



TEKNILLINEN TIEDEKUNTA

# **BIODEGRADATION OF SYNTHETIC TEXTILE FIBRES**

Idamaria Romakkaniemi

PROCESS ENGINEERING

Bachelor's Thesis

2018



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Instructor: Sanna Taskila

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# TIIVISTELMÄ

## OPINNÄYTETYÖSTÄ Oulun yliopisto Teknillinen tiedekunta

Koulutusohjelma (kandidaatintyö, diplomityö) Prosessitekniikan koulutusohjelma		Pääaineopintojen ala (lisensiaatintyö)	
Tekijä Romakkaniemi, Idamaria		Työn ohjaaja yliopistolla Taskila Sanna, TkT	
Työn nimi Synteettisten tekstiilikuitujen biologinen hajottaminen			
Opintosuunta Biotuotteet ja bioprosessitekniikka	Työn laji Kandidaatintyö	Aika Elokuu 2018	Sivumäärä 33
<p><b>Tiivistelmä</b></p> <p>Synteettiset kuidut ovat nopeasti yleistynyt materiaali tekstiiliteollisuudessa. Niitä käytetään monissa arkipäiväisissäkin tuotteissa sellaisenaan tai sekoitettuna muiden kuitujen kuten puuvillan kanssa. Niillä pyritään saavuttamaan tekstiilille halutut erityisominaisuudet. Synteettiset tekstiilikuidut koostuvat polymeereistä ja eivät pääsääntöisesti hajoa luonnossa sellaisenaan. Niiden kerääntyminen luontoon ja meriin on kasvava ympäristöongelma ympäri maailmaa. Synteettiset polymeerit voivat olla vaarallisia eliöille joutuessaan niiden ruuansulatukseen.</p> <p>Tämän ongelman ratkaisemiseksi on pyritty löytämään biologisia menetelmiä hajottaa synteettisiä kuituja. Erilaisia mikro-organismeja on tutkittu, jotta löydettäisiin organismeja, jotka pystyisivät hyödyntämään synteettisiä kuituja metaboliassaan.</p> <p>Tässä kandidaatin työssä pyrittiin keräämään mahdollisimman kattavasti tietoa tähän päivään mennessä tehdyistä tutkimuksista synteettisten kuitujen biohajoamisesta. Erilaiset kuitulajit ja niiden komponentit määriteltiin ja selvitettiin, millaisella prosessilla niitä valmistetaan. Jokaisen kuidun ja niiden komponenttien biohajoamista selvitettiin mikrobien ja entsyymien avulla. Eniten tutkimustuloksia löytyi polyuretaanikuiduista ja vähiten aromaattisista polyamidikuiduista. Tiedonhankinnan tuloksena voisi todeta, että aihe vaatii yhä tieteellistä lisätutkimusta, jotta löydetään tehokas biologinen keino hajottaa synteettisiä kuituja.</p> <p>Tämä kirjallisuustutkielman tavoite on antaa laaja yleiskuva synteettisten tekstiilikuitujen aiheuttamasta saasteongelmasta ja niiden biologisesta hajottamisesta sekä pyrkiä korostamaan, että kerättyjä taustatietoja tulisi hyödyntää ongelman ratkaisemisessa.</p>			
Muita tietoja			

# ABSTRACT FOR THESIS

University of Oulu Faculty of Technology

Degree Programme (Bachelor's Thesis, Master's Thesis) Process Engineering		Major Subject (Licentiate Thesis)	
Author Romakkaniemi, Idamaria		Thesis Supervisor Taskila, Sanna, D. Tech.	
Title of Thesis Biodegradation of synthetic textile fibres			
Major Subject Bioproducts and bioprocess engineering	Type of Thesis Bachelor's thesis	Submission Date August 2018	Number of Pages 33
<b>Abstract</b> <p>Synthetic fibres are a raw-materials, that have rapidly become more commonly used in the textile industry. They are used in many everyday products as such or as blends with other fibres like cotton. Synthetic fibres are utilized to achieve the wanted special abilities in textiles. Synthetic textile fibres are composed of polymers and mainly do not decompose in nature as such. Accumulation of these fibres is a growing environmental problem across the globe. If digested, synthetic fibres can be dangerous to organisms.</p> <p>To solve this problem, research has been done to find biological ways of degrading synthetic fibres. Different kinds of micro-organisms have been examined to find organisms that can utilize synthetic fibres in their metabolism. In this bachelor's thesis the aim was to gather as much information as possible on researches done on synthetic fibre biodegradation to date. Different kinds of fibres and their components were identified and the production processes were examined. Biodegradation of each fibre and their components by microbes and enzymes was investigated. The majority of results were found on polyurethane fibres while less results related to aromatic polyamide fibres. As a result of this data collection it could be stated, that the topic needs more scientific research to find effective ways to biodegrade synthetic fibres.</p> <p>The goal of this literary research is to give an extensive overview on the pollution problem caused by synthetic textile fibres and the biological methods of degrading them as well as to emphasize that the gathered information should be utilized to solve the problem.</p>			
Additional Information			

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ABSTRACT

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## **SYMBOLS AND ABBREVIATIONS**

HDEP	High-density-polyethylene
LDPE	Low-density-polyethylene
MSM	Mineral salt medium
PAN	Polyacrylonitrile
PET	Polyethylene terephthalate
PETase	polyethylene terephthalate hydrolase
PUR	Polyurethane
PUR-A	Solid PUR media
SDA	Sabouraud agar

# 1 INTRODUCTION

## 1.1 Goals

The objective of this thesis is to collect existing information on biodegradation of synthetic textile fibres. The focus is mainly on microbes that have the ability to biologically decompose synthetic fibres wholly or into smaller less harmful particles or compounds.

