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Re:Mix – Separation and recycling of textile waste fiber blends

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Introductory remark

This project ‘Re:Mix - Separation and recycling of textile waste fibre blends’, referred to as Re:Mix I, was a pre-study and thereby included a start-up and consortium building phase. The approach to formulate and manage this project included 1) gathering a well-represented value circle (from academia & research institutes; to textile manufacturers; to textile producers/retailers; and to textile waste management); 2) interaction in the form of workshops; 3) screening academic work to review the potential behind the idea.

This approach to formulate and manage the Re:Mix I project has been successful and constructive, which also resulted in a second phase: ‘Re:Mix II – material recycling of textile blends with nylon and elastane’, starting up Fall 2017 with funding from RE:Source and inkind-support from partners.

The project was funded by RE:Source (to cover nylon-separation) and by the Strategic Reserve Fund in Mistra Future Fashion (to cover elastane separation). Both funding bodies are acknowledged and contributed equally. Therefore this report covers both recycling separation tracks of nylon and elastane.

All partners are acknowledged for their engagement and contribution which has formed the continuation of the Re:Mix, namely: BOKU, Boob, Faering Ltd, Houdini Sportswear, I:Co, Invista, KTH/SciLifeLab, Radici Group, RISE, Swedish Stockings, Swerea IVF, Texaid.

The project was initiated by Dr. Hanna de la Motte (RISE) and Dr. Sandra Roos (Swerea IVF), who are both acknowledged for their initiative and for the idea behind this.

Project leader
PhD Åsa Östlund
29th of September 2017, Stockholm

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Sammanfattning

Ambitionen med projektet ”Re:Mix - Separation och återvinning av fiberblandningar i textilt avfall” var att svara på frågan om vilka tekniska metoder som krävs för att separera nylon (polyamid) och elastan (polyuretan) från andra fibertyper för att möjliggöra återvinning av dessa textilblandningar.

Målet inom Re:Mix I har varit att utveckla positiva strategier för separation av olika fibrer från en fiberblandning genom kemisk återvinning och bidra till en cirkulär ekonomi. Detta uppnåddes genom en sund och ödmjuk dialog mellan projektpartners som har säkerställt att framtida forskning och utveckling, inom det kommande Re:Mix II, kommer att vara relevant, tillämpbart och unikt.

Projektpartnerna identifierade två separationsmetoder, som även skulle kunna fungera i kombination, 1) en smältning av syntetfibrer, så kallad termomekanisk separation; och 2) design av nya specifika enzymer som kommer att fungera som biokatalysatorer för nedbrytning av en specifik polymer som ytterligare underlättar resyntetisering av polymererna.

Inom den termomekaniska separationen var målet att ta reda på förutsättningarna för återvinning av polyamid- och elastanfiberblandningar utan att först separera materialet. Metoden ger nylon- eller elastanpellets som kan användas som råmaterial i processer som spinning och formpressning.

Inom det enzymatiska spåret var fokus inom förundersökningen att möjliggöra separation av nylon 6,6. Enzymet (polyamidaset) designades med hjälp av datasimuleringar (MD) och man såg att det utformade enzymet skulle kunna interagera på tre specifika ställen längs polymeren. Därmed har enzymet potential att fungera som en biokatalysator för nedbrytningen av nylon 6,6, från andra fibrer i en mild och miljövänlig kemisk återvinningsteknik. Forskarna har tidigare resultat på det första kända klyvningsenzym för polyuretan (t ex elastan) [1], som kommer vidareutvecklas för utformning av enzymer som också är lämpade för återvinning av elastan i textilier.

Tillvägagångssättet att formulera och hantera detta projekt inkluderade 1) samla en välrepresenterad värdecirkel (från akademi och forskningsinstitut, till textilproducenter, till återförsäljare); 2) interaktion i form av workshops; 3) screening av akademiskt arbete för att granska potentialen bakom idéerna (som beskrivs ovan).

Det finns många drivkrafter för att utveckla materialåtervinning, till exempel att nå en cirkulär ekonomi, minskad deponi, ökad efterfrågan på jungfruligt material och kommande lagstiftningar.

Fiberblandningar med nylon (polyamid) och elastan (även känd som spandex eller lycra) är vanliga i textilier för underkläder, badkläder, sportkläder och strumpbyxor. Nylon ger styrka åt dessa produkter, medan elastan tillsätts för sin elastiska natur för att generera stretch och komfort. Dessa produkter kastas vanligen i hushållsavfallet efter användning och hamnar på deponi eller för förbränning.

Erfarenheten från Re:Mix I partners är att de blandningar som innehåller elastan (även vanligt i stretch jeans och leggings) och nylon ökar på marknaden. Branschpartnerna (Boob, Houdini, Swedish Stockings) samlar redan egna produkter efter användning med syftet återanvändning/återvinning, men idag saknas en återvinningsteknik för dessa material. De söker aktivt efter återvinningsalternativ för textilier som innehåller 5-13% elastan blandat med 20-95% nylon förutom polyester, bomull eller lyocell. Dessutom kan I:Co och Texaid, som tillsammans har en erfarenhet av flödena av använt europeiskt textilavfall, redan känna av en ökning av elastan i textilavfall. Behovet av återvinningsteknologi för att skilja både elastan och nylon från andra textilkomponenter är kritisk.

Nästa steg som behövs inom mekanisk smältseparationsteknik kommer att vara en optimering av processparametrar och hårdvara. För den enzymatiska separeringstekniken är syftet att skala upp till laboratorie- och pilotskala, eftersom en av de största utmaningarna med denna process är uppskalning. Omfattningen av Re:Mix II, som påbörjas hösten 2017, kommer sannolikt även att innebära kombinationsförsök av ovanstående tekniker, biokatalys och termomekanisk separation.



Meltspun fibres from post-consumer waste
Photo: Swerea IVF

Summary

This is the final report from the project ‘Re:Mix - Separation and recycling of textile waste fibre blends’, referred to as Re:Mix I.

