Characterisation of Bio-Based Polymers for Use in the Packaging Industry

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Abstract

Six polymer film samples, of unknown composition, were provided by a company called Greenlight Packaging for testing. Although the compositions were unknown to the company, as they were provided by a third party, the materials were believed to be PLA based and compostable. The conditions required and capability of the films to undergo biodegradation were unknown. The company were using the films to encase starch based loose fill in order to create a biodegradable cushion to replace expanded polystyrene within the packaging industry, due to the associated difficulties with disposal. During testing, it was noted that these films also had the potential to replace polyolefin films such as low density polyethylene and polypropylene used within the packaging industry. Therefore, throughout this project the properties of the six film samples provided were compared against these two conventional polyolefin films to identify whether the films had comparative physical and mechanical properties in order for them to be considered as compostable replacements. This study analyses the thermal, mechanical, and degradation properties of the six biobased polymer films along with expanded polystyrene, low-density polyethylene film and polypropylene film as references. The material compositions were unknown as the samples were provided by a third part, and were therefore identified using Fourier-transfer infrared spectroscopy by comparing the spectra to a database, which confirmed that all films were Polylactic acid based. Differential scanning calorimetry was used to identify transition temperatures, which were in the regions of 51-72°C and152-169°C. This identifies the temperature ranges at which the films can be used and gives information regarding processing. DSC was also used to identify the oxidation induction temperatures and oxidation induction time which indicated the materials had low thermal stability. Water and oil permeation tests were conducted to establish the barrier properties of the materials. This gave information on whether the material could be used for packaging applications where good barrier properties are required against water and oil. Results concluded that the polyolefins had much better barrier properties against water compared to the biopolymers, although all polymers tested performed similarly with oil as the permeant. Mechanical properties were measured through the use of tensile and tear tests. Samples were also subjected to various conditioning to simulate environmental factors such as freezing, thawing, and being subjected to elevated temperatures and humidity, during storage. The tear strength was then measured again after conditioning and values were compared to identify if conditioning caused any degradation and therefore reduction in strength. Very small changes were observed in the strength of the polymers after conditioning the samples. The stresses,

strains, and tear and tensile strengths varied throughout the polymers. The film was originally developed to encase starch polymer peanuts to form a cushion to be used as a substitute for expanded polystyrene (EPS). Therefore, the thermal conductivity of the films was measured and compared to values for EPS to ensure there was no interference in the insulating properties of the peanuts by the polymer films. All values were similar to that of EPS. The composting of the films was monitored over periods of 3, 6, 9, and 12 months. Samples were also left for 336 hours in an accelerated weathering tester prior to composting for 180 days to identify how UV exposure affects the degradation of materials.

Declarations and Statements

1. This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

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2. This thesis is a result of my own investigations, except where otherwise stated. Other sources are acknowledged by footnotes giving explicit references. A bibliography is appended.

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3. I herby give consent for my thesis, if accepted, to be available for photocopying and for interlibrary loan, and for the title and summary to be made available to outside organisations.

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4. The University’s ethical procedures have been followed and, where appropriate, that ethical approval has been granted.

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Abbreviations

BDP	Biodegradable Polymer
BOPP	Biaxially Orientated Polypropylene
CPP	Cast Polypropylene
DSC	Differential Scanning Calorimetry
EPS	Expanded Polystyrene
FDA	Food and Drug Administration
FMCG	Fast Moving Consumer Goods
FTIR	Fourier Transform Infrared Spectroscopy
GMA	Glycidyl Methacrylate
HDPE	High Density Polyethylene
HP	Hydrolysate Protein
IR	Infrared
LDPE	Low Density Polyethylene
LLDPE	Linear Low Density Polyethylene
mLLDPE	Metallocene Linear Low Density Polyethylene
OIT	Oxidation Induction Temperature
PBAT	Polybutylene adipate terephthalate
PBSA	Poly(butylene succinate-co-butylene adipate)
PDLA	Poly(D-lactide)
PDLLA	Poly(DL-lactide)
PE	Polyethylene
PEG	Polyethylene glycol
PET	Polyethylene Terephthalate
PGA	Poly(glycolic acid)
PHA	Polyhydroxyalkanoates
PHBV	Poly(3-hydroxybutyrate-co-3-hydroxyvalerate)
PLA	Polylactic Acid
PLGA	Poly(lactic-co-glycolic acid)
PLLA	Poly(L-lactide)
PMMA	Poly(methyl methacrylate)
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
SIS	Styrene Isoprene Styrene
UV	Ultraviolet

Chapter 1. Introduction.

i. Background

Plastic is a very favourable and popular material in the packaging industry. There are various types of plastics, all with varying properties. Rigid plastics are favourable for bottles due to its light weight, ease of processing and its flexibility and has become a common replacement for brittle glass. Films are also common within the packaging industry to increase convenience for customers. Films are lightweight, often clear, but can be easily dyed and antioxidants can be added to increase the shelf life of the product.

A rise in demand for these polymers has resulted in a rapid growth for the manufacturing industry resulting in an increase in production of plastics and films including hydrophobic films Low-Density Polyethylene and Polypropylene. Polyolefins such as these accumulate in the natural environment due to irresponsible disposal and their extensive lifespans.

ii. Motivation

Over recent years, we have seen an increase in plastic pollution and concerns have been raised about the welfare of our oceans and their ecosystems. Increased pressure has been applied to governments and companies to tackle the plastic crisis which we are currently facing. Various policies, laws and commitments have been created to improve the quality of our waters and reduce the effect that plastics have on our environment. One of these policies which many companies within the grocery industry have committed to is The UK Plastic Pact. This is a roadmap for achieving various targets to change the way the UK uses and disposes of plastic with a move to 100% reusable, recyclable or compostable packaging by 2025 as one of these targets.

Reusable alternatives, such as cotton and multi-layer plastic bags for life, are favourable as no waste or disposal is involved. The issue with many reusable alternatives is that consumers are responsible for limiting the number of ‘reusable’ alternatives they purchase. Many reusable alternatives also require more energy and water to produce than single use plastic packaging.

Recycling is another solution to landfill and incineration, but it comes with its downfalls. Issues with contamination, separation and sorting along with the complexity of materials and limited recycling facilities available are currently reducing the rate at which we recycle our waste. The facilities in the UK are not currently equipped to recycle many types of plastic; one of which is PP films. LDPE film is becoming more regularly recycled, but is not currently collected by the majority of Welsh councils.

Due to issues with reusable and recyclable alternatives to conventional petroleum-based plastics, compostable options must be explored. There has been an increase in interest and research into biodegradable polymers (BDPs) in recent years to combat the issues presented by other waste disposal methods. PLA is one of the most popular and researched BDPs (discussed in further detail in the literature review). It is a recyclable polyester produced by the polymerization of lactic acid. It is compostable and produced from renewable sources. It can be produced in rigid form or as a film through a variety of processing techniques including additive manufacturing, thermoforming and blow moulding. Applications of PLA include packaging, drug delivery and textiles. It is a niche material with growing interest and researchers have been focused on creating PLA blends to optimise the materials properties.

Biopolymers such as PLA generally have substandard properties compared to conventional petroleum based films such as lower strength and durability. Due to this, blends are often created in order to benefit from the increased strength of petroleum based polymers and the biocompatibility and biodegradability of biopolymers. Although the addition of the petroleum based polymers can improve the mechanical properties of PLA, it causes issues with the biodegradation process. This leads to issues and conflicting opinions on its benefits to the environment and resulting in the greenwashing of many products. Some biodegradable polymers are also designed to degrade in an industrial facility where elevated temperatures are required, as it is a more aggressive process. This requires energy and also still relies on responsible disposal. For this reason, this project uses a home composting facility to analyse the biodegradability of the materials as this method uses the least amount of energy and is more representative of the natural environment.

iii. Materials Provided

Greenlight Packaging are a Welsh company who develop sustainable packaging including compostable films. Their main product is a box formed by six cushioned panels made from starch based peanuts encased in a polymer film. This was developed as a compostable alternative to expanded polystyrene used within the food delivery services. Greenlight Packaging were looking at developing their products further and were supplied with six films by external suppliers, that had unknown compositions but were believed to be compostable and polylactic acid based. It was noted that these films physically resembled petroleum films currently used within the packaging industry. Therefore, the decision was made to investigate these films further in order to understand their properties and conclude whether they would be suitable compostable replacements to polyolefin films low-density polyethylene and polypropylene. Greenlight Packaging also wanted to understand more about each of the films separately to decide on an optimal film for the development of another project they were working on.

The tests will give insight into the properties of the films in order to characterise the polymers and determine if they are suitable replacements to these conventional petroleum based films for use within the packaging industry.

iv. Testing

The thermal, mechanical and degradation properties were evaluated and compared to materials commonly used within the packaging industry. The degradation of the films were monitored over various time periods. Films were also left in a composting facility for a period of 6 months following 336 hours (2 weeks) of accelerated weathering, to determine if UV irradiation had an effect on the degradation of the polymers.

Fourier transform infrared spectroscopy was used to determine the composition of the materials and the functional groups present which confirmed the materials provided were Polylactic Acid. Once the materials had been identified, Differential scanning calorimetry (DSC) was used to identify key state changes in the material and results were compared to literature. This provided the transition temperatures of the materials which were compared with literature of PLA. DSC was also used to determine the oxidation induction temperature and oxidation induction time. These are the temperature at which oxidation starts to occur and the time until oxidation begins, at this defined temperature, consecutively. This gives further information about the materials stability and resistance to oxidative decomposition. It is used to predict the thermal-oxidative performance of material and also as a test method for the identification of biodegradation.

Thermal conductivity of the films was measured to identify the insulating properties of the films and two different arrangements of the starch loose fill. As the films had the potential to be used within an application allowing it to replace EPS, it was essential that the films had very little interference with the insulating properties of the structure. The liquid barrier properties of the films were tested through permeation tests using water and oil as the permeant. This allowed the identification of which films had comparable barrier properties to the polyolefins also tested, which were collected from supermarket packaging. This gave insight into whether the films could be used in food packaging applications where good barrier properties to moisture are required to protect food from external factors. The films ability to prevent diffusion was also measured for applications where retention is required, for example to prevent the leaking of foods containing liquids such as oil. The weight loss over a given period of time was monitored. This indicated the percentage of permeant that had diffused through each of the films.

Mechanical properties were tested through a series of tensile and tear tests. Tensile tests were conducted on each of the eight films. Tensile testing gives an insight into the forces the films are able to withstand before failure and therefore the masses of the products they can package. This is a comparative study and results will be compared with LDPE and PP films that are currently on the market for the packaging industry. Tear tests were conducted on plain, unconditioned samples. Samples were also subject to various conditioning, including low temperatures, high temperatures and humidity, to simulate environmental effects. Tear tests were then conducted on these conditioned samples. Results were compared to those from the unconditioned samples to understand the effect of environmental conditions on the mechanical properties. Conditioning the samples gave insight into how environmental factors such as freezing, heating and storing food at high temperatures could impact the mechanical properties. Tear tests allow the identification of the force required to propagate an existing tear, which indicates the force required to cause further damage to the packaging if a defect were to occur. The packaging of foods often endure low stresses due to their usage, and are more likely to be affected by defects in the materials. Therefore, tear testing was chosen to measure any changes in the mechanical properties post conditioning.

The films were placed in a home composting facility to gain more representative results and to determine whether the films would degrade if users were to compost the packaging at home, or potentially if the films were to enter a natural soil environment. As industrial composting facilities operate at elevated temperatures, it can be assumed that if the polymers degrade in the home composting facility, they will degrade in an industrial composting facility. Samples were subject to composting conditions and various methods were used to determine the level of degradation. Samples were inspected at a number of intervals and images were taken. FTIR was used to identify any changes in the functional groups present in the polymers. DSC was used to determine any changes in the glass transition temperatures, melting temperatures and oxidation induction temperatures as a result of degradation due to composting. Samples were also tear tested after 180 days in the home composting facility to determine any changes in the mechanical properties.

Samples were artificially weathered by exposure to UV and humidity in an accelerated weathering machine and placed into the compost facility. After 180 days, samples were removed and the level of biodegradation was measured using the same methods as stated for 180 days composting without UV-irradiation. Results were compared to the results from non-irradiated samples to understand the effects of UV on the polymers degradation. Accelerated weathering speeds up the effects that UV and humidity, from the natural environment, have on the polymers. This enables the identification of the films which are most affected by these factors and therefore are more likely to degrade in the natural environment. It also gives an insight into whether UV pre-treatment could be considered as an option to increase the rate of biodegradation.

For each of the tests conducted, PP and LDPE were also tested and results were compared.

Chapter 2. Literature Review

Within this literature review, an overview of polymers is given along with how their production has increased and the influence they have on the world we live in today. Their applications in industry will be discussed, focusing on their applications in packaging, following on to further details about plastic films within the packaging industry. Key materials and their properties will be covered, focusing on low density polyethylene, polypropylene and polylactic acid. Their properties will be discussed and the issues they cause to the environment has been documented. Solutions to the issue have also been cited including action in which the government are taking to resolve the problem, recycling as a potential solution, leading on to compostable and other 'environmentally friendly' polymer alternatives. The issues of greenwashing are also discussed and the differences between various adjectives used to describe polymers have been concluded and explained. As the focus of this project is PLA based polymers, further details on PLA have been researched and included within this review including its properties, blends and applications. The degradation of biobased and petroleum based polymers have been researched and cited, with in depth explanations of various methods used to measure and record biodegradation.

2.1 Polymers

Plastics are organic polymers consisting of long carbon chains. (1) The word plastic is derived from the Greek word "plastikos", meaning able to be moulded. (2) The first synthetic plastic was invented in 1869 and while initially developed as a substitute for elephant ivory used in the manufacture of piano keys and billiard balls, the versatility of the material allowed for production in other sectors including toys and glasses frames. (3) Although the polymer became superior to ivory, it had one major defect. It burned easily. Due to its flammable nature, a new gap in the market was created which was later filled in 1907 by Bakelite.(4) Due to a lack of raw materials during WWII, plastic became a material of interest with chemists all over the world working to create new polymers.(5) Since then, global plastic production has grown exponentially with 368million tonnes produced in 2019 compared to the 1.5 million tonnes in 1950.(6)

The monomers present and their arrangements determine the material and its corresponding properties. Most common polymers are comprised of carbon backbones, such as polyethylene, and are derived from non-renewable feedstock such as crude oil.(7,8) It is estimated that approximately 4% of gas and oil production is for the use of plastic production, with a further 3-4% of these resources used for the energy involved in the manufacturing of the polymers. (9) Some authors argue that due to their low weight compared to other materials, the environmental impact is outweighed due to reductions in transport emissions and costs, lower production costs and reduction in space required during storage and transport. (9,10) They have a variety of properties that also make them more favourable than other materials, boosting their use and production.

2.1.1 Properties of Plastic

Plastics have a combination of properties which make them superior to other materials, resulting in their widespread applications. (11,12) They are produced with different compositions providing each material with varying properties allowing for applications in multiple sectors. Generally, they are lightweight, can be coloured or transparent, easy to process and resistant to corrosion. (13)They are cheap to produce and can be processed through a wide variety of techniques including extrusion, injection moulding and rheology. (14) They are durable and flexible making them a good substitute for heavy, brittle materials such as glass, with plastic replacing glass in many applications including bottles, screens and packaging. (15) Due to their low weight, plastics are also starting to replace metals such as aluminium in aerospace designs, with the Boeing 787 Dreamliner fuselage and wings made almost entirely from plastic composite.(16)

Plastics can be divided into thermosets or thermoplastics. Thermosets are crosslinked polymers which form 3D networks on curing. (17,18) The formation is an irreversible process and therefore they have higher temperature resistance to heat.(19) They are strong and rigid and once cured, cannot be re-melted, hence they are difficult to recycle. (20–22) Commonly used thermosets include unsaturated polyesters and epoxy resins.(23)

Thermoplastics on the other hand are made through condensation or addition polymerization. (24) They can be softened and melted when exposed to heat and hardened on cooling, allowing them to be easily recycled.(25) They can be crystalline, semi crystalline or amorphous and depending on the level of crystallinity, they will exhibit a glass transition temperature, a melting temperature or both. (26,27) The crystallinity of a polymer can affect the hardness and strength of a material and is governed by density.(28) Increased crystallinity results in a more rigid polymer as crystallinity can restrict the movement of the polymer chains. (29)

The melting point of plastics range widely with polycaprolactone having a melting point at just 60°C to polytetrafluoroethylene with a melting point of 346°C . (30,31) Mechanical properties also vary among plastics allowing for applications in various sectors.

2.1.2 Applications of Plastic

Plastic is a very versatile material and is encountered every day due to its wide variety of applications in all areas thanks to its various properties. Industries include packaging, building and construction, technology, textiles and transportation. (32–34)

Due to their wide range of melting temperatures and availability of thermosetting polymers, plastics deliver adverse versatility for applications at a range of temperatures including for plastic cooking equipment, packaging that requires sterilizing at high temperatures and aircraft parts. (10,35,36) Due to vast amounts of research that has gone into the development of plastics over the years, advances have also enabled them to be used in the medical industry with uses for surgical equipment, drips, the 3D printing of prosthetics and packaging for medication.(37,38) Plastics also have high thermal and electrical insulation properties allowing materials such as expanded polystyrene (EPS) to be used as insulation in construction. (39) Conducting polymers possess excellent characteristics including high environmental stability and tuneable electrical properties and can be combined with other materials to overcome limitations. These properties allow for various applications within the electronic industry.(40) Polyethylene and nylon fibres are used in construction to reinforce concrete, improving its properties. Tensile strength, crack resistance and ductility of concrete have all been found to increase with the addition of polymer fibres.(41,42) Due to advances, fibre reinforced concrete is widely used in structures such as bridges, foundations and buildings. (1) Within construction, plastics can be used for plumbing, window and door frames as well as other fixtures due to their lightweight, corrosion resistance and durability. (1,8,43) Due to its versatility there are various applications for this material in all sectors with a large portion of its use in packaging.

2.2 Packaging Materials

The UK packaging industry is estimated at £10 billion with 3% of the total UK workforce working in the industry.(44) 42% of all non-fibre plastics manufactured are produced for the packaging industry with packaging accounting for 70% of UK plastic waste.(45) Primary packaging is the packaging that is in direct contact with the product and most commonly used by consumers.(46) It is often single use and is discarded immediately after use.

Plastics are extensively used within the packaging industry with applications in pharmaceuticals, food, groceries, cosmetics and toys. (47) Packaging serves a variety of purposes. Some purposes are necessary and some unnecessary. Firstly is containment. This is when an item is packaged to provide convenience for the consumer and is usually unnecessary packaging.(10) Secondly is protection. Some packaging is required to protect the product from environmental factors such as dirt, oxygen or chemicals. It is often used to preserve food and adding antioxidants to plastics can increase the shelf life of products. (48) Certain products require instructions or additional information and so necessary packaging is used as a method to provide this. Plastic films can be easily printed allowing essential information such as ingredients to be conveyed. Finally, unnecessary packaging that is used to attract customers to the product through the use of colours and imagery to stand out from competitors.(49)

2.2.1 Properties of Packaging Materials

Many different materials are used within the packaging industry including plastics, glass, metals and paper.(50,51) Each material has different properties and is used for different applications. Metallic cans are used to package long life food items as metal has excellent barrier properties against moisture and gas, has excellent mechanical properties and easily recyclable. (52) Due to its tendency to react with certain chemicals found in tinned foods such as tomatoes and fish, cans are often lined with a polymer coating to reduce the risk of corrosion.(53) Glass also possess excellent barrier properties and is used to store items such as alcohol.(15) Paper has very weak barrier properties due to its hygroscopic structure. (54) Therefore, paper and cardboard is often coated with plastic to improve the barrier properties against water and air. (55)

Plastic is a favourable material within the packaging industry as it is tough, durable, cheap and easy to produce with a lower production energy than glass and metal.(56–58) Flexible packaging such as films are commonly used in the packaging industry due to its extreme lightweight and high strength to weight ratio.(10,59)

2.2.2 Plastic Films

Plastic films are defined as a thin continuous plastic of thickness less than 10mil, 0.254mm. (10) The same as rigid polymers, films can be made with a variety of resins including polyethylene and polypropylene, or a combination of resins. (60–62) Single layer films are films that have a single layer consisting of a single resin. In the food packaging industry, they are commonly made from LDPE and are sealed with a tie such as a bread bag.(10) Multilayer films can be different materials that are combined via coating or lamination, or due to advances in technologies, through coextrusion.(63) Multilayer films are often used to package ‘boil in the bag' food items as the polyethylene terephthalate layer provides strength at high temperatures and the polyethylene layer is required for heat sealing. (63) Multilayer packaging also allows for improved mechanical properties. (64)

Flexible packaging such as films require less energy to produce compared to rigid plastics and due to its low weight to strength ratio, less material is required. This reduces the costs and impact on the environment, although a large portion of food packaging materials are multilayer plastics which are not yet recyclable in a cost-effective manner and pose problems for the recycling process.(56)

In the UK, around 290,000 tonnes of plastic bags and wrapping are thrown away every year but a mere 6% of this is recycled.(65) The main types of polymers used to manufacture films are low density polyethylene (LDPE) and polypropylene (PP) with these flexible plastics accounting for 25% of all consumer packaging.(65) It is estimated that around 1.2 million tonnes of plastic film from packaging arises in the UK waste stream every year. Of this, two thirds are post-consumer and one third from commercial, industrial, and agricultural.(66)

i Polyethylene

Polyethylene is the most common plastic and in 2017 accounted for 34% of the total plastic market. (67,68) It is structurally simple and is made up of a long chain of carbon atoms with two hydrogen atoms attached to each carbon atom and are produced by low-pressure polymerization. (69,70) There are several different types of polyethylene, depending on the degree of branching. Each type has varying properties including densities, mechanical properties and molecular weights. (70–72) The most sold grades of polyethylene are high density polyethylene (HDPE), low density polyethylene (LDPE) and linear low density polyethylene (LLDPE). (68) LDPE is soft and flexible, and has a high elasticity. (73) These properties make it ideal for use in the production of films. The density values range between 0.91 g/cm^3 and 0.925 g/cm^3 compared to HDPE which has densities greater than 0.95g/cm^3.(74) The branching in LDPE results in lower densities and makes it more susceptible to crosslinking during extrusion. (75) It is a low cost, recyclable material and is widely used within the packaging industry due to its barrier properties, strength and flexibility. (76,77) Processing LDPE is among the easiest of the plastics, with the potential for injection moulding, rotational moulding, film blowing and blow moulding.(78). It has a semi crystalline structure relatively low melting points of 105-112$^{℃}$ and low temperature impact resistance.(79) It can also be blended with a variety of materials including other polyolefins, biodegradable polymers, fillers and pigments to alter its properties.(80,81)

Although it is a thermoplastic, LDPE is not commonly recycled due to an increase in crosslinking and molecular weight during reprocessing.(82) Therefore the main method of disposal is landfill.

ii Polypropylene

Polypropylene is a low cost linear hydrocarbon thermoplastic.(83) It is formed through addition polymerization of the alkene monomer propylene and processing techniques include extrusion and injection moulding.(84) It was founded by Giulio Natta after he utilised the catalyst developed for PE. It became very popular in the film industry and soon replaced the organic biopolymer cellophane due to its lower associated costs.(85)

Polypropylene accounts for 23% of total plastic production.(86) It can be produced as a rigid polymer or as a flexible film and has a wide range of use in medical devices, textiles and for packaging.(87) Polypropylene films have excellent barrier properties to moisture but average gas barrier properties and so it is often coated or copolymerized where longer shelf life is required. (88) It also has excellent chemical resistance and high clarity. (89) It has a higher melting point than PE films of around 170°C making it a more desirable material for applications requiring higher temperatures, although the heat sealing properties are not as good as PE.(90) It also has high gloss and good tensile strength.(91)

There are two common types of polypropylene film. These are cast unoriented polypropylene (CPP) and biaxially orientated polypropylene (BOPP).(92) CPP is less commonly used in industry with BOPP making up about 80% of polypropylene film packaging. (93) Biaxially orientated polypropylene is stretched in two directions; machine and transverse. This produces molecular chain orientations in two directions, increasing tensile strength and stiffness along with lowering elongation. (91)

During the Great British Beach Clean Up, 718 pieces of litter were found for every 100m stretch of beach, with rubbish from food and drinking accounting for at least one fifth.(94) Food wrappers, plastic grocery bags and other plastic bags were 3 of the top 10 items found in coastal clean ups in 2017. (95) These items have limited disposal methods, hence end up in landfill.