First task is to identify what synthetic textile fibres are, what are they made of, where are they used, how do they end up in nature and marine environments, and what can be done to solve this issue. This thesis aims to answer these questions and to survey the literature on topic. Aim is to gather sufficient quantity information to map the rate of the pollution problem and ways to fix it.

## 1.2 Background

Plastic is one of the most common raw-materials for the present-day commodities. We use plastics everyday more than we even realize, namely more than 240 million tonnes per year (Browne et al., 2011). Even some of our clothes are derived from plastics. Nowadays many of the plastics can be reused, but still most of them end up in landfills and take a lot of time to decompose, if they ever do. Approximately 10% of the plastics produced end up in the marine environments. (Astrom, 2016)

But not all plastics are that easy to clean, or even detect. Microplastics are plastic particles smaller than 5 mm (in some cases specified as even smaller than 1 mm). Compared to macroplastics, they have a larger surface-to-volume-ratio. Due to this, they concentrate non-degradable organic pollutants and absorb metals. (Taylor et al., 2016)

Microplastics originate from degradation of larger plastic particles and are just as harmful for the environment. These little plastics have been detected all around the world, even in the deepest parts of the ocean. (Van Cauwenberghe et al., 2013)

Even though we are trying to decrease the amount of plastic emission, it is impossible to clean up all the plastic particles from the oceans. Plastics will continue their slow lifecycle until they end up at the bottom of the ocean. (do Sul et al., 2014)

And still at the bottom of the ocean, the plastics are causing harm. In (Fischer et al., 2015) microplastic waste was found in the sediments from deep waters between 4869 and 5766 m. Inside deep-sea organisms microplastics were found at the depths of 334-1783 m (Taylor et al., 2016).

The marine wildlife is suffering from the plastic emissions. Vertebrate and invertebrate marine species have been examined and for example many fish and seabirds have been found with plastic particles in their digestive systems. Plastic waste can transfer pollutants from all over the ocean, and by eating plastic particles the organisms become exposed to pollutants. These pollutants may even transfer further to the predators eating the primary exposed species. If the microplastics do not get eaten, they can eventually float to various destinations and become available for even larger variety of species. The plastic waste can cause physical damage to the intestines of the organisms. (do Sul et al., 2014)

It has been investigated, that microplastic fibres are shedding from synthetic textiles for example fabrics during washing. In the research "Shedding of synthetic microfibers from textiles." (Astrom, 2016), more than 90% of the debris found on the west coast of Sweden consists of textile fibres.

A short research and discussion article (2016) by U. Pirc et al. investigates fibre shedding from polyethylene terephthalate (PET) polyester. It is commonly used in fleece fabrics. The article concludes that the approximate shedding of PET fibres during each washing is about 0.0012 wt-%. During tumble drying the amount of fibre shedding was approximately 3.5 times higher. (Pirc et al., 2016)

Other studies have also shown that the amount of fibres lost in one litre of washing effluent is about 100-300 fibres. In case of fleece fabrics the number is even higher, namely 1900 fibres per one washing. (Astrom, 2016)

Astrom also reports that the greatest shedding amounts are discovered from the fleece fabrics. Astrom speculates this to be derived from the difference in the production method of the textile: the woven fibres are cut on the surface to achieve the special features of



fleece. In addition, the impact of using detergent in the washing was examined and resulted in even more shedding of fibres. Astrom suggests that this could be due to the surfactants that are used in detergents. (Astrom, 2016)

Microplastics are so tiny that they are practically impossible to collect from nature. A solution needs to be created to decrease the amount of fibre shedding to nature and to clean the polluted areas eco-friendly. This thesis focuses on biological solutions involving microbes and micro-organisms that have the ability to degrade synthetic textile fibres.

## 2 SYNTHETIC TEXTILE FIBRES

Synthetic fibres, also called man-made fibres, are chemically synthesized fibres that are widely used in manufacturing different kinds of goods. The fibres are engineered to have the wanted special qualities that naturally occurring fibres do not have. These abilities can be fire-resistance, extreme elasticity, durability or so on. Synthetic fibres can be blended with natural fibres like wool or cotton to achieve the required features: texture, appearance, strength, and so on. (CIRFC, About man-made fibres)

Synthetic fibres consist of small hydrocarbon units that are synthesized together into bigger polymers. Many of these fibres are derived from fossil fuels like oil and coal. This makes them non-biodegradable. Nowadays many of them are made from recycled and even renewable raw materials, which is a step towards more ecological direction. (Hallett et. al., 2014)

There are also biodegradable man-made fibres, for example viscose, modal, lyocell and acetate. These fibres are derived from cellulose which makes them natural polymers. (CIRFC, Product and Production Process)

Hallett's book *Fabric for Fashion* (2014) defines these kinds of fibres as artificial fibres, which means the material requires some sort of chemical treatment to become a fibre. These artificial fibres can be either natural or regenerated. Natural fibres have undergone less engineering and chemical modification than regenerated ones. Biopolymer fibres are also classified as artificial. They are made from some other bio-based raw-materials for example corn, soybeans or milk. (Hallett et. al., 2014)

In this research we concentrate on synthetic fibres which are non-biodegradable. Most common synthetic textile fibres are presented in the table 1.

