

Full length article

Analysing the potential of the selective dissolution of elastane from mixed fiber textile waste

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ABSTRACT

Textile products are composed of various blends of synthetic or natural polymers. Elastane increases the functionality during use phase, but impedes high quality recycling. This study investigates the selective chemical dissolution of elastane from blended textile. Hansen solubility parameters and COSMO-RS were applied for solvent screening. The most recommended biobased solvents were experimentally validated with polyester, polyamide, cotton, wool and elastane for which solubility limits were determined and hence, their selectivity towards elastane dissolution. A TGA-corrected gravimetric method was developed as quantification tool and showed that tetrahydrofurfuryl alcohol and γ -valerolactone have comparable elastane dissolution capabilities to classical solvents (5 mg elastane/g solvent). Polyester/elastane and polyamide/elastane blends were subjected to this process as case studies. The LCA study showed that this selective solvent-based dissolution process saves 60% CO₂-eq./kg textile waste compared to incineration. This interdisciplinary work can set the benchmark for further developing and upscaling physical/dissolution recycling processes for blended textiles.

1. Introduction

The textile life cycle consumes quite some non-renewable resources (98 Mt per year worldwide) such as oil for the production of synthetic fibers, fertilizers to grow cotton and resources to produce dyes and auxiliary products. Consequently, the global textiles and clothing industry are responsible for consuming 93 billion cubic metres of water annually, good for 4% of the global freshwater withdrawal; and a greenhouse gas emission of 1.2 billion tonnes of CO₂ equivalent, representing about 5% of the global greenhouse gas emissions (Morlet et al., 2017; World Economic Forum, 2021). Textiles can be divided into 3 categories: clothing or apparel (i.e., shirts, trousers, socks, etc.), home textiles (i.e., bedcovers, curtains, carpets, etc.), other products for industrial, technical and medical purposes and footwear. In the current clothing value chain, less than 1% of the material used to produce clothing is recycled into a similar quality application (closed-loop

recycling), whereas 12% of the material used for clothing ends up being recycled in lower-value applications (open-loop recycling), while 73% of the materials are mostly sent to landfill or to incineration. Other losses from the value chain occur during clothing production (12%) and during the collection and sorting of used clothes (2%). It is thus clear that there is a huge challenge to achieve a circular textile industry (Haslinger et al., 2019; Morlet et al., 2017).

Towards a circular economy for textiles, once produced, textiles can be reused or repaired, but at a certain moment, they need a recycling technology. Textiles can be mechanically recycled as fiber or reggranulate, physically recycled to polymers, or chemically recycled from polymers to monomers. Especially mechanical recycling is hardly able to process blends (e.g., cotton/polyester (PET) with elastane for wearing comfort) and it is not able to remove dyes and contaminants, typically leaving a poor quality material which can only be used in a limited amount of applications (Morlet et al., 2017; Payne, 2015). Chemical

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recycling by depolymerization is promising, but might require energy for depolymerization and later repolymerization. As an intermediate option, dissolution based recycling can be applied for recycling purposes, which allow the selective dissolution of fibers in suitable solvents and adjoining removal of additives.

Selective dissolution of polymers is controlled by two transport processes, i.e., the disentanglement of polymer chains and solvent diffusion at the polymer-solvent surface layer. However, a particular solvent is able to dissolve particular polymers at certain process conditions (Miller-Chou and Koenig, 2003). This selectivity implies that solvent molecules show a potentially unique affinity for the target compound. This property is interesting for recycling in which it is necessary to isolate pure polymers out of blends, which results in the fact that interest in dissolution recycling for treating mixed textiles is gaining attention (Opperskalski et al., 2022).

Currently, synthetic fibers dominate the fiber market (Fig. 1A) with polyester having a market share of 54% of the total global fiber production. The second most used synthetic fiber is polyamide with 5%, followed by propylene, acrylics and elastane, with a combined market share of 5.2%. Plant fibers compose the second largest group of fibers with cotton (22%) and jute, linen, hemp and other plant-based fibers (6%). Animal fibers such as wool, down and silk only consists of 1.6% (Opperskalski et al., 2022). One particularly interesting fiber to selectively dissolve is elastane. Elastane, also known as Spandex or Lycra, is a polyurethane elastomeric fiber. It is frequently added to give mainly clothing a better elasticity (Fig. 1B). Elastane is made of block copolymers consisting of strong, rigid polymer segments alternated with more weaker flexible segments (Broadbent, 2001). It is typically used in concentrations around 5–15% but can also be used up to 49% (Penn Textile Solutions GmbH, 2022). Yet, these elastic fibers hamper the recyclability of the major compound composing the blend. Some typical elastane-containing blends are panties (polyamide/elastane: 88/12) (Zeeman textielSupers, 2022), sportshirts (PET/Elastane: 85/15) (Decathlon, 2022) and fabrics or swimsuits (polyamide/elastane: 51/49) (Penn Textile Solutions GmbH, 2022). Removing the elastane of such blends would free their main polymers such as polyester and polyamide.

Dissolution based recycling methods for textile fibers rely of course on solvents, which can be toxic, flammable, contribute to air pollution, etc. Currently, processes that are reported for selective dissolution of fibers from mixed textiles use solvents like N-methylmorpholine N-oxide (NMMO) in case of the Lyocell process for cellulose (Rosenau et al., 2001), dimethylformamide (DMF) or dimethylacetamide (DMAC) in case of the removal of elastane from polyamide/elastane blends (Gong et al., 2021; Yin et al., 2014). Alternative to the classical synthetic

solvents are a whole range of biobased solvents (BBS). Biobased solvents are referred to as solvents produced from biomass sources (preferably from organic waste). Examples are solvents originating from lignocellulosic biomass (e.g., 2-methyl-tetrahydrofuran, cyrene, etc.) or natural sources (e.g., limonene from citrus waste) (Byrne et al., 2016; Henderson et al., 2011; Prat et al., 2016). Without claiming here that BBS would have no downsides or toxicity, some of these emerging BBS have the potential to make dissolution recycling of textiles 'greener'.

In order to develop tailor-made solvent based recycling procedures for textile blends, theoretical tools can be applied to screen (novel biobased) solvents towards their affinity towards certain polymers. These tools reduce the amount of trial-and-error experiments, which are laborious and costly. Common tools are phase-solubility studies, quantitative-structure property relationships (Das et al., 2020), Hansen solubility parameter calculations via the group contribution method (Hansen, 2007; Van Krevelen and Te Nijenhuis, 2009), analysis of the Flory-Huggins interaction parameter (χ_{12}) (Hansen, 2007), density functional theory calculations with implicit solvation models (Marenich et al., 2009), conductor-like screening model for real solvents (COSMO-RS), COSMO segment activity coefficient model (COSMO-SAC) (Bell et al., 2020), etc. Molecular dynamics simulations allow the investigation of polymer-solvent interactions quantitatively and qualitatively via descriptors such as the solvation free energy, the solvent accessible surface area and the non-covalent interaction (NCI) index (Denayer et al., 2021). Currently, COSMO-RS has been mainly applied to find the best ionic liquid for cellulose (Casas et al., 2012; Liu et al., 2016), keratin (Liu et al., 2018) dissolution and solvent screening for recycling multilayer plastic packaging (Walker et al., 2020). Eventually, the results obtained from theoretical models should preferably be validated by experiments, especially also when the study focuses on waste polymer samples.

For experimental validation of selective dissolution from mixed textiles, the gravimetric approach is frequently applied in order to determine efficiency (Yin et al., 2014). However, this can come along with ambiguous data caused by solvent diffusion into the remaining fibers and leaching of additives disturbing the overall mass balances. This can be partly resolved by the use of drying methods such as described by ISO1833-1. Yet, since biobased solvents exhibit high boiling points, this drying procedure can take a large amount of time, or might even be unable to remove all solvent traces. Furthermore, the validation of selective dissolution from mixed textiles is even more challenging, as also the non-targeted fiber might be partially dissolved. The use of UV-VIS spectrophotometry can cause spectral overlap of the polymers and solvents within the UV-region. Furthermore, this method is more laborious when investigating the solubility behavior at different

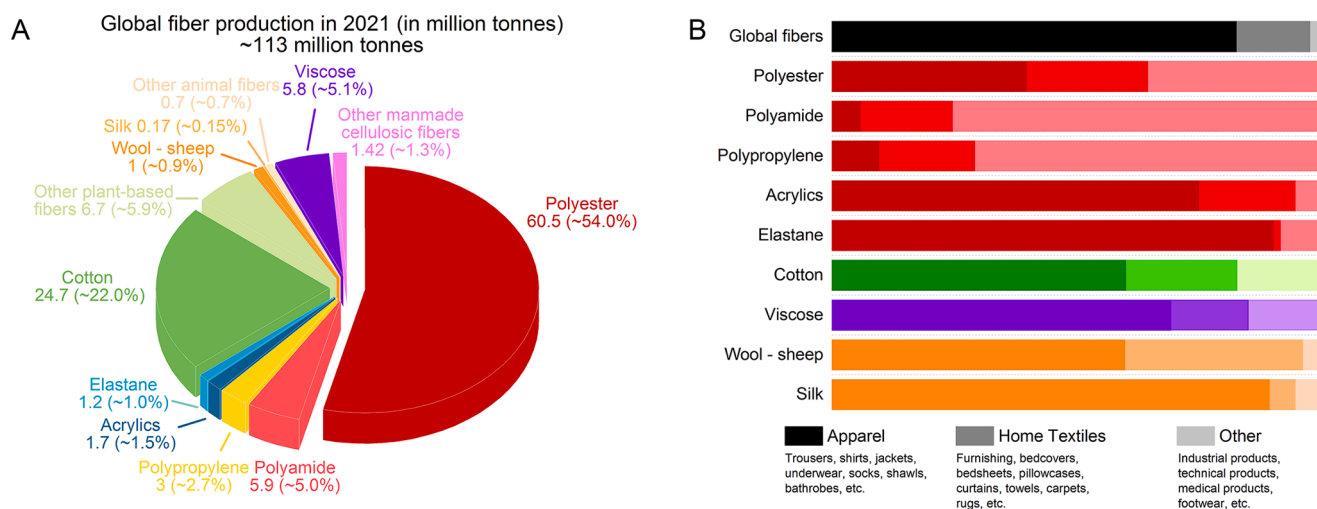


Fig. 1. A: Overview of the global fiber production in 2021 (in million tonnes) and B: the amount of fiber used in various applications (Opperskalski et al., 2022).

temperatures. Therefore, temperature dependent calibration curves are also required. For this reason, in this paper Thermogravimetric Analysis - Fourier Transform Infrared (TGA-FTIR) is proposed as a suitable technique to resolve the aforementioned analytical obstacles. This technique monitors the change in sample weight as a function of temperature and time in a controlled atmosphere (N₂ or O₂). This is accompanied by an evolution of gasses caused by sample decomposition or volatilization. These evolved gasses can be identified with FTIR to determine the sample characteristics. Hereby, this technique allows the simultaneous detection and quantification of multiple components in one sample with a single measurement.