2.2.3 Environmental Issues with Plastics

Globally, around 13.5% of plastic waste is disposed of through recycling and 5.5% through composting. The remaining is disposed of through landfill, incineration or is openly dumped.(96) Lower developing countries, where various waste disposal methods are not available, are a growing concern for the leakage of waste plastic into the natural environment. (95) An increase in plastic consumption and poor waste collection services and disposal methods have resulted in governments exporting their waste to less developed countries. The landfill sites in these countries are often located near water ways such as rivers, which act as a medium for leakage of marine plastics into the ocean. Developing countries account for around 80% of leakage into the oceans.(97) Reports of plastics in the ocean date back to the early 1970s, and have since accumulated at an alarming rate with Lebreton et al estimating that 1.15 to 2.41 million tonnes of plastic waste enter the ocean each year. (97) It is evident from numerous images of floating debris that plastic waste is an issue in the ocean. Garbage patches have formed due to the estimated 5.35 trillion pieces of floating plastic equating to a weight of 268,940 tonnes with the largest garbage patch, Great Pacific Garbage Patch, spanning over 1.6 million km^2. (97,98) Surveys by Barnes et al also revealed that plastic debris has been found on the shores of the remotest islands. (99)

The quantity of plastics entering our oceans is forever increasing with increased plastic production.(100) Plastics are very persistent and many contain additives and chemicals which are toxic to animals and humans.(97,101) Many studies have found that plastic pollution in the ocean has the potential to impose a risk on human health.(102–104) One study showed that between 400,000 and a million people in less developed countries die each year from diseases attributed to plastic pollution, such as diarrhoea, malaria and cancer. (105)

Plastic pollution also has devastating consequences for marine life and birds. 100 million marine animals die each year from plastic waste alone. Recent studies showed that 100% of turtles, 90% of seabirds, 59% of whales, and 36% of seals had plastic in their stomach with the first record of plastic ingestion by sea birds dating back to the 1960s. (97,106) It was found that 1-2mm plastic particles were able to pass through the digestive systems of Northern Fulmars. (107,108) Over a period of 4 years, 93% of Northern Fulmars surveyed had traces of plastic with an average of 33 pieces per bird.(109) A study by Desforges et al found microplastics in two species of zooplankton, which are organisms located at the bottom of the food chain, presenting the risk of introducing plastic into the food chain. (110) Microplastics are defined as plastics with a particle size of 1 μm^5 and are a growing concern for the world's oceans due to their ease of ingestion by filter feeders and other marine species. (111–113) Bans have been put in place to reduce the use of microplastics in industries such as cosmetics but microplastics also accumulate due to the partial degradation of larger plastics.(101)

Marine environments cover 71% of the earth's surface and is home to more than 226,000 eukaryotic marine species and is the source of 17% of edible meat, with this figure set to rise by 2050. (114–116) There are growing concerns and increased research into the potential for microplastics ingested by marine species to reach humans through the food chain.(97) A study by Hurt et al found that 100% of bass monitored in two agricultural reservoirs in midwestern USA had concentrations of microplastics, thus introducing plastic into the food chain. (117) Rochman et al studied various seafood for sale for human consumption in Indonesia and the US and found anthropogenic debris in both.(118) The Austrian Environment Agency published an article which disclosed the presence of microplastics, including polypropylene, in human stool samples. The method of ingestion was undefined and could be due to inhaled fibres or oral ingestion or a combination.(97) A study by Deng et al highlighted the effects that microplastics had on mice. Microplastics were found to accumulate in the kidney, liver and gut, dependent on particle size, and altered metabolisms, induced oxidative stress and indicated toxicity. (119)

2.3 Recycling of Plastics

Recycling is an alternative waste disposal method and solution to landfill. It is an essential contributing factor to a circular economy and has the potential to reduce the release of waste into the natural environment. (73) After decades of exponential growth in the production and consumption of virgin plastic, use of virgin plastic has peaked with a reduction in consumption of 0.6% (2018-19) and 1.2% (2019-20). (120)

Recycling is a process that allows a material to be processed and converted into a new product. Various technologies allow collected polymers to be sorted by colour, density, melting point and various other categories. (121) There are various methods of recycling polymers depending on the type and grade; primary, secondary, tertiary , quaternary and biological. (122)

2.3.1 Mechanical Recycling

Primary mechanical recycling is a basic method of recycling, often conducted by manufacturers, and is used for the recycling of uncontaminated polymers without affecting its properties. (122) The process requires the polymer to be shredded into fragments where they undergo processes to eliminate impurities such as paper labels. This makes the material more homogeneous and eases the blend with additives and other polymers. The material is then melted and often extruded in the form of pellets which are then sent to manufacturers and used to manufacture new products.(123) Other processing techniques such as injection moulding and heat pressing are also used, therefore only thermoplastic polymers can be mechanically recycled. Benefits of mechanical recycling include ease of impurity removal, ease of processing and allows for quick integration back into the production cycle. (124)

Secondary mechanical recycling involves the separation and purification of polymers as content and purity are often unknown. Chain scission occurs resulting in a reduction in molecular weight and often a reduction in mechanical properties. (125) To combat this issue, various stabilizing additives are often used along with intensive drying or vacuum degassing. (126) A decrease in mechanical properties can also be caused by the contamination of other polymers as most polymers are not compatible with each other.(127) Polypropylene is the most relevant contaminant for mechanically recycled polyethylene, due to sorting faults and packaging components, and cause a reduction in the mechanical properties compared to pure polyethylene. (128,129) This reduction in properties result in a less valuable end product. (9) Fourier Transform and Near Infrared can be used to identify the polymer matrix which reduces the risk of contamination through the sorting process. (9,128)

Some bioplastics such as polylactic acid (PLA) can be mechanically recycled a few times without significantly reducing the properties although the lack of available bioplastics for recycling make the process less viable.(130)

Mechanical recycling capabilities are currently controlled by cost, quality of recyclable plastic and degradation of mechanical properties although due to its lower cost compared to other recycling methods and low impact on the environment, it will remain the most effective and commonly used method. (73)

2.3.2 Chemical Recycling

Tertiary recycling, also known as chemical recycling, converts polymer chains to smaller molecules. (131) Processes include pyrolysis and hydrolysis, and typically use liquids and gasses as feedstock for the production of new polymers. (132) It is generally used to recycle polymers that are no longer suitable for mechanical recycling and is used as a last resort due to complexity and cost. (73)

Polymers including polylactic acid (PLA), formed through polycondensation, can be efficiently depolymerized and reprocessed through chemical recycling.(133) This method is expensive due to the depolymerization of the polymers.(134)

2.3.3 Issues With the Recycling of Plastics

There are currently seven categories of recyclable plastics which all require different recycling processes. These are Polyethylene Terephthalate (PET #1), High Density Polyethylene (HDPE #2), Polyvinyl Chloride (PVC #3), Low Density Polyethylene (LDPE #4), Polypropylene (PP #5), Polystyrene (PS #6) and the seventh is categorised as 'other'(#7). This seventh category includes all other thermoplastic materials including Polylactic Acid (PLA). (135) These numbers are used to sort and reprocess the polymers. A study in Wales showed that all 22 councils accept and recycle PET, HDPE and rigid PP.(136–155) Only Carmarthenshire also accepts LDPE, and none of the councils accept PP film or #7. This is due to factors such as the ease of the recycling process, the economics involved and the market for recycled polymers.

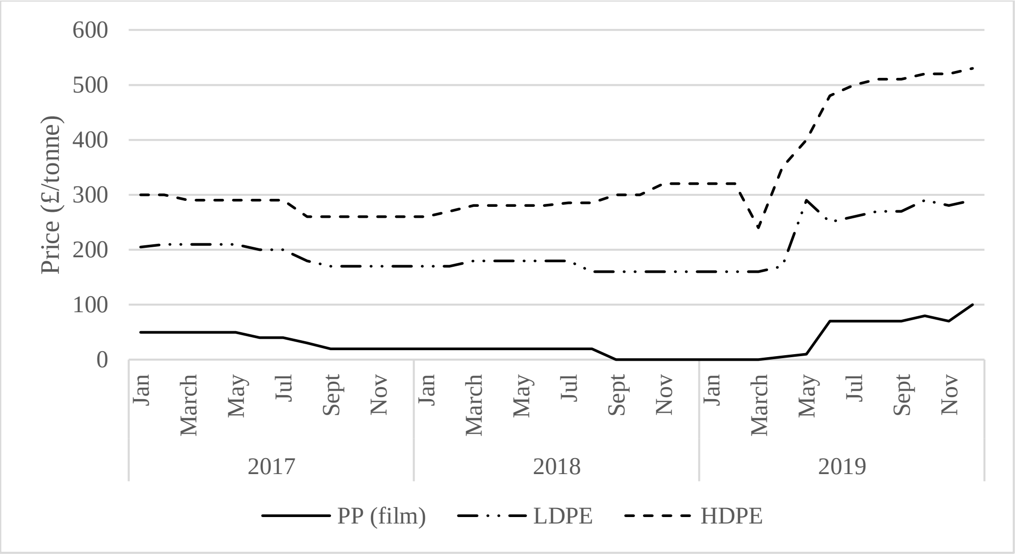

Figure 1 Price paid for a tonne of recycled LDPE, HDPE and PP film from 2017-2019.(156)

Figure 1 illustrates how the market for these recycled plastics has increased recently, but the value of PP film is considerably lower than other polymers. The average price for post-consumer plastics is on the rise with transparent, rigid plastics yielding the best price. Generally, films generate a much lower price. This is due to the problems incurred during the recycling process. Films require different machinery as they often become entangled in recycling and sorting equipment due to their high tensile strength.(157) Therefore, it must be collected separate from rigid plastics. Other factors also contribute to the low market value of recycled films such as colouring, contamination and difficulty cleaning and layering if films.(158) Polypropylene film is relatively cheap to produce and has a greater melting point than LDPE and so requires more heat and energy to recycle. (159) Its higher tensile strength also causes complications making it a less desirable material to recycle. (160)There are currently opportunities for consumers to take their LDPE waste to local supermarkets for reprocessing, but due to inconvenience and insufficient knowledge of this service, the system is not fully utilised.

Many factors affect the desirability of a material including the presence of additives, purity, as certain mixtures can have negative effects on recycling, and availability of the waste material so that it can be recycled in bulk. Availability of processes such as collection, separation technology, purification and storage also affects the rate of recycling. (131) The price difference between virgin and recycled polymers must also be great enough for the recycled polymer to be attractive to buyers.

In order for recycling to be beneficial and part of the solution to the plastic crisis, availability of certain polymers such as PLA need to increase for recycling to be viable, collection of thermoplastics needs to be improved and purity of recyclates needs to be increased through improved sorting methods and accurate labelling.(161)

Until these improvements are made, the consumption and design of plastics must be reviewed. As well as designing packaging that is easy to sort and recycle, alternative materials must be explored because if the issue isn't stopped or at least slowed, by 2050 there could be more pieces of plastic in the ocean than fish. (120)

2.4 Government Action

Generally packaging is designed for short term use and is discarded after use.(9) In recent years there has been a growing concern for the impact plastic, especially single use packaging, has on the environment with an increase in literature demonstrating the issues and emergency with regards to environmental damage. In a survey conducted by Hoffman et al, the issue of plastics in the ocean was ranked as the highest environmental issue out of 9 listed issues with 48% of respondents ranking this higher than climate change.(162) This may be due to the increase in popularity after David Attenborough's Blue Planet II, whose main topic was plastic pollution, was the most watched TV show of 2017 with more than 14 million viewers.(163) Public concerns and growing interest on the topic has put pressure on governments and companies to change their habits and move towards a circular economy.

Households, governments, and businesses all play a role in contributing to the recycling rates and waste produced in Wales. Since 1999, Wales has had its own government which has allowed it to become a global leader in recycling. Wales are now first in the UK, second in

Europe and third in the world for household recycling rates. This has been achieved by setting statutory recycling targets for local authorities since the start of the new government, resulting in an increase in household recycling from 5.2% (1998-99) to 60.7% (2018-19).(164)

In 2018, the UK government published their 25 year plan to improve the environment. Government actions regarding the improvements to the management of waste, plans to minimise plastic consumption and tackling plastics in the ocean are among some of the policies presented in the document. A number of goals have been set by the British government, one of which is to eliminate avoidable plastic waste by the end of 2042.(94) This is in order to reduce carbon emissions, reduce plastic production and to tackle marine pollution. The government aims to reduce global reliance on plastics and have committed to doing more to help developing countries also tackle pollution. Since the start of the 25 year plan, the UK has seen a 46% reduction in problematic plastics, 70% of plastic packaging is reusable or recyclable and the recycling rate for plastic packaging has increased from 44% to 52%.(45) It is evident that these policies can have major impacts but as 80% of the plastic packaging market does not come under the Global Commitment, the impacts are not likely to happen on an international scale. Therefore, the UK government are working to tackle the issue at an international level working through the UN, G7 and G20, along with organisations such as WRAP and the Ellen MacArthur Foundation.(120)

A review by Nielsen et al on government policies indicated a move to tackling more problematic plastic items such as food packaging, after rigorous research into combating microbeads and single use carrier bags.(165) The Packaging and Packaging Waste Directive have set a 50% plastic recycling target which is to be met by 2025 which will increase to 55% in 2030.(166) Various schemes and laws will help to achieve these targets with over 90% of grocery retailers signing up to ‘The Plastic Pact’, committing to making 100% of plastic packaging reusable, recyclable or compostable by 2025. (167)If this target is met, 8 million tonnes of virgin plastic would be prevented from being produced every year resulting in 40 million barrels of crude oil staying in the ground, each year.(120) Members of The Plastic Pact will eliminate unnecessary packaging and reduce the amount of packaging in supermarkets and therefore entering the waste stream. This will be done by developing new business models, focusing on innovation and helping to improve recycling systems within the UK. (168)

Since 2011, approximately £54 million has gone into government research into plastic innovation with encouragement of the development of biobased, biodegradable and environmentally friendly plastics. Currently there are no set standards for biodegradation and so the UK government have committed to work with research councils in order to develop and define a standard for biodegradable plastic bags. After the successful introduction of the 5p charge on carrier bags, resulting in a decrease of use of 83%, governments are confident that policies can make a difference to improving and implementing a circular economy. (94)Fast food restaurants have introduced recycling facilities and are swapping out plastic products such as straws and coffee stirrers for eco-friendly alternatives. After implementing the ban on microbeads, the government are looking into whether other problematic materials can be banned where suitable alternatives are available. For this to be implemented, further research and development needs to be focussed on finding suitable alternatives.

A new tax came into place in April 2022 which will also help to reduce plastic consumption. The tax requires companies to pay £200 per tonne of plastic packaging which contains less than 30% recycled plastic.(169) As this tax is charged by weight, companies are moving from heavier, higher density materials to films and pouches. As we see an increase in the use of films in the grocery packaging industry and have identified the issues with disposal of materials such as LDPE and PP, degradable alternatives must be explored.

2.5 ‘Environmentally Friendly’ Polymers

There are a variety of terms used to describe ‘environmentally friendly’ polymers which are often misleading and used by manufacturers to greenwash plastic packaging and products. The most commonly used terms are: bio-based, biodegradable, and compostable. It is imperative to acknowledge that not all biobased polymers are biodegradable and not all biodegradable polymers are compostable.(130) It is equally important to remember that without viable end of life disposal on behalf of the consumer and appropriate waste management facilities, the environmental benefits of these biodegradable and compostable polymers remain wasted.

2.5.1 Bio-Plastic

Generally, bioplastics are a category of polymer which are made from biological matter rather than traditional petroleum. They can be made from any renewable natural sources such as plants, animals or micro-organisms, proteins and lipids. (170) They have the advantage of renewable resources and reduces the reliance on finite resources such as crude oil but their end of life disposal is complicated. ‘Biobased’ and ‘Bioplastic’ are more terms commonly used to greenwash packaging. (171)These terms simply mean they are derived from biological matter and they are not necessarily biodegradable. It is estimated that half of the bioplastics currently on the market are not biodegradable. (172) It was originally thought that all bioplastics were biodegradable but recent studies have proven some misleading limitations. (173) Common bioplastics include bio-polyethylene and bio-polypropylene and are produced from raw materials such as bioethanol. Although these synthetic polymers are made from these renewable sources, their biodegradability is not affected.(174,175) Alkenes are still alkenes and exhibit very similar behaviours whether bio-based or petroleum based. (176)

2.5.2 Biodegradable Polymers

Biodegradable polymers (BDPs) are materials designed to degrade due to micro-organisms such as bacteria and fungi. The polymers can be biobased or petrochemical based and some BDPs combine petroleum based and biobased polymers to reduce cost and alter the performance.(130,177,178) Biodegradation is a term frequently used by companies to greenwash packaging and products. Biodegradation is governed by various factors including the characteristics of the material, temperature, type of organisms present and nature of pre-treatment.(179–181) Environmental factors such as time, contact type, humidity and pH all affect the level of biodegradation, depending on the conditions preferred by different microorganisms.

There are various properties of a material which affects the rate and ability of biodegradation. The structure of the polymer has a large impact on the biodegradation of a material. Polymers with different chemical structures require different microorganisms to degrade them. Certain functional groups such as esters found in natural polymers are more likely to be degraded by existing enzymes. Hydrophilic functional groups including hydroxyl groups and carbonyl groups, also increase enzyme degradation due to an increase in water absorption. An increase in molecular weight and crystallinity decreases the rate of biodegradation due to the reduced ability of enzymes to penetrate the polymers.(182) Cross linking reduces crystallinity but also causes an increase in density. Therefore the higher the complexity of the structure, the lower the ability and rate of degradation. (183)

There are no defined time constraints for a material to be labelled as biodegradable and some polymers are designed to fragment via natural processes over long periods of time.(184) Designing hydrophobic polyolefins such as polyethylene to partially degrade is often more harmful than if they were left non-degradable as they break down into micro-plastics where they easily enter water systems to be ingested by marine life.(185–187) Micro-organisms attach to the surface of a polymer if it is hydrophilic and since PP and PE have only CH2 groups, these micro-organisms find it difficult to attach.(188) Pre-treatment including corona discharge and UV have indicated an increase in degradation as it either decreases the hydrophobicity of the polymer or introduces functional groups which are more prone to degradation.(189)

2.5.3 Compostable Polymers

A subcategory of biodegradable polymers are compostable polymers. These are materials that are designed and certified to break down completely into water, carbon dioxide and biomass under specified conditions. (190) As the CO_2 is already part of the biological carbon cycle, the CO_2 produced does not contribute to greenhouse gasses. (130) Compostable is a similar concept to biodegradable, but has defined time constraints and conditions and must leave no visible contaminants or toxic residue. According to EN 13432 standard: ‘Requirements for packaging recoverable through composting and biodegradation’, for industrial composting a minimum of 90% biodegradation must occur within 180 days at temperatures of 58 °C for the material to be classified as compostable. Vinçotte is one of the accredited inspection and certification organisations and has introduced five conformity marks. These are OK compost, which guarantees compliance with EN 13432, OK compost HOME, which certifies compostability at ambient temperatures under home composting conditions. A minimum degradation of 90% must occur within 12 months. (130) This label is not based on a standard, but on a certification scheme defined by Vinçotte. OK biodegradable soil is the third mark and certifies the product will completely biodegrade in soil. This label also does not refer to a standard and is based on a scheme defined by Vinçotte. OK WATER guarantees the material will biodegrade in natural fresh water environment and OK biodegradable MARINE certifies biodegradation of at least 90% within 6 months based on ASTM D7081. (191) Plastics that degrade in the marine environment have an increasingly low market share and are not currently available on the consumer market.(176)

2.6 Polylactic Acid (PLA)

Polylactic acid (PLA), a polyester synthesized from monomer lactic acid, is a commonly used biopolymer. It is produced from renewable feedstock such as corn starch and sugar. (192) The six biopolymers provided by Greenlight Packaging that will be used in this project are believed to be PLA based and derived from natural sources. PLA is a thermoplastic and so has the ability to be recycled. (193) Claessen found that PLA can be mechanically recycled a few times without significantly reducing the mechanical properties although due to its higher cost and lack of supply of waste, recycling is not currently an economically viable disposal method.(130)

PLA is a leading biomaterial in medicine and industry, with the potential to replace common petroleum based plastics. In recent years, several PLA based technologies have emerged with an emphasis on achieving similar chemical and mechanical properties equivalent or superior to conventional plastics. It is the most extensively researched and utilized biodegradable polymer due to its renewable nature and many advantageous characteristics.(194)

2.6.1 Properties of Polylactic Acid

PLA is a renewable thermoplastic which is recyclable, biodegradable and compostable. Lactic acid can be produced by fermentation of sugars obtained from renewable resources such as sugarcane or corn starch. It was first synthesized by heating lactic acid and removing condensed water by Carothers, Dorough and Natta in 1932, which later led to the synthesis of polypropylene in 1954.(195,196) Today there are various methods to synthesize PLA but they are complex and can be expensive due to the meticulous control required over conditions such as temperature and pressure. It can be prepared through a variety of polymerization processes with the most common methods being direct condensation polymerization and ring opening polymerization.(197,198) As water is also produced during direct condensation of the lactic acid, ring-opening polymerization is used in industry to produce solvent-free high molecular weight PLA. (199–201)

It is easily processable on standard plastic equipment and can be processed through a variety of processes including injection moulding, film extrusion, blow moulding, thermoforming and 3D printing. PLA has better thermal processability compared to other biopolymers and requires 25-55% less energy to produce than petroleum based polymers.(202) Its production also consumes carbon dioxide and in some cases, properties have been comparable to conventional plastics. (203) Glass transition temperatures are around 55°C and melting temperatures around 165°C (203–205)

Many properties of PLA are dependent on its isomers; laevorotatory (L) lactide and dextrorotatory (D) lactide. These isomers can also be mixed to give DL-lactic acid. Both PLLA and PDLA are crystalline polymers, hence have similar characteristics and melting temperatures. The blended PDLLA has a lower crystallinity than its counterparts and the degree of crystallinity can be adjusted by altering the ratio of isomers. The reduction in crystallinity means that PDLLA is more prone to degradation and many solvents that do not react with PLLA and PDLA have been proven to dissolve PDLLA. It should also be noted that PDLA does not degrade in the presence of certain enzymes that can hydrolyse PLLA and PDLLA, impacting the biodegradation of various PLAs. (206)

Properties of PLA are also dependent on characteristics including molecular weight and degree of crystallinity. Semi crystalline PLA can combine the degradation properties of a more amorphous PLA and the mechanical properties of crystalline PLA. It has a relatively high strength although it has poor toughness and can be quite brittle. (207) Plasticisers can be added to improve the elongation and reduce the toughness and therefore stress levels although caution should be taken to ensure the degradation properties are not affected too drastically.

A disadvantage of composting PLA is that it can have a slow degradation rate. The degradation rate of the polymer depends on the crystallinity, molecular weight and its distribution, morphology and water diffusion rate into the polymer and the complexity of the structure. PLA degrades through hydrolysis in which water molecules break down the ester groups. (208,209) Hydrolysis at elevated temperatures can reduce the molecular weight, improving properties for microorganisms. When PLA is disposed in the environment, these elevated temperatures are not reached and so chain scission does not occur and the polymer remains resistant to microbial attacks. In industrial composting facilities, these temperatures are reached and the materials are able to biodegrade into water, carbon dioxide and biomass. (210) Decreasing the molecular weight and crystallinity along with the addition of additives can improve the rate of biodegradation and reduce the temperatures and conditions required.

2.6.2 Applications of Polylactic Acid

Polylactic acid (PLA) is a very versatile material and can be rigid or flexible, hence its various applications in various sectors.(210) Due to its degradation properties and high biocompatibility, it is commonly used in the medical industry.(211) It has shown the ability to replace conventional plastics in areas including medical equipment, implants and prosthetics as well as paving the way for new advances in tissue engineering, regeneration and drug delivery. (207) Due to its biodegradable nature, PLA removes the need for surgical procedure required for the removal of devices made from non-biodegradable materials. (212)Research in this area has accelerated since approval by the food and drug administration and is continuing as it finds more and more applications within the industry. (213)

PLA with its copolymer PGA (PLGA) have seen a large increase in popularity in orthopaedics due to its bioabsorbable properties allowing it to replace traditionally used materials such as metals in application including fracture fixation devices and tissue growth implants. (214–216) Apiawan et al conducted research into 3D printing an implant for cranioplasty using PLA and found that its production was cost effective with good cosmetic satisfaction. (217) Eppley et al found that resorbable PLA and PGA plates and screws are an effective in the use of facial fixations in children. (218) PLGA is also favourable in drug delivery due to its hydrophilicity. Advancements in PLA copolymerisation has also allowed for application as nanocarriers in the controlled release of drugs in therapies including cancer treatment and dermatotherapy. (219–225) Due to its good mechanical properties and biological response, PLA is also widely used in tissue engineering. Gremare et al successfully produced scaffolds made from PLA for bone tissue regeneration of various pore sizes.(226) PLA and PLGA are both effective antimicrobial polymers.(227) They have proven to be effective in mimicking antimicrobial peptides which are used by the immune system to kill bacteria. Polymers are synthesized by attaching an antibacterial drug to the polymer backbone.(212) Antimicrobial PLA spray coatings have also been used for the reduction of the population of pathogens such as E Coli and salmonella on the surfaces of apples.(228)

PLA and its blends are also extensively used for packaging in the fast moving consumer goods (FMCG) sector due to their comparable mechanical and physical properties.(229) Due to its biodegradation properties, research into its ability to replace single use petroleum based polymers in the food packaging industry is growing. Danone announced a switch from polystyrene to PLA in their Activia packaging which will reduce the carbon footprint of the yogurt packaging by 25% and the use of fossil fuels by 43%. (230)

It has also been deemed as safe to use in the food packaging industry due to its nontoxicity as it is chemically inert. (231) Due to its antimicrobial properties, PLA film is widely used in the food packaging industry as a barrier against microorganisms to improve the shelf life of products. (232) Additives and antioxidants can be added to improve properties. Wang et al added cinnamaldehyde to PLA/PBAT blends to improve the antimicrobial properties. The film effectively slowed the growth of E-coli.(233) He et al created a PBAT/PLA blend which successfully maintained the quality of shitake mushrooms as well as extending their shelf life by more than 14 days. (234) Lupu et al investigated the effect of caffeic acid and vanilla acid stabilizers on the properties of a PLA/styrene-isoprene-styrene (SIS) blend which were irradiated for sterilization for use in the packaging industry. (235)

2.6.3 Polylactic Acid Blends

Polylactic acid has shown positive results through the copolymerization with polymers such as glycolic acid to improve the rate of degradation as well as blending with petroleum based polymers such as LDPE to increase thermal properties. (236,237) Weng et al observed the biodegradation behaviour of poly(butylene adipate-co-terephthalate) (PBAT), PLA and their blend. IR spectroscopy showed a decrease in the carbon content of the molecular structures and an increase in the oxygen content. Visual interpretations indicated that over time PBAT, PLA and PBAT/PLA degraded although pure PLA and pure PBAT had faster degradation rates than their blend.(238) Lee et al improved the biodegradability of PLA with the addition of Polybutylene succinate-co-adipate (PBSA), an aliphatic biodegradable polyester. They found that increasing the amount of PBSA increased the biodegradability at a temperature of 20 °C .(239)

PLA is known to be relatively a brittle material.(240–242) Copolymerization with other bio-polymers, petroleum based polymers and elastomers can adjust the properties of PLA. (243) Burzic et al increased the toughness, tensile and impact properties of PLA through the addition of amorphous and low crystallinity polyhydroxyalkanoates (PHA). (244) Chaiwutthinan et al reported that blending PLA/PBAT at a weight ratio of 70/30 improved toughness.(245) Kumar et al further increased the impact strength of PLA/PBAT blend prepared at a weight ratio of 75/25 by incorporating 5 wt% glycidyl methacrylate (GMA). (246) Fernandes et al reported an improvement in the toughness of PLA/PBAT blended at 70/30 with the addition of nitrile rubber. (247)

PLA production comes at a higher cost than petroleum based materials and so blends are created to reduce this.(248,249) Starch is an inexpensive material which has also proven its ability to enhance the mechanical properties of PLA. (250,251) Hu et al described the effect of PLA/starch blends with citric acid and polyethylene glycol (PEG) as additives. It was found that the elongation at break and impact strength of pure PLA was increased with the addition of PEG acting as a plasticizer, enhancing the mobility of the polymer chains.(252) The addition of small molecules of PEG can control the rate of biodegradation, increasing the possible applications in fields such as medicine and packaging. Results showed from a study by Wang et al showed an increase in the elongation of PLA with the addition of thermoplastic starch.(253) Ayana et al also reported an increase in mechanical and thermomechanical properties of PLA blended with in-situ gelatinized potato starch.(254) Park et al also increased the mechanical properties and toughness of PLA/ starch blends through the gelatinization of the starch. (255)

Although certain properties can be enhanced and controlled through blending, end of life disposal becomes more difficult and if they enter the municipal waste stream, can cause contamination. (256)

2.7 Polymer Degradation

2.7.1 Overview

The stability and durability of plastics is what makes them such a favourable material. The degradation and therefore lifetime of a polymer is dependent on the polymer characteristics and its surroundings. Environmental degradation is caused by a variety of factors, mainly photo-oxidation, thermo-oxidation, humidity, weathering and micro-organisms.(257) Degradation is due to a change in the structure of the polymer chain which causes alterations to the polymer properties including colour, molecular weight and mechanical strength.(258) The structure of the polymer determines its ability and level of degradation. (259) The type of polymer, morphology, molecular weight and crystallinity all have an effect on a materials degradation properties.(260)

During biodegradation, amorphous regions are broken down first due to the need of water to support the hydrolytic process and support microbial activity.(261,262) Generally, biodegradation of biopolymers occurs through hydrolysis of the ester groups hence, the higher the water permeability and absorption, the faster the rate of biodegradation. (263) Due to this, hydrophobic polymers have a severely low rate of biodegradation if any.(264) Higher molecular weights also slow down the rate of biodegradation. (263) Polymers containing stronger bonds such as covalent bonds show little to no biodegradation.(264)

2.7.2 Petroleum Based Polymer Degradation

Generally, polyolefins such as polypropylene and polyethylene are inert hydrophobic petroleum based polymers.(265,266) Enzymes find it difficult to attach to materials with hydrophobic backbones consisting of long carbon chains.(189) The addition of antioxidants improves the polymers ability to resist microbial attacks.(267) Potts et al reported no weight loss of a polyethylene sheet that had been held in moist soil for a period of 12 years.(268) A study by Otake et al showed degradation of thin low density polyethylene films after incubation of 32 years in soil. (269) Andersson et al also noted an increased in the carbonyl peak when studying the degradation of LDPE in an abiotic environment, indicating oxidative degradation.