**Table 1.** Characteristics of different kinds of synthetic textile fibres.

Synthetic fibre	Chemical compounds	Usage and properties	References
Polyester	Polyethylene terephthalate (PET)	The most common synthetic fibre. Fabrics, packaging materials. A large variety of blends and qualities. Wrinkle-free texture.	CIRFC, Polyester Hallett et. al., 2014 Lancashire, 2011
Polyamide (nylon)	Composition differs: Linear macromolecules with amide linkages and aliphatic or cycloaliphatic units joined to them.	Flexible, durable, silky. The first prospering synthetic fibre, known from nylon stockings.	CIRFC, Polyamide Hallett et. al., 2014 Lancashire, 2011
Acrylic	Composition differs: Linear macromolecules with repeating acrylonitrile units. Copolymers of polyacrylonitrile (PAN).	Durable, soft, water-repellent. Common substitute and extension for wool.	CIRFC, Acrylic and modacrylic Hallett et. al., 2014 Lancashire, 2011
Aramid fibres (Nomex, Kevlar)	Aromatic polyamides, poly-m-phenylene isophthalamide patented as Nomex and poly-paraphenylene-terephthalamide patented name Kevlar. Also some variations by other manufacturers.	Strong, fire-resistant, cut-resistant, soft, lightweight, flexible. Used in firefighting wear, bulletproof vests and other protective clothing.	CIRFC, Aramid Hallett et. al., 2014 Lancashire, 2011
Olefin fibres (polyolefin)	Polypropylene and polyethylene. Also variations of these, for example high-density-polyethylene (HDPE).	Lightweight, thick, water-repellent, chemical-resistant. Sportswear, plastics, carpets, geotextiles.	CIRFC, Polyolefin Hallett et. al., 2014 Lancashire, 2011
Polyurethane (spandex, elastane)	Polyurethane and long polymer chains.	Elastic, returns to its form after stretching. Sportswear and other clothing where fit is important. Fibres are mixed with others (eg. cotton or wool) to make fabrics.	Hallett et. al., 2014 CIRFC, Elastane

## **2.1 Polyester fibres**

Polyethylene terephthalate (PET) can be produced from dimethyl terephthalate or terephthalic acid. Transesterification of dimethyl terephthalate can be performed either as batch or as continuous process. In the batch process dimethyl terephthalate is first esterified with ethylene glycol and then polycondensation is carried out. Continuous process has similar unit processes that are executed with several back-to-back reactors.

With terephthalic acid the process can be made a little straighter forward via direct esterification with ethylene glycol. Smaller oligomers are formed first and then with polycondensation bigger polymers. Catalysts are needed in these processes.

In both production pathways the process ends with an extruder that moulds the polymers into continuous filaments. (Sattler et. al., 2011)

## **2.2 Polyamide fibres**

From the group of polyamide fibres nylons are known the best. There are many types of nylon, but the two most common are nylon 6,6 and nylon 6. The production of these two differ a little. Nylon 6,6 originates from the reaction between adipic acid and hexamethylenediamine. These compounds form a nylon salt that is then polymerized to form nylon 6,6. Nylon 6 is also called polycaprolactam. It is formed when caprolactam is polymerized. The number code defining the nylon fibre marks the amount of carbon atoms in a repeating unit: nylon 6 has 6 carbon atoms and nylon 6,6 has two times six thus 12 carbon atoms. (Hallett et. al., 2014) Polymers are pushed through a spinneret at 300 °C to produce fibres, and then cooled down and gathered. (CIRFC, Polyamide)

## **2.3 Acrylic fibres**

The production of acrylic fibres starts with propylene and ammonia, which react with oxygen with the help of catalysts to form acrylonitrile. Acrylonitrile is then polymerized and becomes polyacrylonitrile. (CIRFC, Acrylic and Modacrylic) Polyacrylonitrile is spun into fibres using either wet or dry spinning. In wet spinning the fibres are in a chemical solution and go through a spinneret becoming solid. In dry spinning after the

spinneret fibres go through hot air that evaporates the liquids and thus solidifies the fibres. (Hallett et. al., 2014)

## **2.4 Aramid fibres**

Aramid fibres are aromatic polyamides. There are many variations of these fibres by different producers, but the most significant ones are known as Nomex and Kevlar. Nomex is a meta-aramid polymer. It is produced by a reaction of m-phenylenediamine and isophthaloyl chloride. Dry spinning rather than wet spinning is more commonly used in the making of meta-aramids. (CIRFC, Aramid) Kevlar is a para-aramid polyester and it is produced by a reaction of para-phenylenediamine and terephthaloylchloride. It is then wet spun using sulphuric acid as the chemical solution. (Hallett et. al., 2014)

## **2.5 Olefin fibres**

Olefin fibres, also known as polyolefins, are saturated polymers. Two most common polyolefins are polypropylene and polyethylene. They are linear hydrocarbon polymers with the basic structural formula of  $C_nH_{2n}$ . Polyethylene has hydrogens on every carbon and polypropylene has a methyl group on every other carbon. Polyethylene has a more simple structure than polypropylene and its density can be altered from 0.91 to 0.97  $g/cm^3$ . These different kinds of polyethylenes are named according to their densities: high- or low-density-polyethylene (HDPE and LDPE), linear low-density-polyethylene (LLDPE) and so on. HDPE is more linear than LDPE, which has random branching. (Arutchelvi et. al., 2008)

Polyethylene can be produced in two ways: High- and low-pressure polymerization processes. Ethylene is the most widely used raw-material for these processes. Raw-materials need to be extremely pure to achieve quality polyethylene.

