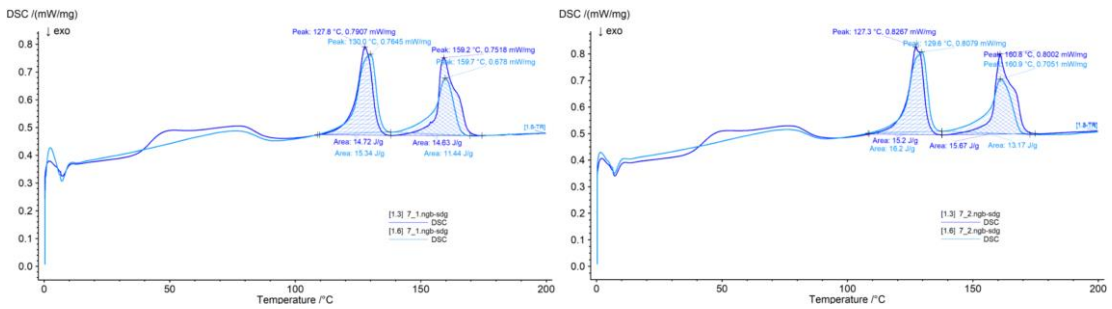
# APPENDIX A: DSC MEASUREMENT RESULTS



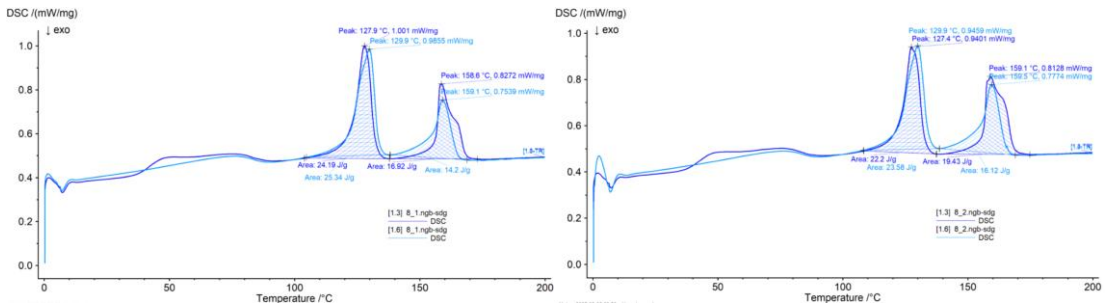
**Figure A.1.** DSC curves for sample 1 on both sample sets.



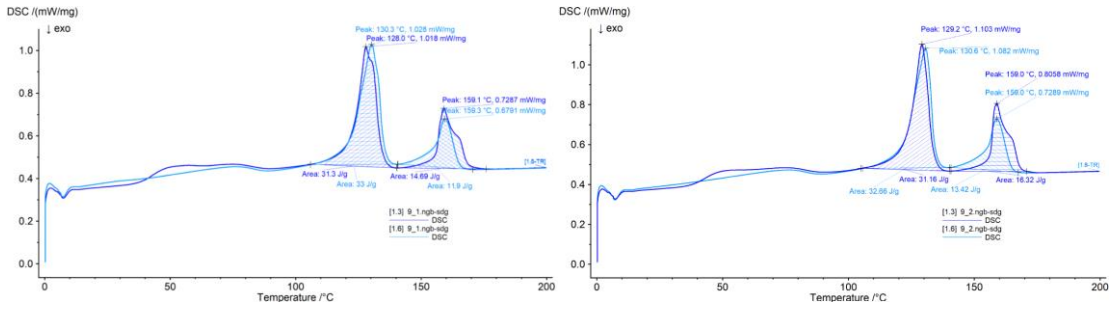
**Figure A.2.** DSC curves for sample 2 on sample set 1.



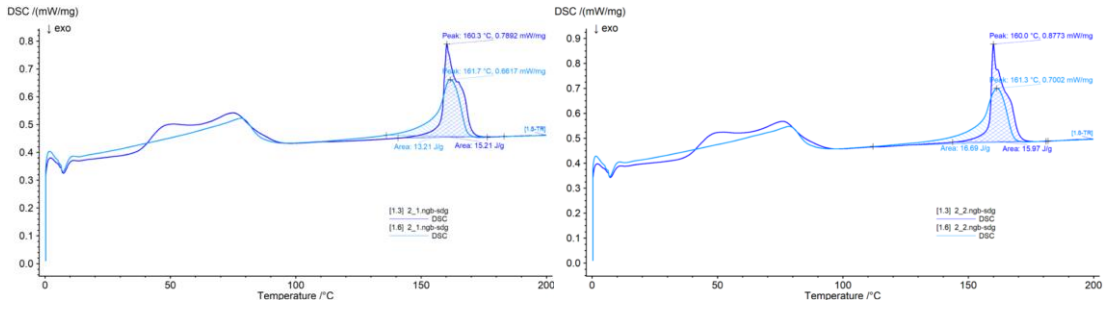
**Figure A.3** DSC curves for sample 3 on sample set 1.