The ambition with the project was to answer the question: Which technical methods are required to separate nylon (polyamide) and elastane (polyurethane) from other fibre types, in order to enable recycling of these textile blends.

The goal within Re:Mix I has been to develop positive strategies for separation of different fibres from a fibre blend prior to chemical recycling, according to a circular economy. This was achieved by a sound and humble dialogue ensuring that the future research and development will be relevant, applicable and unique.

The project partners identified two separation methods, which might work separately or in combination, 1) a melting process of the synthetic fibres, so called Thermomechanical separation; and 2) a design of new specific enzymes that will act as biocatalysts for degradation of a specific polymer which will further facilitate the re-synthetization of the polymers.

Within the thermomechanical separation the aim was to find out the prerequisites for the recycling of polyamide and elastane fibre blends without separating the materials first. The method gives nylon or elastane pellets, which can be used as raw material in processes such as spinning and moulding.

To ensure the viability of the enzymatic track the focus within the pre-study was on the separation of nylon 6,6. It was shown by computer simulations (MD) that the enzyme designed (the polyamidase) to interact and act as a biocatalyst for the degradation of nylon 6,6 most likely interacts at three specific sites along the polymer in water. This is a first positive indication that the designed enzyme can act as a biocatalyst to separate nylon 6,6 from other fibres in a mild and sustainable chemical recycling technology. The researchers have previous results on the first known polyurethane (e.g. elastane) cleaving enzyme [1] to be further developed for the design of enzymes suited also for the recycling of elastane in textiles.

The approach to formulate and manage this project included 1) gathering a well-represented value circle (from academia & research institutes; to textile manufacturers; to textile producers/retailers; and to textile waste management); 2) interaction in the form of workshops; 3) screening academic work to review the potential behind the idea (as described above).

There are many driving forces for material recycling, such as reaching a circular economy, reducing the negative effects of landfills, an increased demand for virgin materials, and upcoming legislations.

Nylon (polyamide) and elastane (also known as spandex or Lycra) fibre blends are common in textiles for underwear, swimwear, active wear and pantyhose. At end-of-life, these products are commonly thrown in the household bin and ends up on landfills or for incineration. Nylon gives strength to these products, while elastane is added for its elastic nature to generate stretch and comfort.

The experience from the Re:Mix I partners are that blends containing elastane (also common in stretch jeans and leggings) and nylon are increasing on the market. The industry partners within Re:Mix (Boob, Houdini, Swedish Stockings) are already collecting their own products after use with reuse/recycling, but a recycling technology for these materials is missing. They are actively searching for recycling options for textiles containing 5-13% elastane mixed with 20-95% nylon in addition to polyester, cotton or lyocell. The Re:Mix partners I:Co and Texaid, who together have an intrinsic understanding of the flows of used European textile waste, can already recognize an increase of elastane in textile waste. The need for recycling technology to separate both elastane and nylon from other textile components is critical.

The next steps needed within the mechanical melting separation technique, will be an optimization of process parameters and hard-ware. For the enzymatic separation technique the next step will be based on the initial work in Re:Mix I scaled up to lab and pilot scale tests performed on waste mixtures, since one of the main challenges with this process is upscaling. The scope of Re:Mix II, starting fall 2017, will most likely include a combination of the above techniques, biocatalysis and thermomechanical separation.



1 Introduction and Background

In a circular economy where discarded materials are recycled in resource efficient manners, a successful shift of textile waste recovery, from incineration to high quality recycled textile fibres, is necessary. Textile material recycling has been identified as a bottleneck in the Swedish textile waste management [2, 3] and large scale recycling plants are requested globally. To overcome this hurdle, the challenge of fibre separation from blends needs to be solved which is addressed herein by enabling recycling technology for separation of fibre blends containing nylon and elastane. To shift textile recycling from current low value products to a circular economy, the key issue of fiber blends must be addressed and it is thus clear that the lack of chemical recycling techniques, in particular for fiber blends, is substantial.

Increasing worldwide populations together with a limited access to virgin fibers cause a great concern to the society, as we understand that future demand will be impossible to respond to. At the same time much of the textile material can be found in waste streams. In 2013, 121 000 tons new clothes and household textiles were put on the Swedish market and 30 000 tons were separately collected for reuse. The major part of textiles today consists of complex blends of fibres containing for instance elastane and/or nylon for comfort. Within Sweden annually each citizen buys 13.1 kg textiles (2016) [4] and put 7.5 kg of textiles in the unsorted bin for household waste (2016) [5], where instead these quantities could have been sorted for reuse or recycling. Incineration is currently the most common waste management treatment in Europe for collected materials (>50 percent), and the low 3-5 percent that are recycled reflect the highly linear textile value chains [3]. By 2020, the Swedish Environmental Protection Agency (EPA) recommends that 25 percent of the textile products introduced on the market should be recycled into new textiles [2], and according to Elander and Ljungkvist [3], as much as 40 percent of the textile waste is suitable for recycling.

A majority of the clothing manufactured today contains at least a small percentage of elastane (e.g. 5%) to provide comfort. Some clothing contains significant fractions of elastane and interesting to note from the experience of the Re:Mix I partners: to replace elastane with eg polyesters would require at least 4 times the material. Moreover, the Re:Mix I partners see the trend that blends containing elastane (common in both stretch jeans and leggings) and nylon are increasing on the market. The Swedish industry partners within Re:Mix (Boob, Houdini, Swedish Stockings) are though already collecting their own products after use with the reuse/recycling purpose awaiting a recycling technology. They are actively searching for recycling options for textiles containing 5-13% elastane mixed with 20-95% nylon in addition to polyester, cotton or lyocell.