In high-pressure processes the reactors are usually autoclave or tubular reactors. Low-pressure processes can be carried out in a suspension, solution or gas-phase. The required catalysts and obtained products also differ between the processes. All these alternative processes have their advantages and disadvantages and the used process depends on the producer. (Jeremic, 2014)

Propene is the main raw-material for the production of polypropylene. Alternative processes can be liquid-phase, gas-phase or hybrid processes. Liquid-phase method is the oldest commercially used process for polypropylene production but the gas-phase technique is an economical and energy-efficient option. (Gahleitner et. al., 2014)

The fibres themselves are made by melts spinning or by cutting strips from a film and winding them. In melt spinning process the molten polymer material is pushed through an extruder and the outcome is long filaments of polymer fibres. (CIRFC, Polyolefin)

## **2.6 Polyurethane fibres**

Polyurethanes can be polyester or polyether polyurethanes. They are made from three basic compounds: polyglycol, di-isocyanate and prepolymers as chain extenders. In the production of polyurethane, these three components form crystalline and non-crystalline segments. The crystalline part is made by di-isocyanate and the chain extenders. The non-crystalline part is composed of either polyester or polyether. The urethane bond is in between these two segments. (Loredo-Treviño et. al., 2012)

The most common polyurethane fibre, known as Spandex, is produced with a two-step process. First polyol reacts with isocyanate to form a pre-polymer. The pre-polymer is then dissolved into a solvent, usually dimethylformamide. The mixture is then reacted with a diamine, for example ethylene diamine. Solution is then put through a dry-spinning process to form filaments. (Sonnenschein, 2014)

Polyurethane fibres are usually mixed with other fibres to achieve the wanted texture and elasticity for the textile. The secret behind the stretchability of spandex is in its structural form: long polymer chains randomly in a cluster. When the fabric is stretched the polymer chains stretches out three times the size they were at rest. When the stretching force is removed, the polymer chains return in coils and the fabric remains back in its form. (Hallett et. al., 2014)

### 3 BIODEGRADATION

As we have established, synthetic fibres are part of the growing waste and pollution problem. The fibres do not degrade in the environment on their own. They accumulate and cause all sorts of problems in the nature and marine-environment. Flora and fauna are suffering due to this and even humans are affected.

In this chapter we discuss the methods of bioremediation that have been investigated to solve the problem of synthetic textile fibre waste. The main focus is on the microbial, fungal and enzymatic methods.

Biodegradation aims to transform pollutants (in this case synthetic fibres) into metabolites that organisms can include to their metabolic pathways and systems. This means that organisms use the fibres as energy and nutrient sources and transform them into harmless natural compounds like water, carbondioxide and methane. (Loredo-Treviño et. al., 2011)

Physical and chemical properties of the synthetic polymer affect the biodegradability of it. Many properties like surface texture, molecular weight, density, crystallinity, melting point and other properties are important factors in bioremediation. Polymers with more complex structure, side chains and greater molecular weight are less likely to be degraded. Enzymes attack the amorphous parts of the polymer chain more likely than the crystalline parts, which means that morphology is also an important factor. The chemical structure is associated with the melting temperatures, which refers that the higher melting point means less degrading. (Tokiwa et. al., 2009)

Synthetic fibres do not have a constant microflora like natural fibres do. This also means that only microbes with the highest adaptability are able to biodegrade synthetic materials. These organisms need to adapt to using the synthetic fibres as their energy source. Micro-organisms try to adsorb themselves onto the fibre structure, attach to the surface and adapt to the conditions to start growing. Synthetic materials are quite promising energy source due to their abundant carbon content. Chemical and physical structure and electrical charge of the surface affect the adsorption to the surface of the fibre. Positively charged fibres adsorb more than negatively charged ones. For the organism to spread further into the fibre there needs to be space inside the structure. Inhomogeneity of the polymer also furthers the degradation process. (Zaikov et. al., 2012)

Complete biodegradation of synthetic materials can require various micro-organisms: One that breaks the polymer apart and others that can break these smaller polymers and use the excreted monomers in their metabolism. Plastics are foreign material in the environments which means nature has not yet perfected a method of utilizing this new resource. It may take year for nature to achieve a comprehensive method of degrading and exploiting synthetic materials. That is why research on the matter is very important and with good results solutions to the pollution problem can be found. (Loredo-Treviño et. al., 2011)

Many of the biodegradation experiments are done on synthetic fibres that have already gone under some sort of pretreatment to enhance the degradation process. Also various blends of synthetic fibres with pro-oxidants, starch or other components that raise the biodegradation levels are examined. However, this research is mainly based on the literature published about the biodegradation of purely synthetic fibres and their compounds.

### **3.1 Polyester biodegradation**

The most common polyester fibre is polyethylene terephthalate (PET). In a research by Yoshida et. al. (2016) biodegradation of PET was investigated. Different kinds of PET containing debris samples were gathered from waste accumulations (soils, wastewaters, sediments, etc.). These samples were examined for micro-organisms able to use PET (with low crystallinity) as their main carbon source. One sample rose above others: it caused morphological changes in the PET film and degraded the surface of the film. 75% of the carbon from the degraded PET film was catabolized into CO<sub>2</sub>. After six weeks in the culture fluid, the PET film was almost completely degraded. This sample was taken into further investigation and turned out it consisted of a mixture of yeast-like cells, protozoa and bacteria. A bacterium named *Ideonella sakaiensis* was isolated from the culture. This bacteria was the one degrading PET in the sample. The enzyme recognized to have a major effect on degrading PET was designated as PET hydrolase (PETase).