In the context of selective dissolution of fibers towards increasing recycling rates for (mixed) textiles, this study targets the selective dissolution of elastane. Yet, to highlight the selectivity, also the other typical fibers are included such as cotton, wool, polyamide and polyester to see if the solvent has any effect on the non-targeted fibers. Therefore, this study aims (i) to screen 150 classical and green solvents to investigate the solvent selectivity towards elastane by means of Hansen solubility parameters and COSMO-RS (ii) to develop and apply an analytical technique to follow the performance of the selective dissolution process based on TGA-FTIR (iii) to experimentally validate the recommended green solvents from the theoretical tools for selective elastane dissolution (iv) to prove the effectivity of this systematic approach on two case study textile blends, being polyester/elastane and polyamide/elastane and (v) to gain insight in the environmental performance of a selective dissolution process for elastane by performing a Life Cycle Assessment (Fig. 2).

2. Materials and methods

2.1. Theoretical tools

2.1.1. Hansen solubility parameters (HSP)

One of the most widely used approach to calculate the solubility of drugs, dyes, solvents, polymers, oils and surfactants is the Hansen solubility parameters (HSP) (Hansen, 2004; Phan et al., 2022). The affinity between polymer and solvent is described by three partial solubility parameters: dispersion (δ_d), polar (δ_p) and hydrogen bonding (δ_h). The individual HSPs were calculated based on the group contribution method of Hoftyzer and Van-Krevelen: $\delta_d = \frac{\sum_i F_{di}}{V_m}$, $\delta_p = \frac{\sqrt{(\sum_i F_{pi}^2)}}{V_m}$, $\delta_h = \frac{\sqrt{\sum_i E_{hi}}}{V_m}$, where i represents the structural moiety within the molecule, F_{di} and F_{pi} are the total group contributions for dispersion ($J^{1/2} \text{ cm}^{3/2} \text{ mole}^{-1}$) and polar ($J^{1/2} \text{ cm}^{3/2} \text{ mole}^{-1}$), respectively, E_{hi} represents the group contribution to the hydrogen bonding energy ($J \text{ mole}^{-1}$) and V_m is the molar volume of the molecule ($\text{cm}^3 \text{ mole}^{-1}$) (Phan et al., 2022; Van Krevelen and Te Nijenhuis, 2009). Table 1 shows the Hansen solubility

Table 1

Hansen solubility parameters (δ_d , δ_p and δ_h) and interaction radius R_0 of the selected textile fibers.

Fiber	δ_d (MPa ^{1/2})	δ_p (MPa ^{1/2})	δ_h (MPa ^{1/2})	R_0 (MPa ^{1/2})	Reference
Elastane	17.6	6.0	9.0	9.0	(Ramphal et al., 2019)
Cotton	24.3	19.9	22.5	17.4	(Hansen, 2007)
Wool	17.7	20.9	18.6	12.7	(Hossin et al., 2016)
Polyester	18.8	11.9	4.5	16.8	(Gressier et al., 2019; Ügdüler et al., 2020)
Polyamide	16.0	11.0	24.0	8.2	(Hansen, 2007)

parameters of the various textile fibers and their interaction radius (R_0), which is the highest, experimentally determined, difference for a good interaction between polymer and solvent. The solubility parameter distance R_a in the Hansen space gives a qualitative view for the miscibility between polymer and solvent. The smaller R_a , ideally below R_0 , the higher the miscibility between the two compounds (Hansen, 2007).

$$R_a = \sqrt{4(\delta_{d,A} - \delta_{d,S})^2 + (\delta_{p,A} - \delta_{p,S})^2 + (\delta_{h,A} - \delta_{h,S})^2} \quad (1)$$

where $\delta_{i,A}$ and $\delta_{i,S}$ (MPa^{1/2}) are the i^{th} parameter of the polymer and solvent, respectively.

2.1.2. Molecular modeling-Conductor-like Screening Model for Realistic Solvent (COSMO-RS)

Textile fibers are polymers, that in our study are modeled by optimizing three monomers (e.g., polyamide, polyester and elastane) or by optimized polymer representations from literature (e.g., cellulose (Liu et al., 2016) and wool (Liu et al., 2018)). The structures were optimized with the B3LYP (Becke, 1993; Lee et al., 1988) functional along with the 6-311++G(d,p) basis set, which has already proven its reliability in describing organic systems (Tirado-Rives and Jorgensen, 2008). The calculations were carried out with Gaussian16 (Frisch et al., 2016) by means of density functional theory (DFT). The COSMO files of the polymers and (biobased) solvents were generated by Gaussian 16 and processed with Biovia CosmoTherm 20.0.0 (Dassault Systèmes). The database consists of 150 classical solvents and green solvents (Table S1) (BTC Europe GmbH, 2022; Prat et al., 2016). The influence of the number of monomers were investigated by using the COSMO-RS function “weight string”. Specific atoms of the studied polymer model can be switched on or off, being “0” or “1”, respectively (Section 2 of supporting information). The reported COSMO-RS values are relative molar solubility values ($\log S_{RS}$ with $[S_{RS}] = \text{mol L}^{-1}$) obtained after conversion of

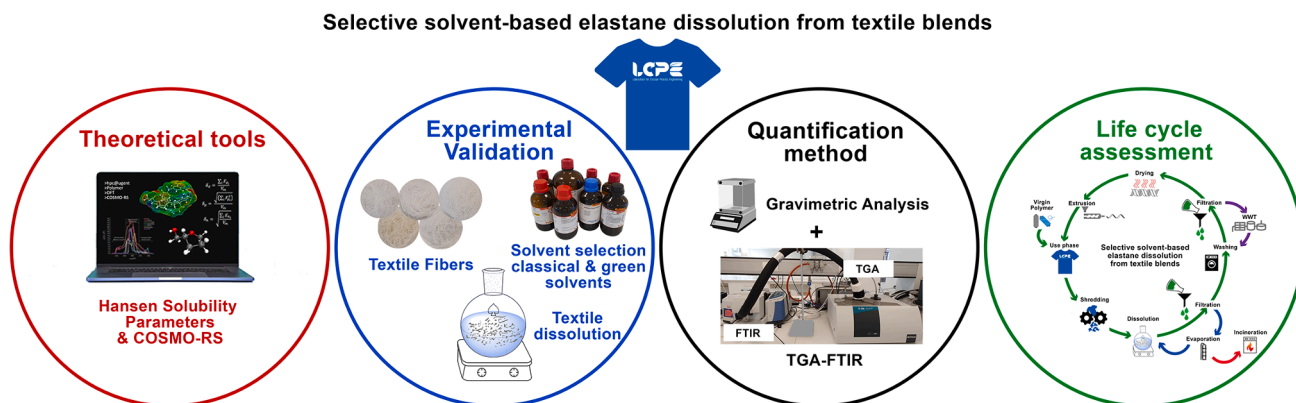


Fig. 2. Schematic overview of the selective dissolution process of textile blends in this study.

the mole fraction solubility values ($\ln x_s$) via the molar volume of the solution. Overall, COSMO-RS is able to obtain solvation free energies in pure and solvent mixtures and avoid the more lengthy explicit solvent treatments.

2.2. Experimental section

2.2.1. Thermogravimetric analysis with Fourier transform infrared spectroscopy (TGA-FTIR)

In order to determine the maximal solubilities, a NETZSCH TG 209 F3 Tarsus thermogravimetric analyzer coupled to Nicolet™ iS20 FTIR Spectrometer was applied to perform the analyses. The FTIR allows the detection of the evolved gasses derived from the sample decomposed within the TGA. A general temperature profile was developed for the analyses. An inert environment was established with nitrogen gas at 20 mL min⁻¹ throughout the entire analysis. The experiments started at a temperature of 35 °C and heated till 250 °C with a heating rate of 15 °C/min and kept isothermally for 15 min at 250 °C. The thermal decomposition step of the polymer (250–500 °C) was carried out at a heating rate of 100 °C/min. Afterwards, a heating rate of 15 °C/min has been applied to rise the temperature till 750 °C with additional oxygen gas (2 mL min⁻¹) to ensure complete oxidation of the sample content. This general method can be applied to all textile fibers, since cotton and wool did not achieve complete degradation in a nitrogen-only atmosphere.

2.2.2. TGA-FTIR method development

This section will first discuss the parameters of the TGA-FTIR that can influence the identification and quantification of the evolved degradation products. The discussed parameters are the heating rates at the point of thermal decomposition of the polymer (250–500 °C), the nitrogen or oxygen atmosphere, and limit of quantification. The influence of the heating rates could play a role in the degradation reaction mechanisms and thus, the formation of degradation products. The degradation mechanism of polyurethane foam has already been described by Jiao et al. (2013). The developed method will be applied to investigate the dissolution of pure textile fibers within elastane dissolving solvents (Fig. 3). Furthermore, the method will also be applied for the two case studies.

Different heating rates were assessed to find the most accurate procedure for polymer degradation and to investigate the peak resolution in the FTIR spectrum (Table 2). Table 2 shows the influence of various heating rates on the TGA-FTIR measurements of 10 wt% elastane dimethylacetamide solution. The values solely obtained from TGA show that the heating rate of 100 K/min shows the smallest deviation to the standard solution concentration. Overall, a heating rate of 100 K/min

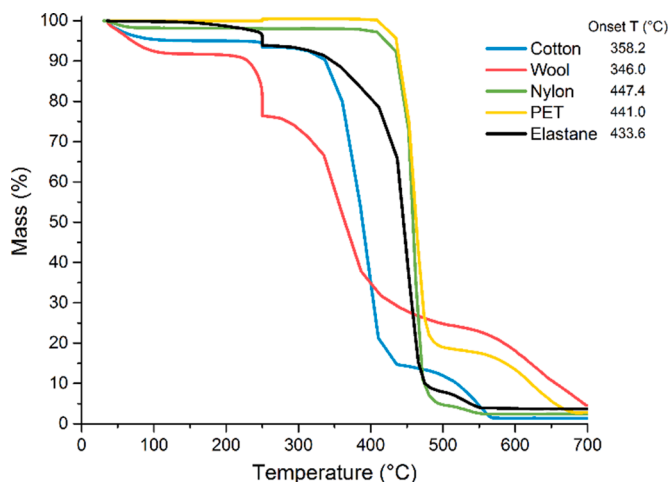


Fig. 3. The thermograms of pure cotton, wool, polyamide, polyester and elastane fibers along with their onset temperatures.