It was also noted that this peak decreased when subjected to a biotic environment although there was an increase in double bonds related to weight loss.(270) During the microbial degradation of polyethylene, chain scission occurs resulting in the formation of fragments with low molecular weights. Due to the decrease in molecular weight, microbes attack the compound using the polymer as a carbon and energy source. (271)

The structural complexity of the polymer also affects its ability to biodegrade with the presence of branching assisting with microbial attacks. (189) The branching of LDPE makes it more susceptible to biodegradation.(272) Due to its structural complexity, PP is not easily degraded. Polypropylene also exhibits a greater resistance to stress cracking compared to polyethylene resulting in a decrease in the susceptibility to biodegradation.(273) Some researchers have isolated microorganisms that have the ability to partially biodegrade polypropylene. Cacciari et al established that the fungus A. Niger can degrade PP.(274) Andriani et al observed the biodegradation of polypropylene film and beads by bacteria from a coastal area in Jakarta. After 50 days of incubation, both the polypropylene films and beads experienced a reduction in weight. (275) Lee et al reported the ability of Pseudomonas aeruginosa strain isolated from the gut of a superworm to depolymerize four plastics. It was noted that out of the four polymer types, the biodegradation rate for PE was highest and was lowest for PP. (276)

To enhance the biodegradability of these petroleum based polymers, blends can be created. One of the most common biodegradable blends is polymers and thermoplastic starch. (277) Starch is a natural polymer with comparable mechanical properties and biodegrades by enzyme scission due to low crystallinity but causes complications during recycling.(278–281) Foust et al created a LDPE/cellulose blend to improve the biodegradability of LDPE by common fungi. Results found 100% surface growth of Trichoderma virdie on the LDPE blend in 90 days and Gliomastix within 150 days when blended with 8% cellulose. (282)El-Shafei et al blended polyethylene with 6% starch and noted a change in the films mechanical properties and weight.(283) Saha modified metallocene-based linear-low-density polyethylene with(mLLDPE) with protein hydrolysate (HP). With 20% HP, 35% biodegradation was observed. Increasing this percentage to 40% HP resulted in 50% biodegradation after 21 days of incubation at 25°C.(284) Franechetti et al investigated the effect of blending poly(hydroxybutyrate-co-hydro-xyvalerate) (PHBV) with PE and PP separately. They reported that the PE/PHBV blended at a ratio 80/20 was more susceptible to microbial attack than PP/PHBV blended at the same ratio.(285) Goncalves et al also studied the biodegradation of

PP blended with PHBV at a ratio of 4:1. They reported an increase in degradation on PP with the addition of PHBV after 120 days of soil burial. They also noted that the PHBV sample biodegraded completely within 30 days.(286)

2.8 Pre-treatment of Polymers for Enhanced Degradation

The pre-treatment of polymers is another method of enhancing its degradability. Methods including UV irradiation, sterilization and thermal pre-treatment have proven to increase the degradation of various polymers. Depending on the treatment method, properties of the polymer such as hydrophobicity and functional groups are adjusted. (189) Th effect of UV irradiation on the biodegradation of LDPE is widely reported. Albertsson et al irradiated LDPE films for periods of 0, 7, 14, 26 and 42 days before burial. IR spectroscopy confirmed oxidative degradation of LDPE and indicated an increased rate of biodegradation from 0.2% to 8.4% by irradiating the samples with UV light before abiotic treatment.(287) Pandey et al monitored the degradation of UV irradiated isotactic PP, LDPE and their ethylene-propylene copolymer in a soil environment. FTIR spectra found an increase in carbonyl and hydroxl regions for samples exposed to 0-100 hours of UV.(288) Sivan et al also observed the formation of carbonyl groups in polyethylene after 60 hours of UV irradiation and 30 days soil burial.(289) El-Rehim et al accelerated the biodegradation of LDPE/starch blend through UV pre-treatment. They reported a decrease in tensile strength by 20-66% for UV-irradiated samples and 10-26% for non-irradiated samples.(290) Joen et al reported a reduction in molecular weight and tensile properties of PLA after UV exposure. Increasing the UV to 8 hours improved the biodegradability of the PLA. After 8 hours, the ability of PLA to biodegrade was reduced. They experienced the formation of a brittle white solid during UV irradiation which was poorly assimilated by microorganisms.(291)

2.8.1 Methods for Measuring Degradation

There are various methods for the measurement degradation reported in literature. Common measurements include visual inspections, FTIR, DSC and mechanical strength.

i FTIR

FTIR is a widely cited method of degradation. The formation of functional groups and changes in peak intensity have the ability to illustrate the degradation of materials. Scans are run from 4000-400cm^{-1} to include all functional groups. (246) Jitendra et al monitored the evolution of hydroxyl and carbonyl groups through the use of FTIR of irradiated and non-irradiated PP and LDPE film samples which had been subjected to a composting environment. Their results found that polypropylene was more susceptible to microbial attacks compared to polyethylene and exposing the samples to longer periods of UV increased the presence of hydroxyl and carbonyl groups.(292) FTIR showed a strong absorbance in the 1650-1860 cm^{-1} region of biodegraded LDPE due to the formation of carbonyl groups indicative of various oxidative processes.(293) Matsunaga et al also reported the formation of carbonyl groups in LDPE after corona discharge treatment indicative of degradation. (294)

ii DSC

DSC can be used to measure and detect any alterations in a materials transition and oxidation temperatures of potential degradation. The glass transition temperature is expected to change with increased level of degradation due to biodegradation starting in the amorphous regions. The glass transition and melting temperatures of PLA remained at 60°C and 153 °C respectively after soil burial. (238) Tsai et al irradiated PLA poly(methyl methacrylate) (PMMA) blends and reported the UV irradiation to have no effect on the glass transition temperature. (295) Rodrigo et al composted HDPE/PP blends with additive. There were no significant changes in the melting temperatures and so it was concluded that biodegradation started in the amorphous region. (296) Gulime et al reported a broadening of the endothermic melting peaks after UV irradiation of LDPE. This is due to changes in the crystallite size caused by secondary crystallization and chain breaking.(297)

iii Mechanical Properties

There are various reports on the change in mechanical properties in materials after experiencing degradation. As the polymer degrades, chain scission occurs, decreasing the mechanical properties. Nakamura et al reported a reduction in the tensile strength and elongation of a LDPE/starch blend after immersion in activated sludge.(298) Bikiaris et al blended LDPE and starch. After 8 weeks of immersion in activated sludge, the elongation was reduced by 85%. (299) Orhan et al observed the biodegradation of plastic compost bags made from HDPE, LDPE and LDPE/starch blend. The PE/starch blends saw an 82.76% decrease in tensile strength, LDPE 13.04% and HDPE 5.33% after 15 months of soil burial.(300)

Mechanical testing is also used to quantify the effect of UV irradiation on the degradation and mechanical properties. Kackmarek created polypropylene/cellulose blends at various ratios. Samples were then irradiated and composted in a soil environment. The addition of cellulose reduced the mechanical properties of pure PP but increasing the cellulose content above 5% didn’t show a significant effect on the properties. UV-irradiation caused a decrease in the stress at break and elongation in all samples with varying cellulose content.(301)

Due to the environmental issues stated caused by the production and irresponsible disposal of plastics, along with the lack of sustainable disposal methods available, it is imperative that alternatives are researched. Compostable alternatives have the potential to replace conventional petroleum based plastics. They are a viable solution to the plastic crisis and have the ability to reduce the footprint we leave on the planet, environment and its ecosystems.

Although PLA is a widely researched BDP, the research available is heavily focused on PLA in its rigid form rather than film form. There are also gaps in research regarding PLA film degradation within a home composting facility. Focus is on the biodegradation for use in the medical industry rather than compostable polymer packaging. The properties of PLA films in comparison to polypropylene and low density polyethylene for use in the packaging industry is also rarely cited.

Therefore, this project aims to investigate the mechanical, thermal and degradation properties of renewable PLA based films in order to form conclusions on their suitability to replace conventional polymer films used within the packaging industry. Conclusions will be drawn with regards to suitability of the biobased polymers to replace polyolefin films within the packaging industry through the analysis of the physical and mechanical properties. The films ability to degrade in a home composting facility will also be measured to determine whether the films have the ability to degrade in a home composting in environment. These properties will then be compared to polypropylene and low density polyethylene film samples.

Chapter 3. Methodology

Within this methodology section, further information about the materials used within this project, their origins and their physical properties are discussed. The tests conducted within the project are discussed further, with explanations about why each test was chosen. Test methods are described in detail and the relevant equipment and software used is also noted. The method of analysis of results is also discussed prior to the results and discussions section. Any sample preparation required is cited, including relevant dimensions and masses. Any issues that were faced and the relevant adjustments made to resolve these are also included.

3.1 Materials

Figures 2 and 3 show the cushioned panels developed by greenlight Packaging which is used in the construction of a biobased insulating box. This box will be used to for the transportation of items which need to be kept at a low temperature such as food. Greenlight Packaging were supplied with six polymer films, which were believed to be PLA based and compostable, by external suppliers. The purpose of this project was to identify an optimal compostable film to encase soluble starch peanuts. During testing it was noted that properties were similar to that of LDPE and PP film. Therefore, focus on using these films as compostable replacements to polyolefin packaging was also investigated.

Figure 2. Insulation panels developed by Greenlight Packaging.
Figure 3. Greenlight's insulation in box.

Greenlight Packaging had been provided with six films by third party suppliers. The six films provided had unknown compositions but were believed to be compostable and PLA based. As the films were supplied with unknown compositions, FTIR was used to produce a spectra which could be compared to a database for identification. The film IDs provided by Greenlight and used throughout this paper are Polylink, Biax, EF5LKO, EF51V2, EF61A2, and BioFlex.

These films have an array of properties and vary physically with differing thickness, density and transparency. Figure 4 shows the samples supplied by Greenlight Packaging.

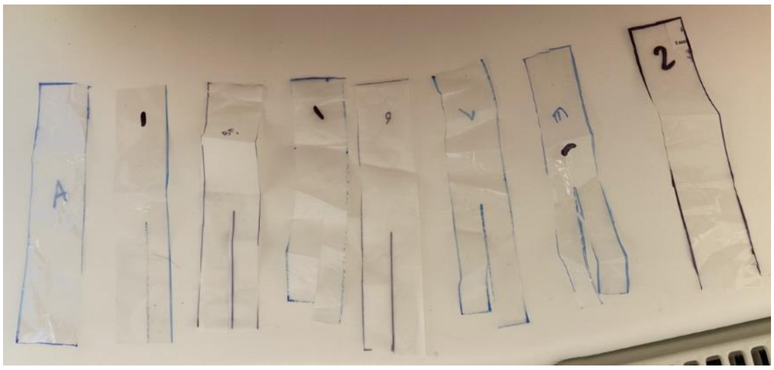

Figure 4. Samples left to right: A2, Biax, BioFlex, EF5LKO, Polylink, V2, LDPE and PP

Low Density Polyethylene and Polypropylene films were collected from supermarkets and used to compare results. As a standard, Polypropylene from apples, dry leaf salad, peppers and tomatoes were collected. Low density Polyethylene from potatoes, cabbage, carrots and onions were collected and analysed. (302). The thickness of each of material was measures using a micrometre and recorded in Table 1.

Table 1. Material ID and Thickness

Material ID	Thickness (mm)	Supplier Name	Supplier Country
Polylink	0.04	Unspecified	Unspecified
Biax	0.06	Bi-Ax	Canada
EF5LKO	0.05	Novamont	Italy
BioFlex	0.06	FKuR	Germany
A2	0.07	Novamont	Italy
V2	0.11	Novamont	Italy
LDPE	0.05	N/A	N/A
PP	0.04	N/A	N/A

3.2 Fourier Transform Infrared Spectroscopy

IR Spectroscopy studies the interaction between matter and IR radiation by passing IR light through the sample material. Depending on the energy level, vibrations of specific molecular bonds are triggered. Some light is reflected, while the remainder travels through the sample. Specific wavelengths of light are absorbed by the sample and the remaining light is transmitted and collected by a detector to produce an electronic signal. Data is collected as a raw signal and is Fourier transformed into a plot of transmission against wavenumber which can be used to identify the material. The spectra can then be split into two sections, the functional group region and the fingerprint region. The functional group region is where degradation and changes in the material can be identified. The fingerprint region contains a large number of peaks associated with the individual compounds, enabling it to be identified.(303)

Various analysis techniques can be used to extract data from the spectrum. In this paper, FTIR will be used to identify the materials, determine degradation caused by UV exposure and composting, and analyse the barrier properties with regards to water and oil permeation. This will be done by analysing the emergence and change in intensity of various peaks relating to different functional groups, for example the presence of water in the polymers was identified by an increase in intensity of an OH peak.

Each of the films were placed onto the crystal of a PerkinElmer Spectrum Two FTIR Spectroscopy and infrared light was passed through to achieve wavelength spectra. Each spectrum was obtained by taking 4 consecutive scans within the range of 4000–400cm^{-1} with a resolution of 2 cm^{-1}. Three repeats were taken, moving the material, so another section is on the crystal to ensure reliability. These results were then compared to a database of reference spectra using the Bio-Rad KnowItAll software. Before each test, a background test must be run to account for any atmospheric interferences caused by the carbon dioxide and water in the air. This is a non-destructive test, so no sample preparation was required.

3.3 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) is a thermo-analytical technique that measures physical and chemical changes within a material in response to temperature or oxygen. The equipment provides qualitative and quantitative information about endothermic and exothermic processes, or changes in heat capacity. A thermogram is produced and from this, glass transition temperatures (T_g) and melting temperatures (T_m) can be determined, indicated by an endothermic change in the enthalpy. This gives the range of temperatures at which the material will be in its viscous and rigid state and indicates the working range. The T_g and T_m also give useful information with regards to processing techniques where heat is involved such as injection moulding and thermoforming.

Samples of 5-10mg, measured to one decimal place, were weighed and placed in an aluminium crucible of diameter 6.65mm. The sample and empty reference crucible were placed on the sample holder with integrated temperature sensors, located in a temperature-controlled chamber of a Perkin Elmer Jade heat flux DSC. Samples were then subjected to pure nitrogen or pure oxygen, depending on the test method used, at a rate of 19.8ml/min. Prior to all tests, the instrument was pre-purged with the relevant gas for 5 minutes and the pressure was monitored. The Pyris Series software was used to collect the data and produce heating and cooling curves.

3.3.1 Glass Transition and Melting Temperatures

When an amorphous or semi crystalline polymer is heated, the temperature at which the polymer structure turns viscous is called the glass transition temperature. At this temperature, the material takes on glassy-state properties such as brittleness and rigidity. Above the glass transition temperature, amorphous and semicrystalline polymers become soft and flexible. Semi crystalline and crystalline polymers have a melting temperature which describes the transition of solid to liquid phase and can be defined as the temperature at which entire chain movement occurs.

Glass transition temperatures are present in amorphous and semicrystalline materials. It is indicated on the thermogram as a small endothermic step change in enthalpy on the heating curve. Glass transition temperatures are affected by a variety of factors including the molecular weight, measurement method and heating rate. (304) Increasing the heating rate has shown to increase the glass transition temperature. (305,306) Melting temperatures are definite and occur when the ordered crystalline phase loses its laminar structure. It is indicated by larger endothermic peaks and are present in materials with a degree of crystallinity. Slower cooling rates have been shown to improve crystallinity, which leads to higher melting temperatures. (307) The length of the polymer chains also increases the melting temperature as the chains become entangled and become harder to break.(308)

It was noted that running the test with materials in sealed crucibles produced more repeatable results than unsealed and so samples in a sealed pan and a sealed reference crucible were heated from ambient temperature, at a rate of 10°C min, to 250 °C under a nitrogen atmosphere. The sample was then held isothermally for 1 minute, then cooled back to ambient temperature at a rate of 30 °C /min. The heat flux DSC heats the sample and reference to the specified temperature and the difference in heat flow into the sample and reference is measured. The difference in heat output is then recorded against time to form a thermogram. This method was used to identify glass transition temperatures and the melting point and are indicated by peaks in the heat flow due to the heat absorption as the sample undergoes the endothermic phase transition. These were then compared to literature to identify whether the materials are PLA based.

3.3.2 Oxidation Induction Temperature

The oxidation induction temperature (dynamic OIT) is a measure of the response of polymers to pure oxygen and its resistance to oxidation, when subject to a defined heating rate in an oxygen atmosphere. The temperature which is then derived from the thermogram produced, indicated by a sudden increase in enthalpy, is the temperature at which oxidation begins. This gives detail about the thermal-oxidative stability of the material and can also be used as a method for identifying biodegradation.

Samples were contained in an open crucible to allow the material to have full exposure to the oxygen. The sample and reference crucible were heated in nitrogen from room temperature to 180 °C just past the melting point of PLA, at a rate of 5 °C /min. The gas was then changed to oxygen and the samples were heated further to 280 °C at a rate of 2 °C/min. Crucibles were then cooled back to room temperature at a rate of 30 °C/min. The oxidation induction temperature is indicated by a precipitous increase in the heat flow. Using the offset method, the oxidation induction temperature was identified. Three repeats were taken for each material and the average dynamic OIT was calculated.

3.3.3 Oxidation Induction Time

Oxidation induction time (Isothermal OIT) is a measure of the response of a material to pure oxygen and its decomposition due to oxygen, when subjected to a defined temperature in an oxygen atmosphere. It is measured as the time between the introduction of oxygen and the onset of oxygen.

The sample and reference were heated to the oxidation induction temperature, which had been defined via the method previously stated in 3.3.2, at a constant rate of 10°C/min in a nitrogen atmosphere. Once this temperature had been reached, heating was discontinued and the sample and reference were held isothermally for 2 minutes to allow the temperature and material to settle. The atmosphere was then switched to oxygen at a constant flow rate of 19.8ml/min where the sample was held at a constant temperature for 60 minutes. The gas was then changed back to nitrogen and the instrument was cooled to 30 °C at a rate of 50 °C/min. If the material was yielding an oxidation induction time of greater than 60 minutes, the test was rerun at a higher temperature. Tests were repeated until the graph displayed an abrupt increase in heat flow which could be reproduced.

3.4 Thermal Conductivity

Thermal conductivity measures the ability of a material to transfer heat. There is the potential for these films and loose fill, shown in Figure 5, to replace expanded polystyrene (EPS) and other materials used for insulation. Greenlight Packaging focus on developing an alternative to EPS for the insulation of food during transportation. Therefore, the insulation packaging used by the two largest meal kit delivery companies, along with EPS was used to compare the results of the PLA based films.

This is a non-destructive test, but the test analyses the effect of the loose fill arrangement as well as the films, so sample preparation was required. The test was conducted in order to test the thermal conductivity of the starch peanuts and ensure they had comparable insulating properties to EPS. The thermal conductivity of the films were also analysed to ensure the addition of the film had little to no interference on the insulation properties of the starch peanuts.

Originally a metal ring was used to contain the test specimen but it was noted after comparing the results to literature that the metal was interfering with results and causing an increase in thermal conductivity. Therefore a PLA ring, diameter 50mm, height 10mm and thickness 0.5mm was 3D printed and used instead. Starch based loose fill, which was also provided by Greenlight Packaging, was positioned within the ring and wrapped in each of the films, as shown in Figure 5. Two separate loose fill arrangements were used, sparsely and densely packed, and are shown in Figure 5. The thickness of the samples were measured to 2 decimal places using a calliper gauge micrometre. Silicon oil was then applied to the upper and lower surfaces of the specimen before placing the sample onto the bed of the FOX 50 heat flow meter. The samples were then subjected to high pressures and nitrogen and a value for thermal conductivity (λ) was defined using the WinTherm 50 software. The test was run at ambient temperature by setting an upper temperature of 33 °C and lower temperature of 13 °C to give an average of 23 °C and delta value of 20. Two repeats were taken, flipping the sample between tests to improve reliability, and an average was calculated. This was repeated for each of the films for both loose fill arrangements.

Figure 5. Loose fill arrangements. Left is the loose packing and right is dense packing.

3.5 Water Permeation

A water permeation test is used to investigate the materials barrier properties by analysing its resistance to water diffusion and absorption. Small pots, shown in Figure 6, were filled with water. The amount of water added to each pot was measured by eye, adding roughly the same amount to each, with any variance in water volume having a negligible effect on permeation. Prepared circular film samples were then placed across the top of the pots, positioning the films between two rubber seals to reduce any slip, before securely bolting a metal lid. It was essential that the bolts were fastened in a certain order, fastening the bolt opposite next, to evenly distribute the pressure caused by tightening. The time was noted and pots were weighed to 4 decimal places. The pots were then turned upside down. Masses and times were noted daily and bolts fastened to reduce the risk of leaking. Once the rate of water loss began to slow and reached a point where no more water could be absorbed or diffused, indicated by a flat line on the graph, the test was terminated. FTIR was then used to assess whether the films retained any water, indicted by the emergence or increase in intensity of OH peaks. Water loss rate was also calculated and plotted against time for each material.

Figure 6Water Permeation Pot.

3.6 Oil Permeation

30 ready meals from the top 4 supermarkets were analysed and the oil most commonly used and present in 69% of ready meals was rapeseed oil. This data was collected by analysing the ingredient lists of 30 randomly selected own brand ready meals from the top 4 largest supermarkets. Each oil type was noted and a tally system was used to calculate the most commonly used oil. The same method as stated in 3.5 was used for oil permeation, replacing water with rapeseed oil as the permeant.

3.7 Tear Test

A tear test is designed to measure the force required to propagate the existing cut along the length of the sample. This is then used to establish the mechanical strength of packaging and indicate the force required to propagate a tear if the packaging material were to become damaged. Using a Hounsfield 1kN electrically operated tensile test machine, a tear test was conducted on specimens made from each of the films. The trouser leg method was adopted to analyse the tear properties. Samples with a length of 120mm and width of 25mm were cut and a 50mm slit was cut along the centre of the specimen to create the ‘trouser legs’. One of the ‘legs’ of the sample was clamped in the bottom static grip and the other in the top dynamic grip. Once the test was started, the top grip began to move and the tear began to propagate. The test is terminated once the tear propagates through the sample. Testing was run at 100mm/min taking measurements every 0.2seconds. Three repeats were taken for each material and an average was taken and is reported in the results.

Samples with the same dimensions as stated above were subject to various conditions, prior to tear testing, to simulate the environments they may be exposed to in the food packaging industry. Samples were then left at room temperature for a minimum of 2 weeks prior to testing. Tests were then conducted at ambient temperature. Results were compared to samples that had not been subject to artificial aging.

3.7.1 Freeze-Thaw Cycle

Two freezing cycles were used to simulate the effects low temperatures may have on packaging used for frozen goods. The first subjected the samples to -18°C for 720 hours (30 days). The samples were then removed and left at room temperature for 1 week. Once fully thawed, samples were tear tested. This cycle was used to simulate home freezing.

The second freeze-thaw cycle subjected the samples to -18 °C for 24 hours, then room temperature for 1 hour. This cycle was repeated 4 times. Samples were then left at room temperature for 1 week to thaw fully. Tear tests were then conducted at ambient temperature.

This cycle was used to simulate the freezing and slight thawing that occurs in stores during replenishment.

3.7.2 High Temperature Conditioning

Heated food at instore hot food counters should be held at temperatures no less than 63 °C for no longer than 2 hours. Samples were therefore subjected to temperatures of 60°C , 80 °C and 94°C for 2.5 hours in a Genlab oven. Samples were then tear tested at ambient temperature to investigate whether heat had an effect on the mechanical properties.