PETase hydrolyzes PET into smaller monomers and was even able to have an effect on commercial highly crystalline and dense PET. (Yoshida, 2016)

In other research PET was reported to be hydrolysed by cutinases from filamentous fungi *Fusarium oxysporum*, *Fusarium solani* and *Penicillium citrinum*. The hydrolysing effect was mostly seen to be quite superficial and did not have an effect on the bulk structures. These enzymes released terephthalic acid while hydrolysing PET fibres. (Guebitz et. al., 2008)

Modified polyethylene terephthalates are mentioned in "Biological degradation of plastics: a comprehensive review." by Shah et. al. (2008). These are PET based applications with co-monomers that reinforce the degradability of the fibre. These monomers can be aliphatic, amide or ether monomers. The idea is that these linkages are weaker and more exposed to biological attacks which increases the degradability of PET. The most common modified PETs are polybutylene adipate/terephthalate, polytetramethylene adipate/terephthalate and commercially known Biomax. (Shah et. al., 2008)

### **3.2 Polyamide biodegradation**

Polyamide fibres are usually mixed with natural fibres, like wool and cotton, which possess their own microflora. In this kind of mixed textile, nylon 6 is exposed to the micro-organisms from natural fibres. Different kinds of mixtures cause different kinds of changes in the nylon 6 fibres: fading, decomposition and streakiness. (Zaikov et. al., 2012)

Micro-organisms known to degrade polyamides were studied by extracting them from damaged fibres. The fibres were degraded in active sewage sludge or soil. These bacteria were already adapted to using polyamides as their nutrient and energy sources, thus they differ from their original strains. The extracted micro-organisms contained strains of *Bacillus subtilis* and *Bacillus mesentericus*. (Zaikov et. al., 2012)

There are also enzymes that can hydrolyse polyamides: different kinds of amidases cutinases and proteases. Amidase from *Nocardia sp.*, cutinase from *Fusarium solani* and Protease from *Beauveria sp.* have been reported to have an effect on polyamide fibres. This hydrolysis can be amplified with solvents. (Guebitz et. al., 2008)

In an older research "Nylon biodegradation by lignin-degrading fungi." by Deguchi et. al. (1997) it was established that micro-organisms with lignolytic activity are also able to degrade nylon 66. White rot fungus IZU-154, *Phanerochaete chrysosporium* and *Trametes versicolor* were used in the experiment. Nylon 66 membranes were incubated with IZU-154 fungi at 30 °C in agar media. The media contained of glucose, ammonium tartrate,  $\text{KH}_2\text{PO}_4$ ,  $\text{NaH}_2\text{PO}_4$ ,  $\text{MgSO}_4$ , agar, thiamine-HCl,  $\text{CaCl}_2$ ,  $\text{FeSO}_4$ ,  $\text{ZnSO}_4$ , and  $\text{CuSO}_4$  dissolved into water. After 20 days of incubation the nylon was washed, dried and dissolved. Molecular weight of the membranes was examined and the results showed that nylon degradation had happened in the media. This suggests that nylon could be degraded the same way as lignin.

The effect of *Phanerochaete chrysosporium* and *Trametes versicolor* was examined in almost same media (without ammonium tartrate). This experiment also concluded that both fungi were able to degrade nylon 66. In addition to these *Flavobacterium sp.* strain K172 has been reported to degrade nylon oligomers. This organism has been isolated from nylon waste.

Examining the nylon membranes after the treatment showed that the white rot fungi had degraded nylon into four groups:  $\text{NHCHO}$ ,  $\text{CONH}_2$ ,  $\text{CHO}$  and  $\text{CH}_3$ . This indicates that oxidation was the main method of degradation, not hydrolysis.

Additionally the impact of added manganese was reported to have accelerated the degradation process. The best results were obtained with 1 mM of  $\text{MnSO}_4$ . (Deguchi et. al., 1997)

"Biodegradability of plastics." by Tokiwa et. al. (2009) has gathered information on plastic biodegradation. It reports that nylon 4 strains have been degraded quite well in activated sludge and soil. From these experiments strains of *Pseudomonas sp.* were identified to be the ones degrading nylon. (Tokiwa et. al., 2009)

### 3.3 Acrylic biodegradation

Only a few results on acrylic biodegradation were found. Most of the results are not specifically about the fibre biodegradation, but the acrylonitrile and acrylic polymer biodegradation. These results still indicate the biodegradability of acrylic fibres due to the biodegradability of the compounds that are used in the making of the fibres.