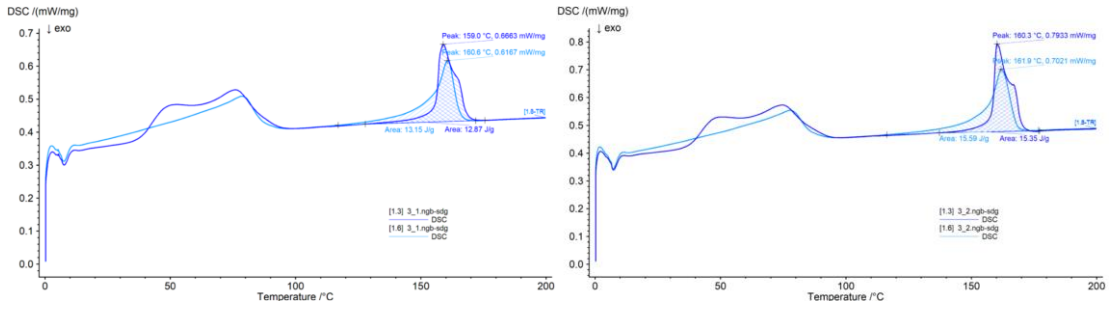
**Figure A.4.** DSC curves for sample 4 on sample set 1.



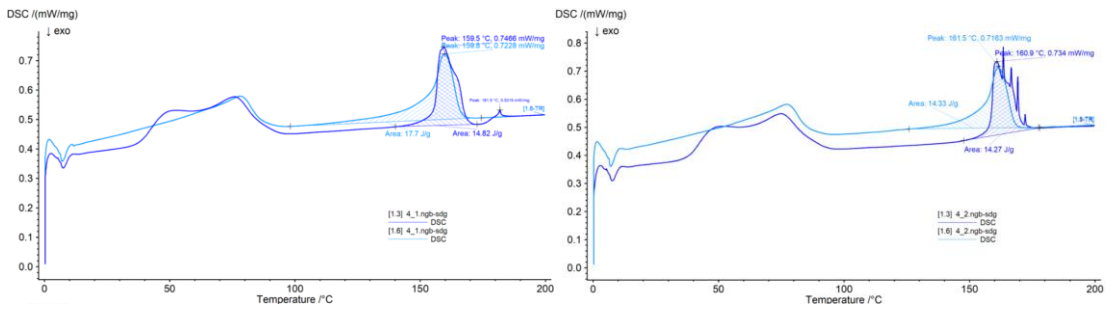
**Figure A.5.** DSC curves for sample 5 on sample set 1.



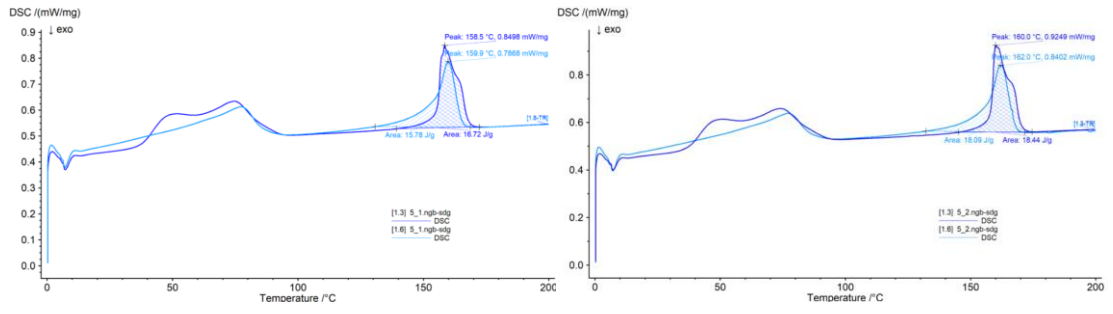
**Figure A.6.** DSC curves for sample 2 on sample set 2.