3.7.3 Hight Temperature and Humidity Conditioning

Samples were subjected to temperatures of 60°C, 80 °C and 94°C for 2.5 hours at a humidity of 90%, as this was the highest level of humidity the machine could reach, to simulate the moisture created by steam. 94 °C was also the highest temperature a Binder humidity chamber could reach. To make direct comparisons with the humidity conditioned samples, 94 °C was used for the test runs with heat without humidity too. Samples were then dried in the Genlab oven at 50 °C and weighed hourly, until constant mass to ensure all moisture was removed and samples were tear tested at ambient temperature.

3.8 Tensile Test

The tensile strength of a material is dependent on a variety of factors. The ability of a material to withstand a greater force depend on properties such as crystallinity, molecular weight and crosslinking. A higher degree of crystallinity results in higher tensile strength due to the strong intermolecular bonding within the crystalline phase.(309) Higher crystallinity also results in an in increase in properties such as density, hardness and Young’s modulus.(28) Many factors affect the crystallinity of a material including the molar mass, purity, thermal history and cooling rate.(310,311) Larger molecular weights result in more, large polymer chains becoming entangled, causing an increase in tensile strength.(312,313) At lower molecular weights, chains are bonded by weak van der Waals, resulting in a lower force required to break these bonds.(314) Cross linking also increases the tensile strength due to the restriction of motion of the polymer chains.(315,316)

Using the Hounsfield 1kN electrically operated tensile test machine, a tensile test was conducted using a similar method as that stated above for the tear test.

Solid 120x25mm samples were cut, and 25mm^2 pieces of cardboard were attached either end, sandwiching the film using epoxy resin. This reduces slip while the test was conducted and allows for true deformation of the film. The ends were then clamped into the tensile grips and the test started. Preliminary tests were run and it was noted that due to the forces applied, the samples were susceptible to slip and therefore were not able to deform fully prior to failing.

Therefore, cardboard squares were attached at both ends, sandwiching the film, before clamping in the grips. Initially, UHU adhesive was used to attach the carboard but it was noted that the samples were still slightly susceptible to slip. Therefore, a test using epoxy resin as an adhesion was run and it was noted that samples experienced very little or no slip during tension.

The test was run at 5mm/min for the first minute, then increased to 100mm/min for the remainder of the test. This is a standard test speed for the elastic region to determine the stiffness. This allows the material to adjust and fully react to the force. Once the material is in the plastic region, the speed of the test has less of an effect and so the test speed is increased to 100mm/min to reduce the time taken to reach failure. Three repeats were taken for each material.

3.9 Composting

3.9.1 Method of Composting

Composting is a subcategory of biodegradation and is defined as an aerobic process which uses microorganisms to break down material into water and biomass. For this project, composting was conducted in a home composting facility outside of the laboratory, to give organic and realistic results, without relying in ideal conditions. The home compost facility, situated outside of the laboratory, consisted of food waste, manure, dry leaves and established compost. Temperatures ranged from -1 °C to 30 °C Over the period of a year and average outdoor and compost facility temperatures are shown in Table 2. Home composting facilities have a lower and less constant temperature than industrial facilities, especially in winter months, with temperatures achieved only slightly greater than atmospheric temperatures. It should therefore be noted that materials that do not degrade in a home composting facility may have the ability to degrade in an industrial facility that has elevated temperatures. For this project, a home composting facility was selected over an industrial facility. This is because industrial facilities require ideal, elevated conditions for biodegradation to occur, which requires excess energy. Responsible disposal and council collection would also be required when using industrial facilities. Home composting is a process where the parameters are much less controlled and therefore the results produces in this study are more representative of real life situations. Although home composting requires the availability of a facility as well as responsible disposal, it requires much less energy than industrial composting. Home composting can also be an indicator of whether the material will degrade in the natural environment due to irresponsible disposal.

Temperature affects the humidity in the facility and the rate of degradation hence why home composting generally takes longer than industrial. Vinçotte are the certification agency responsible for certifying home compostable materials. Their OK compost HOME certification states that for biodegradation, materials must show a of 90% degradation within 365 days at temperatures 20-30°C For disintegration, samples must fit through a sieve with 2mm mesh, with more than 90% of dry weight material passing through, within 180 days after being held at temperatures ranging 20-30°C.(130) Depending on the method of determining degradation, samples of various dimensions were added to the compost facility for varying periods of time. Once the samples were removed, they were washed thoroughly with deionized water and dried in an oven at 50 degrees with silica desiccant until constant weight. All composting began in October 2020 and was held for various periods of time.

Table 2 Temperatures of the compost facility and outdoor temperature for October 2020-21

Month	Average Outdoor Temperature		Average Temperature of Composting Facility (°C)
	Low (°C)	High (°C)	
October 2020	4	17	12
November	1	17	10
December	-1	13	7
January 2021	-2	11	5
February	-2	14	6
March	-3	20	7
April	-2	18	9
May	22	3	12
June	26	6	16
July	12	29	17
August	10	22	18
September	7	28	17
October	19	5	13

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3.9.2 Methods to Determine Degradation

i. Visual Interpretations

Samples were added to the compost facility and left for 3, 6, 9 and 12 month periods. Visual inspections of the samples were conducted and images were taken at each time interval. This is a simple method to determine any initial degradation but is not a quantitative method.

ii. FTIR

Spectra were generated using the method previously stated for samples subject to 3, 6, 9 and 12 month periods of soil burial. Please refer to 3.2 for test method. The formation of peaks indicative of degradation were monitored.

iii. DSC

Heating and cooling curves were generated using the method stated in 3.2.1 for samples subject to 3, 6, 9 and 12 month periods of composting. Oxidation induction temperatures was also used to measure the extent of degradation. It is expected that the OIT decreases with increased degradation. The method used for this is described in 3.2.2.

iv. Mechanical Testing

Samples of dimensions stated in 3.7 were added to the compost facility for a period of 6 months. The trouser tear method was used to analyse the effect of degradation on the mechanical properties. Trouser tear method has previously been described in 3.7.

3.10 Accelerated Weathering

A QUV accelerated weathering tester was used to irradiate samples for 336 hours prior to soil burial. Samples were then placed in the composting facility for a period of 180 days. The accelerated weathering tester is used to reproduce the damage caused by environmental factors such as sunlight and humidity. This gives insight into whether UV can be used to accelerate the degradation of these polymers along with which polymers would withstand UV better if subjected within the natural environment. UV-irradiation can also be used as a form of pre-treatment to enhance the biodegradability of certain materials. The effect of UV pre-treatment on the biodegradability of the materials were therefore also tested. will also be tested. Samples with dimensions noted in 3.7 were added to the chamber, consisting of 4 bulbs, and were held at 45°C The intensity of the light was 0.75W/m^2/nm. Samples were tested using the methods of determining degradation as previously described in 3.9.2. Results will be compared to those of samples without UV-irradiation to determine the effect of artificial weathering on degradation.

Chapter 4. Results and Discussion

4.1 FTIR

FTIR spectra were generated using a Perkin Elmer FTIR and the BioRad KnowItAll ID expert software was used to identify the material compositions. Four readings were taken and compared to ensure reliability. The spectra are shown in Figure 10. The KnowItAll software analyses and matches the peak locations of the materials to a database of known materials. The material composition identified by the BioRad KnowItAll software is displayed in Table 6, along with the wavelengths associated with the main peaks. The lower limit for absorption is 0 but negative absorbance can occur due to the difference in the sample scan and background scan. Baseline correction can be used to adjust this but the focus of these spectra was the location and intensity of the main peaks. It was also noticed during testing that altering the force gauge had an effect on the peaks in the 400-500cm^{-1} range. All peaks at this range were very small and so were negligible when noting peak locations.

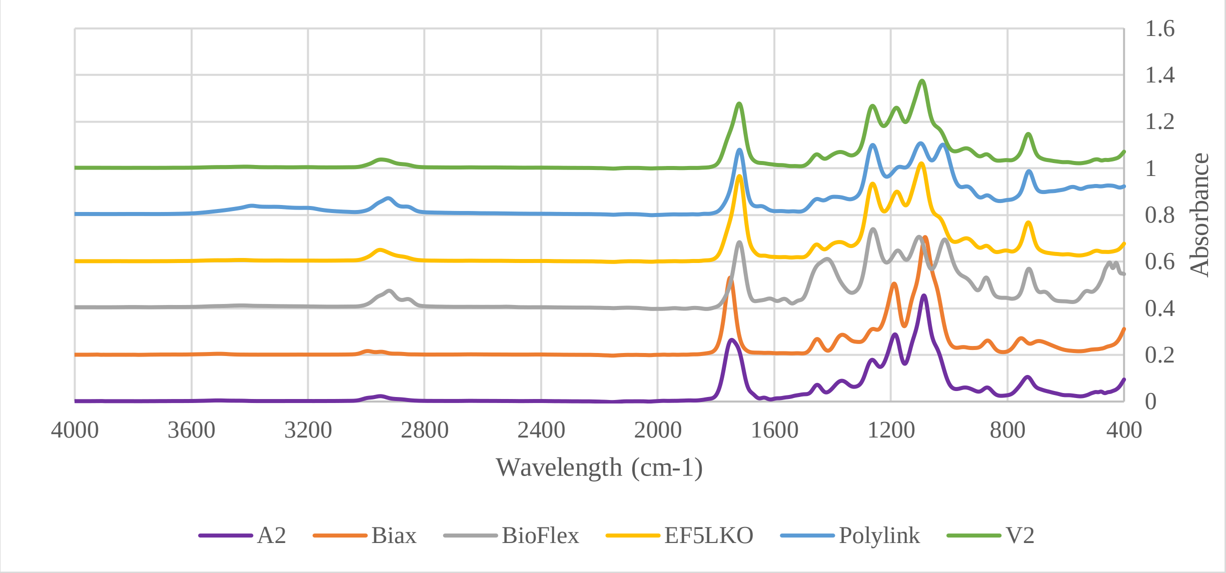


Figure 7 FTIR Spectra of all polymers

After comparing the spectra and main peak locations of the six biopolymers in figure 10, it was noted that all six materials exhibited peaks at similar wavelength with different intensities, indicating these are very similar materials. Table 6 displays the wavelengths of the main peaks for each of the materials. The overall spectra of all polymers are very similar with the same characteristic peaks at 1720-1755 cm^{-1}, 1230-1290 cm^{-1}, 1090-1105 cm^{-1}, 720-760 cm^{-1}. All polymers also exhibit an OH peak associated with PLA, at different magnitudes. The spectra for the biopolymers are very similar with all spectra displaying a small peak corresponding to the -CH stretching vibration, a strong C=O stretch, C-H bending vibrations and C-O stretching vibrations which are all associated with PLA. (317–319) Comparing the spectra and peak locations to literature, it was noted that these peaks corresponded with PLA and different copolymers which confirmed that the six biopolymers provided by Greenlight Packaging were PLA based.

The BioRad KnowItAll software was able to distinguish some of the co-polymers within the matrix of some of the films. By comparing the spectra to the KnowItAll database, it was confirmed that A2, Biax, EF5LKO and V2 contained PLA, which confirms these films are indeed biobased. Although the KnowItAll software did not identify PLA in the material composition of Polylink or BioFlex, the spectra were compared to literature along with the spectra of the other biopolymers that were confirmed as PLA, to confirm these materials also contained PLA.

Table 3 Material Compositions identified by the KnowItAll software with Peak Locations

Material ID	Material Composition	Peak Locations (cm^{-1})	
Polylink	Cellulose acetate propionate	3400 2950 1720 1260	1090 1020 725
Biax	Alpha-Hydroxy omega Vinyl ether PEG-block-PLA	1755 1460 1360 1230 1190	1095 875 760
EF5LKO	Poly(DL-lactide)co-glycolide 80:20	2950 1740 1290	1180 1100 730
BioFlex	No Result	2950 1720 1425 1290 1180	1105 1010 875 730
A2	Polylactide-block-poly(ethylene glycol)-block-polylactide	2960 1755 1455 1390 1275	1190 1090 875 730
V2	BOC Aminopropyl polylactic acid	2960 1720 1455 1260	1180 1090 720

i Polylink

Comparing with the other spectra and literature, it can be assumed that due to the peak locations, the sample is of PLA origin. The BioRad software was able to identify that some of the peaks on the Polylink spectra correlated to Cellulose Acetate. These peaks were around 2950cm^{-1} and a broad peak around 3400 cm^{-1}, associated with OH stretching.(320) The spectra were also compared to literature to confirm results (321–323). Cellulose acetate is a mixed thermoplastic cellulose ester. (324) It is tough and easy to process with little to no odour. It also has good grease resistance compared to other film-formers.(325) Cellulose acetate (CA) has been found to be biodegradable in soil and seawater although the biodegradation rate is greatly affected by the degree of acetyl-substitution (DS) with a lower DS resulting in a higher level of biodegradation.(326–328)

ii Biax

The *KnowItAll software denoted that the* peaks derived from the spectra for Biax corresponded to the spectra for Alpha-Hydroxy omega Vinyl ether PEG-block-PLA, with this making up 100% of Biax. Comparing the spectra to FTIR spectra of PLA-PEG block copolymers, it can be seen that the corresponding peaks occur around 1755 cm^{-1}, 1460 cm^{-1}, 1360 cm^{-1}, 1190 cm^{-1}, 1095 cm^{-1}. The C-H bending frequencies at 1460 cm^{-1} and 1360 cm^{-1} have been observed in PEG and copolymers. (317,329) Alpha-Hydroxy acids are carboxylic acids that have one hydroxyl group attached to the alpha position, and includes glycolic acid, salicylic acid and lactic acid.(330) Polyethylene glycol (PEG) is a synthetic biocompatible polyether. It is flexible, hydrophilic and highly soluble in water allowing it many applications in various sectors.(331,332) Due to its biocompatibility, PEG is frequently blended with biopolymers such as PLA, in order to increase the elongation, softness and impact strength of the PLA. (333,334) Many authors have noted that the addition of PEG causes a reduction in the tensile strength of PLA, with increased concentrations decreasing the tensile strength further.(335–337)

iii EF5LKO

The *BioRad* software was able to identify EF5LKO as Poly(DL-lactide)co-glycolide (PLGA) which is a biodegradable polymer frequently used in the medical industry. Comparing the results to literature, it is confirmed that the peaks are in similar ranged to those reported. (338,339) Increasing the glycolide content offers control over the degradation of the polymer and reduces the glass transition temperature, altering the temperature at which the material exhibits ‘rubbery’ behaviour.(340,341)

iv BioFlex

Although the BioRad software was unable to identify the material composition, research into the material confirmed that the material was a PLA blend, although the full composition was not reported. It was also noted that BioFlex had the peaks associated with PLA which are present within all the polymers. The spectra contains additional peaks with a medium OH bending vibration and CH bending. After comparing the spectra to literature, it was noted that these peaks correspond with the peaks present on the spectra for polybutylene adipate terephthalate (PBAT).(342–345) PBAT is another biodegradable thermoplastic polymer. The addition of PBAT to PLA hosts the ability to improve the properties due to the flexibility and toughness of PBAT, without compromising biodegradability. (346) The improved mechanical properties of PLA due to the addition of PBAT, including increased impact and tensile strength, has been widely cited. (347–350) It has also been reported that the addition of PBAT increases the elongation at break of PLA. (351–353) BioFlex also has more intense peaks around 2950cm^{-1} and 3400 cm^{-1} compared to the other biopolymers, similarly to Polylink. BioFlex also has a small peak at 1010cm^{-1}, similarly to Polylink indicating that these materials have a similar structure and that BioFlex potentially also contains cellulose acetate.

v A2

Polylactide-block-poly(ethylene glycol)-block-polylactide accounted for 49% of A2 material composition with corresponding peaks at 1755cm^{-1}, 1455cm^{-1}, 1190cm^{-1} and 1090cm^{-1}. Similarly to PLGA, Polylactide-block-poly(ethylene glycol)-block-polylactide (PLA-b-PEG-b-PLA) is biodegradable polyester.(354) The addition of PEG increases the hydrophobicity and degradation rate of the PLA. (355,356) Alpha-Methoxy-alpha-(trifluoromethyl)phenyl acetonitrile constitutes 13% of the polymer matrix. Hexafluoro-2-methylisopropanol, also known as flurothyl, accounts for the remaining 11% of the composition. Very little data and research is available on these compounds.

vi V2

BOC Aminopropyl polylactic acid accounted for 32% of the matrix. It is a white powder derived from propylamine and is used in the production of films. (357) The effect of aminopropyl triethoxysilane on the mechanical properties of PLA have reportedly increased, with studies showing increases in tensile strength, flexural strength and impact strength. (358,359)

4.2 Differential Scanning Calorimetry (DSC)

4.2.1 Glass Transition and Melting Temperatures

Figure 7 shows the thermograms produced through the use of DSC for each of the materials studied. From these thermograms, any glass transition temperatures and melting temperatures can be derived. The glass transition temperature is denoted as a characteristic step in the endothermic direction and the melting temperature is indicated by an endothermic peak. The temperature of the peak can then be identified using the Pyris software. Table 3 shows the melting temperatures for each of the materials. All thermograms for the biobased materials, excluding Polylink, exhibited a large endothermic peak in the 150-170 °C region associated with the melting temperature. Smaller endothermic peaks are also exhibited relating to the melting temperatures of other materials possibly present in the films. A2 and V2 both have smaller endothermic melting peaks at 67°C and larger melting peaks at 169 °C. EF5LKO has slightly lower melting temperatures of 64°C and 166 °C. EF5LKO and V2 also exhibited an exothermic peak around 80 °C and A2 around 100 °C related to cold crystallization. Cold crystallization occurs above the glass transition temperature but below the melting point as the chains have gained enough energy to move and fold into a more ordered structure. These exothermic peaks can occur due to the thermal history and processing of the polymer. These three materials are provided by the same external supplier and so were expected to have similar compositions and properties due to processing techniques. In future, thermal scans should be run, heating the samples to just above the melting point to erase any thermal history.

BioFlex had one of the lowest melting points out of the biobased plastics with a temperature of 152°C along with two lower melting temperatures of 51°C and 63°C . The intensity of the melting peak of BioFlex is much lower than the other polymers. After FTIR revealed that BioFlex may also contain PBAT, the lower melting temperature may be due to the addition of this polymer as research has shown that the addition of PBAT to PLA causes a reduction in melting temperature.(360,361). Polylink didn’t show any definitive peaks to indicate glass transition or melting temperatures and just gradually softened over a range of temperatures, indicating low crystallinity. A test was also run at a heating rate of 10°C/min, heating a Polylink sample from room temperature to 350°C to account for possible higher transition temperatures. There were no further peaks indicated on the curve and at 300°C the polymer began to combust.

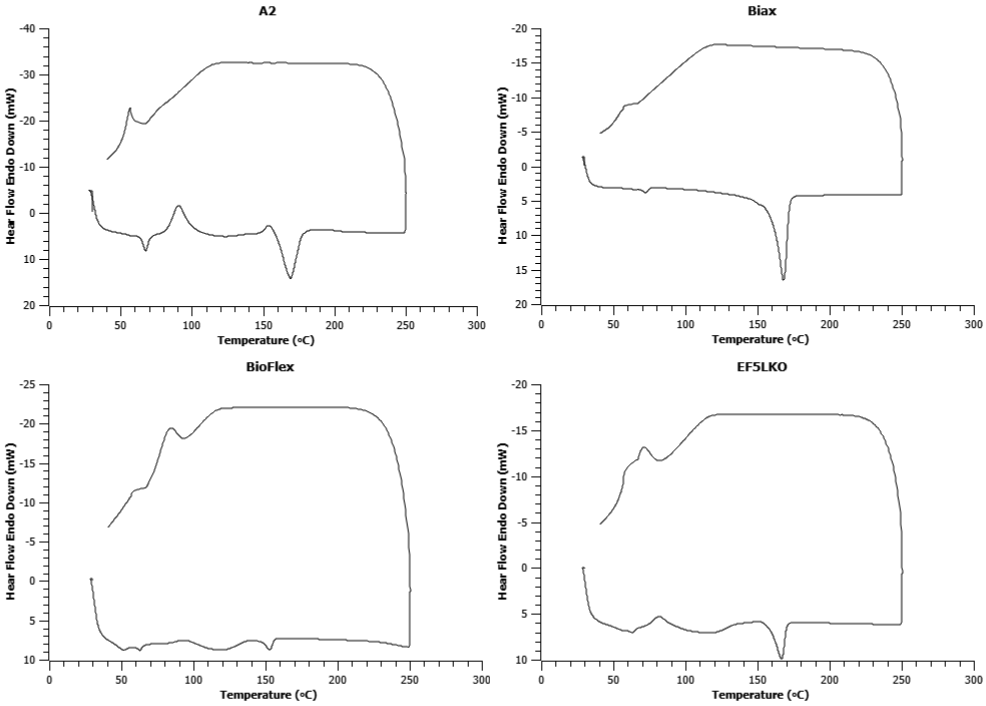
LDPE showed a low melting temperature of 123°C This is low temperature is due to the level of branching involved in the structure as defects are produced due to branching.

Polypropylene had a melting temperature of 168°C Neither polyethylene nor polypropylene indicated glass transition temperatures. This is because they have much lower glass transition temperatures of below 30°C (362–365)

Test were run from room temperature up to 250°C as it was believed that the materials were PLA based. Glass transition temperatures of 50-80°C and melting temperatures of around 170°C were noted in literature, hence why this temperature range was chosen. (246,366–369) Comparing these results to literature, it can be assumed that A2, Biax, EF5LKO and V2 contain PLA. Exact material compositions were determined through the use of FTIR.

Table 4 Melting Temperatures derived from thermograms produced by DSC.

Material	Peak 1(°C)	Peak 2 (°C)	Peak 3 (°C)
A2	-	67	169
Biax	-	72	168
BioFlex	51	63	152
EF5LKO	-	64	166
LDPE	-	-	123
PP	-	-	168
V2	-	67	169



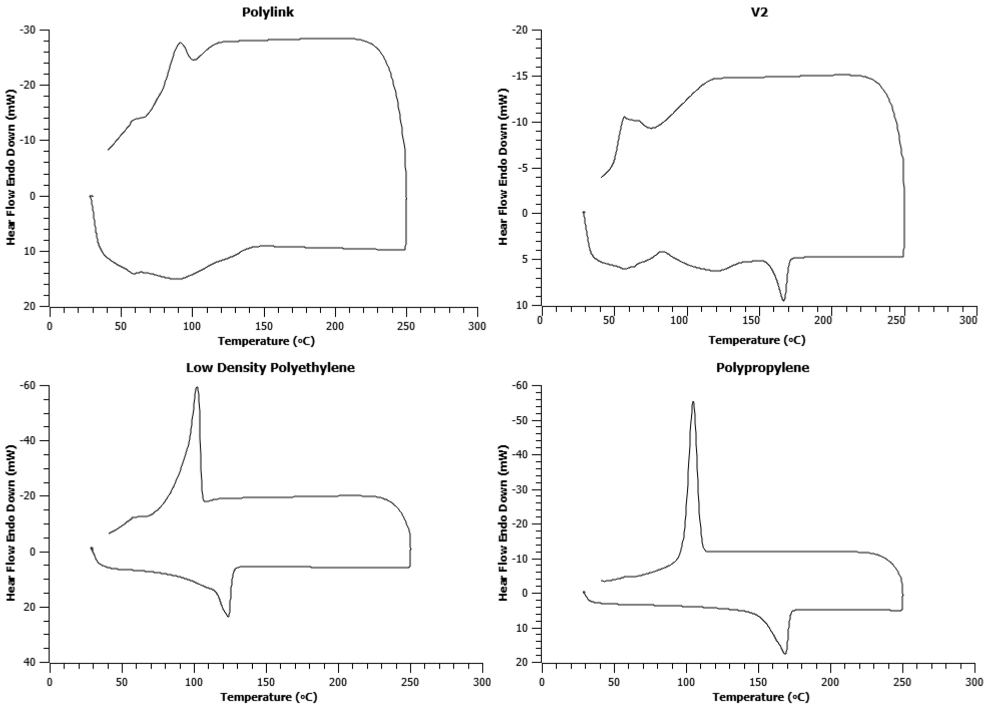


Figure 8. Thermogram of all polymers, heated in a nitrogen atmosphere.

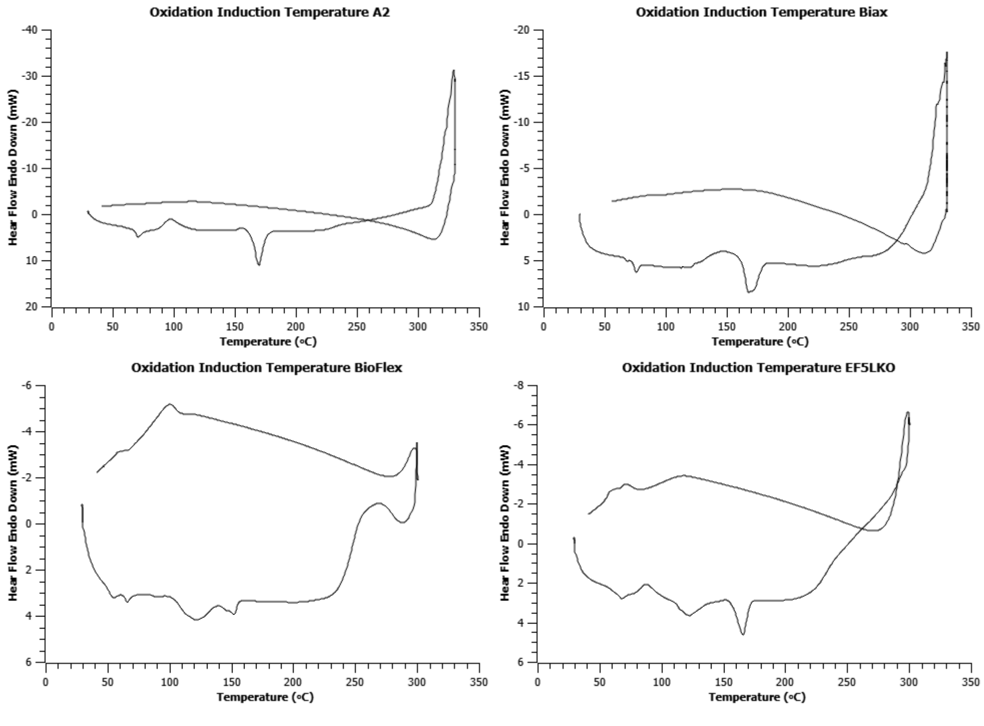
4.2.2 Oxidation Induction Temperature

The oxidation induction temperature (OIT) is the temperature at which oxidation begins to occur when the sample is held in an oxygen environment, and is indicated on the thermogram by a steep change in enthalpy. The sample is heated to 180 °C just above the melting temperature, in an inert nitrogen environment. The purge gas is then changed to oxygen and the temperature continues to increase at a constant rate until oxidation occurs, indicated by a sharp sudden increase in enthalpy. *This* decomposition has shown to deteriorate the mechanical strength and electrical properties of materials and plays an important role in the aging of polymers.(370) Depending on the application of the polymer, materials need to be stabilised against environmental factors such as oxidation.(371) This is an essential property for polymers used within the food packaging industry. Both the oxidation induction temperature and oxidation induction time can be used as a method of quality control for new polymers emerging onto the packaging market. It was noted from literature that increasing the heating rate causes an increase in oxidation induction temperatures. (372,373) This test was carried out a standard heating rate of 10°C/min. This study focusses purely on the heating of the polymers and the cooling process is beyond the scope of this paper.