Table 2

First two columns: the influence of the heating rate on TGA results of a 10 wt% elastane dimethylacetamide solution and last two columns: the accuracy of the TGA method for various elastane dimethylformamide solutions with a heating rate of 100 K/min with a 20 ml N₂/min flow rate ($n = 3$).

Heating rate (°C/min)	TGA mass loss (wt%)	Standard solutions (wt %)	TGA mass loss (wt%)
15	8.98 ± 0.05	5	3.82 ± 0.11
30	8.91 ± 0.23	2.5	1.59 ± 0.24
50	8.95 ± 0.34	1	0.65 ± 0.12
100	9.06 ± 0.05	0.75	0.47 ± 0.09
		0.5	0.22 ± 0.17
		Level of Detection (LOD)	0.15 wt%
		Level of Quantification (LOQ)	0.50 wt%

with a 20 ml N₂/min flow rate was chosen. Various standard solutions were analyzed and the concentrations were determined by TGA only to assess the level of detection and level of quantification. Furthermore, Table 2 provides an overview to what extent the deviations can be attained with this TGA method.

TGA-FTIR spectra provide insight into the various degradation products that originate from the fiber (Fig. 4). The unique profile for each fiber as a function of temperature is useful to identify the efficacy of treating textile blends. The FTIR spectrum of elastane shows degradation compounds around 3700–3550 cm⁻¹, which can be ascribed to the presence of compounds containing hydroxy groups (OH). Stretching vibrations are detected at 2860 cm⁻¹ and 2940 cm⁻¹ and are caused by CH₃ and CH₂ (a)symmetric stretches. The double peak at 2310 and 2359 cm⁻¹ indicates the formation of carbon dioxide, which appears for each studied fiber. The combination of peaks at 1739, 1265 and 1111 cm⁻¹ is characteristic for aromatic esters and represent C=O stretch, C-C-O stretch and O-C-C stretch, respectively. The peak at 1370 cm⁻¹ from tert-butyl groups (-C(CH₃)₃), the peak at 1625 cm⁻¹ shows the presence of vinyl ethers. The absorption band near 1513 cm⁻¹ evolves from the bending vibrations of N-H in aromatic secondary amine (Jiao et al., 2013). For PET, the most pronounced peaks are found at 3580 and 1760 cm⁻¹ from carboxylic acid products. The FTIR spectrum for cotton displays a broad absorption band at 3600 cm⁻¹ can be allocated to chemically adsorbed water. Smaller peaks between 3000 and 2750 cm⁻¹ and 2180–2112 cm⁻¹ correspond to C-H stretches and CO, respectively. Broad peak at 1750 cm⁻¹ would come from C=O bond, very broad peak at 1500–900 cm⁻¹ can be ascribed to in-plane vibrations of C-H and the skeletal vibration of C-O and C-C (Wang et al., 2007). The FTIR spectrum of polyamide shows C-H stretches of alkanes between 2850 and 3000 cm⁻¹, constrained carbonyls and cyclopentanone give rise to the peak at 1760 cm⁻¹ and N-H in-plane bend vibrations result into absorption bands at 1500 cm⁻¹. Peaks at 930–965 cm⁻¹ are C-O stretches originating from cyclic acid anhydrides (NETZSCH-Gerätebau GmbH, 2019). The FTIR spectrum for wool is very similar to that of polyamide due to the nitrogen-containing protein structure of wool, with the only main difference visible at 1750 cm⁻¹ from C=O stretch of esters. These TGA-FTIR spectra are a semi-qualitative tool to assess the presence of one polymer within textile blends when they consist of polymers with similar degradation temperatures, which cannot be resolved with TGA solely. The recommended, and interference-free, peaks to check the presence of elastane within polyester-elastane blends before or after solvent treatment are the doublet at 2860 and 2940 cm⁻¹ (CH₃ and CH₂ (a)symmetric stretches) and the peak at 1111 cm⁻¹ (O-C-C stretch). A summary of the interference-free peaks is given in Table 3 depending on the composition of elastane-containing blend.

2.2.3. Samples and reagents

Pure textile fibers were obtained to investigate the selectivity of the solubilization process. Worsted wool gabardine (187 g m⁻²), polyamide (190 g m⁻²) and polyester (150 g m⁻²) were provided by Center for

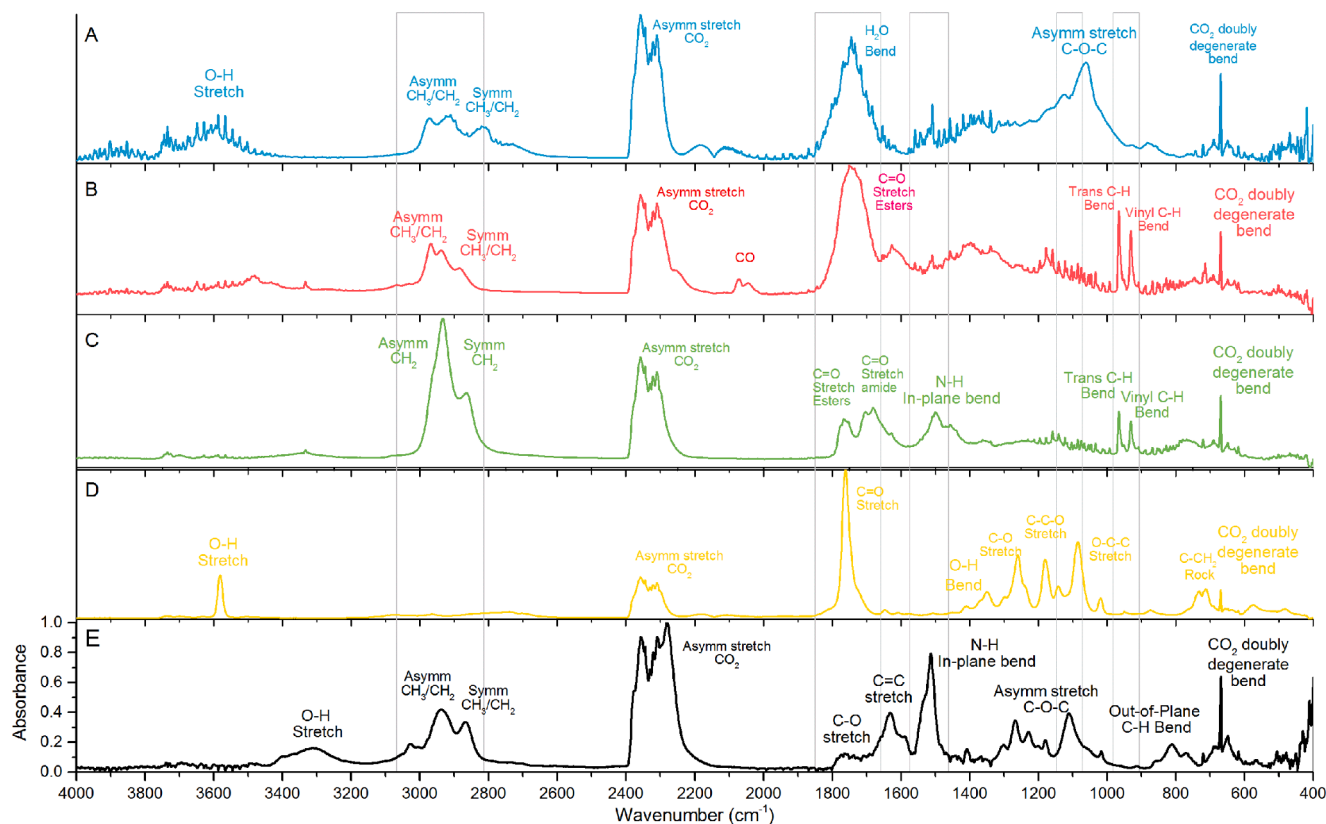


Fig. 4. The normalized TGA-FTIR spectra of pure cotton (A), wool (B), polyamide (C), PET (D) and elastane (E) fibers at their onset temperature.

Table 3

Interference-free wavelength regions of degradation products from textile fibers to investigate the presence of a fiber within the blend.

Textile blend	Unique peak for elastane (cm^{-1})	Unique peak for other fiber composing the blend (cm^{-1})
Cotton-elastane	1513 & 3350	1050
Wool-elastane	1513	930–965
Polyamide-elastane	1110	930–965
PET-elastane	1513, 1625, 2860 & 2940	3580
Cotton-PET-elastane	3350	cotton: 669 PET: 1260

Testmaterials B.V. Pure cotton (190 g m^{-2}) and elastane filaments were supplied by Utextel. Dimethylacetamide (DMAC, Sigma-Aldrich), dimethylformamide (DMF, Chemlab), n-methylpyrrolidone (NMP, VWR Chemicals), cyrene (CYR, Sigma-Aldrich), tetrahydrofurfuryl alcohol (THFA, Sigma-Aldrich) and γ -valerolactone (GVL, Sigma-Aldrich) were the main organic solvents of interest.

2.2.4. Dissolution of pure textile fibers: determination of maximal polymer solubility

In order to determine the maximal solubility of the five fibers within solvents, excess solid material was used. A solid/liquid (S/L) ratio of 1:100 was applied for cotton, wool, polyamide, polyester and elastane. The fibers and solvent were put in a flask and placed in a heating mantle (Joanlab). This setup is further equipped with a magnetic stirrer, temperature controller and a reflux condenser as cooling mechanism. The extraction time and temperature were 4 h and 100°C , respectively. After the dissolution process, the solution was filtered with a 5–13 μm particle retention filter paper (VWR). The remaining fibers were dried for 4 h at 100°C . For elastane, the solution has been centrifuged for 2 h at 4000

rpm to obtain the excess solid phase from the solution. The amount of dissolved polymer is calculated by the gravimetric-thermogravimetric analysis corrected approach. The gravimetric approach is frequently applied in order to quantify the amount of dissolved polymer. However, this can come along with ambiguous data caused by solvent diffusion into remaining fibers and leaching of additives disturbing the overall mass balances as shown in the equations (Fig. 5). The contribution from absorbed solvent and inorganic additives within the remaining fibers can be corrected by means of TGA. The addition of IR to the TGA allows the analysis of dissolved polymer within the filtrate.