Furthermore, a Life Cycle Assessment (LCA) previously presented [6], shows that chemical recycling of blends in Scandinavia has a large potential to reduce climate impact as renewable energy is accessible to a larger extent than in the rest of Europe. The report also presents a preliminary study on the separation of polyester and cotton, showing that it is generally better to separate and recycle both materials, than to incinerate one of them. When reviewing the feasibility of existing chemical recycling within the textile sector, it is apparent that for polyesters (not blends), recycling is fully motivated from an environmental viewpoint [7], although depending on oil prices the cost profile of recycled polyester can be higher than the price for virgin polyester [8]. A similar cost-scenario is expected within recycling of nylon and elastane together with the challenge with cost-worthy separation technology of fibre blends.

These studies can be used as a motivator for ReMix (if assuming that recycling of polyester might be similar to recycling elastane and nylon), as the ambition is to find milder processes, ie more sustainable and less energy demanding, for recycling via enzymatic treatments. Biotechnology is already used in textile industry (e.g. laccase enzymes) so

there might be less hinders in embrace a new biotech processes, as the suggested Re:Mix approach.

Today the recycling of nylon/elastane blends is very costly and limited in scale through the process of solvent extraction (dissolution of elastane in dimethylformamide or dimethylacetamide) [9]. There are though other processes, like Nilit Ecocare fibre: a recycled nylon 6,6 through re-melting of the virgin material, which can be found commercially. Both nylon and elastane fibres today are fossil based, but can also be derived from bio-based sources. Eg Invista has a product with bio-derived elastane from corn [10], and RadiciGroup is producing nylon 6,10 from castor oil plant seeds [11]. In general biobased material is of course positive from a climate perspective, though the main benefit is if they also ought to be recycled to be included in the circular mindset.

1.1 Scope

The ambition with the project was to answer the question: Which technical methods is required to separate nylon (polyamide) and elastane (polyurethane) from other fibre types, and enable recycling of these textile blends.

The goal is thus to develop positive strategies for separation of different fibres from a fibre blend prior to chemical recycling, according to a circular economy.

The overall goal will be reached by three sub targets:

- 1) Mapping of knowledge, demands and methods in the field of polymer separations and recycling
- 2) Establishment of an efficient and relevant project group
- 3) Summary of fibre separation activities, and the developed strategies in a report

The experimental setup has been based on tracks, and to show their viability and potential in a future recycling process. Within the thermomechanical separation the aim was to find out the prerequisites for the recycling of polyamide and elastane fibre blends without separating the materials first. Moreover, the second track included computer simulations for the enzymatic design concept to be performed and selected enzyme to be further synthesized and tested in small scale.

The developed strategies should further be the basis for a subsequent larger project, which besides technical solutions, will contribute to the global revolution needed for the textile industry to become more sustainable and circular.

2 Execution of work

This project, Re:Mix- Separation and recycling of textile waste fibre blends, referred to as Re:Mix I, was a pre-study and thereby included a start-up and consortium building phase.

The approach to formulate and manage this project included:

- 1) a well-represented value circle (from academia & research institutes; to textile manufacturers; to textile producers/retailers; and to textile waste management)
- 2) interaction in the form of workshops
- 3) academic work to review the potential behind the idea.

2.1 Project constellation and actors

2.1.1 Management

The project has been coordinated by Dr. Åsa Östlund (RISE), assisted by Marie Syrén (RISE) supporting with her scientific competence regarding polymeric surface chemistry. Dr. Christina Jönsson (Swerea IVF) has supported the project with the expertise of life cycle assessments in order to incorporate this in the next project phase.

The main responsibility from the project management has been to:

- foresee that the overall goal for the project is fulfilled (see 1.1 Scope) with its three sub targets:
 - o mapping of knowledge, demands and methods in the field of polymer separations and recycling
 - o establishment of an efficient and relevant project group
 - o summary of fibre separation activities, and the developed strategies in a report
- deliver three workshops (see more under '2.2 Interaction between stakeholders and researchers')
- coordinate (and support) in applications for further founding

The points of interaction during the project have been through the workshops (see Figure 1) where all partners have met.

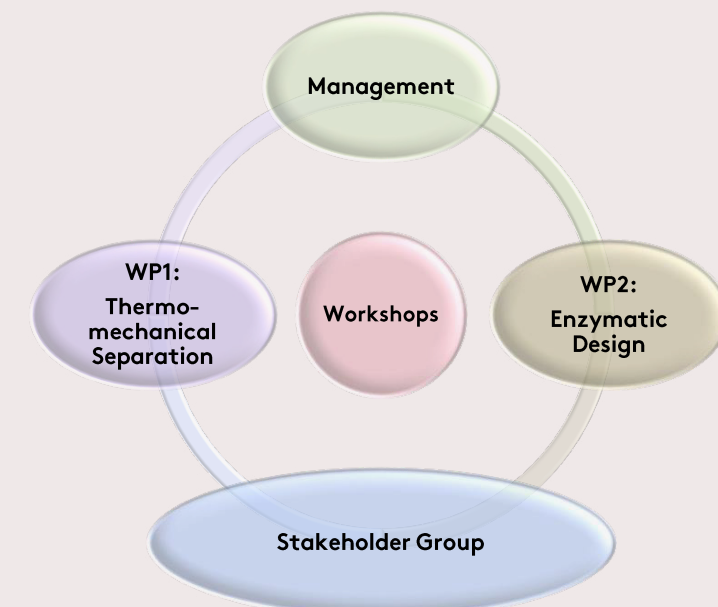


Figure 1: Working organization for Re:Mix during the pre-study phase, where the workshops held was the centre of interaction, where the Stakeholder Group was active together with the researchers. The academic experimental work on Thermomechanical Separation of textile blends (work package 1) and Enzymatic Design (work package 2) has been conducted in order to find out the potential of these approaches.

2.1.2 Stakeholder group

Stakeholders from industry were well-represented throughout the project, which facilitated in giving the most relevant direction based on the needs and relevance indicated by the industry. To align the studies and reach understanding between the different steps

in the Re:Mix value circle (academia & research institutes, textile manufacturers, textile producers/retailers, and textile waste management) the stakeholder group was activated more intensely during the workshop sessions. Representatives from the following companies have been active during the project: Boob, Faering Ltd, Houdini Sportswear, I:Co, Invista, Novozymes¹, Polykemi¹, Radici Group, Swedish Stockings, and Texaid.