Figure 8 show the thermograms for each of the materials tested. The tangent method was used to determine the value for OIT. This method uses two tangential lines where the intersecting point dictates the value. Table 4 shows the oxidation induction temperature values for each material, determined by this method. Values for the oxidation onset temperature range between 220-284°C Biax yields a much greater oxidation induction temperature compared to the other materials along with Polylink which also yields a relatively high OIT. The ability of radicals and molecules to diffuse through the polymer matrix alter during thermal degradation due to oxygen and occur above the melting temperatures. EF5LKO yielded the lowest oxidative temperature of the biobased materials, with a temperature similar to that of LDPE. A low OIT of 223°C for LDPE is confirmed by work conducted by Babaghayou et al whom found that LDPE induced oxidation at temperature ranges of 219-236 °C . (374) Comparing the results of the biobased films to literature, the oxidation induction temperature for PLA is approximately 230°C (375,376)

Table 5 Oxidation Induction Temperatures.

Material	Oxidation Induction Temperature (°C)
A2	230
Biax	284
BioFlex	240
EF5LKO	220
LDPE	223
PP	225
Polylink	250
V2	230



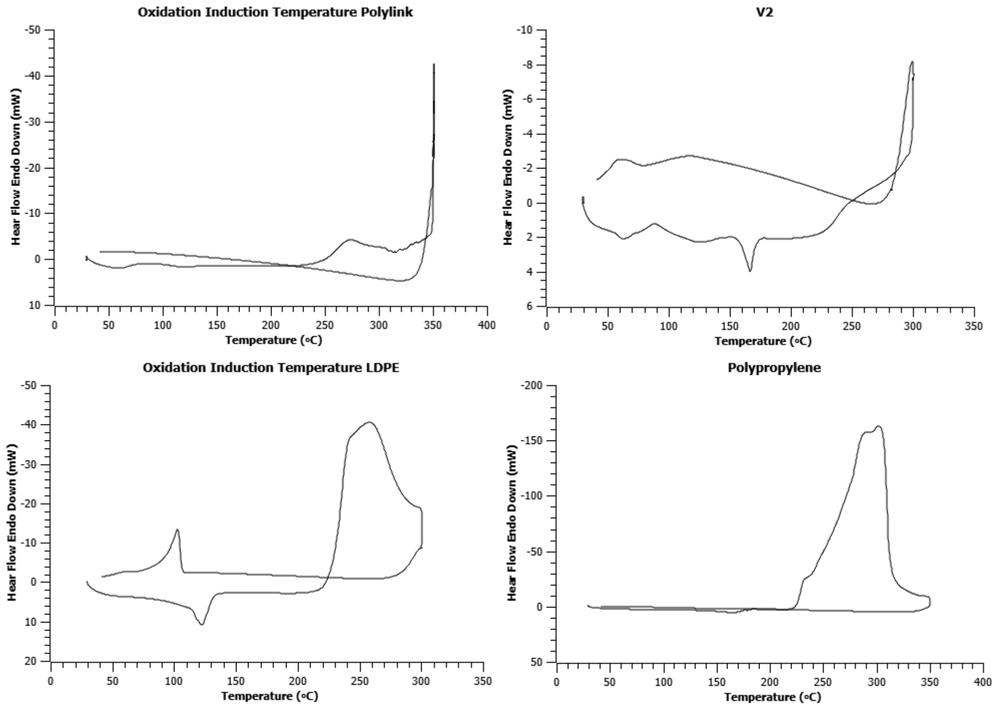


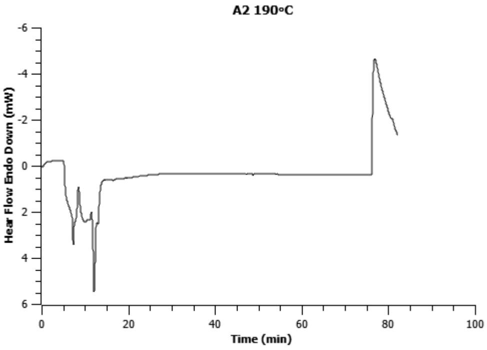
Figure 9. Oxidation Induction Temperature thermogram of all polymers

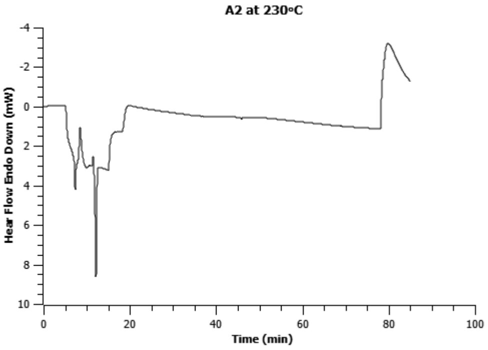
4.2.3 Oxidation Induction Time

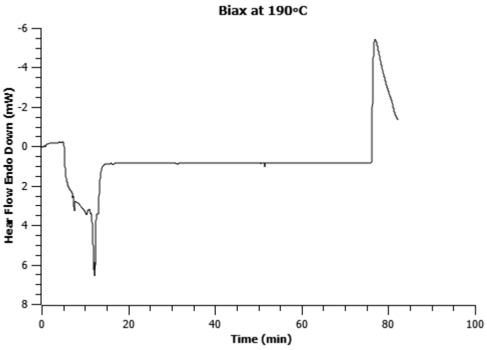
Oxidation induction time (OIT*) is the measure of the time it takes for oxidation to occur at a given temperature and is more widely cited than oxidation induction temperature. The temperature used for these tests are the defined oxidation induction temperatures as stated in Table 4. Samples are heated in nitrogen to the oxidation induction temperature. The inert gas is then switched to oxygen and the sample is held isothermally for 60 minutes. Tests were also conducted at 190°C for comparison of results at a lower temperature. The calorimetric curves for each material at their oxidation temperatures and 190°C are shown below in Figure 9. Similarly to oxidation induction temperature, oxidation induction time is indicated on the graph as a steep change in enthalpy. The tangent method was also used to determine values for the oxidation induction time. Table 5 shows these figures. At 190°C , polypropylene was the only polymer to indicate a sharp change in enthalpy giving an oxidation induction time of 13.47 minutes. A2, Biax, Polylink and V2 showed no signs of thermal degradation. BioFlex, EF5LKO and LDPE showed very slight bowing in heating curve which indicates slight oxidation. Samples showed slight discolouring, changing from white to beige. At the given oxidation induction temperature, polypropylene was the only material to yield a time of greater than 1 minute. This indicates that these materials are very unstable and begin to oxidise as soon as oxygen is introduced at these temperatures. Comparing these results to literature, Pérez et al found that the oxidation induction time of PLA was less than 1 minute but its stability was improved with the addition of stabilizers increasing the Oxidation induction time to almost 12 minutes. (375,376) It was evident that samples had fully oxidised after testing as the samples were dark in colour and samples appeared desiccated.

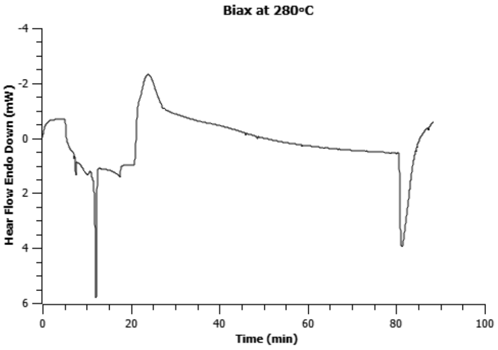
Table 6 Oxidation Induction Times

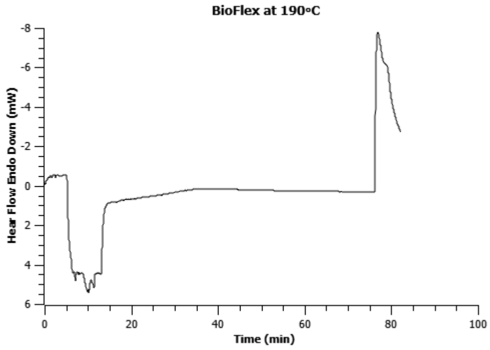
Material	Oxidation Induction Time (min) at 190°C	Oxidation Induction Time (min) at OIT
A2	No Oxidation	<1
Biax	No Oxidation	<1
BioFlex	Slight Curve	<1
EF5LKO	Slight Curve	<1
LDPE	Slight Curve	<1
PP	13.47	2.52
Polylink	No Oxidation	<1
V2	No Oxidation	<1



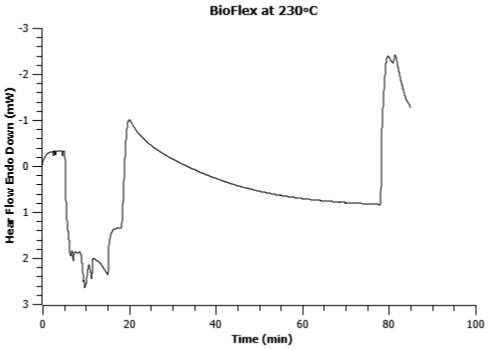




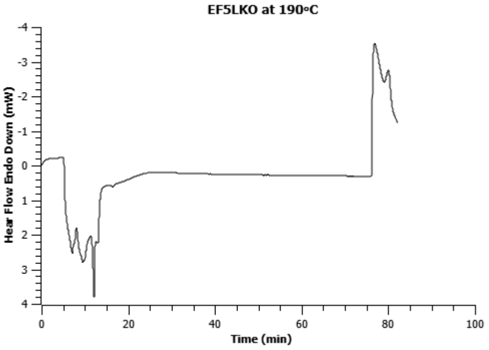




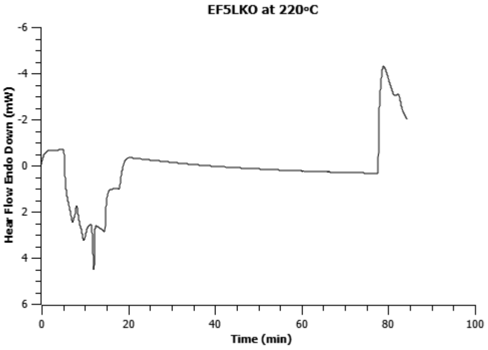
BioFlex at 190ᵒC



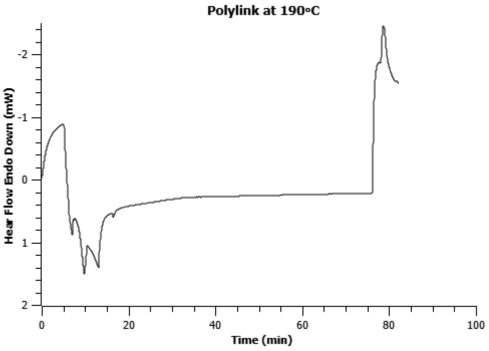
BioFlex at 230ᵒC



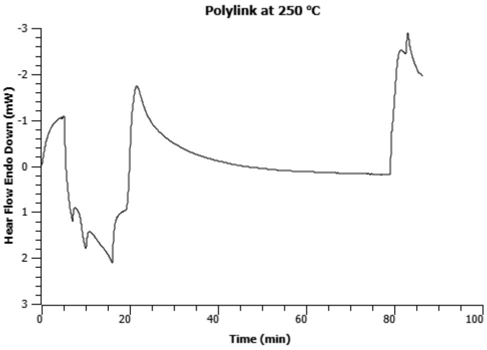
EF5LKO at 190ᵒC



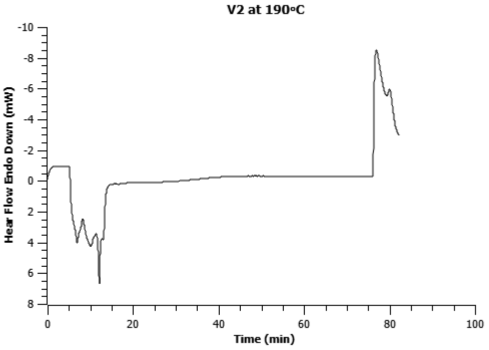
EF5LKO at 220ᵒC



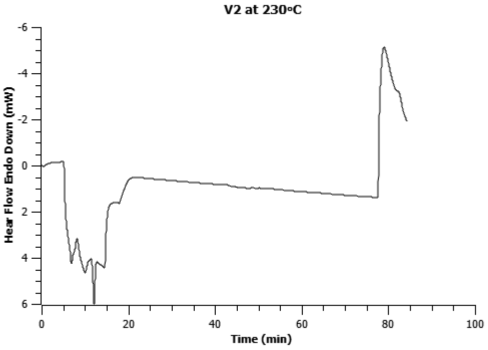
Polylink at 190ᵒC



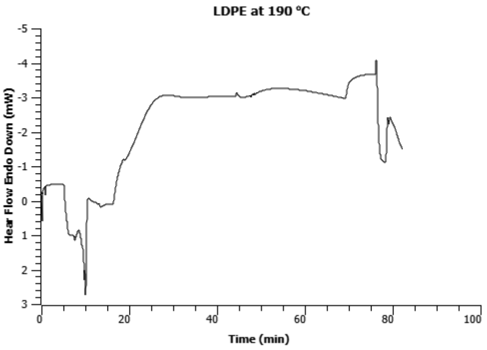
Polylink at 250 ºC



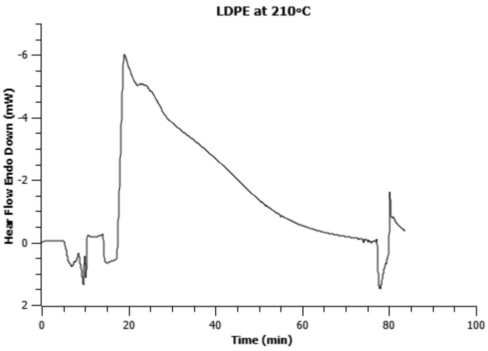
V2 at 190ᵒC



V2 at 230ᵒC



LDPE at 190 ºC



LDPE at 210ᵒC

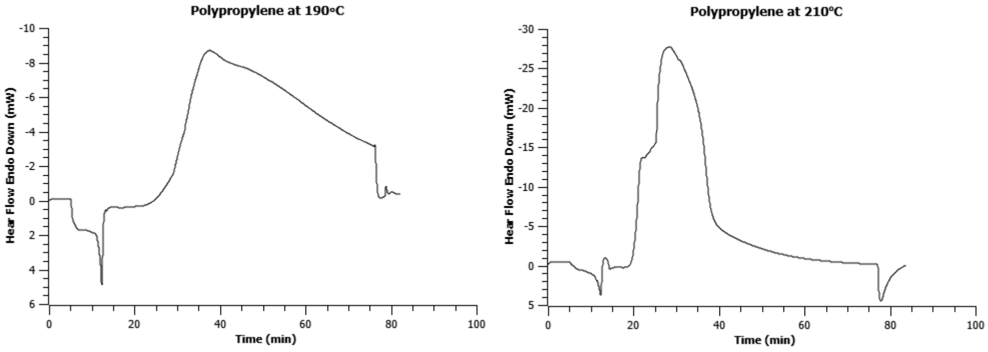


Figure 10 Oxidation Induction Time thermogram of all polymers

4.3 Thermal Conductivity

A low value for thermal conductivity is essential when choosing a material to be used as insulation. Various factors affect a materials ability to conduct heat. Parameters include density, thickness, moisture content and aging. (377) Table 7 shows the thermal conductivity results for two of the most popular food preparation delivery companies along with EPS. Delivery Company 2 had a material with insulating values very similar to EPS and was made from plastic fibres. Delivery company 1 used thick corrugated cardboard which gave a thermal conductivity value much greater than all other materials. For these films to be considered as a replacement to EPS, thermal conductivity values must be around 0.037W/mK.

The pressure the FOX thermal conductivity meter applied to the sample caused the PLA ring to reduce in height due to compression. Thickness values were measured using a calliper and were noted. Once the value for thickness became lower than 8mm, the new ring was used along with a new, identical arrangement was used. Results shown in Table 8 show that overall, the loose fill packing density resulted in a lower thermal conductivity. This was expected as this arrangement allowed for more air to be enclosed, reducing heat flow through conduction. Further research could be conducted into the packing densities to determine an optimal arrangement for the loose fill and improve insulation quality further. Polylink and A2 showed lower λ values with the densely packed arrangements. Results are reliant on temperature and the quality of cooling. Anomalies may be due to the lack of cooling provided by the chiller if the chiller hadn't had enough time to fully activate.

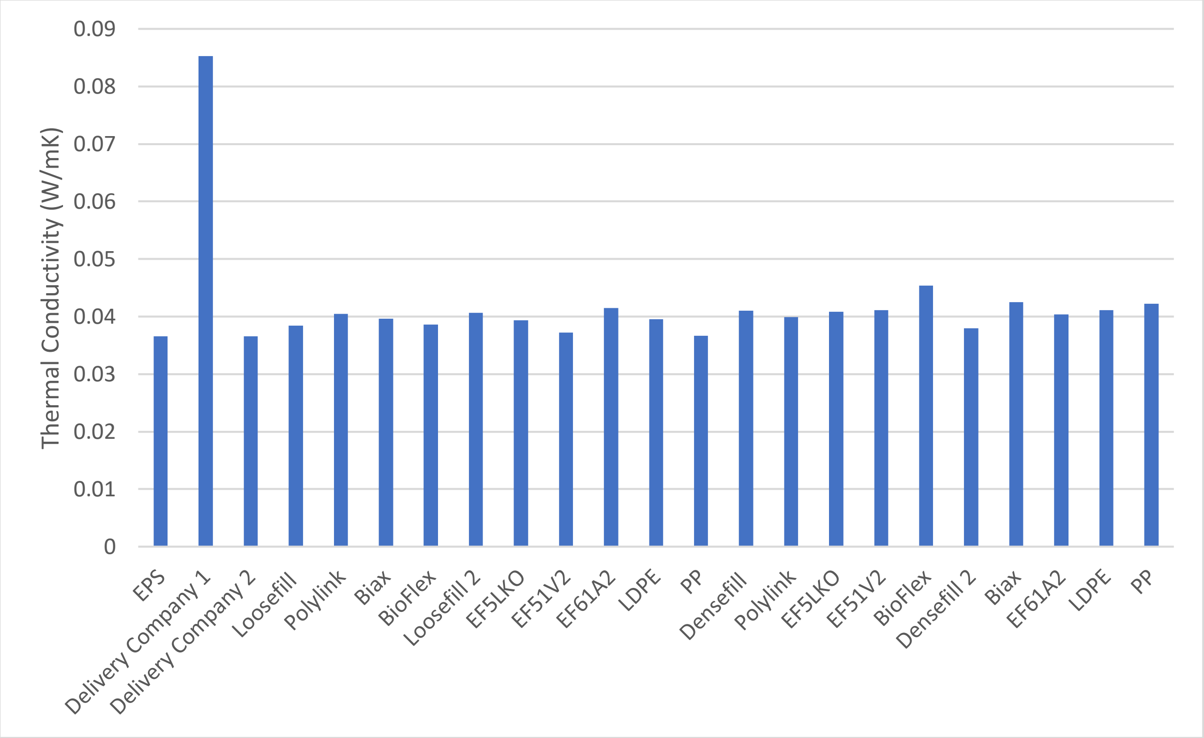


Figure 11 Thermal Conductivity of EPS, Food Delivery Packaging, Biopolymers and Polyolefins

PP had the lowest value for thermal conductivity in the loose fill arrangement for all films tested. V2 had the lowest value for thermal conductivity in the loose fill arrangement out of the biopolymers. Bioflex had the highest value for thermal conductivity out of all the films tested. This was expected as BioFlex has a high density. A higher density results in more tightly packed molecules. This increases collisions, increases internal energy and therefore heat is transferred more efficiently. (378)

Overall, it can be seen from figure 11 that the thermal conductivity values for all samples tested, excluding delivery company 1, are very similar to that of EPS. Therefore it can be concluded that the films have very little interference on the insulating properties of the loose fill peanuts. It can also be concluded that, as the insulating properties are comparable, the cushions shown in figure 2 and 3 would be suitable compostable replacements to EPS in applications where good insulating properties are required, provided that the polymer films are compostable.

Table 7 Thermal Conductivity Of Packaging From Food Two Delivery Services and Expanded Polystyrene

Material	Thickness 1 (mm)	Thickness 2 (mm)	λ1 (W/mK)	λ2 (W/mK)	Average
EPS	9.94	9.96	0.037	0.037	0.037
Delivery Company 1	17.02	17.04	0.085	0.086	0.085
Delivery Company 2	9.89	9.99	0.037	0.037	0.037

Table 8. Thermal conductivity of bio-based materials with both loose fill arrangements.

Material	Thickness 1 (mm)	Thickness 2 (mm)	λ1 (W/mK)	λ2 (W/mK)	Average
Loose fill					
Polylink	10.08	9.94	0.039	0.041	0.041
Biax	9.59	10.14	0.037	0.042	0.039
BioFlex	10.17	9.47	0.039	0.038	0.039
Loose fill 2					
EF5LKO	9.06	8.76	0.039	0.039	0.039
EF51V2	8.59	8.88	0.038	0.036	0.037
EF61A2	10.77	9.93	0.043	0.040	0.041
LDPE	10.16	9.5	0.040	0.039	0.039
PP	9.62	8.55	0.037	0.037	0.037
Dense fill					
Polylink	10.13	9.78	0.040	0.039	0.039
EF5LKO	10.15	9.64	0.042	0.040	0.041
EF51V2	10.04	10.34	0.042	0.040	0.041
BioFlex	10.27	10.27	0.045	0.046	0.045
Dense fill 2					
Biax	10.86	10.72	0.043	0.042	0.042
EF61A2	10.79	10.39	0.041	0.039	0.040
LDPE	10.94	10.01	0.043	0.039	0.041
PP	10.81	9.89	0.043	0.042	0.042

4.4 Water Permeation

Barrier properties depend on a variety of factors. Properties of the permeant and the film both affect the rate of diffusion. The permeant, in this case water, will move from areas of high concentration to areas of low concentration. The permeant will either be absorbed, adsorbed or will diffuse through the film and evaporate. FTIR was used to determine the level of absorption. Density and crystallinity are two of the most determining properties of water permeation. A lower density results in water vapours passing through easily and so has a high permeability. This is the same with crystallinity. The structured polymer chains do not allow water molecules to pass and so a higher crystallinity results in greater barrier properties.(379)

The mass of each water container was measured every day until the water loss rate levelled out. Figure 12 shows the water loss rate over a period of 197 days. After this period, all polymers showed either water loss or leaking which resulted in termination. Starting masses of 180g-248g were measured. The amount of water added to the pots was measured out by eye, hence the differing starting masses. V2 had the greatest difference in starting mass between the two samples with starting masses of 208g and 180g. It can be seen from Figure 12 that the water loss rate for these two samples were consistent and at a very similar rate, indicating that the amount of water added to each pot did not have an effect on the water loss rate. Due to this difference in starting volumes of water, the tests terminated at different speeds due to the complete diffusion of the water although conclusions on loss rate can still be drawn.

Polylink had the largest water loss rate over the time period and the water loss rate stopped earlier than other samples. This is because all the water in the pot had permeated through the film. Any large changes in the water loss rate indicated on the graph as a significant decrease were due to leaking. Leaking occurred in Biax, EF5LKO and V2 to the extent that tests were terminated. Although leaking occurred in Biax, it had the most consistent trend and one of the lower water loss rates out of the biobased polymers along with BioFlex. BioFlex is a very dense material and so it was hypothesised that it would have a low permeability. A2 had a very inconsistent water loss rate with average values of 0.1873g/day for one samples and 0.2548g/day for the other.

Overall, the biobased polymers had a much greater water loss rate than the petroleum based polymers.

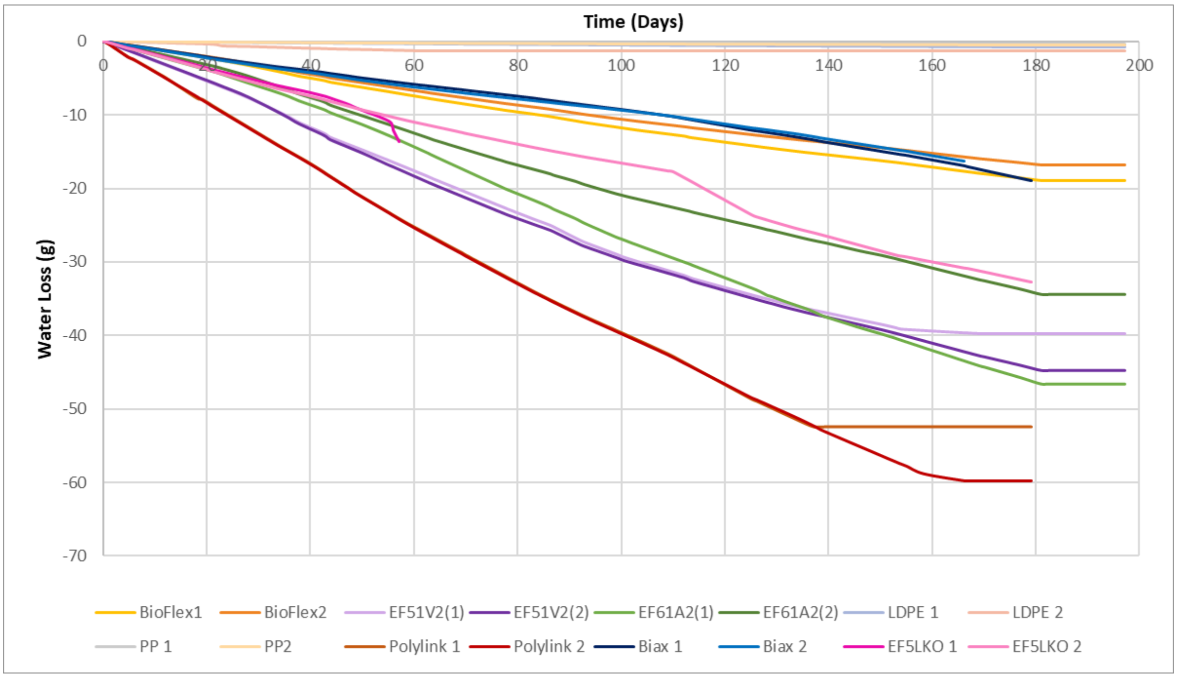


Figure 12 Water Loss Rate over a period of 197 days.