**Figure A.7.** DSC curves for sample 3 on sample set 2.

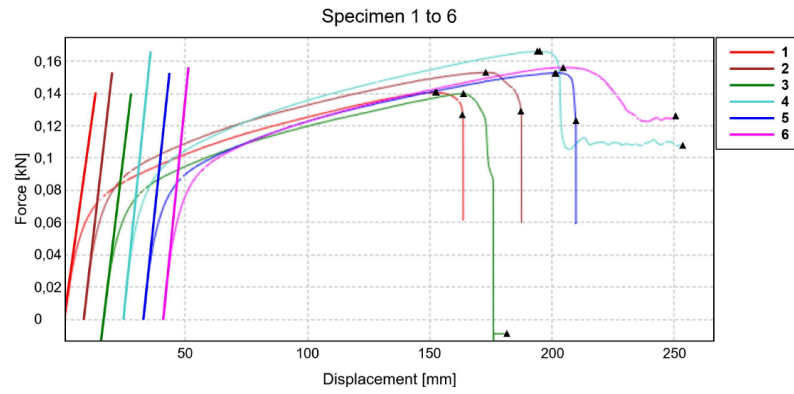


**Figure A.8.** DSC curves for sample 4 on sample set 2.

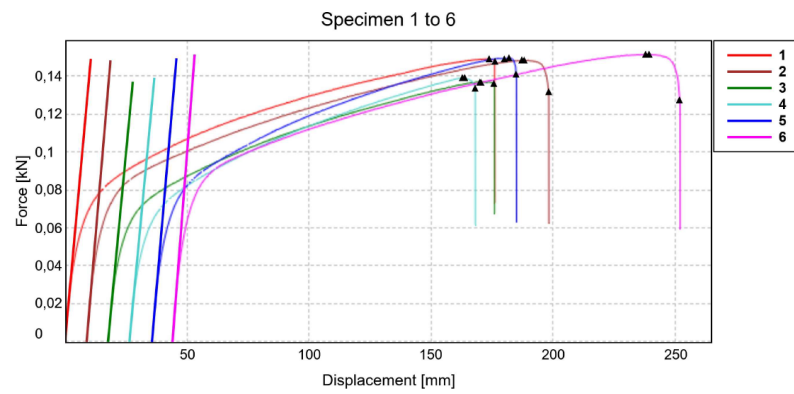


**Figure A.2.** DSC curves for sample 5 on sample set 2.

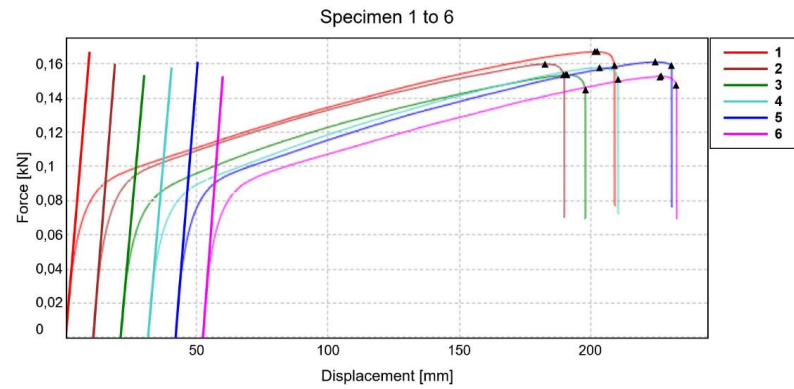
## APPENDIX B: TENSILE TEST RESULTS



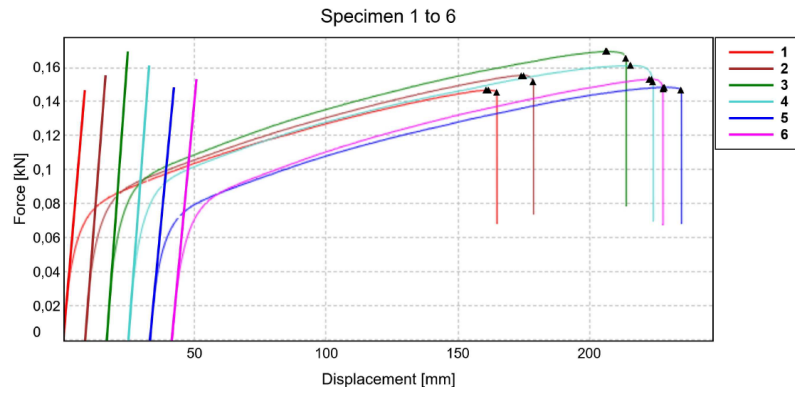
**Figure B.1.** Tensile test results for sample 1 on both sample sets.



