



Cite this: DOI: 10.1039/d6su00426a

Circular processing of post-consumer textile waste via selective PET depolymerisation and reprocessable sulfur composites

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The increasing accumulation of post-consumer textile waste poses major challenges for sustainable materials management, particularly for blended fabrics. In this work, an integrated valorisation strategy combining selective chemical recycling with subsequent material upcycling is presented. Poly(ethylene terephthalate) (PET) was selectively depolymerised via glycolysis, achieving conversions of up to 99.7% and yielding bis(2-hydroxyethyl)terephthalate (BHET) with high purity. The remaining textile fraction, consisting predominantly of cellulose (67%), was directly repurposed as a reinforcing filler material. Inverse vulcanised polymers based on elemental sulfur and waste cooking oil were synthesised and their mechanical properties were tailored through fibre incorporation and additional crosslinking using dicyclopentadiene (DCPD). The combined approach yielded tensile strengths of up to 0.9 MPa, which corresponds to an eight-fold increase relative to the neat polymer. Reprocessing experiments demonstrate vitrimer-like behaviour, with DCPD-containing composites retaining their mechanical performance over multiple cycles. E-factor analysis highlighted the benefit of the integrated valorisation concept, demonstrating improved resource efficiency through the combined utilisation of both PET and non-PET textile fractions.

Received 22nd July 2026
Accepted 21st September 2026

DOI: 10.1039/d6su00426a

rsc.li/rscsus

Sustainability spotlight

Post-consumer textile waste is a growing environmental challenge due to rising consumption and complexity of fibre blends that hinder recycling. Here, selective PET depolymerisation was combined with upcycling of the remaining textile fibres into reprocessable inverse-vulcanised composites from elemental sulfur and waste cooking oil. Elemental sulfur as a by-product of petroleum refining, is often stockpiled due to overproduction, while waste cooking oil is an abundant waste stream from gastronomy and the food industry. By valorising multiple waste streams within a single material platform, this approach enhances resource efficiency, reduces waste, and supports circularity. E-factor calculations showed that incorporating textile fibres improved overall material efficiency compared to PET depolymerisation alone. These findings contribute to UN Sustainable Development Goals 9 and 12.

1 Introduction

The global textile industry relies on large amounts of resources. In 2023, 125 million tons of synthetic and natural fibres were

consumed.¹ Less than 1% of these fibres were made from recycled textile waste,¹ about 60–80% of the produced fibres end up as textile waste, that is being landfilled, incinerated or disposed in the environment.² This waste causes environmental and social challenges in addition to the large resource gap for a circular textile economy.³ This underscores the necessity of effective recycling strategies.⁴ Waste fractions consisting of one material can be recycled mechanically. However, contaminations from additives like dyes or chemical finishers can complicate the mechanical recycling process. In addition, about 45% of the overall textile waste consists of blended textiles containing multiple polymer materials like cotton, polyester, nylon, acrylic, and spandex. These blends are typically incompatible with mechanical recycling.^{5,6}

For these waste streams, chemical recycling technologies can be an interesting alternative. Respective technologies focus on a (selective) cleavage of polymeric bonds to obtain molecular

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building blocks, which can be purified and processed into recycled polymers with virgin-like properties.⁷ Especially polyesters like poly(ethylene terephthalate) (PET), accounting for 54% of all the produced fibres, are suitable for chemical recycling.⁸ Using chemical recycling technologies by depolymerisation with a suitable nucleophilic solvent, PET can be degraded into its constituent monomers. Depending on the solvent employed different process parameters and product compositions can be achieved.⁹