2.1.3 Work package 1: Thermomechanical separation

The work on thermomechanical separation was led by Dr. Christina Jönsson (Swerea IVF). Most of the work within this work package was delivered as a diploma work by Ida Marklund, at Swedish School of Textiles, 2017 [12]. The background knowledge is based on studies with the Vinnova funded project 'Spill till guld' [13], wherein one of three areas involved waste from textile production.

2.1.4 Work package 2: Enzymatic design

This biotechnology track has been led by Dr. Per-Olof Syrén at SciLifeLab/KTH (Stockholm, Sweden) and the work has been conducted as a joint effort between BOKU- University of Natural Resources and Life Sciences in Vienna and SciLifeLab/KTH. Dr. Doris Ribitsch at BOKU recently contributed to the discovery of the first known polyurethane (e.g. elastane) cleaving enzyme [1]. KTH/BOKU together has also developed the first designer enzyme capable of nylon hydrolysis to be further developed for the design of enzymes suited also for the recycling of elastane in textiles.

2.2 Interaction between stakeholders and researchers

During the prestudy three workshops was arranged, with the purpose of sharing information and experience within the field, and to define next steps.

2.2.1 1st workshop, November 2016

At this workshop representatives from the relevant Re:Mix value circle was invited broadly and representatives from 12 different stakeholders participated in the discussions. The purpose of this workshop was to shape the project group and to formulate the focus and framing on research and development on new solutions for nylon and elastane separations.

2.2.2 2nd workshop, January 2017

This was a work meeting to formulate the basis for an application for further funding. The ambition for the meeting was to:

- overview what is needed and allocate work within the Re:Mix partners for the Re:Source application to be submitted Wednesday 20170208.
- formulate the Key Performance Indicators (KPI) and to build up a Project Plan to be included in the application.
- divide the work between the core group members for preparation of the application.
- briefly plan the continued work within the prestudy.

¹ Passive partner, not present at workshops.

2.2.3 3rd workshop, June 2017

The 3rd and final workshop of the prestudy of Re:Mix was held in June 2017, after the notification of acceptance of further founding from RE:Source to be started in Oct 2017. During this workshop the project members got a presentation of results regarding the on-going development within the Re:Mix prestudy, how this will continue into the next phase of the project, and a workshop for the future. During the last point the opportunities and project design for a larger project (eg EU funded project) was explored and discussions on how to develop the work further based on the same idea and innovation that Re:Mix brings us.

2.3 Experiments & Methods

Within the experimental development of the Re:Mix Pre-study the two tracks of enzymatic design and thermo-mechanical processes were explored and the methodologies are below described more in detail.

2.3.1 Thermomechanical separation

Most of the work conducted on thermomechanical separation was published as a Diploma Thesis by Ida Marklund, June 2017 [12]. The materials studied in this work can broadly be divided into three primary groups:

- Model system: known material composition was created to map how different amounts of elastane affect the thermomechanical processing and the properties of the nylon6 and elastane-recyclate. In total, four different ratios are included in the model system; 0, 5, 10, and 15% elastane. A pure PA6 quality (0% EL) was processed in the same way as the blends to provide comparable reference values. For further details see Diploma Thesis, Ida Marklund, 2017[12].
- Post-industrial waste: Two different warp knitted, Nylon6 fabrics; containing 10% and 22% Elastane respectively, correspond as post-industrial waste. The type of Elastane was not specified. The fabrics were provided by Boob design and are normally used in their swimwear collection.
- Post-consumer waste: Bags with sorted post-consumer waste, containing polyamide and elastane garments, were provided by Human Bridge. The waste included swimwear, sport socks, underwear and a great variation of panty hose and active wear. The material composition of product groups used is described in Table 1.

Figure 2 shows a schematic over all the materials processed including the material compositions.

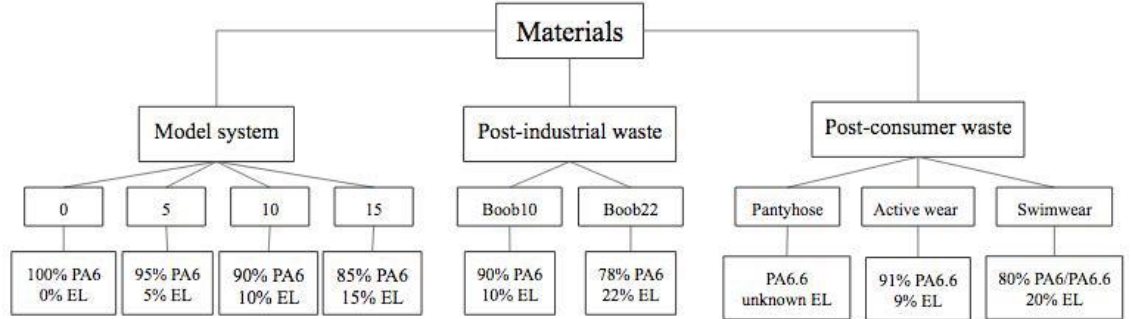


Figure 2: schematic over the materials processed by thermomechanical treatment.

Product group	Polyamide [%]	Elastane [%]	Weight [kg]
Swimwear	80	20	8
Active wear	91	9	25
Pantyhose	unknown	unknown	10

Table 1: Description of the material composition of product groups used within the field of post-consumer waste with thermomechanical treatment.

The materials in the three primary groups have gone through the same main processes (see Figure 3).