Figure 13. Image of samples after water permeation. Top row left to right: A2, LDPE, EF5LKO, Biax. Second row left to right: V2, Polylink, BioFlex and PP

FTIR was conducted to determine the level of water absorption. Four scans were taken and peak locations were averaged. Figure 13 shows the samples after testing. Pots were cleaned before use using isopropanol but a thin layer of unidentified substance coated most of the materials. It was noticed that the bolts at the bottom of the pots had started to rust which caused a layer to form on some of the polymers. During FTIR, the beam was located in areas where this occurred least but it was noted that this may cause interferences with the spectra.

Peaks in the hydroxyl group region indicate the presence of water and therefore absorption.(380) Large O-H peaks in the 3000-3500 cm^{-1} region are associated with the presence of water.(381) There were no notable differences in the peak locations on the A2 spectra. Biax showed slight alteration in the absorbance at some peaks resulting in a misalignment. This may be due to issues caused by background noise and so baseline correction needs to be used to resolve this issue. The magnitude of the peaks on the spectra for BioFlex changed drastically. The absorbance at all peaks was reduced significantly and a broad peak at around 3000-3500cm^{-1} emerged. This is due to the stretching O-H bond present in water. EF5LKO saw a slight alteration in the absorbances around 1720 cm^{-1}, 1450 cm^{-1}, 1260cm^{-1} and 1180 cm^{-1}. A broad peak corresponding with O-H stretching was also observed indicating water retention. Absorbances fluctuated around the 500-400 cm^{-1} region but it was noted that these changed readily when adjusting the force gauge and so are negligible. Similarly to BioFlex, Polylink showed a large decrease in absorption across all wavenumbers. This is due to a reduction caused in the purity of the substance. V2 also showed a slight reduction in absorbance but generally peak locations remained the same apart from the emergence of an O-H peak. Generally, when polymers become swollen due to the absorption of fluids such as water, the peak intensities alter. Due to the increased hydrophilic nature of PLA, enhanced for increased biodegradation, the PLA based polymers absorbed a greater amount of water compared to the polyolefins, indicated by altered spectra and emergence of OH peaks.(382,383) Cellulose also has a greater absorption rate than PLA, LDPE and PP which was confirmed by the large water loss rate by Polylink.

LDPE remained unchanged with slight discrepancies likely due to background noise and the force applied to the crystal. PP showed a reduction in absorption overall but no changes in peak location were identified. Repeats were taken but all data showed negative absorptions. This is again likely due to background noise and baseline correction would be required for quintessential spectra.

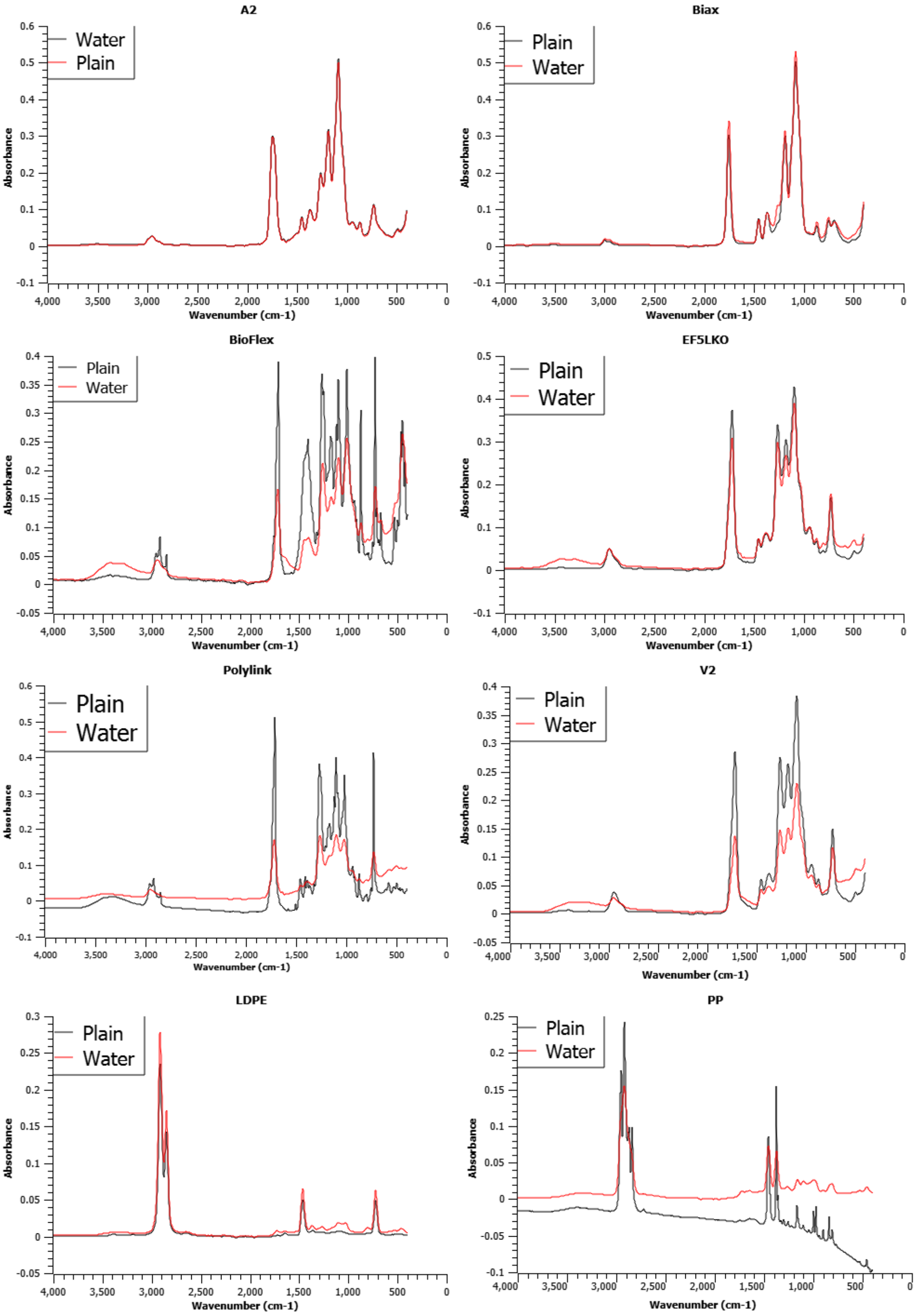


Figure 14 FTIR comparing spectra of plain samples and samples after water permeation.

4.5 Oil Permeation

After noting that rust was an issue during the water permeation, sealant needed to be added to reduce the effect the rust had on samples. The areas were cleaned with isopropanol and adhesive was applied in one pot and silicone sealant in another. Oil was added and the pots were left for 48 hours to see if the oil reacted with either. It was noted that the oil reacted with the glue and caused it to come away from the bolt. Therefore silicone was used to seal the bolts on all of the pots.

Figure 15 shows the rate of loss of oil over a period of 80 days. Leakage occurred in many of the samples. Leaking occurred in EF5LKO within the first week and so a new sample was prepared and the test for this sample was restarted. By day 14 and 49, EF5LKO experienced severe leaking and tests were terminated. At this point it was assumed that the oil was reacting with the polymer. One of the V2 samples and one of the A2 samples also experienced leaking and so were terminated. Leaking was the only indication of weight loss during the test.

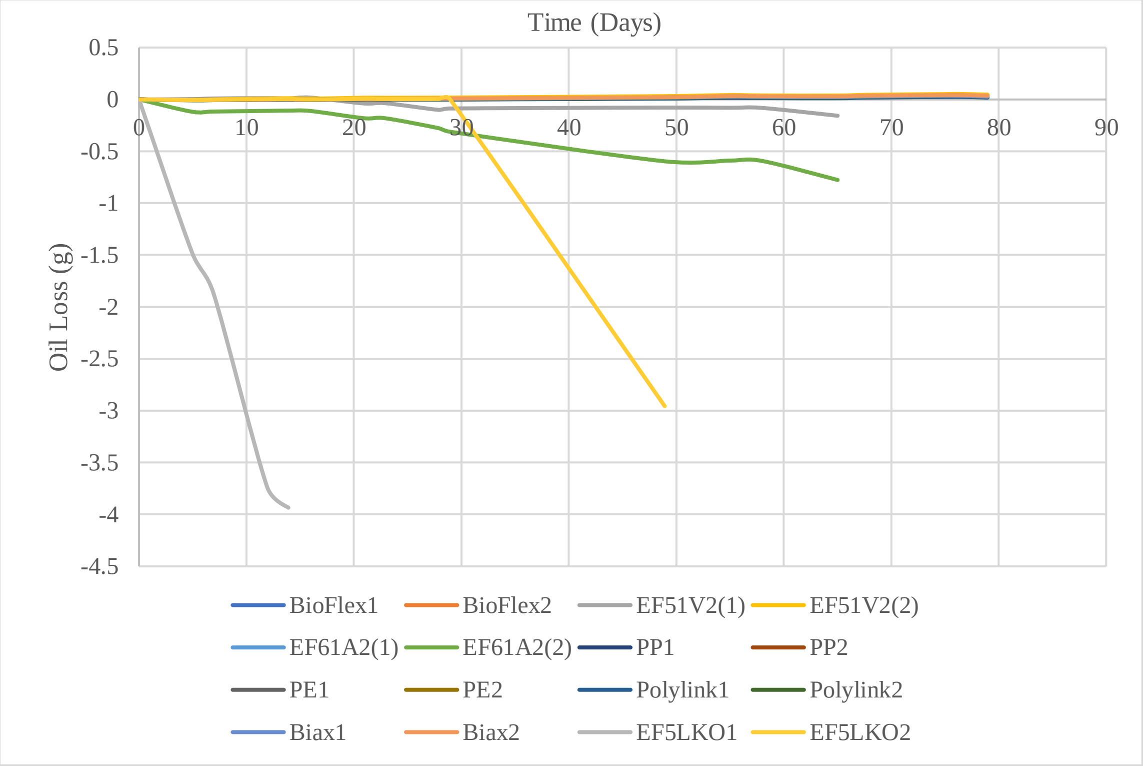


Figure 15 Rate of oil loss over a period of 78 days.

Figure 16 shows the same graph but without the samples that experienced leaking allowing an in depth view. It can be seen that the weight fluctuates but over time, gradually increases. These increases were very small and ranged from 0.0289g to 0.0514g at the highest point. The data shown in Figure 16 follows the same trend for all polymer films and the changes in mass are very minimal. This is likely due to environmental factors affecting the test and are therefore negligible. Greater changes in mass would be noted if the oil were permeating the polymers, and so it can be assumed that oil does not permeate these polymers.

Apart from the samples that experienced leaking, no samples showed any indications of weight loss or diffusion. It is evident by comparing Figure 12 and Figure 16 that the water loss rate with water as the permeate is much greater. The rate of diffusion of the permeate is affected by factors including temperature, concentration and particle size. An increase in temperature causes an increase in kinetic energy causing the particles to move faster, increasing the rate of diffusion. As diffusion takes place due to the difference in concentration and occurs down the concentration gradient, a greater difference in concentration results in faster molecular movement and so increases the rate of diffusion. The size and mass of the particle also affects the rate of permeation. Heavier and larger molecules move more slowly and therefore diffuse slower. Rapeseed oil has a much larger molar mass than water and a lower density resulting in a greater molar volume.(384–387)

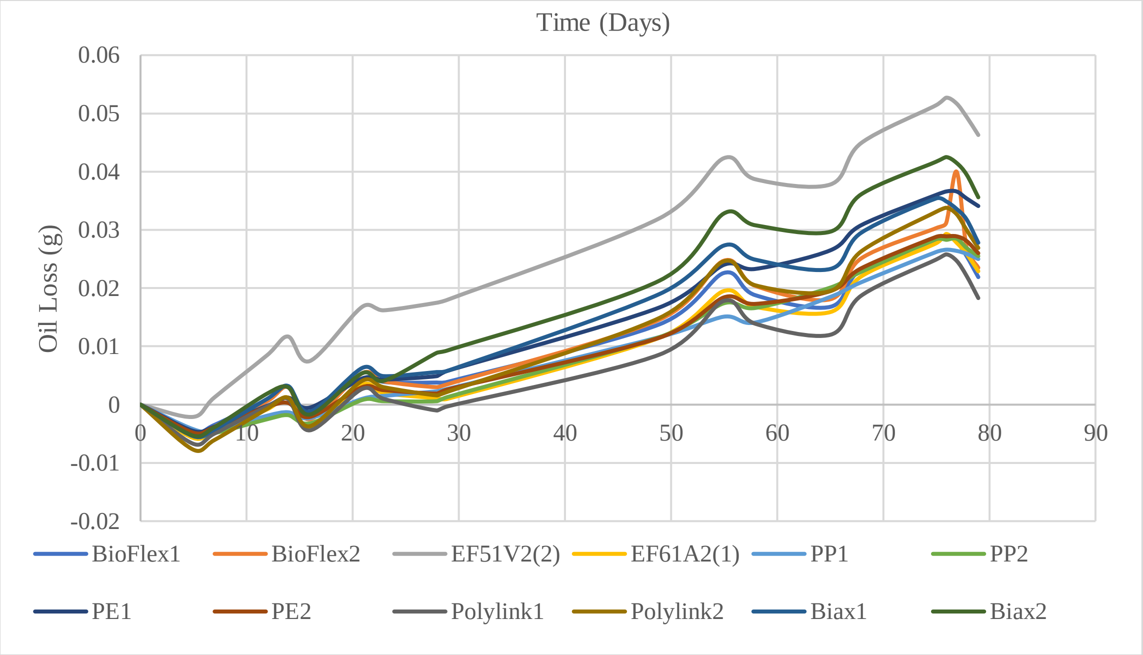


Figure 16 Rate of oil loss over a period of 78 days focussed on samples without leaking.

Rapeseed Oil has a variety of peaks associated with it with C-H stretching, C=O stretching and C-H bending being common indicators of the presence of rapeseed oil. (388) The absorbance of peaks on the spectra of A2, Biax, BioFlex, EF5LKO, V2 and LDPE increased after oil permeation as shown in Figure 17. Samples were washed in deionized water and dried in the GenLab oven at 50 °C until constant weight. Due to the hydrophobicity of oil, it was difficult to fully clean the samples and oil residue may have remained on the polymers. A2, Biax, EF5LKO, V2 and LDPE saw an increase in absorbance at 3000cm^{-1} due to the C-H stretching indicating the presence of alkanes. These materials also showed an increase in absorbance around 1460 cm^{-1} corresponding to C-H bending of the alkane group. A2, Biax, EF5LKO and V2 also showed an increase in absorbance at 1750cm^{-1} which corresponds to the C=O stretching of esters. The absorbance at the peak location 1150 cm^{-1} increased in A2 and Biax due to C-H bending. A2, EF5LKO, V2 and LDPE also saw an increase in absorbance around 700 cm^{-1} due to C=C bending associated with alkenes. Out of plane vibrations also occur in this region.(388) It is difficult to form conclusions from the PP spectra due to the absorbance differences in the two spectra. Repeats were taken and all spectra showed negative absorbances. It was noted throughout this project that many of the PP spectra showed a negative absorbance. Baseline correction is required to give absolute results and to draw precise conclusions.

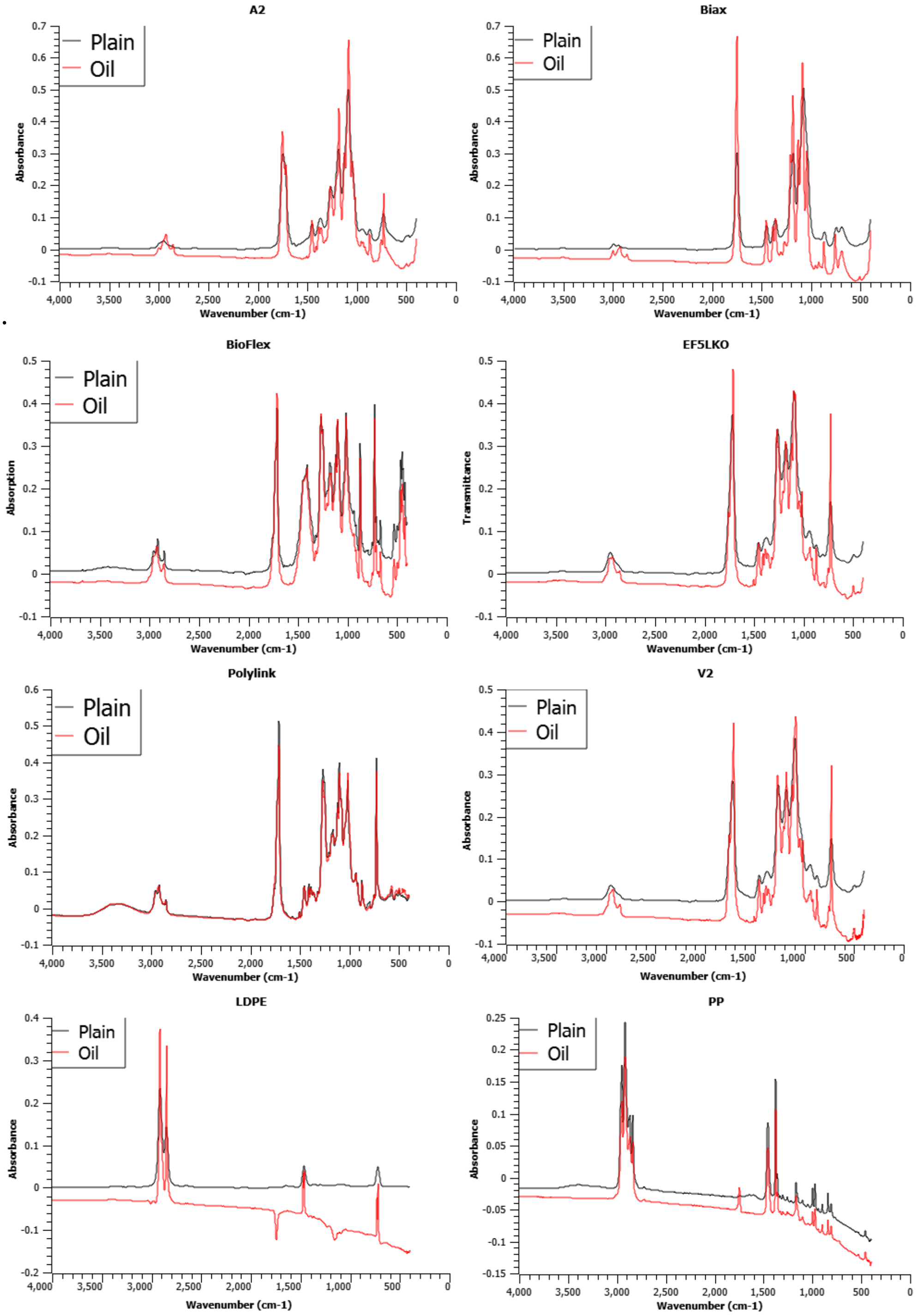


Figure 17. FTIR Spectra of samples after Oil Permeation.

4.6 Tear Test

4.6.1 Un-conditioned Tear Samples

Performing a tear test gives insight into the mechanical properties and performance of a material once damage has been caused. A tear is created in the sample to replicate possible damage that could be caused, and the maximum force withstood during the propagation of this existing tear was measured. The load and time were recorded during loading and tearing of the sample. Two different behaviours were observed when analysing the characteristic curves. Polypropylene is a low or non-extensible film whereas LDPE is a highly extensible film. (389)

The maximum force exhibited during loading was recorded for each test and an average of the three readings was calculated for each material. It can be seen from Figure 18 that highly extensible films were able to withstand a greater force. Bioflex withstood the greatest force out of all polymers tested with a maximum force of 6.64N This is due to the regular, laminar structure of the chains and intermolecular bonding. Bioflex and Polylink were the only bio-polymers to display a greater force than LDPE. BioFlex had a value 58% greater, and Polylink had a value 26% greater than LDPE. EF5LKO had a relatively low tear strength of 1.62N. A2 and Biax had very low tear strengths, both with values of 0.17N These forces were very similar to maximum tear force of PP which had a value of 0.18N. It should be noted that the thicknesses of the materials ranged from 0.04-0.11mm and therefore the mechanical properties of the materials are inevitably going to differ. The tests were conducted with the samples with thicknesses as provided by the company.

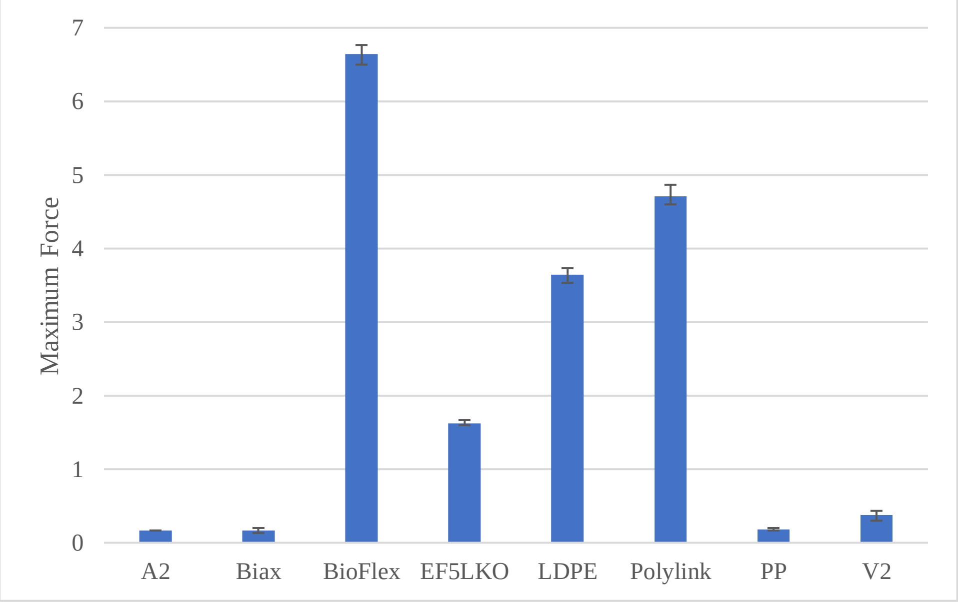


Figure 18. Tear Test Results

Figure 19 shows the specimens after the tear tests were conducted. It can be seen that the two A2 samples tore differently. The first sample propagated the existing tear along the centre of the sample. The second sample shows the tear propagates to the left, following the grain of the polymer, detaching the ‘trouser leg’. This occurs due to anisotropy in the material. Due to biaxial orientation during processing, the material is stretched in two directions; the machine direction and transverse. (390) The machine direction is the direction the film is fed through the machine. The transverse direction is perpendicular to this.(391) Tear strength is usually lower in the machine direction due to the orientations of the amorphous and crystalline regions (392–395) Biax also created a tear to the right of the sample indicating a difference in the molecular orientation within the polymer. The tears occur against the chains, where bonds are broken and with the chains, where chain separation occurs. Tearing where bonds are broken require a greater strength resulting in a higher force withstood.

A rough tear on the BioFlex sample showed a large amount of stretching and resistance during tearing which is reflected in the high tear strength as bonds were broken rather than separated. Polylink and LDPE also exhibited this due to the breaking of the bonds. EF5LKO and V2 both tear straight down the middle, simply extending the existing tear. This may be due to the simple chain separation rather than bond breaking. The tear in the PP sample tore further than the A2 and Biax samples but still veered slightly to one side due to the orientation of the polymer chains, these samples exhibited clean tears and there were no indications of stretching.

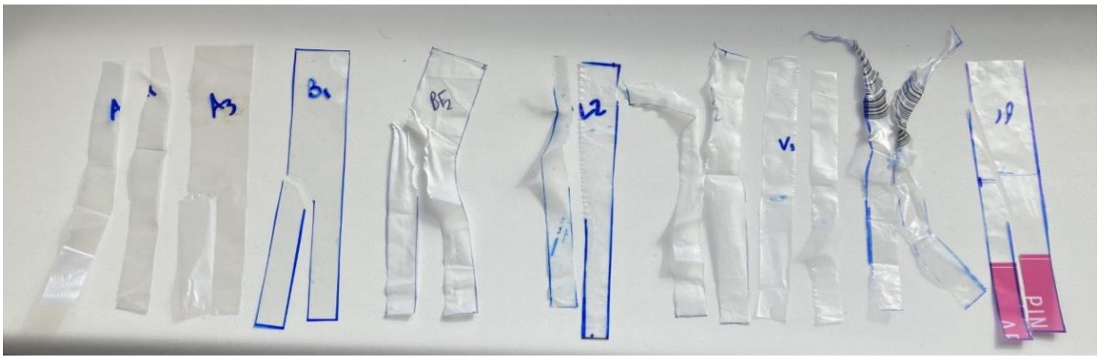


Figure 19. Image of samples after tear test. Left to right: A2 (1), A2 (2), Biax, BioFlex, EF5LKO, Polylink, V2, LDPE and PP.