Multiple depolymerisation techniques are known for PET obtained from packaging waste, which can theoretically be transferred to pure PET fibre waste.¹⁰ However, only a few depolymerisation processes for PET fibres from blended textile waste are reported in literature so far. L. Both *et al.* used an alkaline hydrolysis to depolymerise PET at 140 °C for 5 minutes to terephthalic acid (yield up to 69%) and monoethylene glycol.¹¹ Tanaka *et al.* reported a catalysed methanolysis process at 50 °C and 2 hours to obtain dimethyl terephthalate with a yield up to 94%.¹² Yang *et al.* have demonstrated a catalytic glycolysis process using ethylene glycol at a temperature of 160 °C and for 24 hours resulting in the monomer bis(2-hydroxyethyl)terephthalate (BHET) with a yield up to 43%.¹³ Shukla *et al.* developed an aminolysis process for the depolymerisation of PET staple fibres using ethanolamine in the presence of a metal acetate catalyst yielding 85% of monomer bis(2-hydroxyethyl)terephthalamide (BHETA).¹⁴

Despite most of the described procedures used polyester/cotton blended textiles as waste fractions, none of them reported the combined valorisation of both waste fractions.^{11–13} Consequently, a significant portion of the material remains underutilised. The residual textile fraction obtained after selective depolymerisation of PET represents a mixture of natural and synthetic fibres and additives, which is difficult to separate into its individual components. Instead, these residual fibres can be repurposed as reinforcing fillers in polymeric materials, providing an alternative strategy that enables the utilisation of otherwise low-value waste streams.

This approach can be realised in inverse vulcanised polymers, where elemental sulfur is incorporated as a major component *via* copolymerisation with unsaturated organic comonomers, yielding sulfur-rich polymeric materials.¹⁵ Elemental sulfur is generated in large quantities as a by-product of petroleum refining,^{16,17} with global production reaching approximately 85 million tons annually. However, only a fraction of this sulfur is consumed in established applications, primarily in the production of sulfuric acid, fertilisers, and rubber vulcanisation,^{18,19} resulting in the accumulation of substantial stockpiles worldwide.²⁰

In addition to their waste-derived feedstock basis, inverse vulcanised polymers can exhibit vitrimer-like behaviour due to the presence of dynamic sulfur–sulfur bonds within the polymer network. These bonds undergo reversible exchange reactions (S–S metathesis) at elevated temperatures, enabling reshaping, repair, and recycling of the material.^{21–23} To effectively utilise this excess sulfur, suitable organic comonomers are required. In this context, unsaturated vegetable oils are commonly employed due to their renewable origin and

comparatively low environmental impact.^{20,24–27} However, the use of virgin vegetable oils raises concerns regarding competition with food production, potentially affecting resource availability and cost. As an alternative, waste cooking oil (WCO), generated during domestic and industrial food preparation processes such as frying, represents an attractive feedstock that is widely available, low-cost and does not interfere with food supply chains.²⁸ Global WCO generation is estimated to reach approximately 50 Mt annually, with around 4 Mt produced in Europe alone.²⁹ At present, WCO is predominantly used as a feedstock for biodiesel production, with additional applications including thermal valorisation and incorporation into construction materials.^{30–32} Despite these applications, only about 28% of the generated WCO is effectively recycled, leaving a substantial fraction underutilised.²⁹ Improper disposal of these waste streams can lead to severe environmental issues, including soil and water contamination, blockage of sewage systems, and harmful effects on aquatic ecosystems.^{33,34} These challenges highlight the need for efficient valorisation strategies, positioning WCO as a promising comonomer in inverse vulcanisation for value-added polymer production.

However, inverse vulcanised polymers derived from vegetable oils typically exhibit limited mechanical performance, which restricts their applicability in mechanically demanding applications.³⁵ To overcome these limitations, reinforcement strategies based on filler incorporation have been explored.