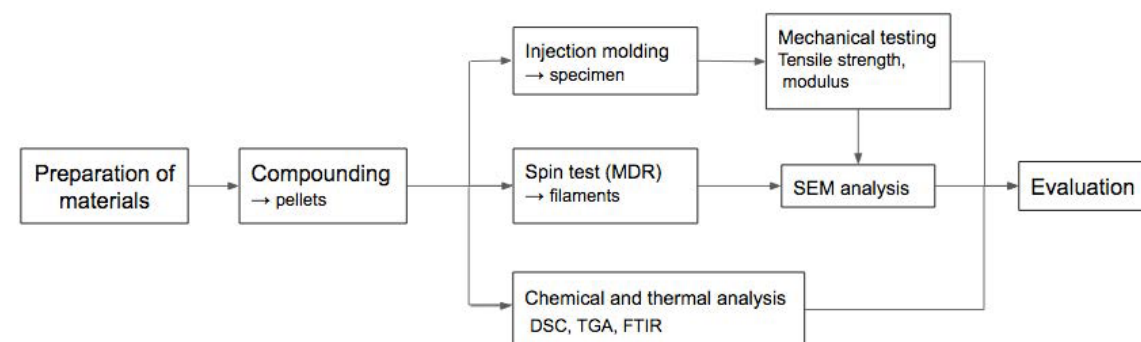


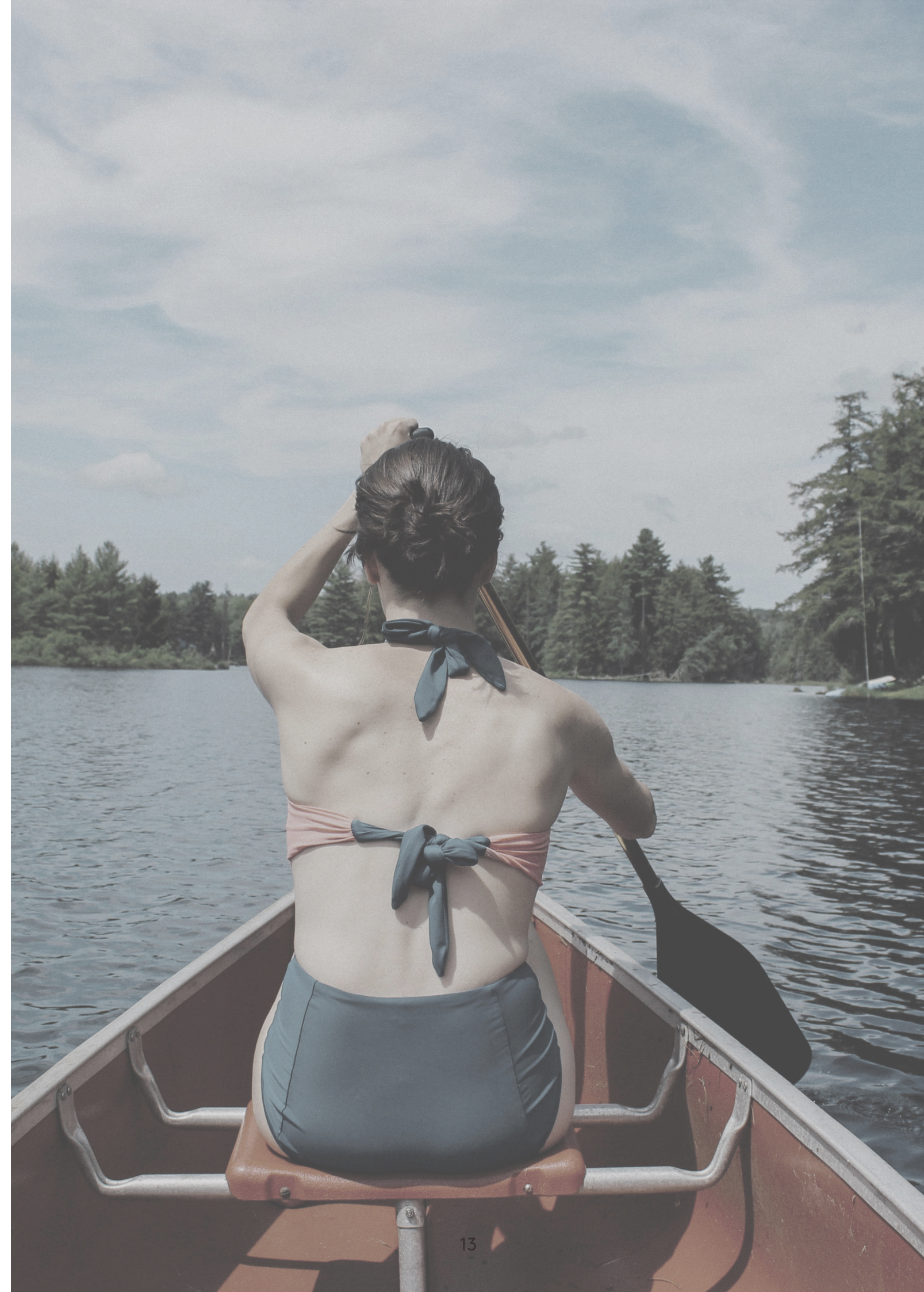
Figure 3: Schedule of the main process steps performed on the Nylon/elastane fibre blends.

2.3.2 Thermomechanical processing

All four ratios (0, 5, 10 and 15%) of elastane were processed both at 240°C and 280°C. The elevated temperature, 280°C, was used to simulate the process temperature for PA6.6. The three fractions of post-consumer waste (swimsuits, active wear and pantyhose) were cut into smaller pieces by hand, before being grinded in a Rapid granulator, G150-42-DT. The milled garments were fed by hand into zone three of the extruder. A melt filter was attached at the extruder's outlet to separate out the other fibres contaminating the polyamide and elastane blend. A first trial with a 250 µm filter was performed, before it was changed to 500 µm filter.

A melt draw ratio (MDR) test was performed on a Rheoscope 1000, CEAST 6742/000, capillary viscometer to investigate the spinnability of the compounds. A cone capillary was used, with a diameter of 10 mm and a height of 19 mm. The extrusion hole diameter was 0.78 mm.

A melt spinning trial was conducted on material L10 (PA6, 10% elastane). The pellets were dried in the dehumidifier for 72h at 80°C before being fed to the melt spinning machine. Spinning equipment from Extrusion Systems Limited (ESL, Leeds, England) equipped with two single screw extruders was used.