2.2.5. Characterization of extracted elastane

The quality of the extracted textile fibers was assessed by liquid ^1H NMR at room temperature (400 MHz). ^1H NMR was preferred over ^{13}C NMR because if 10% of the carbamates were split to anilines, this would go undetected in ^{13}C solid state NMR. In liquid phase ^1H NMR, the spectra do reveal meaningful distinctions, since the carbamates and also free methylenedianiline (MDA)-type anilines can be detected giving shifts that cannot be mistaken. For this, spectra of 64 scans were recorded on a Bruker Avance III HD 400 device. Firstly, the pure elastane fiber (2 mg) was dissolved in d^6 -DMSO (0.5 mL, stirred at 120°C for 10 min) (Fig. 11, spectrum A). To improve the limited solubility of elastane in pure d^6 -DMSO, a solvent mixture of DMF:DMSO (1:1 volumetric ratio, 0.5 mL) was used to dissolve pure elastane fiber (5 mg, stirred at 120°C for 10 min) (spectrum B). Next, aliquots of the solvent-extracted pure elastane and the sportshirt were measured by mixing the recovered solvent layers (0.3 mL of recovered DMF or THFA, stirred at room temperature) with d^6 -DMSO (0.3 mL) for ^1H NMR. To improve signal quality of these dilute samples of fibers, phase and baseline correction by Whittaker smoothing were done for each spectrum. Spectra of solvent-extracted pure elastane and fiber of the sportshirt in DMF (spectra C and D, respectively) showed major DMF peaks (δ 7.95 ppm (CH, s), 2.89 ppm (CH₃, s), 2.73 ppm (CH₃, s)) and spectra of solvent-

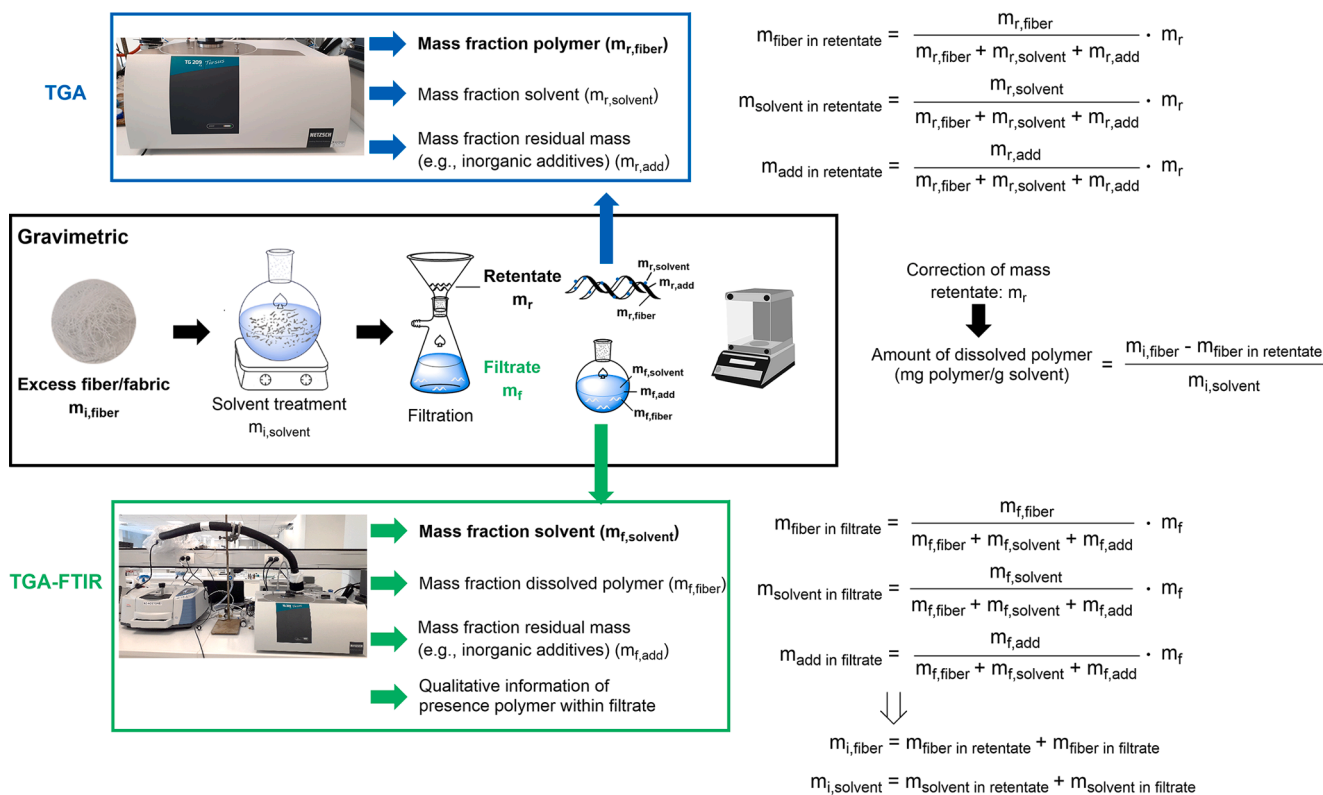


Fig. 5. Overview of the quantification method used for determining the amount of dissolved polymer (gravimetric and TGA as correction). The TGA-FTIR was used to qualitatively analyze the dissolved polymer within the solution.

extracted pure elastane and fiber of the sportshirt in THFA (spectra E and F, respectively) showed major THFA peaks (δ 4.52 ppm (OH, b), 3.75 ppm (CH₂, m), 3.59 ppm (CH, q), 3.34 ppm (CH₂, s), 1.78 ppm (CH₂, m), 1.56 ppm (CH₂, m). The quality of the fibers after solvent treatment was monitored in the chemical shift range of 7.40 – 5.70 ppm, which is a domain that does not coincide with solvent peaks.

2.2.6. Tensile properties

The tensile tests of the pure original and the solvent treated fibers were performed with Textechno Statimat M. The gage length was set on 100 mm. A load cell of 10 N was used for cotton, wool and pet fibers, while a load cell of 100 N was applied for polyamide fibers. The test speed was set at 100 mm/min with a pretension of 0.50 cN/tex. The one-way analysis of variance (ANOVA) was carried out to investigate the difference in tensile properties between the solvent treated fibers with respect to the original fibers by comparison of means. The significance level for the statistical analyses is 0.05. The p-values obtained lower than 0.05 are regarded as significantly different. All statistical analyses were performed with SPSS Statistics Version 25 (IBM) software.

2.2.7. Carbon footprint calculations

A carbon footprint is calculated for the dissolution-based recycling process presented in this study based on a preliminary flow scheme as shown in Fig. 12. The different processes included in the flow scheme are shredding of the material, dissolution of the elastane, filtration, post-washing step, a second filtration step, drying, extrusion of remaining fiber, wastewater treatment and solvent recovery by evaporation. From previous studies (O'Rourke et al., 2022; Ügdüler et al., 2022), the recovery rate and S/L ratio of the waste material in the solvent were found to be key contributors to the environmental impact of physical recycling processes. Therefore, a sensitivity analysis is performed for these two process parameters. The S/L ratio is based on the obtained solubility limited for elastane (Fig. 7), and is varied between 0.01 and 0.1 kg textile/L solvent and a solvent recovery rate between 98.00 and 99.99%.

Data for the elastane dissolution process were adapted from the results obtained at lab-scale, i.e., dimethylformamide and γ -valerolactone was used at a temperature of 100 °C to achieve complete dissolution of the elastane. Energy calculations are based on the specific heat capacity and additionally, Aspen Plus simulations were performed to optimize the solvent recovery for both solvents (section 11 of the supporting information). Hereby, the dissolution process is simulated in a batch reactor while the solvent recovery stage is considered continuously by using buffer tanks. Heat losses were not included and the compressor efficiency is set at 0.7. An elastane content of 10 wt% was assumed to be present in the input stream. In the current analysis, it is assumed that the dissolution residues containing elastane and additives is incinerated after downstream processing of the solvent. However, recovery of elastane from the solvent and recycling it in new products could be investigated in further research. As benchmark scenario, incineration of the textile blend and replacement of the incinerated textile by virgin textile is assumed. The carbon footprint for the different processes was extracted from the Ecoinvent database v3.1 in OpenLCA 1.9. The results of the carbon footprint are expressed as kg CO₂-eq./kg textile waste. An overview of the data inventory, the assumptions made in the current analysis for the preliminary process chain, and the datasets used from the Ecoinvent database is shown in Table S11.

3. Results and discussion

3.1. Solvent screening and selection

Theoretical screening tools such as Hansen Solubility Parameters and COSMO-RS can provide insights into the fundamental understanding of the intermolecular interactions for a broad set of compounds at a low computational cost. Sections 1 and 2 of the supporting information display the solvents considered for screening and the ten most recommended solvents based on HSP and COSMO-RS, respectively. Elastane is typically produced from a methylene diphenyl diisocyanate and a

polyol. Therefore, results of the screening methods suggest aromatic-based solvents and electron-donating compounds, more specifically polar aprotic solvents. Yet, from preliminary experiments, it was found that aromatic-based solvents were not able to dissolve elastane. Therefore, the aromatic based solvents were excluded in further calculations and experiments. Fig. 6 illustrates the 2D plot of Hansen solubility parameters for the different fibers. The selected solvents in this figure were mainly concentrated on elastane dissolution. For elastane, solvents with a R_a distance $< 9.0 \text{ MPa}^{1/2}$ are considered good potential elastane dissolving solvents (Table 1). Some suggested green solvents from HSP are 1,3-dioxolane ($1.20 \text{ MPa}^{1/2}$), dimethyl isosorbide (1.86), tetrahydrofurfuryl alcohol (THFA, 2.54), ethyl levulinate (3.08), etc. (Fig. 6) These results are corresponding well with the study of Ramphal et al. (2019) in which HSP were used to identify biobased solvents for polyurethane synthetic leather dissolution. In textiles, elastane is typically added to blends consisting of other polymers such as polyester, polyamide, etc. In order to obtain the selectivity of the dissolution process, the aforementioned solvents used for elastane dissolution should leave other textile polymers untouched (Tables S2-S5). For cotton, HSPs show the lowest R_a -values for ethanolamine (14.79), formamide (14.87) and glycerol carbonate (15.92). Sulfur-containing solvents such as dimethyl sulfoxide (17.40) and sulfolane (17.92) are also potential candidates for cotton dissolution. According to the HSPs, the aforementioned solvents for cotton dissolution would also dissolve wool. Furthermore, DMF (10.27), a good-elastane dissolving solvent, can jeopardize the wool. HSPs show that polyamide are more prone to alcohols or glycols such as propylene glycol (2.37), ethylene glycol (2.83) or 1,3-propanediol (3.07). Polyester fibers can be dissolved by dimethyl isosorbide (1.28); 1,3-dioxolane (1.58), tetrahydrofuran (1.79) and THFA (2.60). Based on HSPs, biobased solvents such as dimethyl isosorbide, THFA and ethyl levulinate thus have the potential to replace classical polar aprotic solvents such as DMF, DMAC and NMP. Furthermore, wool and polyester would suffer most from these polar aprotic solvents as well.