Two researchers had used actual acrylic fibres in their experiments. Battistel et. al (2001) found stains of *Brevibacterium imperiale* and *Corynebacterium nitrilophilus* that segregated nitrile hydratases. The micro-organisms were commercial and the polyacrylonitrile fibres contained 10% of acetate groups. The CN groups of the polymers were hydrolysed into amides in consequence of the enzymatic attack. (Battistel et. al., 2001)

The other research by Tauber et. al. (2000) examined *R. rhodochrous* impact on polyacrylonitrile fibres, granules and fabric. The organism was able to hydrolyze nitrile groups on the surface of the fibres and granules but seemed not to have an effect on the fabric. The nitrile groups were hydrolyzed into amides and released ammonia. The amount of the released ammonia was smaller with high-molecular-weight polyacrylonitrile which indicates less hydrolyzing. The nitrile groups of granules were at least partially hydrolyzed to acids whereas the fibre nitrile groups only to amides. Seems that *R. rhodochrous* is quite sensitive of the geometry of the used substrate. The nitrile groups of acrylic fibre surface were hydrolyzed up to 16%, which does not seem very effective for bioremediation. (Tauber et. al., 2000)

Polyacrylonitrile polymers were metabolised as a sole carbon source by a strain of *Micrococcus luteus*. Gudrun Fischer-Colbrie et al. (2007) discovered that *M. luteus* produced nitrile hydrolysing enzymes that transformed polyacrylonitriles nitrile groups into acrylic acid. The micro-organism was obtained from soil and sewage samples in Austria and incubated in a media with polyacrylonitrile. The experiment was carried out with different kinds on polyacrylonitrile polymers and the best results were when the crystallinity of the polymer was at its lowest. (Fischer-Colbrie et al., 2007)

Sutherland et. al. (1997) investigated acrylic biodegradation by *Phanerochaete chrysosporium*. Two crosslinked polymers with high molecular weight were incubated with fungus. One of the polymers was polyacrylate and the other copolymer of acrylamide and acrylic acid. The incubation happened in a standard liquid media in temperature of 37 °C. The experiment was done with limited and sufficient nutrients. Under the limited nutrient conditions the fungi seemed to degrade polymers more sufficiently. When the degradation rates decreased additional glucose was added to the media, which increased the degradation rates again. The results showed that the polymers were degraded into carbondioxide by the fungus. (Sutherland et. al., 1997)

Nawaz et. al. (1991) found a gram negative bacterium that was able to use acrylonitrile as the sole nitrogen source. The bacterium was obtained from industrial waste and it was identified as *Klebsiella pneumoniae*. The maximum growth and therefore the maximal rate of acrylonitrile biodegradation was obtained in a phosphate buffered medium with temperature of 28 °C and pH of 7.5. The growth rate grew even higher when glucose was added as the carbon source for the bacterium. The results of a gas chromatographic analysis showed that after 24 h of incubation all acrylonitrile had disintegrated into acrylamide and then after 96 h into acrylic acid. (Nawaz et. al., 1991)

Yamada et. al. reports in "Microbial utilization of acrylonitrile." that an isolated strain of *Arthrobacter sp.* could use acrylonitrile as the exclusive nitrogen and carbon source. The bacterium oxidises acrylonitrile to acrylamide and then acrylic acid. (Yamada et. al., 1979)

These researches and some others with the same topic seem to be concentrating on the rather on the production of the metabolite product chemicals, than on the biodegradation process. Nawaz et. al. mentions that this biological method is an alternative way to produce amides from nitriles (Nawaz et. al., 1991). Thus the effective biodegradation of acrylic fibres needs to be researched further focusing on the aspect of bioremediation.

### 3.4 Aramid biodegradation

Aramids are aromatic polyamides. This would suggest that they might be susceptible to biodegradation similarly as polyamides, but the research shows otherwise. Poly-m-phenylene isophthalamide (Nomex) is said to be non-biodegradable and no differing research was found (DuPont, 1999).

One old article on biodegradation of Poly-paraphenylene-terephthalamide (Kevlar) by by Takashi Watanabe (1991) was found. The original research was written in Japanese but in this summary Kevlar biodegradation by *Aspergillus flavus* was reported. After 30 days of incubation at temperature of 30 °C swelling and splitting was observed on the fibres. (Watanabe, 1991)

### 3.5 Olefin biodegradation

Most of the research on polyolefin biodegradation is conducted on polymer blends, with starch or some sort of biodegradable additives. More of the experiments also seems to be on polyethylene rather than polypropylene.

Cacciari et. al. (1993) gathered soil samples from different sites with plastic wastes. The obtained micro-organisms were first grown on polyethylene-starch blend and then inoculated with pure isotactic polypropylene. Only one of the four communities was able to grow on polypropylene and three organisms were recognised: *Pseudomonas chlororaphis*, *P. stutzeri* and *Vibrio sp.* Biodegradation of polypropylene was detected, but quite slowly and in a long period of time. (Cacciari et. al., 1993)

Alariqi et al. (2006) investigated the effect of  $\gamma$ -sterilisation on biodegradability of isotactic-polypropylene and high-density-polyethylene (HDPE). The degradation was done under fungal environment with *Aspergillus niger* and composting. The biodegradation experiment lasted six months with monthly observation. The  $\gamma$ -sterilisation seemed to accelerate the degradation process in both polymers but polypropylene was in both cases more susceptible to biodegradation than HDPE: after five months the polymer film was so damaged it could not be obtained for observation. (Alariqi et al., 2006)

Volke-Sepúlveda et al. (2002) examines thermally oxidized low-density-polyethylene (LDPE) biodegradation with and without additional ethanol. Incubation with the additional ethanol enhanced the biodegradation and morphological changes on the LDPE with both *Penicillium pinophilum* and *Aspergillus niger*. *P. pinophilum* seemed to cause a higher rate of biodegradation than *A. niger*. This is probably due to the difference in the enzymatic activities of the organisms. (Volke-Sepúlveda et al., 2002)

Hadad et. al. (2005) found a polyethylene degrading micro-organism in soil. Branched low-density-polyethylene (LDPE) was incubated with isolated strain of *Brevibacillus borstelensis* for 30 days in temperature of 50 °C. *B. borstelensis* was capable of utilizing LDPE as a sole carbon source degrading it up to 30%. (Hadad et. al., 2005)

In research by Rajendran Sangeetha Devi et. al. (2015) samples of polyethylene waste were gathered from marine environments in India. Isolated fungi were incubated with high-density-polyethylene (HDPE) for one month in temperature of 30 °C. The most successful fungal strains were identified as *Aspergillus tubingensis* and *A. flavus*. These organisms were able to grow on the HDPE film and degrade it. Degradation was observed as surficial chanced (crackling, roughness) and weight loss.