4.6.2 Freeze Thaw Test

Figure 20 shows the maximum strength of the polymers prior to exposure to low temperatures, after being conditioned at -18°C for 720 hours (30 days) and a freeze-thaw cycle described in 3.7.1. No significant changes were observed when changing the exposure environment for materials EF5LKO, PP and V2. Slight changes can be noticed on the graph but these are likely due to environmental factors during testing such as temperature, as tests were run at ambient temperature in an uncontrolled environment. This could also be due to slight differences in the specimens. Both A2 and BioFlex saw an increase in maximum load when the period of exposure to low temperatures was increased and thawing was not introduced. BioFlex had an increase of almost 0.4N when exposed to 30 days, although the error bars suggest this change is due to variance in results rather than the aging of the polymer. FTIR analysis of the samples after the one week freeze thaw cycle and 30 days at -18°C for A2 and BioFlex saw the formation of an O-H peak around 3500cm^{-1}. This indicates the presence of water. (396) Freeze-thaw cycles have been reported to increase the moisture uptake in polymers, that can result in a decrease in the flexural properties, increasing its susceptibility to crack. (397,398) When a material is frozen, the molecules lose energy and rearrange. When the materials are then thawed and exposed to air, the molecules regain energy and moisture from the environment can be absorbed.(399) When the materials are then refrozen, the moisture in the pores of the material change from the liquid state to the solid state and expand.(400) This expansion causes the pores to stretch and in some cases cause cracking. This allows for more water absorption at the next thawing cycle and this continues and escalates. This can create defects in the material, reducing the mechanical properties. (401,402) A freeze-thaw study on HDPE and PP samples reported a reduction in flexural strength and stiffness. (403) It was also observed that exposing Polylink and LDPE to these low temperatures caused a significant decrease in maximum strength with reductions of 56% and 22.5% respectively.

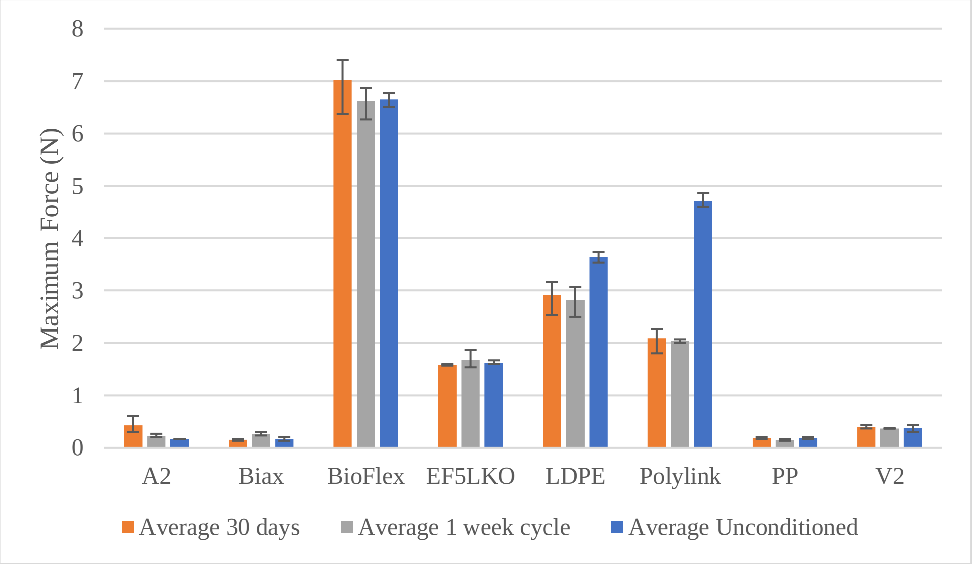


Figure 20. Tear Test Results of the two freeze-thaw cycles and unconditioned samples.

4.6.3 Response of Plastics to Heat Treatment

Figure 21 shows the maximum tear strength for each of the polymer samples after conditioning at temperatures of 60°C 80°C and 94°C. The overall order of materials with the greatest forces remained the same. There were no consistent trends identified when subjecting Biax or V2 to each of the temperatures. The introduction of heat caused the maximum tear strength for Polylink and EF5LKO to drop by almost half. For A2 samples conditioned at 80°C, the maximum force withstood dropped. This also happened when humidity is introduced. This is likely due to the transitions experienced by the material at temperatures of 70-90°C. The introduction of heat may change the structure and/or the crystallinity of the polymer leading to a slight change in mechanical properties. BioFlex had an increase in maximum strength when conditioned at 94°C compared to conditioning at 60°C and 80°C as well as room temperature. As the temperature at which the LDPE was exposed to increased, the maximum force also increased. This is also true for PP but with much lower forces. It is evident from these results and results in 4.6.2 that LDPE performs better when conditioned at higher temperatures with the maximum tear strength increasing with increasing conditioning temperatures from -18°C to 94°C. As all conditioned tear tests were run at room temperature, this suggests that conditioning certain polymers at different temperatures may alter the structure and therefore properties of the polymer, although the changes are minimal.

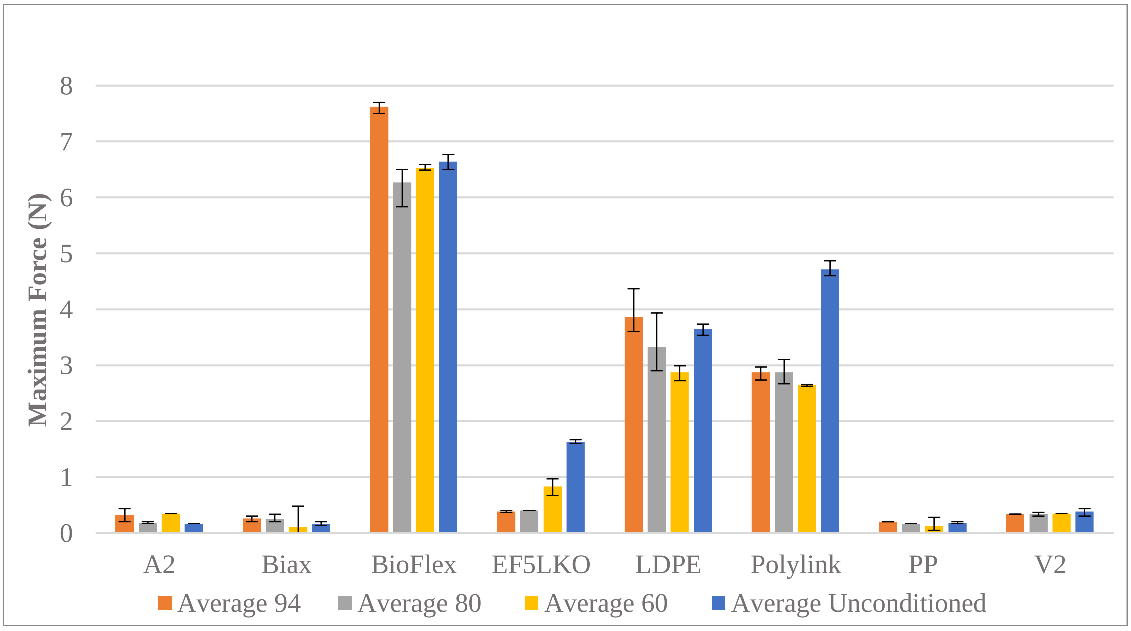


Figure 21. Tear Test Results After conditioning at 94 °C 80 °C and 60°C.

4.6.4 Response of Plastics to Heat and Humidity

Different polymers have different capabilities in terms of absorbing moisture. The effect moisture has on the mechanical properties also differs in different polymers depending on characteristics such as crystallinity as water passes through amorphous regions more easily.(404) Absorbed water can reduce the glass transition temperature and strength and cause dimensional effects including swelling.(405) Increasing the moisture of a sample results in the formation of hydrogen bonds, increasing the distance between chains and decreasing the glass transition temperature, and therefore the temperature at which materials become brittle. (406)

Figure 22 shows the maximum force withstood after conditioning at 90% humidity at 60°C 80°C and 94°C There are no notable changes in the mechanical properties for Biax over the temperature range. There are also no notable changes in the mechanical properties with the introduction of humidity for Biax. PP and V2 show slight increases in mechanical strength with an increase in temperature. The same phenomenon of a drop in tear strength in samples conditioned at 80 °C occurs in A2, as stated in 4.6.3, with the introduction of humidity. For EF5LKO and Polylink the maximum force decreases with the introduction of heat and humidity compared to the unconditioned sample. The introduction of heat and humidity may lead to slight degradation of the polymer which therefore may have small effects on the overall strength. There were also no noticeable changes in the mechanical forces at each of the temperatures with the introduction of humidity for A2, Biax, PP or V2.

Bioflex has a similar maximum force when conditioned at 60°C and 94°C. For the samples conditioned at 80°C, the maximum force increases. The introduction of humidity causes an increase in tear strength for BioFlex samples conditioned at temperatures of 60 °C and 80°C The maximum strength when conditioned at 80 °C increases when humidity is introduced with values increasing from 6.26N to 7.34N. For the samples conditioned at 94°C, the maximum force decreases from 7.62N to 6.53N with the introduction of humidity. No major changes are exhibited for the BioFlex samples conditioned at 60°C with the introduction of humidity. Similar behaviours are noted in Polylink with regards to the introduction of humidity with an increase in maximum force for samples conditioned at 60 °C and 80°C but decrease in tear strength at 94°C The changes noted in maximum tear strength after the films had been conditioned are very small. The load cell used was 100N and so using a load cell of smaller magnitude such as 10N may give more accurate results.

The tear strength for LDPE remains relatively constant over the given temperatures compared to when samples are not exposed to humidity. At higher temperatures, LDPE is in its ‘rubbery’ state due to its low glass transition temperature. This increases the materials permeability and therefore water absorption. An increase in water absorption leads to an increase in chain mobility, hence reducing the mechanical strength (407) Although the tests are run at ambient temperature, exposure to high heat and humidity may cause permanent changes to the structure of the polymer, affecting the mechanical properties.

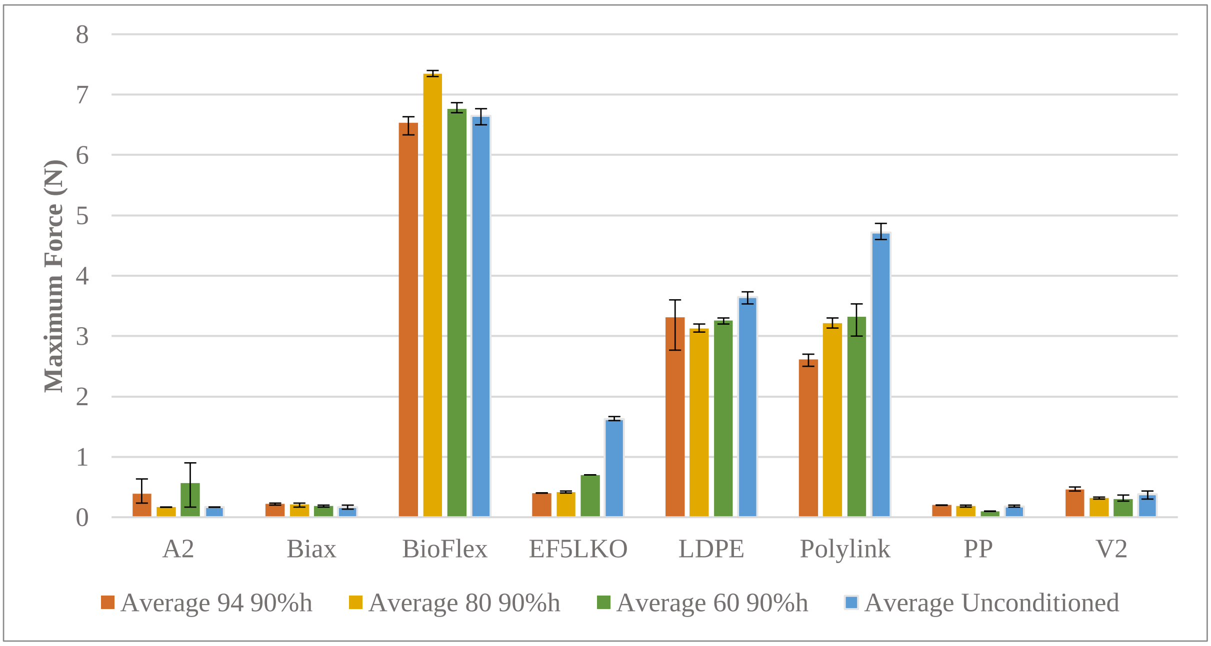


Figure 22 Tear Test Results After 94 °C 94%h, 80 °C 94%h and 60 °C 94%h

4.7 Tensile Test

A tensile test measures the maximum force a material can withstand before breaking. The first part of the test was run at a slower speed of 5mm/min for one minute as at a slower rate, chains are able to rearrange and flow plastically.(304) After one minute, the speed was increased to 100mm/min which caused the polymers to deform until failure.

From Figure 23, it is evident that Biax has a much greater tensile strength than all polymers tested. It was noticed that under tension, Biax turned from transparent to translucent. This is due to the aligning of the amorphous chains when under tension, giving the material a more crystalline structure and increasing its strength.(408) Polypropylene had the second highest maximum tensile force with a value of 37.8N, 79% less than Biax. These films are very stiff and exhibit low elastic behaviours and it can be seen in Figure 24 that Biax and PP have straight, clean breaks and don’t exhibit any signs of stretching. A2, BioFlex, and EF5LKO all have very similar tensile forces. Polylink and LDPE had the lowest tensile strength of all the polymers tested. The error bars on this chart indicate very small variance in the results. During the tensile test, the materials are subjected to much greater forces compared to when tearing. This may be the reason for the reduction in error and suggests that perhaps using a load cell with smaller magnitude may reduce any errors.

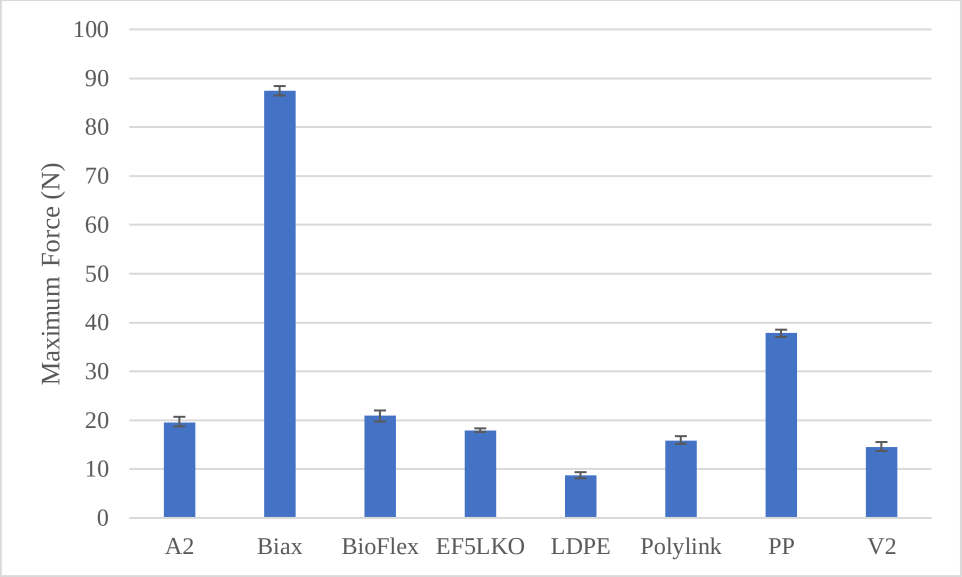


Figure 23 Tensile Test Results.

Figure 24 shows the samples after being subjected to a tensile force. A2, Biax, V2, EF5LKO and PP all showed clean breaks perpendicular to the force due to the direction of the chains. Bioflex showed a large elongation and necking due to its ductility.(409) During FTIR analysis, it was noted that BioFlex contained PBAT. This is often added to PLA to improve the flexibility of the PLA as it has a high elongation of >500%. (410) Polylink and LDPE also showed a small amount of elongation and necking. Elongation is the measure of a materials ability to deform before failure and is a measure of ductility. (304)

Figure 24. Image of samples after tear test. Left to right: A2, Biax, BioFlex, V2, EF5LKO, Polylink, LDPE and PP.

Figure 25 shows the stress-strain curves for the biopolymers and the two polyolefins tested. Stress strain curves were generated to give more detail about the polymers performance under tension as well as to give further information about the material as samples had various thicknesses. Comparing the characteristic curves, it can be seen that BioFlex, Polylink and LDPE have similar curves, while A2, Biax and V2 have similar curves. As previously mentioned, this is due to the extensibility of the materials. Polymers exhibit a combination of elastic and plastic deformation with the proportion of each dependent on the viscosity of the polymer. (304)

LDPE and V2 have a much lower maximum stress compared to the other materials. A2, BioFlex, EF5LKO and Polylink all have relatively similar maximum stress values. PP and Biax experienced much greater stresses compared to the other films tested, with a maximum stresses of 52.87MPa and 68.4MPa respectively. The stresses within a material are affected by the composition and properties of the material. Generally, materials with high stiffness may experience higher stresses compared to more flexible and ductile materials. They also tend to fracture suddenly, with little observable elongation. This is confirmed by figure 24. Comparing the stresses and strains of these two materials to the other materials, it can be assumed they have similar mechanical properties. Their physical properties are also similar with both materials having high gloss and high transparency.

BioFlex had the greatest %Elongation with a value of 314%. This suggests that this material is ductile and performs plastically without fracturing, indicated by the large plastic region on the stress strain curve. A high %Elongation for BioFlex was expected after it was discovered that it contained PBAT which has high elongations of >500%. Polylink is another flexible material with a %Elongation of 113%. Flexible packaging is essential for the packaging of certain products, usually heavier items, as the films are able to deform before failure to give indication of potential failure, hence reducing the risk of damage to products. Ductile materials also tend to have good tear resistance. Comparing figure 25 to figure 18, it can be seen the more flexible films with greater %Elongation have a greater tear resistance. EF5LKO had the lowest %Elongation although had a greater maximum tear force than A2, Biax, PP and V2. A2, EF5LKO and V2 all have similar %Elongation at failure values.

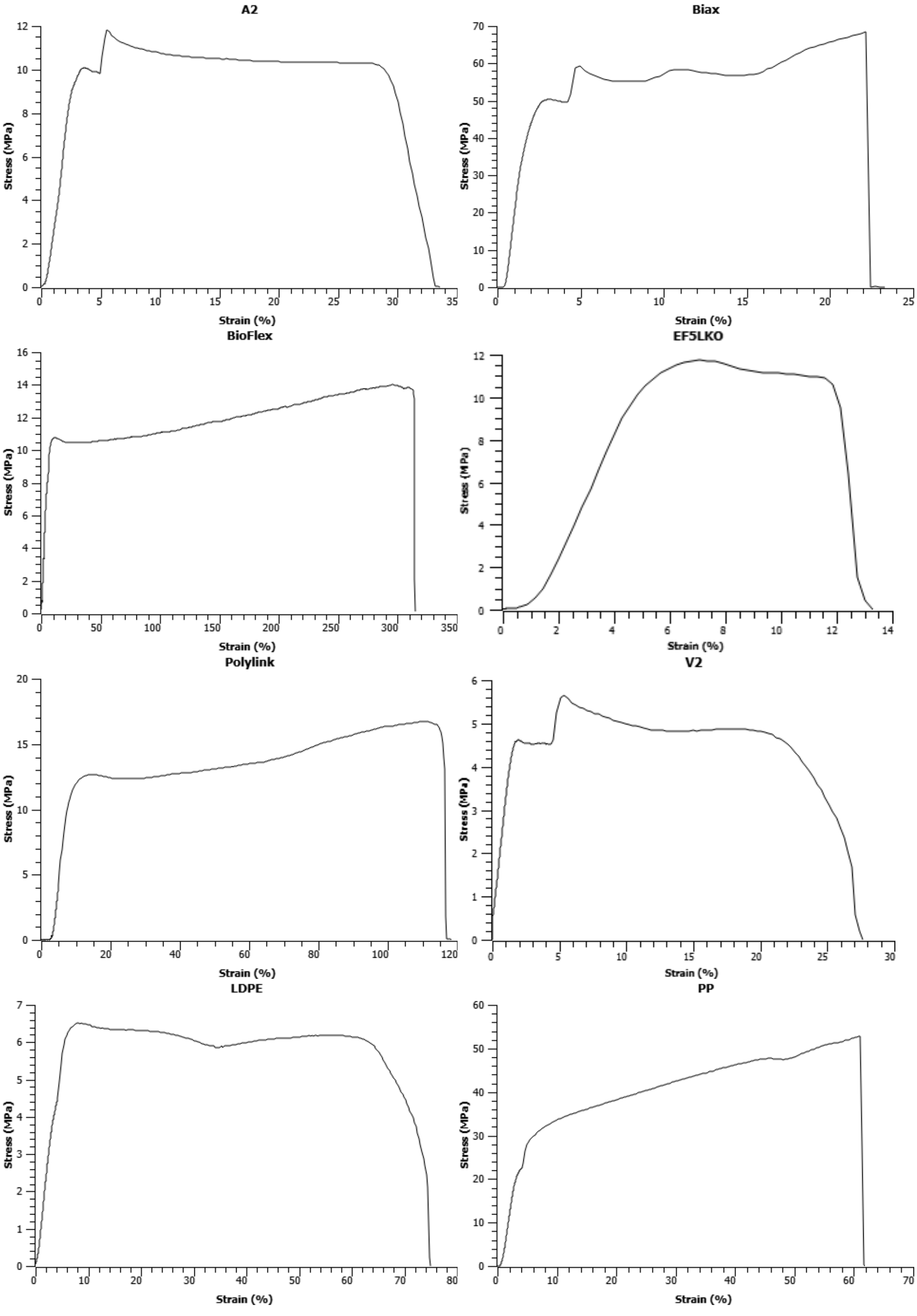


Figure 25 Stress-Strain Curves for all polymers tested

4.8 Composting

4.8.1 Methods to Determine Degradation

i. *Visual Interpretations*

After 6 months of degradation, Polylink was the only film that exhibited physical signs of degradation. Figure 26 shows the 120x25mm Polylink samples after 6 months of degradation. The samples were very brittle and began to fragment during removal from the compost facility and whenever handled. After 6 months of degradation, A2 showed no visual indications of degradation. After 9 and 12 months, there were no traces of A2 in the soil.

Figure 26 Polylink samples after 180 days composting.

ii. FTIR

FTIR is a widely cited method of degradation measurement.(411–416) Degradation is reported as a change in absorbance of peaks at 1750cm^{-1} and 1715cm^{-1}.(417) C=O ester groups will decrease due to degradation, causing an increase in absorbance in peaks around 1715 cm^{-1} relating to carboxyl groups.(417) O-H bonds at around 3400cm^{-1} are also expected to increase due to the formation of hydrogen. (418)

Figure 27 shows the spectra of samples after 3, 6, 9 and 12 months of composting. The overall peak intensity of A2 increased indicating degradation. There was a slight increase in absorbance of the peak at 1715 cm^{-1} and decrease at 1740 cm^{-1} after 180 days of composting A2. Strong peaks also occur at 1086cm^{-1} and 1186cm^{-1} due to C-O stretching after 6 months of soil burial, indicating oxidative degradation. Biax showed no significant changes in absorbance along with EF5LKO indicating that no, or very minimal degradation occurred. V2 showed an increase in the broad O-H stretching peak at around 3400cm^{-1} due to the production of water during biodegradation. V2 also saw a reduction in the absorbance in the C=O region due to the decomposition of ester groups. LDPE saw a large increase in absorbance overall after 3 months, indicating an increased level of degradation. After 6 months of soil burial, the absorbance decreased to below the level of absorbance for samples that were exposed to 9 and 12 months of soil burial. The beam used in FTIR is incredibly small and therefore only monitors a small amount of the material. Due to crosslinking and other factors, results can vary as different regions of a polymer are attacked during biodegradation.

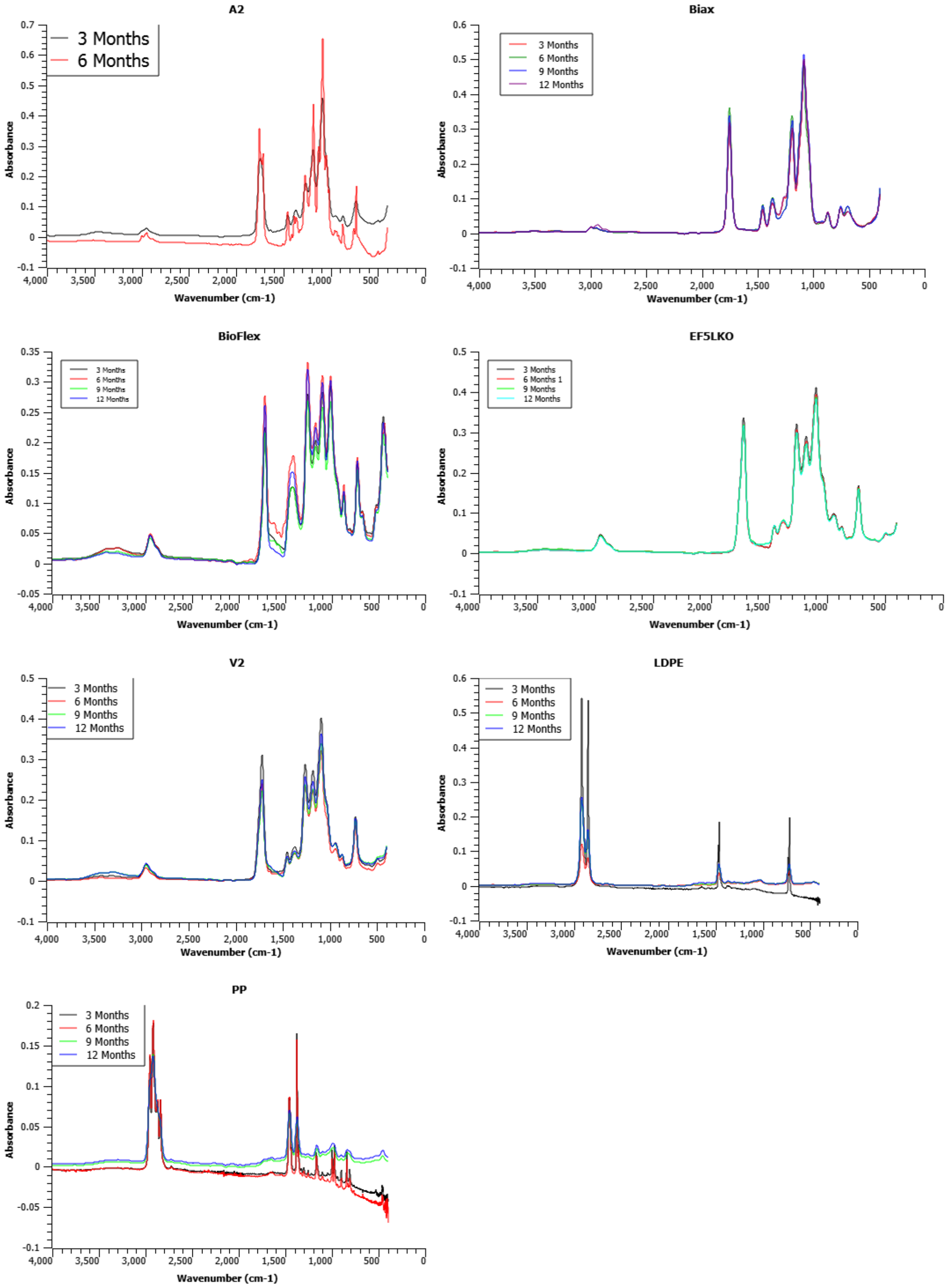


Figure 27. FTIR Spectra of samples after 3, 6, 9 and 12 months of soil burial.

iii. DSC

During biodegradation, the amorphous regions of the plastic are attacked first, resulting in a change in the glass transition temperature. (419) Table 9 displays the melting temperatures of the polymers after 3, 6, 9 and 12 months of composting. Slight fluctuations were noticed due to test conditions but there were no significant changes seen in the melting temperatures of any of the biopolymers or polypropylene. LDPE saw a significant decrease in the melting temperature over the 12 month burial period with a decrease from 123°C to 110°C Branching in a material causes defects which results in a decrease in the melting temperature. The flexibility of the chains also affects the melting temperature with the presence of double bonds and aromatic groups restricting movement and therefore increasing the melting temperature.(304) Materials with lower melting temperatures are more susceptible to microbial attack. (420)

Table 9 Melting temperatures of samples after soil burial.