Based on COSMO-RS, green furan-containing solvents, such as 2-methyl tetrahydrofuran ($\log S_{RS} = 18.64$), 2,5-dimethyl tetrahydrofuran (15.60), tetrahydrofurfuryl alcohol (15.31) and 2-methyl furan (15.27), and ether solvents (e.g., cyclopentyl methyl ether) are recommended for elastane dissolution (Table S2). Classical solvents for elastane dissolution such as N-methylpyrrolidone (NMP), which is

undesirable due to its toxicity (Prat et al., 2016), and dimethylacetamide (DMAC) also emerge as potential solvents. For cotton, only THFA reappears in the COSMO-RS results. For wool, three different models were investigated for their solubilities, it was found that the experimental solubility for wool was presented the best via the oxidized glutathione and polypeptide model (Liu et al., 2018). THFA reappears for all three wool models as a potentially good solvent, which indicates bad selectivity for elastane dissolution. Next to that, classical polar aprotic solvents (e.g., DMF, NMP and DMAC) also appear in the screening results, meaning that wool has a high chance of being co-dissolved as well when treated together with elastane. Polyamide shows mainly alcohols and phenol-containing compounds for dissolution. This fiber should in principle remain unharmed in the presence of dipolar aprotic solvents. Polyester, which shares structural similarities with elastane, are prone to chlorinated solvents (e.g., dichloromethane), but would also be dissolved by green (e.g., THFA; 1,3-dioxolane; 2-methyl furan) and common aprotic solvents (e.g., NMP and DMAC) (Table S4). Fig. S1 shows the theoretical selectivity obtained from the calculated COSMO-RS solubilities. The theoretical selectivity shows in general that cotton would have the least influence during solvent treatment followed by polyamide, PET and wool. For cyrene, which is an interesting biobased solvent, changes to wool, polyamide and then PET might be occurring. As shown in Section 2 of the supporting information, HSP and COSMO-RS recommend various solvents. The solvents that are proposed by both theoretical tools are further considered for experimental validation. Therefore, classical dipolar aprotic solvents (e.g., dimethylacetamide) will be compared against the performance of biobased solvents (e.g., tetrahydrofurfuryl alcohol). In order to obtain the selectivity of the dissolution process, the solvents used for elastane dissolution will also be applied on the other textile fibers.

3.2. Dissolution of pure textile fibers: determination of maximal polymer solubility

To calculate how much polymer can be dissolved per mass of solvent, results from the gravimetric approach have been corrected with results obtained from TGA as explained in Fig. 5. Classical solvents such as DMAC and DMF show the highest capability to dissolve elastane with 5.1 and 5.0 mg/g solvent, respectively. The green solvent THFA shows

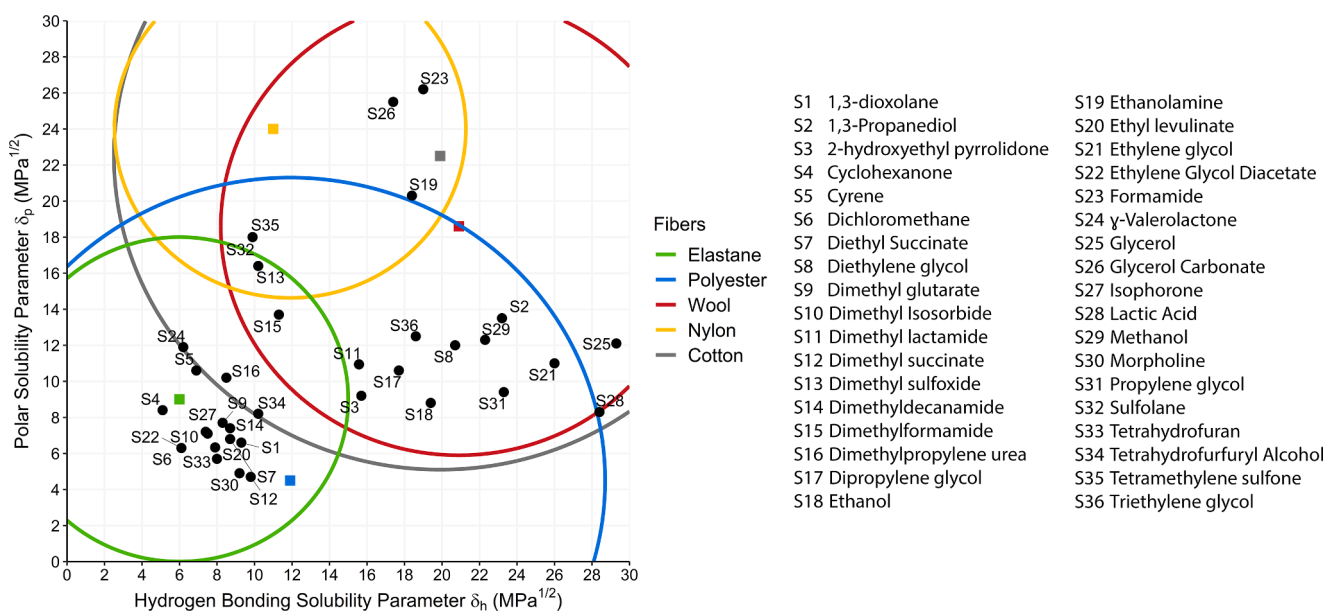


Fig. 6. 2D plot (δ_p and δ_h) of Hansen solubility parameters and spheres of the studied fibers and the selected solvents. The smaller the distance between polymer and solvent R_a ($\text{MPa}^{1/2}$), the more likely the solvent will solubilize the polymers. Additional data are illustrated and tabulated in the supporting information (Tables S1-S6).

comparable performance to the classical solvents (4.9 mg/g THFA), while cyrene and γ -valerolactone show a slightly lower solubility compared to THFA (3.3 mg/g cyrene and 4.3 mg/g γ -valerolactone) (Table S9). Cotton and wool are also prone to DMAC and DMF dissolution, while PET looks to be the most resistant fiber to all these solvents compared to the other textile fibers. Generally, Fig. 7 gives an overview of the most suitable solvent to be used for each specific textile blend in order to minimize the potential dissolution or swelling of the other major textile component within a blend when dissolving elastane. Table 4 displays the selectivity towards other fibers composing the textile blend for each studied solvent attained together with some EHS scores for these solvents based on Prat et al. (2016)

3.3. Tensile properties pure fibers

Since the aim of this study is to obtain a selective dissolution method for elastane, other fibers such as cotton, wool, polyamide or polyester that make up textile blends should not be dissolved. Fig. 7 illustrates that up to 5.1 mg elastane can be dissolved per g of solvent. This codissolution behavior potentially affects the physical properties of the polymer. Therefore, the pure fibers were tested before and after solvent treatment for their tensile properties (Table S10, Fig. 8). The cotton fibers seem to retain their tensile strength after treatment, including in case of using DMAC which shows no significant difference compared to the original fiber ($p > 0.05$) even though there is a codissolution of 1.64 mg/g DMAC. The highest degradation in fiber strength can be observed for wool treated with DMF ($F(1,4) = 765, p < 0.001$), which also showed the highest solubility (2.37 mg wool/g DMF). When wool is treated with CYR or GVL, there seem to be no significant difference in tensile properties ($F(1,4) = 0.616, p = 0.476$ and $F(1,4) = 4.72, p = 0.096$; respectively), although there is a loss noticeable of 1.23 mg/g cyrene, but no loss was observed for GVL. Polyamide showed no loss in tensile strength after treatment of classical solvents, but showed a loss of 22% and 42% for the green solvents THFA and GVL, respectively ($F(1,4) = 5.86, p = 0.073$ and $F(1,4) = 24.12, p = 0.008$; respectively). The treatment of PET fibers with DMAC, NMP and cyrene resulted in significant differences in tensile strength compared to the original PET fiber ($F(1,4) = 9.05, p = 0.040$; $F(1,4) = 8.49, p = 0.044$ and $F(1,4) = 16.95, p = 0.015$; respectively).

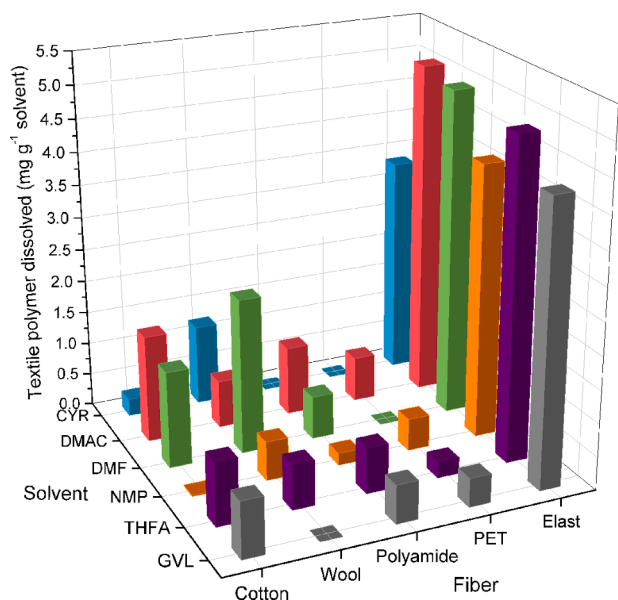


Fig. 7. The maximal solubilities of cotton, wool, polyamide, polyester and elastane in five various elastane-dissolving solvents (CYR: cyrene, DMAC: dimethylacetamide, DMF: dimethylformamide, NMP: n-methylpyrrolidone, THFA: tetrahydrofurfuryl alcohol and GVL: γ -valerolactone) (Table S9).