2.3.3 Characterisation of the thermomechanical study

A number of different characterisation methods were used, for example Fourier transform infrared spectroscopy (FTIR), Thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC). As well as microscopic methods.

Mechanical tests were performed on all specimens as they were produced through injection moulding, measuring tensile strength.

In order to get a better understanding of elastane, various kinds of elastanes and TPUs were characterized with FTIR, DSC and TGA. These materials are listed in table below.

2.3.4 Enzymatic design

Within the enzymatic design track in Re:Mix, in-house computational enzyme design tools developed in Syrén's laboratory at KTH/SciLifeLab were utilized to develop an engineered enzyme towards polyamide man-made materials [14].

Specifically, this project allowed us to pursue our hypothesis that hydrolytic enzymes constitute highly suitable starting scaffolds to introduce polyamidase activity by enzyme engineering. With their solvent exposed active site, we reasoned that cutinases would constitute suitable starting templates to achieve optimized transition state (TS) stabilization by enzyme design [9] to afford polyamide plastic hydrolysis.

The project investigated cutinases from *Humicola insolens* (HiC) and *Thermobifida celulosilytica* (TcC). Based on the two different starting scaffolds, in silico enzyme design was performed using the software YASARA. A model amide oligomer represented industrially relevant polyamides. For starting enzyme structures, missing hydrogens were added and the corresponding hydrogen network was optimized by using Amber03 force field, keeping all heavy atoms fixed. Crystallographic waters were kept. For the Van der Waals interactions, a cut-off of 7.86 Å was used and PME accounted for long-range electrostatics. The structures were minimized through repeated steps of short molecular dynamics and energy minimizations, initially by releasing fixed waters and then on all atoms. The thus obtained structures were finally subjected to simulated annealing. All simulations were performed under standard conditions under the canonical ensemble at 298 K using a Berendsen thermostat (and the Amber03 force field). MD-simulations were performed for 120 ns in duplicate, with a 20 ns equilibration phase and 100 ns production phase. Different initial random seeds were obtained by a slight change in the simulation temperature (by 0.0012 K).

To generate enzyme variants, megaprimer primers were designed carrying the specific mutations and used in two-stage PCR reactions to introduce the mutations. HiC variants were purchased from GeneArt® (Life Technologies, USA). Due to the different expression levels of TcC and HiC, different strategies were applied to obtain sufficient amounts of protein. TcC wild-type and variants were produced in shake flasks whereas HiC variants were expressed in a 5L fed-batch bioreactor (Minifors, Switzerland). All cutinases were purified by nickel-immobilized metal ion affinity chromatography over the C-terminally fused 6xHisTag. Wild-type cutinases and variants were analysed regarding esterase activity towards the soluble substrates para-nitrophenyl acetate (pNPA) and para-nitrophenyl butyrate (pNPB). Kinetic data were calculated by simple weighted non-linear regression of the Michaelis-Menten equation (Systat Software Inc). For investigation of amidase activity, para-nitrobutyranilide (pNBA) was used and kinetic parameters were calculated as described above. Activity on polyamide Nylon 6,6 (Goodfellow, UK) was determined by Gas Chromatography-Mass Spectrometry (GC-MS), to detect hydrolysis products. MALDI analysis of polyamides subjected to enzymatic hydrolysis was performed at KTH, department of Fibre and Polymer technology.

3 Results and Discussion

3.1 Thermomechanical separation

The results from the mechanical separation are below presented as compounding, filament production or injection moulding.

For the different collected waste material (post and pre consumer waste), compounding to pellets was successful for only some materials. The results from these materials were compared to a model system to identify challenges for post-consumer waste.

In summary the results showed:

- Pellets could be produced from all materials
- Injection moulding successful in most materials
- Meltspinning only successful with some materials

In summary, parameters of importance were identified as follows:

- Dependency on drying condition of the material
- Dependency of material content
- Gas formation during processing
- Initial attempts of melt filtration clogged the filter system

All extrudates were relatively even with a diameter between 1 and 2 mm for the materials in the model system. The grinded Boob 22% elastane fabric could be fed automatically into the extruder in zone three. The extrudates from this waste material were also relatively even with a diameter between 2–3 mm and kept their black colour from the fabrics (see Figure 4).



Figure 4: Pellets from post-consumer waste

The swimwear and active wear fractions could be fed automatically with an even flow of material into the extruder. This was not possible for the grinded pantyhose's. All thermal processing of the post-consumer waste generated a distinct odour and gas formation. The gas formation was more prominent for the post-consumer waste than for the model system and the post-industrial waste. See Figure 5 where extrudates from the postconsumer waste is compared to the post-industrial waste, Boob 22%.



Figure 5: Extrudates from Boob 22% at the top and post-consumer waste in the bottom.

All the materials processed at 240°C in the model system could be successfully injection moulded at 250°C. The materials compounded at the higher temperature, 280°C, were 0 and 5% successfully moulded at 280°C. For the materials with 10 respectively 15% of elastane, it was not possible to injection mould specimens at 280°C. Also drying parameters were investigated where longer drying times were preferable for success.

The swimwear fraction could be moulded into specimen while it was not possible for the active wear- and the pantyhose fractions.

3.1.1 Spin test – melt draw ratio

The spinnability was assessed in terms of the melt draw ratio. During the spin test performed on the Boob22% material, it was possible to draw the melt into fibres the first minutes of the feeding of the material, see Figure 6. But eventually gas occurred, the flow got uneven and it was not possible to draw the melt without breakage. When the pellets made from post-consumer swimwear waste, were extruded at 280°C, directly gas occurred together with a distinct odour and a clicking sound. The flow out from the capillary was uneven and the melt could not be drawn into fibres. No result could be measured.



Figure 6: Fibres from the boob 22% fabric

The compounding with melt filtration had to be halted directly. The filters clogged and the pressure got too high in the compounder. What was extruded was very uneven, twisted, brittle and filled with bubbles.