Recent studies demonstrate that the incorporation of fillers can significantly enhance the mechanical performance of inverse vulcanised polymers derived from vegetable oils. For example, the addition of carbon fibres (20 wt%) to sulfur–canola oil polymers resulted in tensile strengths of up to 0.9 MPa.³⁶ Chalker *et al.* further demonstrated that the mechanical performance of wool fibre-reinforced composites based on similar systems is strongly governed by fibre orientation, with aligned architectures achieving Young's moduli of up to 1 MPa.³⁷ In addition, the incorporation of fillers such as carbon black, cellulose microfibrils, and nano clays has been shown to increase stiffness and tensile strength in inverse vulcanised polymers based on conventional petroleum-derived comonomers such as 1,3-diisopropenylbenzene (DIB), with the highest improvements generally observed at increased filler loadings, although often at the expense of ductility.³⁵

In this work, we present a holistic approach for the utilisation of post-consumer blended textile waste by combining selective chemical recycling with subsequent material upcycling. First, PET is selectively depolymerised from the textile mixture to recover BHET, while preserving the remaining fibrous fraction. BHET is purified and obtained in a sufficient quality for repolymerisation. The resulting residual textile fibres are subsequently repurposed as reinforcing fillers in sulfur-rich polymers prepared *via* inverse vulcanisation using elemental sulfur and WCO. The influence of fibre incorporation on the mechanical performance of the resulting composites is systematically investigated by varying the fibre loading and evaluating the tensile properties. Furthermore, the reprocessability of the materials is assessed to determine the effect of fibre reinforcement on the dynamic network behaviour of the polymers.

2 Results and discussion

2.1 Depolymerisation of PET

Post-consumer blended textile waste from old clothing with a PET content of 24% was shredded using two different apparatus: a cutting mill and a cutting machine. The waste was submitted to a PET depolymerisation *via* glycolysis following the known reaction conditions in Fig. 1.³⁸ The reaction was conducted in a batch reactor with an overhead stirrer and on a scale of 50 g waste material. The purity of BHET was analysed using HPLC, FTIR, and ¹H NMR spectroscopy (see SI). For the material shredded with the cutting machine a very high PET conversion of 99.7% can be observed, while for the material from the cutting mill only a moderate PET conversion of 43.1% was observed. In both cases a similar moderate yield (30–35%) and high purity of BHET was measured. PET oligomers were formed as a by-product, resulting in a mass balance of 77–99%. The difference in the PET conversion can be explained by an effect that was also observed visually during the reaction. The material shredded with the cutting mill contained many scattered threads and fibres that partly blocked the overhead stirrer and hindered proper mixing of the reaction solution. This effect was also observed with the material obtained using the cutting machine but to a lower extent. For the used reaction setup with a batch reactor, it therefore is favourable to have small defined particles. The low BHET yield is a consequence from the low PET content in the feedstock material. Despite the low PET proportion high amounts of solvent (ethylene glycol) are necessary to ensure proper mixing of the reaction. This unproportionally high ethylene glycol content complicates crystallisation of BHET and resulted in a low product recovery. This explains also the varying mass balance of the reactions future investigations will target the recycling of textile waste with higher PET content (>60%) including mechanical pre-treatment by pelletising fibre waste to potentially increase the quality of mixing and therefore the yield of BHET in the reaction.

2.2 Processing of residual material

For better processability in subsequent steps, the residual textile fraction obtained after glycolysis was submitted to two

different washing steps. It was first treated using a 10 wt% NaOH-solution for removal of impurities and residues from the depolymerisation reaction.³⁹ Afterwards it was bleached with 0.1 wt% NaOCl-solution, washed and dried for removal of dyes, resulting in a 70.9% recovery rate of the material throughout the washing steps.⁴⁰ It was not possible to completely remove the dyes but nevertheless a significant improvement for the colour was achieved. The cellulose content of the material was determined at 67.48% from the weight of the residual material. The composition of the remaining material was not further analysed but based on the composition of the waste stream, it can be assumed that it contains impurities from other polymer fractions or wool.

To further utilise the purified residual textile fraction was employed as mechanical reinforcements (textile fibre TF) in inverse vulcanised polymers synthesised from elemental sulfur and waste cooking oil. As previously mentioned, their mechanical properties are undesirable for many applications, therefore composites with different fibre loadings were prepared and subjected to tensile testing to evaluate the influence of fibre incorporation on their mechanical performance.