3.4. Case study: textile blends

In the previous section, the solubility behavior for each textile polymer – solvent combination was determined based on pure fibers as well as their solubility limits in the selected classical and green solvents. In order to validate this approach, textile blends were treated with DMF as classical solvent and THFA as green solvent since they both showed high elastane dissolution performance in combination with minimal codissolution of polyester and polyamide. Since elastane is mainly used in apparel, a sportshirt (PET/elastane: 85/15) and panties (polyamide/elastane: 88/12) were selected as textile blends. As shown in Fig. 9A, the performance of both solvents are similar in both case studies. The extraction of various additives results that the amount of dissolved content is higher than the elastane content. In order to verify if the elastane has been entirely removed from the blend, TGA-FTIR spectra were investigated. Hereby, Fig. 9B illustrates the TGA-FTIR spectrum of the original sportshirt, which consists of PET and elastane. The unique peaks of degradation products of PET and elastane were already identified in Fig. 4 and Table 3. The TGA-FTIR spectra of the sportshirt retentate after treatment with DMF and THFA are also displayed. It can be observed that the double peak that represents elastane has disappeared for the sportshirt after solvent treatment, which indicates the successful dissolution of elastane. The TG-IR spectra of the filtrates are shown in section 6.3 of the supporting information. The 2D analysis of the filtrate showed elastane peaks (2940, 2860, 1739, 1265 and 1111 cm^{-1}) that emerge at minute 30 of the spectrum. The unique peak for PET (3581 cm^{-1}) was not observed within the filtrates of DMF (Fig. S16) and THFA (Fig. S17). For the panties, the double peak at 2860 and 2940 cm^{-1} representing elastane is interfering the polyamide peak (Fig. S3B). As explained in the Table 3, in this case the 1110 cm^{-1} peak is more suited for analysis of the solid retentate. The removal of elastane from panties is also proven with TG-FTIR as illustrated in Fig. 9C. The absorption bands that indicate the presence of polyamide (930 and 965 cm^{-1}) were not detected within both filtrates (Figs. S18-S19). The TG-FTIR method is thus useful for (1) correcting for the solvent absorption effect during the dissolution process and (2) identifying the presence of elastane within solvent treated textile blends.

Fig. 10 displays pictures and SEM images of the original, the DMF treated and THFA treated sportshirt and panties. For both cases, it shows by its color removal that the solvent treatment also coextracted additives such as dyes, but potentially also other additives such as finishing agents, UV-stabilizers, antioxidant and other auxiliaries. For elastane filaments, polyamide and polyester textile products, the average amount of these chemicals can attain up to 2.6 wt% (Selling and Houser, 2005), 9.4 wt% (Wu, 2012) and 11.5 wt% (Wu, 2012), respectively, which is in order of magnitude compared to the excess weight loss seen in Fig. 9A. The SEM images for the sportshirt shows the filaments of polyester and elastane panties clearly woven alongside each other, but it is hard to distinguish from one another (Fig. S20). The stereoscope images were able to visualize the two filaments (Fig. 10B). The elastane filament within panties is the more thicker filament compared to the polyamide filaments as shown below. The filaments were less organized or compact after solvent treatment, also indicating the disruption of the internal structure of the remaining fibers.

Additionally, the quality of the extracted elastane was characterized by means of liquid ^1H NMR to assess whether the recycled elastane can be recycled or not. Liquid ^1H -NMR (d^6 -DMSO, 400 MHz) of the pure elastane fiber, before solvent treatment (Fig. 11 spectrum A) shows that this only contains methylene diisocyanate derived-carbamates (blue and white boxes). Since, the solubility of the elastane fiber in pure d^6 -DMSO was found low, added DMF (1:1 with DMSO) improved the detection of these carbamates (spectrum B), by which was seen that both the 4,4'- as 4,2'-carbamate aromatic isomers are present in the elastane. Additionally, a sample of pure elastane after solvent treatment in DMF, showed no breakage of the carbamate bonding (spectrum C). The carbamates bonds are linkages of the aromatic moieties with the flexible polyol

Table 4

The fiber selectivity per solvent during elastane dissolution (green indicates low codissolution, while red high based on internal normalization between the solvents) and safety, health and environment criteria for each solvent according to the CHEM21 solvent guide (scoring from 1 to 10, with 10 representing the highest hazard in each category) (Prat et al., 2016).

Solvent	Selectivity towards other fibers					BP (°C)	FP (°C)	Safety Score	Health Score	Environmental Score
Cyrene	PET PA	[Cotton	Wool]			203	108	1	5	7
Dimethylacetamide		PET	Wool	PA	Cotton]	166	70	1	9	5
Dimethylformamide	PET		PA		[Cotton	153	58	3	9	5
N-methylpyrrolidone	Cotton PA	PET	Wool			202	96	1	9	7
Tetrahydrofurfuryl alcohol		PET		Wool PA	Cotton]	178	75	1	9	5
γ -valerolactone	Wool		PET	PA	Cotton]	207	96	1	2	7

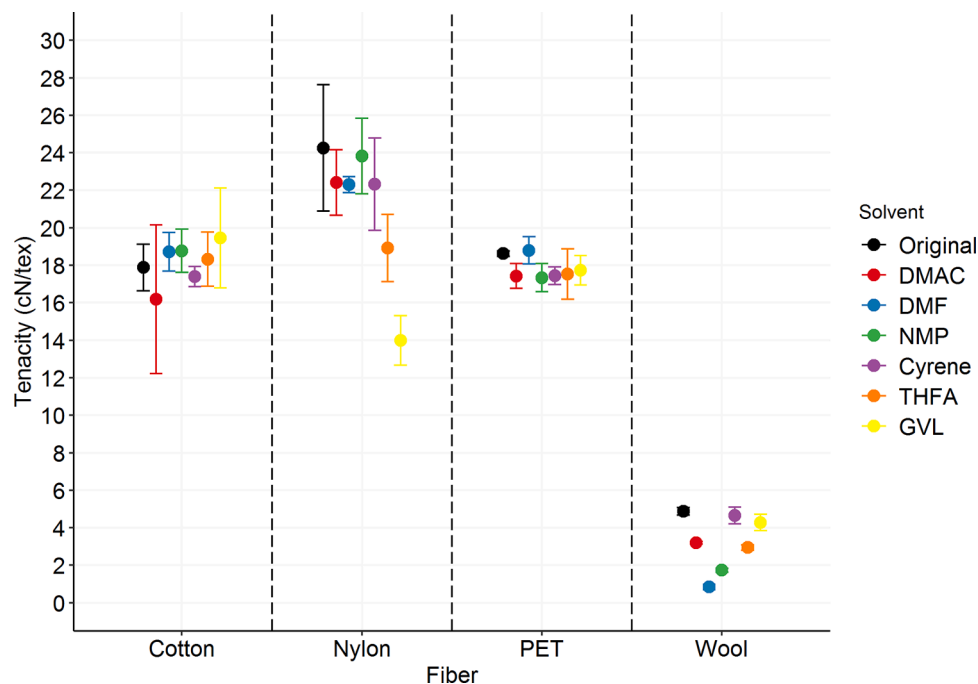


Fig. 8. The tenacity of pure fibers before and after treatment with elastane dissolving solvents (Dimethylacetamide (DMAC), dimethylformamide (DMF), n-methylpyrrolidone (NMP), cyrene (CYR), tetrahydrofurfuryl alcohol (THFA) and γ -valerolactone (GVL)).

chain, and no breakage of these bonds means that the initial polyurethane structure of the elastane remains intact. Additionally, extracted elastane, from the sportshirt in DMF (spectrum D), had primarily carbamate-containing aromatic rings, which shows no quality loss of the fiber. After solvent treatment in THFA however, carbamate bond breakage is observed. Carbamates of the pure elastane and the fiber extract of the sportshirt (spectra E and F, respectively) are converted to methylenedianilines (MDA). Pure elastane in THFA seems to retain some carbamate bonding of 4,4'-aromatic isomers while 2,4'-isomers are mostly converted to 2,4'-MDA compounds (green box). Fiber extracted from the sportshirt retains only a small fraction of 4,4' carbamates, while it is primarily converted to 4,4'-MDA (orange box), and 2,4'-MDA by the solvent treatment. Hence, the solvent-extracted fibres retain their original structure in DMF, while also partial depolymerisation of the elastane fiber happens in THFA. As usually elastane is only used in minor quantities in most textiles, the focus of this paper was on liberating the other polymer types such as polyester or polyamide. Future research could explore if it is possible to also recover the elastane. It should be noted, however, that it is likely that the solvent in which the elastane is dissolved would also contain impurities such as leached additives such as dyes, which might complicate the further purification of pure elastane.

4. Life cycle assessment (LCA) of elastane dissolution from textile blends

Recycling methods should ideally create environmental benefits, which is analyzed in this section by means of a Carbon Footprint. The impact of the recycling process for an elastane/polyester blend is compared with a benchmark scenario in which the textiles would be incinerated with energy recovery and the incinerated textiles would be substituted by virgin material. The preliminary flowchart used to calculate the impact of the recycling process is shown in Fig. 12A. In addition to the dissolution process itself, also other processing steps, such as shredding, washing, drying, and extrusion are included. An important assumption is that the elastane ends up in the bottom fraction of the evaporation together with other potential impurities and this residue is incinerated. However, further research should focus on the valorization of this fraction, which ideally should result in a lower environmental impact. The results of the Carbon Footprint are visualized in Fig. 12B.