3.2 Enzymatic design

For many synthetic materials and fibres, enzymatic activities are poor or even lacking, as evolution did not yet have sufficient time to develop biocatalysts efficient towards man-made polymers. Hence, there is a great need to use emerging state-of-the art enzyme design tools to generate designer enzymes with novel activities and enhanced performance. This pre-study focused on initial concept and method development to contribute in tackling this great challenge. The research group of Syrén at KTH utilized computational enzyme design with the aim to introduce polyamidase activity in two different cutinase enzymes. In order to identify “hot-spot” positions that could be important in dictating interactions between proteinwater and the transition state, molecular dynamics (MD) simulations were performed using 3PA 6,6 as model substrate. MD studies revealed that position Ile179 in TcC and side chains Ile167 and Leu64 in HiC might have an impact in polyamidase activity of the selected cutinases. The amino acid positions were subjected to site-directed mutagenesis. In a first step, variants were characterized by our collaborator BOKU, with respect to detection of hydrolysis products from enzymatic treatment of synthetic polymers. Furthermore, MALDI analysis was performed at KTH (see Figure 7) and for analysis of release of hydrolysis products by GC-MS, see Figure 8.

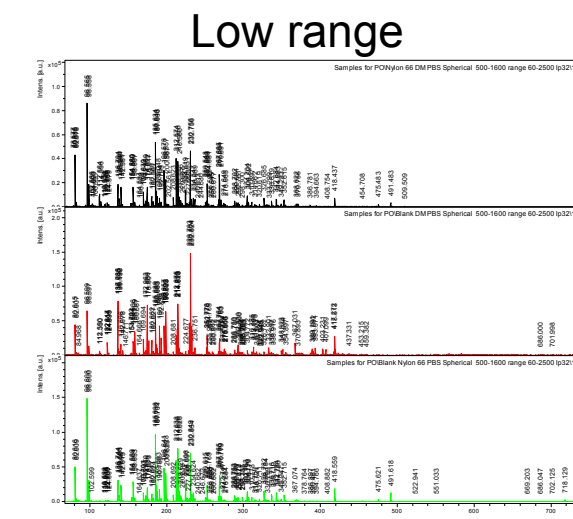


Figure 7: Preliminary results generated from this project. MALDI analysis performed in low mass range showed several potentially interesting mass peaks. The peaks potentially correspond to hydrolysis degradation products from polyamides subjected to first developed designer enzymes in the project. This initial mass spectrometry-based analysis indicates that further experimental development is needed.

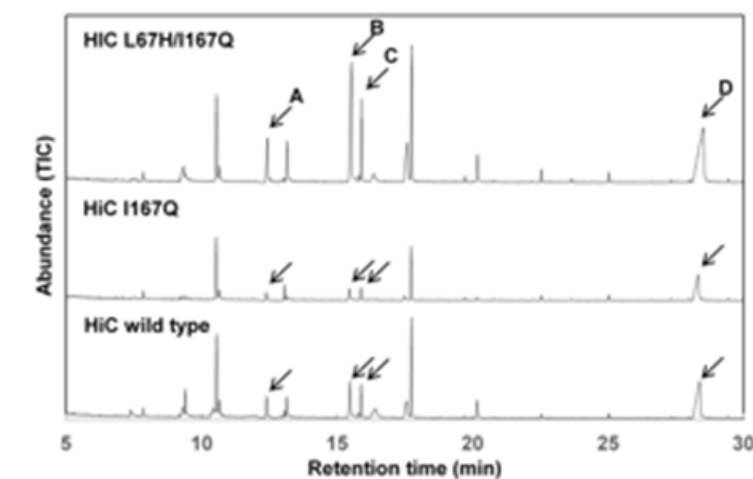


Figure 8: Analysis of release of hydrolysis products by GC-MS. Derivatized compounds were identified through comparison of the respective fragmentation pattern with data in the NIST library. Labelled arrows represent the compounds identified. (A) N-(6-aminohexyl)acetamide; (B) 6-ethoxy-6-oxohexanoic acid; (C) N,N'-(hexane-1,5-diyl)diacetamide; (D) 1,8-diazacyclotetradecane-2,7-dione.

4 Conclusions, implementation and next steps

4.1 Thermomechanical separation

All the materials with elastane were possible to compound both at 240°C and 280°C, but in the next process step (inject moulding or fibre spinning) it was more difficult at 280°C, especially with mixtures with more than 10% elastane. More gas formation and a crackling sound were constant at the higher admixtures of elastane.

Injection moulded test bars of polyamide with elastane processed at 240°C showed good mechanical properties. However, it was not possible to draw a fibre with this mixture without breakage of several filaments even though virgin polyamide was used to make a bi-component fibre.

4.2 Enzymatic design

This project resulted in gained knowledge with respect to biotechnologically-assisted depolymerization of man-made materials. This pre-study has allowed for preliminary material analysis after enzymatic hydrolysis by MALDI, from which we conclude that further optimization of our experimental setup is needed. This is planned for future joint projects. We have shown that enzyme design is a promising method to afford hydrolysis of pure synthetic polymers.

4.3 Next steps

Next step within the thermomechanical track is to evaluate the possibilities with melt filtration more with the materials from the model system to have a more controlled setup. In addition to this it will be important to search for other coupling agents on the market that could help in the process to make elastane more compatible with polyamide in the recycling processes. To increase the shear stress during compounding could be another way to reduce the particle size if it is not possible to separate the elastane from the polyamide.

Within the enzymatic track this pre-study shows the importance of a multidisciplinary approach in tackling complex research problems. The knowledge generated will constitute a cornerstone for further collaboration on catalyst development, with a future focus on textile materials. An important aspect of future research will be the analysis of the performance of designer enzymes in the hydrolysis of material blends, e.g. mixtures of polyamides with other fibre types.