### **3.6 Polyurethane biodegradation**

Polyester polyurethanes (PUR) are one of the few synthetic fibres relatively susceptible to biological degradation. According to research that is due to the ester and urethane linkages in the polymer that make it exposed to enzyme hydrolysis.

In research "Fungal communities associated with degradation of polyester polyurethane in soil." by Lee Cosgrove et. al. (2007) PUR degradation in soil was investigated. PUR was buried in soils (in United Kingdom) with different kinds of acidity for five months and the micro-organisms colonizing on the PUR were then identified. One of the used soils was neutral (pH 6.7) and the other was acidic (pH 5.5). The soils also had different levels of carbon, magnesium, potassium and phosphorus levels. After the five month burial the fungal colonies were investigated on the surface of PUR and in the soil surrounding it. The acidic and neutral soils both had quite the same amount of organisms

in them, but in the acidic soil more of them were growing on the PUR surface than in the soil. In the neutral soil the amount of fungus in the soil and on the PUR was more similar. Denaturing gradient gel electrophoresis was used to analyze the cultures. The both soils had a variety of around 35-40 organisms and they differed quite much between the acidic and neutral soils. Lower variety of organisms was found on the surface of PUR. Internal transcribed spacer was used to identify the organisms on the PUR surface: five from the acidic soil burial and four from the neutral soil burial. These fungi were tested for their ability to clear Impranil, which indicates their ability to also degrade PUR. Only five of them showed to clear Impranil: from acidic soil *Geomyces pannorum* and *Penicillium inflatum* and from neutral soil *Alternaria ps.*, *Neonectria ramulariae* and *Penicillium viridicatum*.

The degree of degradation was examined with a tensile strength test. After five months of burial the PUR in both soils had lost around 15-fold of their tensile strength which is up to 95 %.

In conclusion this research indicates that before mentioned fungi organisms are able to degrade PUR quite effectively in situ. Physicochemical properties seem to have an effect on the colonizing organisms and thus the biodegradation process. (Cosgrove et. al., 2007)

In the research by Russell et. al. (2011) endophytic fungi found inside woody plants in Amazonian rainforest were examined for their ability to degrade polyester polyurethane (PUR). Out of 59 isolated microbial organisms 18 were detected to degrade polyester PUR. The used media was a solid PUR media (PUR-A) with the composition as follows: 19 mM  $\text{NaH}_2\text{PO}_4$ , 33.5 mM  $\text{K}_2\text{HPO}_4$ , 7.6 mM  $(\text{NH}_4)_2\text{SO}_4$ , 250  $\mu\text{M}$   $\text{MgSO}_4$ , 147  $\mu\text{M}$   $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , 14  $\mu\text{M}$   $\text{ZnCl}_2 \cdot 4\text{H}_2\text{O}$ , 12  $\mu\text{M}$   $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , 12  $\mu\text{M}$   $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ , 10  $\mu\text{M}$   $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ , 11  $\mu\text{M}$   $\text{CuCl}_2$ , 12  $\mu\text{M}$   $\text{MnCl}_2$ , 12  $\mu\text{M}$   $\text{H}_3\text{BO}_3$ , 19  $\mu\text{M}$  thiamine, 0.05% casamino acids, 2.5 mM Na-Citrate and 1.8 mM HCl. To one litre of mixture with this composition was added 15 mL of agar and 10 mL of Impranil DLF, an anionic aliphatic dispersion of PUR with 4% N-methyl pyrrolidone, as the substrate. Polyester PUR was incubated in this media with the different micro-organisms for two weeks. The degradation of PUR was observed by the clearing of the medium: opaque medium became translucent if degradation occurred. The 18 organisms identified to degrade polyester PUR were strains of genera *Alternaria*, *Bionectria*, *Plectosphaerella*, *Lasiodiplodia*, *Pestalotiopsis*, *Phaeosphaeria*, *Nectria* and *Pleosporales* fungus.

Degradation rates were also observed with optical absorbance in a liquid media. Top degradation rates were found with the strains of genera *Lasiodiplodia* and four out of six of the *Pestalotiopsis* genus. These five most active organisms were after that tested for their ability to use polyester PUR as a sole carbon source in a liquid culture. The medium for these experiments was prepared with the same composition as PUR-A medium but without agar, casamino acids, sodium citrate and thiamine. The used substrate was Impranil DLN (no N-methyl pyrrolidone). For two weeks optical clearance of the opaque medium was measured every two days and the growth of the fungus was observed by mass. All five tested organisms were able to degrade polyester PUR as a sole carbon source.