Fig. 2 shows representative photographs of the synthesised polymers before hot pressing (left) and SEM micrographs of the corresponding dog-bone specimens after hot pressing (right). In the photographs, the fibres can be clearly identified beneath the surface of the fibre-reinforced samples. Unlike the neat polymers, which exhibited a smooth and homogeneous surface, the fibre-containing samples showed a slightly wavy surface topography caused by the embedded textile fibres.

SEM images were taken from the specimen surface and cross-sections of the hot-pressed dog-bone samples. On the surface of all samples, sulfur crystallisation can be observed on an otherwise flat and dense polymer matrix. In the cross-sections, no voids or air pockets were visible, as expected after the hot-pressing process, indicating good consolidation of the material. Fibres could only occasionally be identified at fractured edges. In these regions, exposed fibres were observed, which appeared to be coated with polymer. No pronounced inhomogeneities or fibre agglomerates were detected within the analysed regions. However, due to the limited visibility of

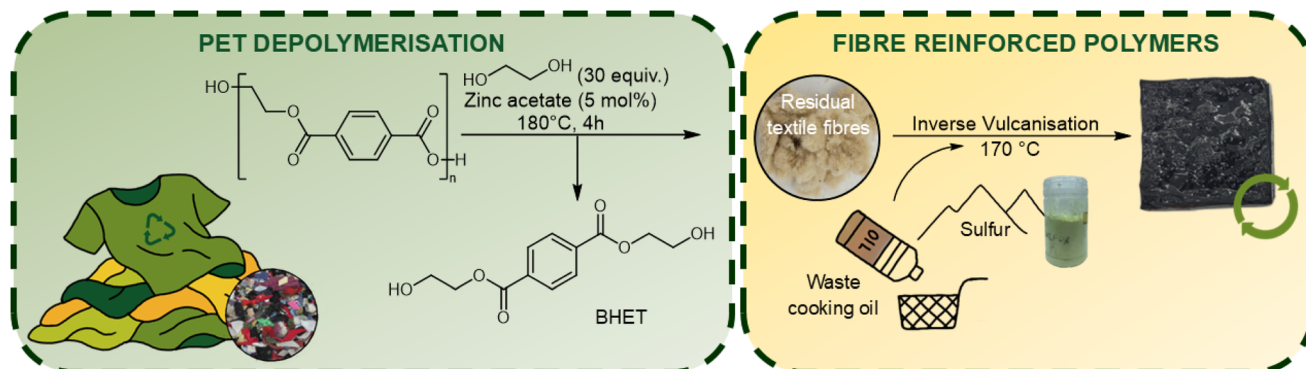


Fig. 1 Schematic illustration of PET depolymerisation followed by conversion into reprocessable and fibre reinforced sulfur polymers *via* inverse vulcanisation of waste cooking oil with elemental sulfur.