Fig. 12B shows that environmental benefit can be created; even assuming that only the polyester fraction would be valorized. Different solvent recovery efficiencies via evaporation are simulated, varying between 98.00 and 99.99%. Especially at low solid-liquid ratios, efficient solvent recovery is key to create a positive impact of the recycling

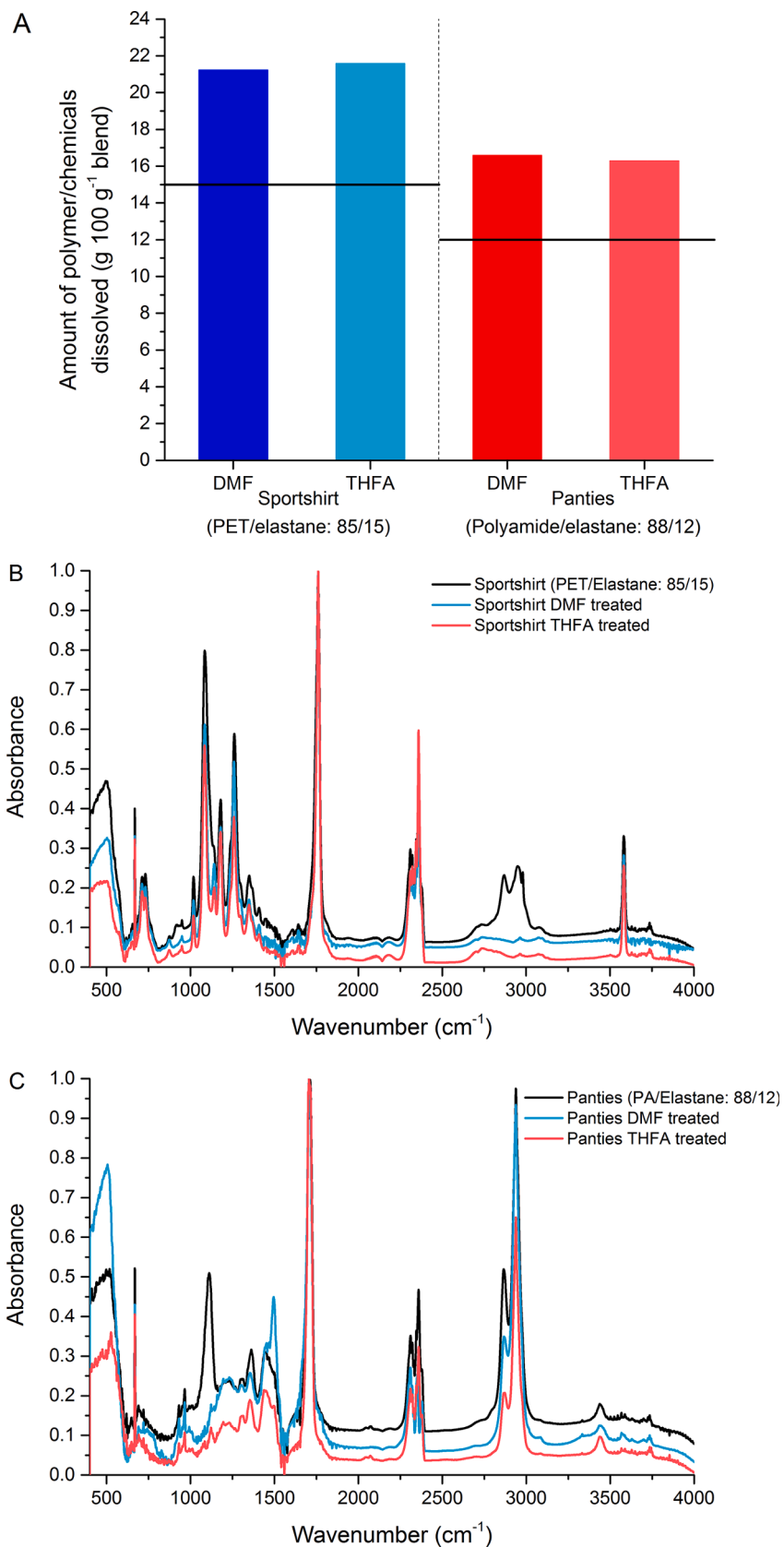


Fig. 9. The amount of textile polymer dissolved (g per g textile blend) with DMF and THFA from the sportshirt and panties calculated via the TGA-corrected gravimetric approach (A). TGA-FTIR spectra of the retentates from the sportshirt (B) and panties (C) before and after treatment with DMF and THFA, respectively.

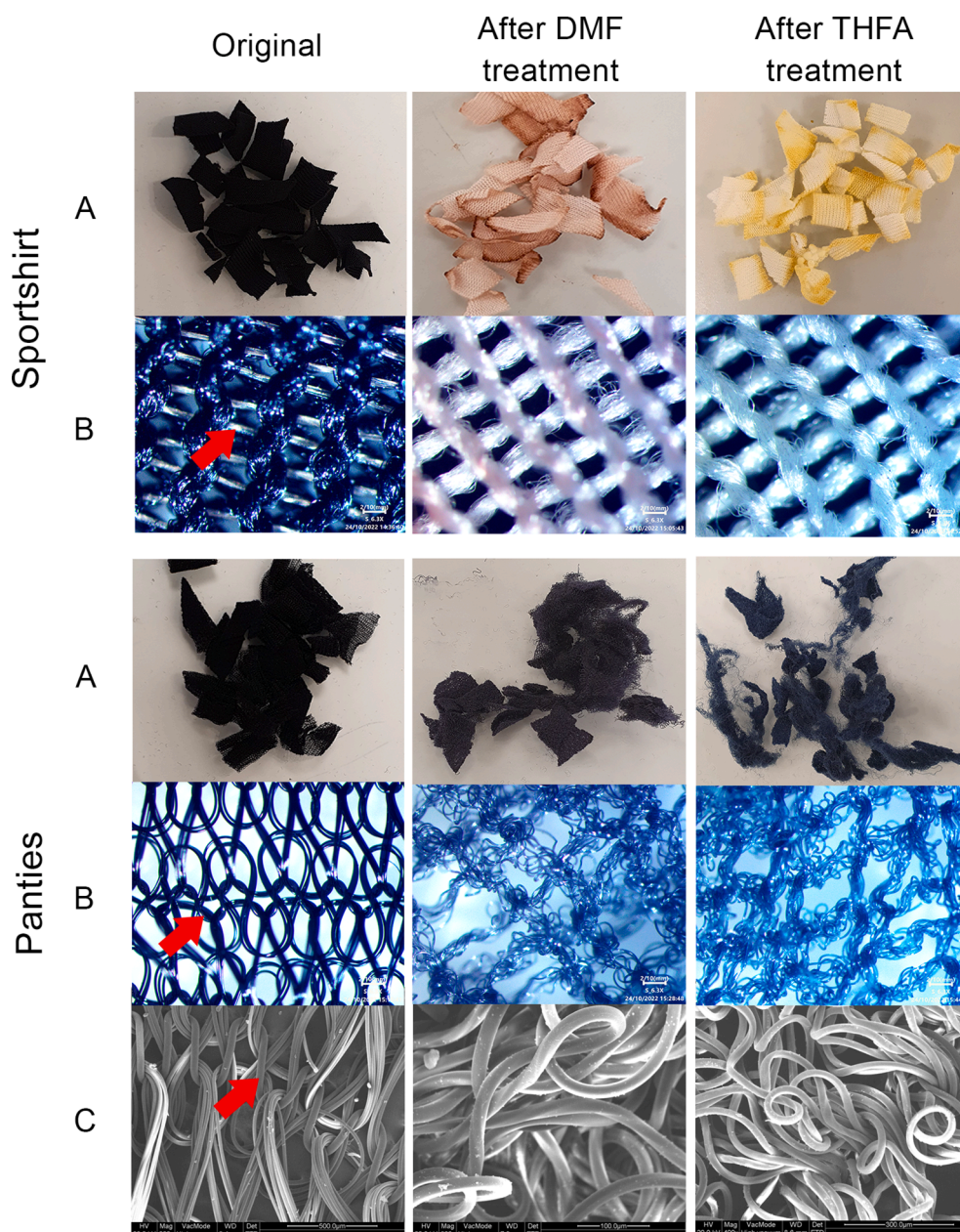


Fig. 10. Photographic (A), microscopic (B) and SEM-images (C) of case study materials (sportshirt and panties) before and after solvent treatment with dimethylformamide (DMF) and tetrahydrofurfuryl alcohol (THFA). The red arrows on the images indicate elastane.

process. The solvent recovery stage was calculated for DMF and GVL based on mechanical compression by means of Aspen Plus simulation. Compared to the reference scenario (i.e., incineration, which has an impact of 3.6 kg CO₂-eq./kg textile waste, at least a S/L ratio 0.050 kg textile/L solvent should be applied if a solvent recovery of 98.00% can be achieved. Based on the performed experiments in this study, the solubility of pure elastane within the green solvents reaches up to 0.046 kg/L. Applying this S/L ratio, the fact that the collected textile waste consists of 10 wt% of elastane and a solvent recovery of 99.99% would result in a Carbon Footprint of 1.19 kg CO₂-eq./kg textile waste. Hence, 2.45 kg CO₂-eq./kg textile waste could be saved by recycling the textile blend compared to incinerating and subsequently substituting the incinerated materials with virgin polymers. Even when a solvent recovery of 98% and 99% is considered, 1.10 and 1.76 kg CO₂-eq./kg textile waste could be saved by recycling the textile blend, respectively. Furthermore, the recycled textile can theoretically be recycled again, which would increase this benefit. Next to that, the impact of other

polymers, such as PA is higher, being 9.3 kg CO₂-eq. per kg PA which would mean additional benefits in case of PA recycling. Thus, the developed recycling process has the ability to have a positive impact on the environment by reducing overall greenhouse gas emissions and can increase the resource circularity in the textile industry.