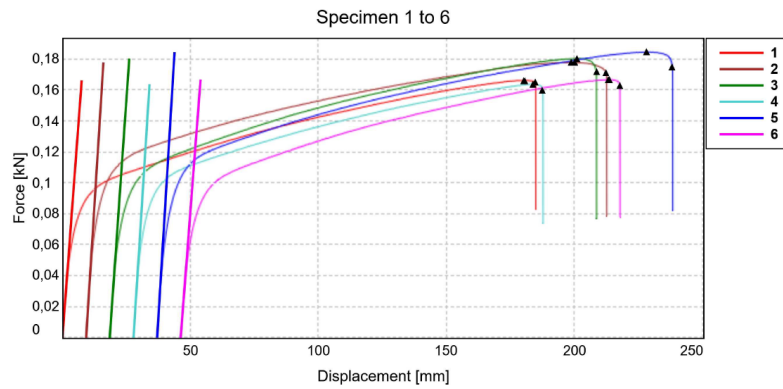
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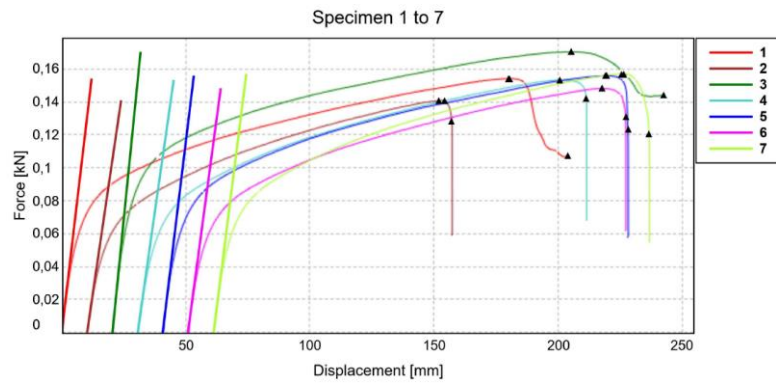
**Figure B.3.** Tensile test results for sample 3 on sample set 1.



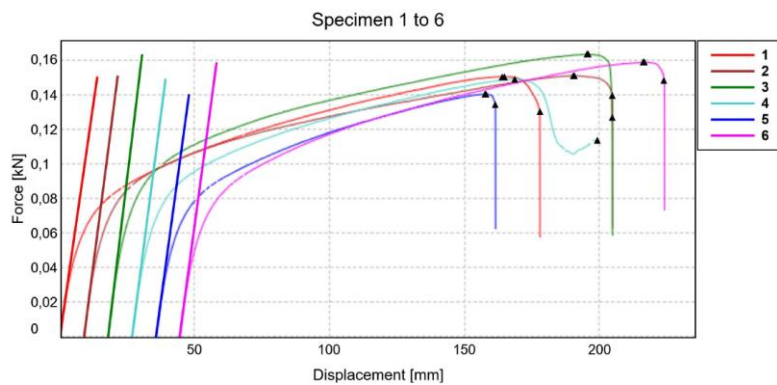
**Figure B.4.** Tensile test results for sample 4 on sample set 1.



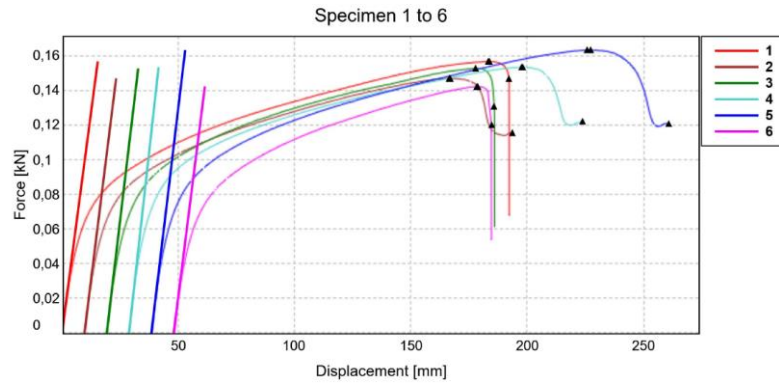
**Figure B.5.** Tensile test results for sample 5 on sample set 1.



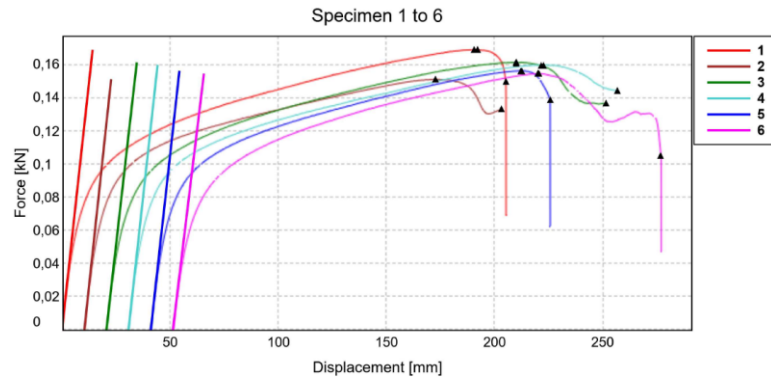
**Figure B.6.** Tensile test results for sample 2 on sample set 2.



**Figure B.7.** Tensile test results for sample 3 on sample set 2.



**Figure B.8.** Tensile test results for sample 4 on sample set 1.



**Figure B.9.** Tensile test results for sample 5 on sample set 1.