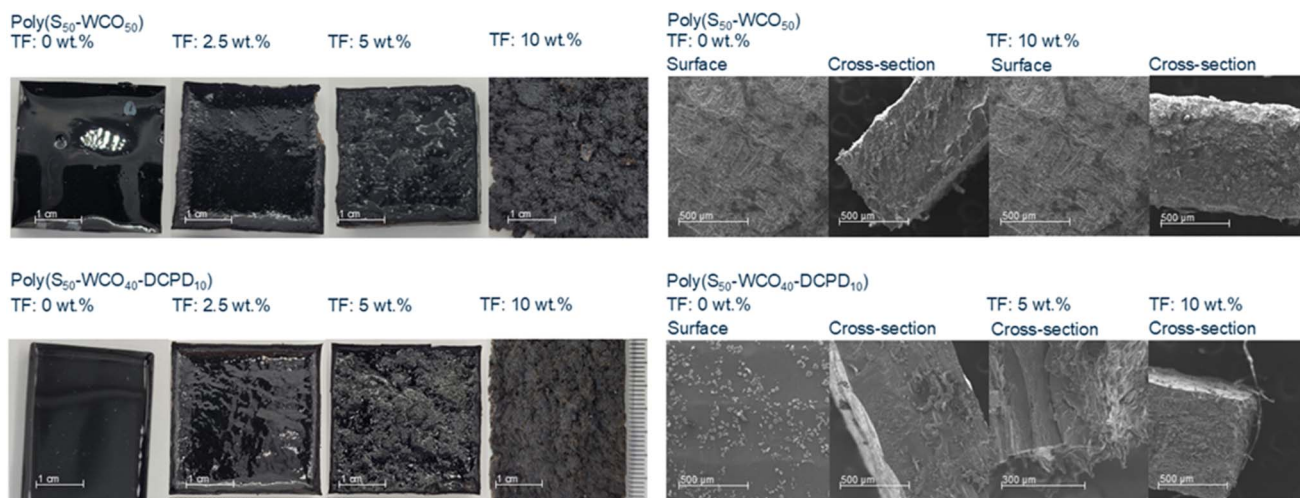


Fig. 2 Pictures of synthesised polymers before hot-pressing (left) and SEM images of selected polymers after hot pressing (right).

individual fibres, no detailed conclusions regarding the fibre distribution could be drawn from the SEM images.

The chemical and thermal properties of the synthesised polymers were characterised by FT-IR, DSC, and TGA. Due to the limited solubility of the inverse vulcanised polymer in common solvents such as chloroform and THF (approximately 30–40% solubility), ^1H NMR spectroscopy was only performed for the initial reference polymer to confirm the consumption of double bonds, as evidenced by the disappearance of alkene peaks at 5.20–5.44 ppm. FT-IR spectra confirmed the reaction of unsaturated bonds through the disappearance of characteristic alkene vibrations at 3007 cm^{-1} and 1655 cm^{-1} , while no significant spectral differences were observed upon fibre incorporation, indicating the absence of new chemical bond formation between fibres and the polymer matrix. DSC analysis revealed minor residual sulfur melting peaks, indicating incomplete sulfur incorporation, which is commonly observed in inverse vulcanised polymers based on vegetable oils at sulfur loadings around 50 wt%, due to the limited availability of reactive double bonds.⁴¹ The glass transition temperature (T_g) remained largely unaffected by fibre loading and was consistently observed at approximately $-33\text{ }^\circ\text{C}$. TGA measurements showed comparable degradation behaviour for all samples, with an initial mass loss occurring between approximately $200\text{ }^\circ\text{C}$ and $300\text{ }^\circ\text{C}$, followed by the main polymer decomposition between $300\text{ }^\circ\text{C}$ and $450\text{ }^\circ\text{C}$. Overall, the combined analyses indicate that fibre incorporation does not significantly alter the chemical structure of the polymer network, and that reinforcement is primarily governed by physical interactions rather than covalent bonding. FT-IR, DSC, and TGA data for all samples are provided in the SI.

To evaluate their mechanical performance, tensile tests were conducted on the synthesised materials. In terms of reproducibility, all polymer composites with fibre reinforcement were synthesised twice. The tensile strength, strain at break, and Young's modulus values shown in Fig. 3 represent the average

of three to four tensile tests performed for each material, with error bars indicating the standard deviation.

The neat polymer exhibits a low tensile strength of approximately 0.1 MPa, which is consistent with values reported for comparable inverse vulcanised systems.³⁷

The incorporation of textile fibres significantly increased tensile strength compared to the unreinforced polymer.