Material	Lower Melting Temperatures (°C)				Melting Temperature (°C)			
	3m	6m	9m	12m	3m	6m	9m	12m
A2	66	65	-	-	167	166	-	-
Biax	72	72	71	71	169	167	167	167
BioFlex	63	62	62	63	152	152	152	153
EF5LKO	62	62	64	64	166	166	166	166
LDPE	-	-	-	-	122	112	109	110
PP	-	-	-	-	169	166	167	168
V2	-	-	-	-	166	166	166	166

Oxidation induction temperature can be used as a measure of the level of degradation. The same method as stated in 3.2.2 was used and thermograms were produced. Again, the tangential method was used to determine the oxidation induction temperature. It was expected that the oxidation temperature would decrease with an increase in the level of degradation, and therefore samples with longer exposure times would have decreased oxidation induction temperatures.(418) Table 10 displays the oxidation induction temperature of the polymers after 180 days of composting. It can be seen that the oxidation induction temperature remained the same for A2, LDPE and PP. Slight increases of 4 °C and 6 °C were seen in the oxidation temperatures of EF5LKO and V2 respectively. This is likely due to slight errors incurred during the deduction of the temperature through the tangential method. Large decreases of 50 °C and 17 °C occurred in the oxidation induction temperature of Biax and BioFlex respectively. Large decreases such as these do not occur due to errors and therefore indicate degradation in these samples.

Table 10 Oxidation induction temperature of samples after 180 days of composting.

Material	Oxidation Induction Temperature prior composting(°C)	Oxidation Induction Temperature post composting (°C)
A2	230	231
Biax	284	231
BioFlex	240	223
EF5LKO	220	224
LDPE	223	220
PP	225	218
V2	230	236

iv. Mechanical Testing

After initial inspection, it was assumed that minimal to no degradation had occurred in the materials. Figure 28 shows the maximum tear force of plain samples compared to samples that have been composted for 180 days. The mechanical properties for all materials excluding A2 has been reduced. Polypropylene showed the largest decrease in tear strength with a 44% reduction in the maximum force withstood. Out of the six samples tested, the deviations in results are very small, as indicated by the error bars on the graph. BioFlex showed the lowest percentage decrease in mechanical properties with a decrease in tear strength of 8%. BioFlex is a very dense material with a much greater tear strength and so it was predicted that degradation would be limited and mechanical properties would remain superior. The increase in tear strength for A2 could be down to initial cross linking causing an initial increase in mechanical properties. Anisotropy in the material may also contribute to the difference in mechanical strength before and after composting. . If the test was to be redone, samples would be cut at a 90^o angle for each of the materials to account for strength differences in the machine and transverse directions. One of the difficulties with composting samples for such long periods of time is that it is difficult to conduct repeats. More samples should be added to the composting facility to allow for further repeats in destructive tests.

The error bars shown in figure 28 display the maximum and minimum values recorded. It can be seen that the error incurred is very small and so results can be assumed to be reliable. Also, the reduction in maximum force post composting is very small, and is reduced further when error bars are added. These changes are all less than 1N and are therefore negligible. A2 and BioFlex display much larger error bars for the data recorded after 180 days of composting.

The difference in results for A2 may be due to the anisotropic nature of the material, as previously discussed. The variance in the BioFlex results may be due to errors caused due to the large magnitude of the load cell or a possible variation in the degradation of the samples.

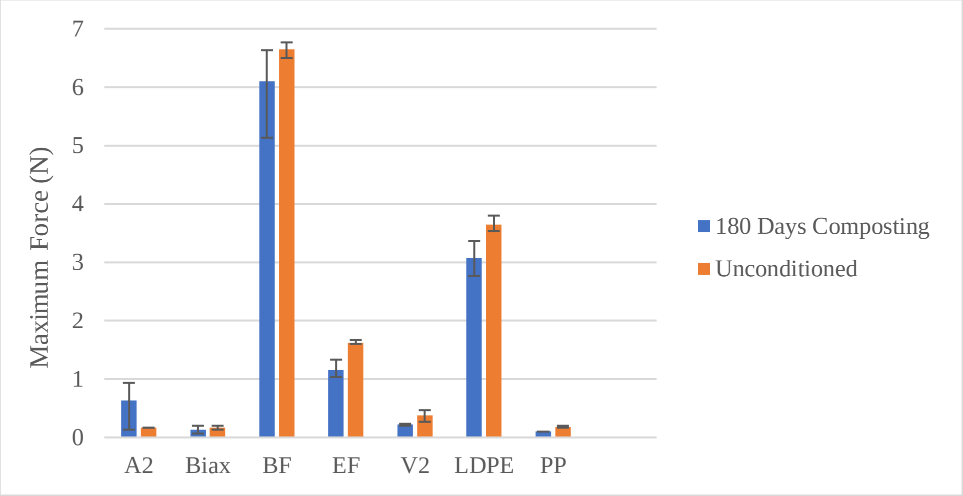


Figure 28. Tear tests after 180 days of composting compared to non-treated samples.

4.9 Accelerated Weathering

After conducting UV weathering for 1500 and 2000 hours, no samples were stable enough to tear test. After this result, it was noted that UV irradiation rapidly decreased the stability and mechanical properties of both the biobased and petroleum films, so it was predicted that tear properties would decrease after UV exposure. After 180 days of composting, the samples were very brittle and needed to be removed carefully. Figure 29 shows the samples A2, Bioflex, EF5LKO and V2. Samples are very difficult to segregate due to the level of degradation. Due to the level of degradation of Polylink without UV pre-treatment, it was assumed that there were no residuals of this material.

Figure 29 Samples after 180 days of composting, post UV irradiation.

4.9.1 Mechanical testing

Figure 30 shows the maximum tear force of UV irradiated samples after 180 days of composting compared to plain samples. Only one A2 sample was stable enough to be put into the Hounsfield and tear tested. This effects repeatability resulting in the reliability of results to be questioned. All samples that were added to the composting facility for Biax, LDPE and PP were recovered, tear tested and an average was calculated. A 62% decrease in tear strength was noted in PP after composting after UV irradiation compared to a 43% reduction in tear strength without UV irradiation indicating that UV pre-treatment has a detrimental effect on the biodegradation of PP. A2 showed a 60% reduction in tear strength after UV irradiation. This decrease further supports the proposition that anisotropy is responsible for the increased mechanical properties stated in 4.8.1. LDPE showed a decrease of 16% and Biax showed the smallest decrease in tear properties with a reduction of 10% in the tear strength post UV irradiation. The introduction of UV had a very minimal effect on reducing the mechanical properties of LDPE after composting, with a 0.5% decrease in tear strength. Materials such as polyethylene often contain stabilisers to reduce thermal-oxidative degradation during reprocessing. These stabilisers also act to improve the stability of the polymer during UV exposure. (421)

There is no error bar indicated for the UV irradiated A2 samples as only one sample was able to be retrieved and tested. The error bar for LDPE is quite large compared to the error bars of the other materials. This is the same for composting after 180 days without UV pre-treatment. This may be an indication that some form of degradation has occurred. Alteration to the properties is further suggested by the change in melting temperature after composting. As previously stated, the magnitude of the load cell may also be responsible for these errors and the difference in forces are a matter of newtons.

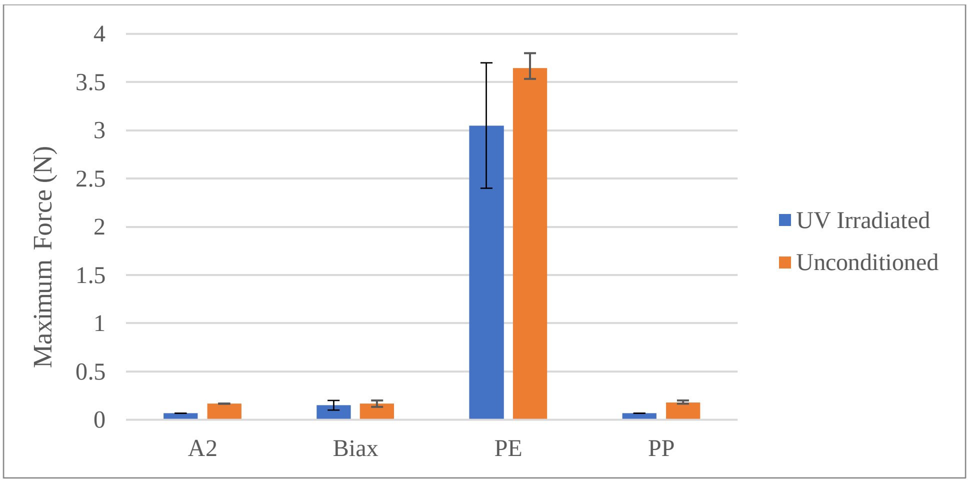
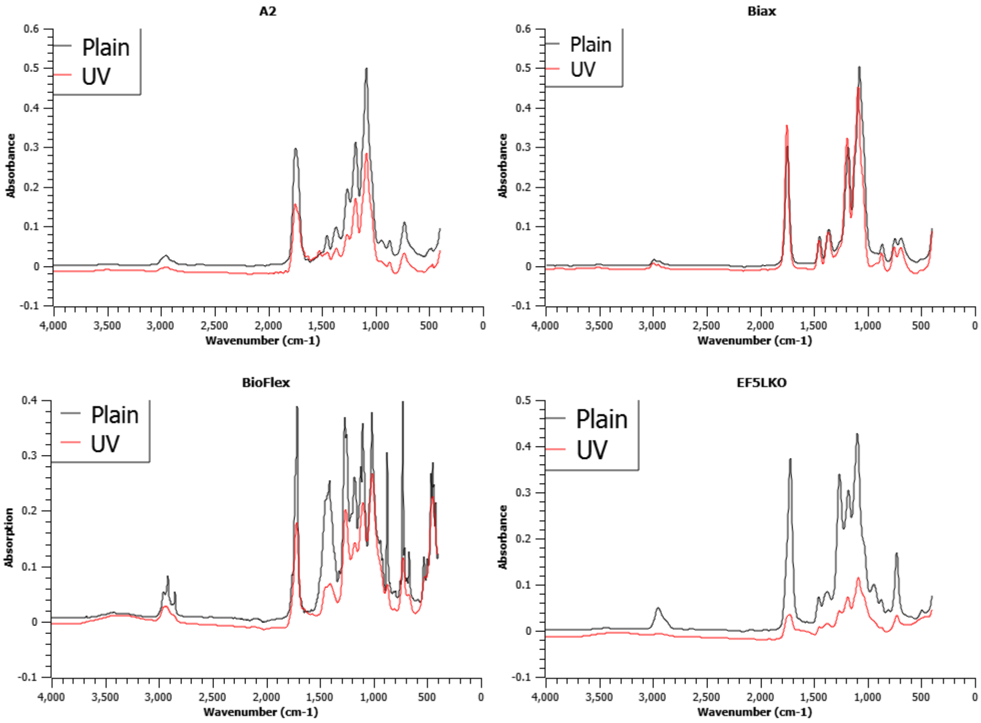


Figure 30. Maximum Tear Force of UV irradiated samples and Plain samples.

4.9.2 FTIR

Figure 31 shows the FTIR spectra of samples exposed to 336 hours of UV irradiation and 180 days composting compared to plain samples. A2, BioFlex, EF5LKO and V2 all experienced a large decrease in the absorbance. Absorbance is frequently reduced due to the concentration of the material. Cross-linking due to UV irradiation can caused a reduction in concentration and therefore absorbance. (422)No changes were noted in the absorbance of LDPE.



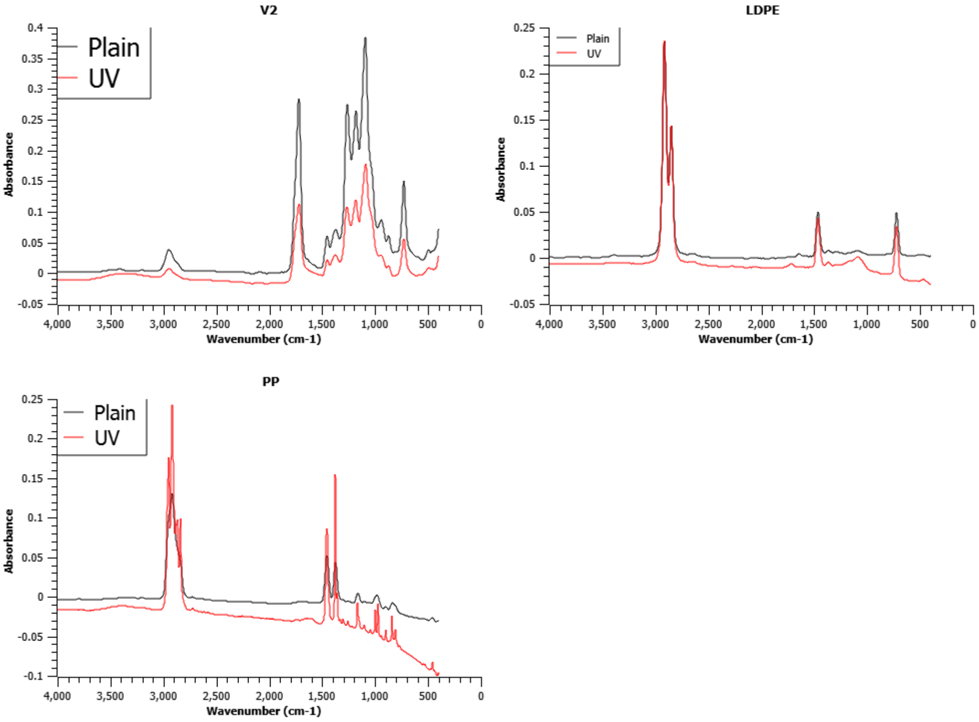


Figure 31. FTIR spectra of plain sample and UV irradiated with 180 days soil burial.

4.9.3 DSC

Table 11 displays the melting temperatures of the polymers after 180 days of composting after 336 hours of UV irradiation. The lower melting temperature of A2 remained unchanged and the melting temperature indicted by the larger peak decreased slightly by 4°C. Biax and BioFlex experienced slight increases in the lower melting temperatures indicated by smaller peaks, due to other materials within the matrix. All other melting temperatures for all other samples remained the same after UV irradiation. All changes were very minimal and may be caused by human error during the derivation of melting temperature from the thermogram. The melting temperature of LDPE remained the same at 122 °C Comparing this figure to the figure for 6 months in Table 9, it can be seen that introducing UV reduces the effect that biodegradation has on the thermal properties.

Table 11 Melting Temperatures of samples after UV pre-treatment and soil burial

Material	Lower Melting Temperatures (°C)	Melting Temperature (°C)
A2	67	165
Biax	75	169
BioFlex	67	152
LDPE	-	122
PP	-	170

It is also expected that oxidation induction temperature will decrease, indicating an increase in the level of degradation. Table 12 displays the oxidation induction temperature of the polymers after 180 days of composting. It was noticed from the thermograms produced that the change in enthalpy indicating oxidation induction were not as strong when UV irradiated samples were tested. This was also noted with the absorbance on the FTIR spectra and is due to the concentration of the of functional groups associated. (423) This made the determination via the tangential method more difficult. Therefore slight discrepancies may occur in the results. The oxidation induction temperature for A2 remained around 230°C The OIT of Biax was again significantly reduced from 284°C to around 230°C BioFlex showed a more significant change in enthalpy allowing the OIT to be determined more easily. A value of 222°C was established which is the same as that of the sample without UV irradiation indicating UV does not have an effect on the OIT. The value for LDPE decreased by almost 20°C indicating slight oxidative degradation. This degradation may just be caused by UV irradiation and not necessarily due to biodegradation. Further testing should be conducted on UV irradiated samples prior to soil burial to established this.

Table 12 Oxidation induction temperatures after 180 days composting and UV pre-treatment.

Material	Oxidation Induction Temperature (°C)
A2	Approximately 230
Biax	Approximately 235
BioFlex	222
LDPE	202
PP	221

Chapter 5. Conclusion

After comparing the FTIR spectra of the biopolymers to one another, it was concluded that the polymers had similar compositions. At the start of the project it was assumed that the biopolymers were PLA-copolymers. FTIR confirmed this was the case and the spectra displayed the main peaks associated with PLA. FTIR was also able to identify some of the other compounds present in the polymers such as cellulose and polyethylene glycol.

Overall, the mechanical properties of A2, V2 and EF5LKO are substandard in comparison to PP and LDPE so would not be suitable substitutions. The water loss rate over the time period was much greater for the bioplastics and so these polymers would not be suitable for applications where good barrier properties are required. the biobased and polyolefin films showed little difference in the results regarding oil permeation. The results for the oil permeation did not draw clear conclusions and results were likely effected by environmental conditions, although if the oil was permeating the polymers and diffusing out, some reduction in mass would be experienced.

Polylink and BioFlex had similar physical properties such as reflectance, colour and opacity. These two polymers also had the greatest maximum tear strength, with values comparable and superior to LDPE. This indicates that these materials would be best at resisting further damage and potentially failure if the packaging were to become defected. The stress-strain curves from the tensile tests also show these films exhibit large plastic deformation and have large %Elongation at failure. Therefore these bioplastics have the potential to replace LDPE in applications where good barrier properties are not required. Further research into the processing of these materials would be required to form full conclusions. Further research into the biodegradation of BioFlex in an industrial composting facility would also need to be conducted in order to draw full conclusions on whether BioFlex would be a sustainable replacement to LDPE.

Similarly to PP film, Biax is a transparent material with a similar melting temperature to PP. Biax also had a much greater tensile strength compared to the biopolymers and polyolefins. The maximum tensile strength of Biax was 79% greater than the tensile strength of polypropylene and had a slightly greater tear strength than PP. a greater tear strength is ideal for reducing the failure of packaging when defects occur although considerations would need to be taken when replacing polypropylene in applications where lower tear resistance is required, such as for packaging lighter items where access via ripping is used. Biax also had a much greater OIT than all polymers tested with a value of 284°C, indicating it has a greater thermal stability and is likely to provide a more effective barrier against oxygen. Further research into this would need to be conducted to draw full conclusions. Biax also had the lowest water loss rate in comparison to the other biopolymers although this was still much greater than for the polyolefins. Therefore Biax would be a practical and sustainable replacement to polypropylene in the packaging industry where good moisture barrier properties are not essential. This research found that Biax showed no physical indication of biodegradation over the 180 day period, even after 336 hours of UV pre-treatment. Therefore further research would need to be conducted into the degradation process of this material in an industrial composting facility along with the origin of the compounds used in the production of the material to establish if it is a sustainable alternative

UV irradiation increased the rate of degradation of A2, BioFlex, EF5LKO and V2 significantly to the extent that it was difficult to isolate the materials. Due to the level of degradation without UV pre-treatment, it was assumed that Polylink fully degraded after UV pre-treatment. Samples were very brittle and needed to be handled carefully. Only one A2 sample was stable enough for tear testing and hence no repeats were taken reducing the reliability of the result. This indicates that UV pre-treatment could be considered as a method of pre-treatment to enhance the biodegradability of these films. It also indicates that over a prolonged period of time, Biax, LDPE and PP would withstand degradation due to environmental factors such as UV more than the other films tested.

Home composting was adopted as the method of biodegradation during this project as this is the most efficient and sustainable method of disposal for compostable films. It should be noted that as industrial composting is a more aggressive process at elevated temperatures, the biopolymers tested within this project that did not compost in the home facility may degrade in an industrial facility. As home composting is the desired method of disposal and the method used within this project, conclusions have been made based on this. Visual inspections were the most accurate method of degradation measurement. Other methods of degradation identification included peak analysis through the use of FTIR, monitoring of Tm and OIT through the use of DSC and tear strength. These methods observed very little indications of degradation and the polymers with properties that altered were different for each method of measuring degradation. Therefore, visual interpretation is the trusted method of degradation identification. From this method, Polylink was the only polymer to show signs of degradation.

As Polylink fully degraded within the 365 days as given by the standard, Polylink is the only film provided by Greenlight Packaging to be considered as a home compostable alternative to the polyolefins tested. It has the potential to replace LDPE in applications where good barrier properties are not required and clarity is not essential as a compostable alternative for single use applications or packaging with short lifespans. Good tear resistance is required in applications where large forces are going to be endured. Therefore, Polylink could be used to replace commonly used LDPE in applications such as carrier bags. Carrier bags are usually opaque, and used at room temperature. They are very rarely exposed to humidity or extreme high or low temperatures post processing. LDPE has a particularly low melting temperature meaning it has good heat sealing properties and so further research would need to be conducted into the heat sealing properties of Polylink due to its undefined melting temperature. Single use carrier bags are a major issue at present and so introducing a compostable alternative could have a profound effect on plastic pollution. (99) Currently, certain supermarkets provide compostable carrier bags and so further research into the optimal material for compostable single use carrier bags could be conducted, comparing the physical and mechanical properties as well as the associated biodegradation process. The origin of the raw materials should also be assessed in order to form full conclusions on the most sustainable and viable material.

With improved waste management infrastructure, advancements in material technologies and correct labelling, compostable polymers have the potential to resolve the issues which plastics are currently causing.

Future Work

As previously stated, Polylink is a material with high tear strength and rate of biodegradation but low stability and tensile strength. Blending the polymer with PE, PP or Biax may improve its properties and enhance the biodegradability of PE, PP and Biax. During the planning of this project, it was intended that blends were to be created, composted and tested using the same methods but unfortunately, the machinery was not available so this could not be done.

Polylink had the highest water loss rate out of all of the polymers tested. Creating LDPE/Polylink and PP/Polylink blends could increase the barrier properties of the Polylink. LDPE saw an increase in the mechanical properties at elevated temperatures and so creating LDPE/Polylink blends may reduce the effect that higher temperatures have on the mechanical properties of Polylink.

Polylink is also an amorphous material which softens gradually over a range of temperatures. Polypropylene has the highest melting temperature of the eight films of 170°C The melting temperature and glass transition temperatures of these blends would be measured and also crystallinity calculated. Polypropylene was also the only polymer to have an oxidation induction time of greater than 1 minute. Creating PP/Polylink blends may increase the stability and tensile strength of Polylink. PP also had a relatively high tensile strength while Polylink had a much lower tensile strength.

Creating blends would reduce the biodegradability of the polymers but would enhance the properties of the biopolymers. Although biodegradability would be compromised, the sustainability of raw materials would be improved as renewable sources are used for the production of PLA, compared to petroleum sources used for LDPE and PP. As some of these biopolymers have comparable, and in some cases inferior mechanical properties to the polyolefins tested, focus should be on improving the barrier properties of biobased materials for use in the grocery packaging industry, where good barrier properties against air and moisture are required.

Further research into the biodegradability of the biopolymers in an industrial facility where higher temperatures are used should also be conducted. This may open the opportunity for these polymers to be collected by councils with other compostable waste, and composted in a facility. Although this still relies on accurate sorting and disposal, it is a better method of disposal for films. There are less issues with contamination meaning there will be less materials rejected from sustainable recovery compared to recycling. Further research into improving the home compostability of these materials should also be conducted.

Biax is a biobased material comprised of PEG-PLA block copolymer. Blending Polylink with Biax would mean the material is still biobased and produced from renewable resources unlike PE/Polylink and PP/Polylink blends. Polylink has an increased tear strength and a relatively low tensile strength while Biax has a substantially higher tensile strength but low tear strength. Creating a Biax/Polylink blend will improve both of these properties. Blending these material at different ratios has an effect on the properties and so different %wt should be used to find an optimal result. Additives can also be included in the matrix to improve properties further. The addition of PBAT to the polymer matrix of BioFlex may be responsible for its high elongation at break and could be used to improve the ductility of materials such as Biax and PP. It should be noted that there are different methods used for the preparation of polymer blends and some blends may be immiscible. Further research into the compatibility, miscibility and how the properties are altered would need to be conducted before full conclusions are drawn.

The level of toxicity would need to be evaluated and approval is required if the polymers are to be used within the food packaging industry to ensure materials comply with legislations. The gas permeability would also need to be investigated for the application of longer life products. Further testing on the polymers surface properties would also be required to determine the ability to print on the polymers as well as the determination of migration.

Further research into the materials behaviours in various marine environments could also be investigated to simulate the effect on materials if they were to end up in the natural environment. Delacuvellerie et al submerged compostable polymers PLA and PBAT along with non-degradable LDPE, PS, PET and PVC in the Mediterranean sea on the sediment and in a water column. After 82 days in the water column, no bacterial degradation was observed. After 80 days in the sediment, PBAT experienced a 12% weight loss due to the presence of bacteria. (424) Throughout this research, it has become evident that the rate of biodegradation is highly dependent on the micro-organisms present and the enzymes they produce.

Compostable biopolymers are relatively new materials and although interest in them has grown significantly recently, very little information and research is available. This paper adds to the current pool of data and research that is currently available for compostable and biobased polymers.

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