Except for the upcoming Re:Mix II, the developed strategies should be the basis for a subsequently larger project, which besides technical solutions will contribute to the global revolution needed for the textile industry to become more sustainable. The expected impact on long term from the project is to accelerate the shift of the Swedish textile industry towards a circular economy as many of the Swedish actors in design and retail of apparel are driving the sustainability questions globally. It is a fact that within the fashion industry, Swedish brands have credibility globally within sustainability issues. We wish that Swedish fashion manufacturers will adept the future Re:Mix technology for recycling of textile blends, and acquire recycled textiles from their suppliers. The upcoming project aims at delivering at least 3 active and specific enzymes and developed separation methods verified in case studies and delivered in scientific publications and public reports.

5 List of Publications

Thesis for the Degree of Master in Science: Ida Marklund, Thermomechanical Recycling of Polyamide and Elastane fibre blends. The Swedish School of Textiles. Report no. 2017.14.06

The aim with this thesis was to map parameters that enable thermomechanical recycling of polyamide and elastane fibre blends. A model system with known raw materials and compositions was developed to analyse how different admixtures of elastane (0, 5, 10 and 15%) and process temperatures affect the thermomechanical reprocessing of these blends. Virgin, light stabilized polyamide 6 granulates were blended with cut elastane threads during compounding at either 240°C or 280°C. These temperatures were chosen to simulate process temperature of polyamide 6 and polyamide 6.6 respectively. It is concluded that limitations in process temperature and composition differs between different processes, due to variations in residence times, the character of the processing and in extension required material properties.

Manuscript: Antonino Biundo, Raditya Subagia, Michael Maurer, Robert Vielnascher, Per-Olof Syrén*, Doris Ribitsch*, Georg M. Guebitz, Nylonidases by Redesign of Water Networks, 2017.

The project has in part contributed to the manuscript that is under review.

6 Dissemination

Press release: from Mistra Future Fashion in Oct 2016. Reach: academia and press.

Poster: at Mistra Future Fashion annual meeting 27th of September 2017. Reach: Mistra Future Fashion consortium of 90 participants.

Final report: The report will be available via Energimyndigheten, RE:Source, and distributed within the Mistra Future Fashion program, using its communication channels, as well to all members of the project group.

7 References

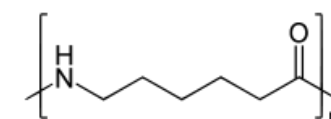
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8 Appendix: Chemical structures

8.1 Nylon (also called polyamide)

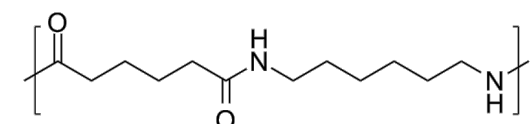
Nylon is a generic name for a group of synthetic polymers, based on aliphatic or semi-aromatic polyamides. Nylon is a thermoplastic material that can be melt-processed into fibres, films or shapes. Nylon is made of repeating units linked by peptide bonds. Commercially, nylon polymer is made by reacting monomers which are either lactams, acid/amines or stoichiometric mixtures of diamines (-NH₂) and diacids (-COOH). Mixtures of these can be polymerized together to make copolymers. Nylon polymers can be mixed with a wide variety of additives to achieve many different property variations.

Nylon 6, or polyamide 6 (Caprolactam)

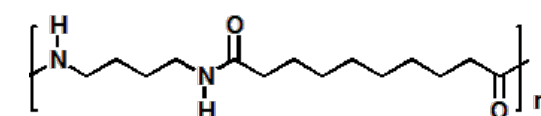


Nylon 6,6, or Polyamide 6,6 (hexamethylenediamine and adipic acid)

Most common



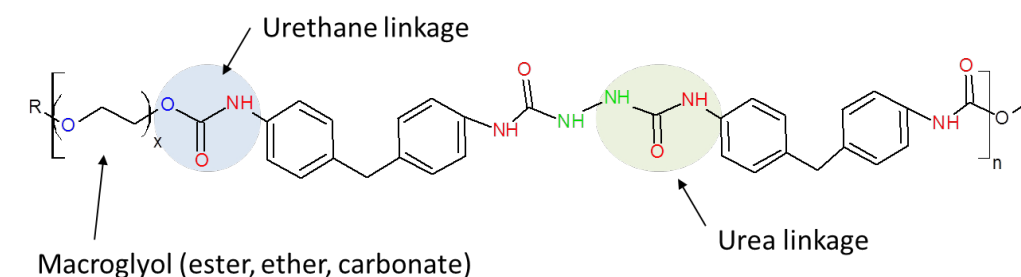
Nylon 6,10, or Polyamide 6,10 (hexamethylene sebacamide)



Nylon-6,10 (PA610) is semicrystalline polyamide commonly used in monofilament form in applications such as bristles and brushes. Due to its low moisture absorption compared to other nylons, it retains its properties better when wet.

8.2 Elastane, or Spandex or Lycra (Polyurethane)

A synthetic fiber known for its exceptional elasticity. It is stronger and more durable than natural rubber and it is a polyether-polyurea copolymer.





Mistra Future Fashion is a research program that focuses on how to turn today's fashion industry and consumer habits toward sustainable fashion and behavior. Guided by the principles of the circular economy model, the program operates cross disciplinary and involves 50+ partners from the fashion ecosystem. Its unique system perspective combines new methods for design, production, use and recycling with relevant aspects such as new business models, policies, consumer science, life-cycle-assessments, system analysis, chemistry, engineering etc.

MISTRA is the initiator and primary funder covering the years 2011-2019. It is hosted by RISE Research Institutes of Sweden in collaboration with 13 research partners.



Sweden will become a world leader regarding minimizing and utilizing waste. To achieve this vision we need new technologies, business models and processes. This is part of RE:Source's task. RE:Source is Sweden's leading research and innovation investment within the resource and waste area. It is a national innovation arena and supports the development of innovations that can contribute to a more efficient use of resources in both society and business.

RE:Source has been appointed by the Swedish Energy Agency, Vinnova and Formas as a Strategic Innovation Program, which means it has great importance for Sweden's international competitiveness, as well as for creating sustainable solutions to global societal challenges.