The reinforcement efficiency is strongly influenced by fibre dispersion and processing conditions. At a fibre loading of 2.5 wt%, pronounced batch-to-batch variability is observed, with tensile strength values ranging from 0.12 MPa to 0.26 MPa. This variability correlates with differences in fibre size and dispersion quality. The first batch was prepared using untreated textile fibres, whereas subsequent batches employed fibres that had been mechanically processed by cryogenic grinding (freezing in liquid nitrogen followed by milling) to reduce fibre size and improve dispersion within the polymer matrix. The processed fibres were used for all further experiments. Composites containing 5 wt% textile fibres exhibit more consistent mechanical behaviour, with tensile strengths of approximately 0.2 MPa, indicating improved reproducibility at intermediate filler contents. At a fibre loading of 10 wt%, the highest tensile strength values of up to 0.35 MPa are achieved. Nevertheless, increased fluctuation is again observed. This behaviour is attributed to limitations in mixing efficiency at high fibre contents. At this loading, the high fibre content hindered effective magnetic stirring. While the first batch was mixed using a magnetic stirrer with limited efficiency, the second batch was mixed more intensively by manual stirring, resulting in more homogeneous fibre dispersion and consequently higher tensile strength. The observed batch-to-batch variability was confirmed by statistical analysis (see SI). Consequently, although fibre reinforcement generally improved tensile strength compared with the unreinforced polymer, statistical comparisons revealed no significant differences between the individual fibre-reinforced formulations. The substantial batch-to-batch variability contributed to the overall