5. Conclusions

Textile products are often composed of polymer blends in order to optimize their application properties. However, recycling elastane containing blends is still one of the obstacles to achieve a closed loop recycling process. This study has shown an in-depth investigation on the selective chemical dissolution of elastane containing blends. The theoretical tools Hansen solubility parameters and COSMO-RS provided biobased alternatives (e.g., cyrene, tetrahydrofurfuryl alcohol (THFA) and γ -valerolactone (GVL)) for classical solvents. The experimental validation showed that THFA and GVL have comparable dissolution

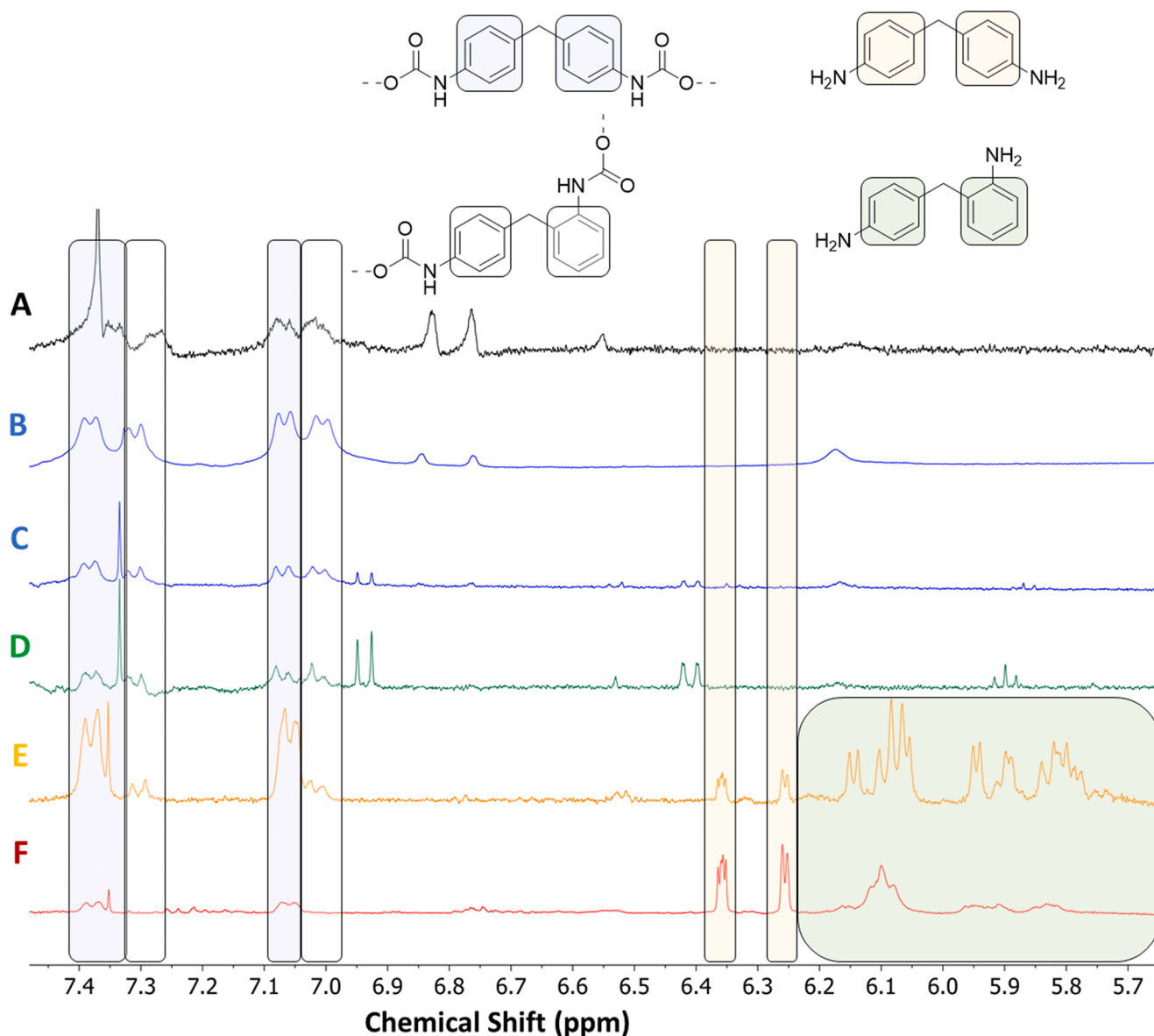


Fig. 11. Liquid ^1H NMR spectra (d^6 -DMSO, 400 MHz) of pure elastane in d^6 -DMSO (A), DMF:DMSO (1:1) (B), and the solvent-extracted pure elastane and fiber of the sportshirt in DMF (C and D, respectively) and THFA (E and F, respectively). The blue and white boxes indicate methylene diisocyanate derived-carbamates, while green and orange boxes highlight the presence of 2,4'-methylenedianilines and 4,4'-methylenedianilines due to solvent treatment, respectively.

capabilities to classical solvents. Tensile properties of pure fibers were performed to investigate the influence of the solvent. Cotton seemed to be inert by the influence of solvent, while polyamide is more prone to THFA and GVL dissolution compared to cyrene. For PET fibers, this is the opposite for which THFA and GVL would be recommended. Wool retained most of its tensile properties after treatment with cyrene and GVL. After treating fibers separate, the most appropriate solvents for elastane dissolution can be applied for various elastane-containing blends. Elastane from polyester/elastane blends can be dissolved selectively with cyrene, dimethylacetamide, dimethylformamide and THFA to remove the elastane part. Polyamide-elastane blends are preferably treated with cyrene, while NMP is the most suitable solvent for cotton-elastane blends. GVL shows the best selectivity in wool-elastane blends. This was evaluated with two case studies: sportshirt (polyester/elastane) and panties (polyamide/elastane). Important in the field of selective dissolution of fibers such as elastane from textile blends is a correct analytical procedure. For example solvent diffusion phenomena take place during textile dissolution, which requires a correction to the established gravimetric approach by means of thermogravimetric

analysis. Also partial removal of additives or even non-targeted fiber might disturb the gravimetric approach. Therefore in this study a combination of a TGA-corrected gravimetric approach, TGA-FTIR and SEM imaging was used. The additional FTIR spectra recorded assists the thermogravimetric analysis to verify the presence of solvent within fiber, dissolved polymer in the filtrate and elastane within the textile blend. The LCA study showed that the selective solvent-based dissolution is more environmentally friendlier recycling pathway compared to the incineration of textile waste. By applying the maximal solubility of elastane for solvent dissolution, considering that the collected textile waste consists of 10 wt% elastane and a solvent recovery of 98%, this solvent-based procedure already saves 60.3% CO_2 -eq./kg textile waste compared to incineration. This procedure can reach a saving up to 67.5% CO_2 -eq./kg textile waste when a solvent recovery of 99.99% is considered. This combined experimental and theoretical work, can form an important basis for further developing dissolution based recycling processes towards closing the textile loop.

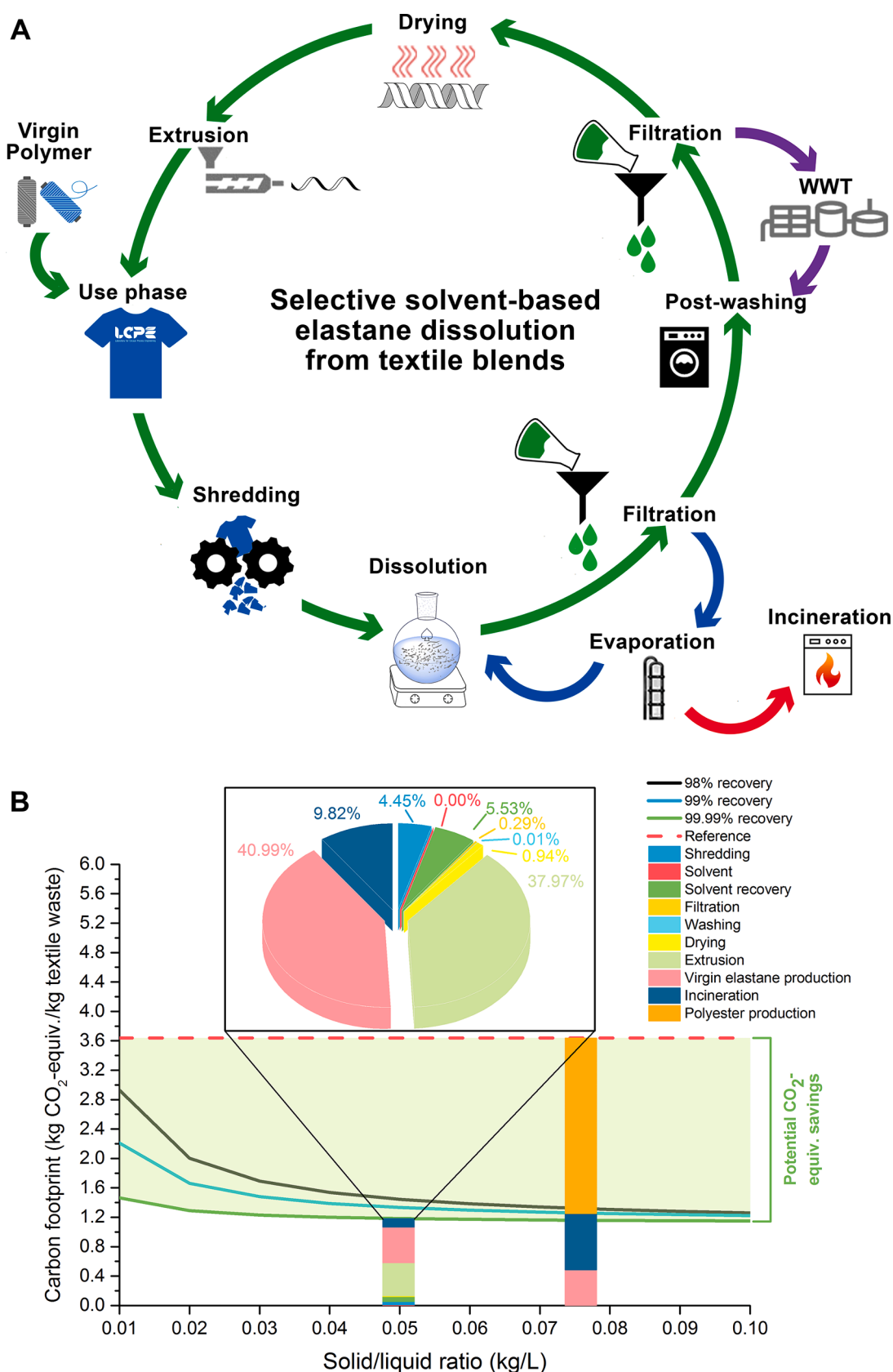


Fig. 12. Carbon footprint of the presented recycling process for polyester/elastane blends. A: Preliminary flow scheme of the developed recycling process and B: the results of the carbon footprint of the recycling process as function of the applied solid/liquid ratio with sensitivity analysis for solvent recovery (GVL) and breakdown of the different steps included in the process at a solid/liquid ratio of 0.05 kg textile/L solvent.

CRedit authorship contribution statement

Kim Phan: Investigation, Writing – original draft, Formal analysis. **Sibel Ügdüler:** Writing – review & editing. **Lies Harinck:** Methodology, Investigation. **Ruben Denolf:** Software, Validation. **Martijn Roosen:** Software, Validation, Writing – review & editing. **Galahad O'Rourke:** Methodology, Investigation. **Dirk De Vos:** Writing – review & editing. **Veronique Van Speybroeck:** Resources, Software, Writing – review & editing. **Karen De Clerck:** Resources, Writing – review & editing. **Steven De Meester:** Conceptualization, Resources, Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.resconrec.2023.106903](https://doi.org/10.1016/j.resconrec.2023.106903).

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