Comparing these two conditions no significant difference was detected when using Impranil DLN and DLF. This suggests that for these organisms fungal growth is possible on polyester polyurethane. (Russell et. al., 2011)

Also the anaerobic degradation of polyester PUR became of interest and was examined. The tests with Impranil DLN medium were repeated under anaerobic conditions. The rates of PUR degradation under aerobic and anaerobic conditions were compared. One of the *Pestalotiopsis* genus fungi showed similar rates of degrading PUR, others appeared with smaller rates of degradation but all tested fungus were able to grow under anaerobic conditions. (Russell et. al., 2011)

The clearance in the culture medium that indicates degradation appeared outside of the fungal growth area. This points to appearance of extracellular enzymatic activity. Using a cell-free filtrate and heat treatment with *Pestalotiopsis microspora* strain created results that suggested that an extracellular serine hydrolase like protein is responsible for the degradation of polyester PUR. (Russell et. al., 2011)

Also *Aspergillus niger* has been reported to have a degrading effect on polyester PUR. *A. niger* was used as a positive control organism in the research "Fungal communities associated with degradation of polyester polyurethane in soil." by Lee Cosgrove et. al. (2007). Its degrading activity is relatively slow and the identified organisms seemed to defeat *A. niger's* abilities. (Russell et. al., 2011)



A recent research by Khan et. al. (2017) gained promising results in polyester PUR biodegradation. Degradation by *Aspergillus tubingensis* was examined in three kinds of environments: burial in soil, in mineral salt medium (MSM) and in sabouraud agar (SDA). The best degradation results were observed on SDA plates, presumably due to the richly nutritious environment that supported the secretion of enzymes. In a liquid medium containing MSM with 2% glucose the degradation levels of polyester PUR were also very high after two months of incubation. The PUR film had decomposed into smaller pieces and almost 90% degradation was observed.

In addition to the genera of the fungi, also the conditions affect the degradation process. The secretion of enzymes from *A. tubingensis* was observed under varying conditions: temperature, pH and carbon source. The optimal temperature for both lipase and esterase was at 37 °C. pH 9 increased esterase activity at first, but it decreased quite fast. Neutral pH of 7 kept the esterase activity levels high. For lipase the maximum activity was found at pH 5: activity was good for first two weeks and after that decreased slowly. Maltose, sucrose, glucose, galactose and glycerol were tested as carbon sources for the enzymes. Lipase liked glycerol the best and esterase preferred galactose, sucrose and glucose all quite equally. Overall the highest enzyme activity levels were found with sucrose as carbon source.

With all this new knowledge and promising results Khan et. al. conclude that *A. tubingensis* could be used in a large-scale polyester PUR degradation process. (Khan et. al., 2017)

As seen above, most of the research about PUR biodegradation is focused on the fungi able to degrade PUR. Quite a few micro-organisms have been reported to have an enzymatic degrading activity on PUR: genera *Bacillus*, *Pseudomonas*, *Comamonas*, *Alicyclophilus* and *Curvularia*. The amount of research done on the subject dating back to the beginning of 20th century seems to be superior compared to other synthetic fibres. Also most of the results of degrading polyurethanes have been about polyester polyurethane, polyether polyurethane is much more resistant to biodegradation. (Loredo-Treviño et. al., 2012)

A research by Y. Matsumiya et. al. (2010) investigated polyether polyurethane biodegradation. A modified Luria-Bertani medium was used to isolate the supposed

polyether PUR degrading micro-organisms. With a differential interference contrast microscope strains of genus *Alternaria* were identified. With further investigation the strain responsible for the degradation was identified as *Alternaria sp.* After ten weeks on agar plate this isolated organism was able to degrade about 27% of polyether PUR. The degradation was observed using a scanning electron microscope and the polyether PUR seemed to have been disrupted and melted by the effect of the fungus. Results indicated that the degradation had happened on the urethane and urea bonds.

Matsumiya et. al. also mentions that *Staphylococcus epidermidis* has been reported to have had a degrading effect on polyether polyurethane. (Matsumiya et. al., 2010)

One of the first researches of PUR degradation Darby et. al. (1968) found a couple of fungi capable of degrading both polyester and polyether PUR: *Aspergillus niger*, *A. flavus*, *A. versicolor*, *Aureobasidium pullulans*, *Chaetomium globosum*, *Penicillium funiculosum* and *Trichoderma sp.* Though it ought to be mentioned, that these all were tested as a mixture which may affect the results. (Darby et. al, 1968)

In conclusion, polyurethane fibres can be biologically degraded. Many micro-organisms have been reported to degrade PUR, the most significant ones mentioned above. This knowledge should be used to come up with a solution to degrade PUR waste in a larger scale to reduce the fibre pollution problem.

## **4 SUMMARY**

This bachelor thesis focuses on the biological ways of degrading synthetic textile fibres. The research was thorough and aimed at getting a comprehensive view into the most used synthetic textile fibres and how their biodegradation has been examined till this day. The main focus was on biodegradation of the purely synthetic fibres and their components rather than the blends, pre-treated and otherwise assisted biodegradation.

Synthetic polymer materials are a big pollution problem worldwide. The extent and severity of the problem was explored to get a view on why bioremediation is an important topic to study. Firstly the composition, production and usage of the synthetic textile fibres was examined. Then the goal was to find out as much as possible on how biodegradation of these fibres has been studied and what were the results. Focus was on microbial, fungal and enzymatic biodegradation. The most results were found on polyurethane fibres and the least on aromatic polyamide fibres.

The results indicated that there are some potential micro-organisms that could be utilized in larger scale bioremediation. The subject still requires more examining to come up with a functional solution for the synthetic polymer pollution problem.

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