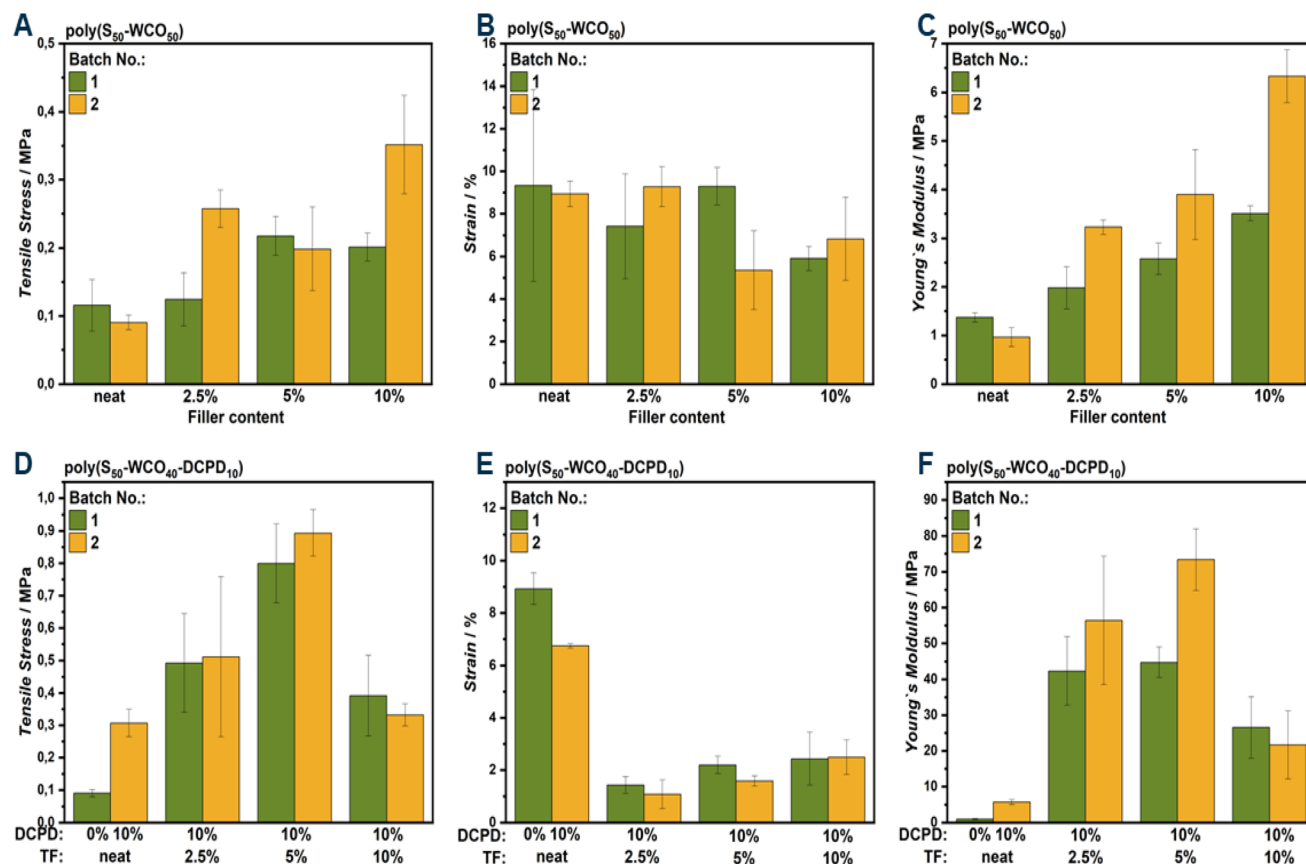


Fig. 3 Mechanical properties of poly(S₅₀-WCO₅₀) (top) and poly(S₅₀-WCO₄₀-DCPD₁₀) (bottom) with increasing fibre loadings. (A and D) Tensile strength, (B and E) strain at break, and (C and F) Young's modulus of the respective composite materials, obtained from tensile testing.

scatter of the data and have partially masked differences between fibre loadings.

Despite the observed reinforcement, the tensile strengths achieved in this study remain comparatively low to literature reports on fibre-reinforced inverse vulcanised polymers (Fig. 4). For instance, tensile strengths of up to 0.9 MPa have been reported for composites containing 20 wt% carbon fibres,³⁶ whereas wool-fibre-reinforced systems with fibre contents of 50 wt% reach values of approximately 0.5 MPa.³⁷ The lower tensile strengths observed here can primarily be attributed to the comparatively low fibre loadings (≤ 10 wt%), as well as the absence of strong interfacial interactions between the textile fibres and the sulfur-rich polymer matrix. Furthermore, differences in fibre properties, dispersion quality and processing strategies are likely to contribute to the reduced reinforcement efficiency.

In contrast to tensile strength, statistical analysis did not reveal a significant effect of fibre loading on strain at break, indicating that the ductility of the polymer matrix is preserved. The absence of a significant decrease in strain suggests relatively weak fibre-matrix interactions, with reinforcement governed predominantly by mechanical interlocking rather than strong interfacial bonding, in agreement with the findings from the chemical and thermal characterisation. Consequently, deformation prior to failure remains matrix-dominated, and the overall chain mobility is not substantially restricted by fibre addition.

Young's modulus, however, increases systematically with increasing fibre content, indicating an enhancement in stiffness. The modulus rises from approximately 1 MPa for the

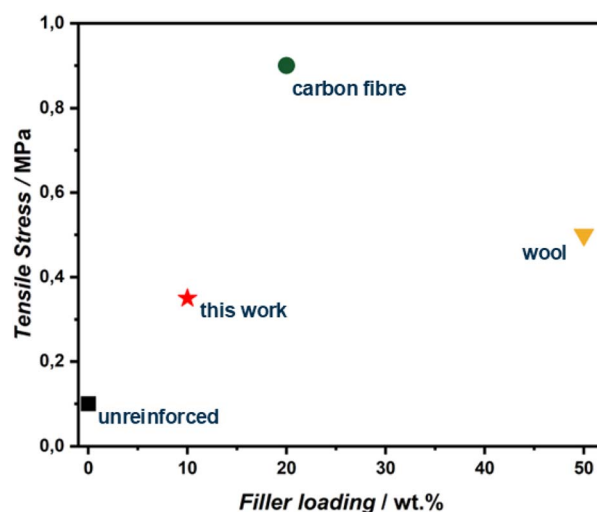


Fig. 4 Comparison of tensile strength as a function of filler loading for inverse vulcanised polymer systems based on 50 wt% sulfur and 50 wt% canola oil, compiled from literature and this work. Data points represent unreinforced polymer, fibre-reinforced systems (wool,³⁷ carbon fibre³⁶) and the composites investigated in this study.

unreinforced polymer to up to 6.3 MPa at a fibre loading of 10 wt%. This increase is attributed to the fibres acting as rigid inclusions within the polymer matrix, which restrict elastic deformation in the initial loading regime and result in a steeper stress-strain response. Given the limited mechanical performance of poly(S_{50} -WCO $_{50}$), even after reinforcement with textile fibres, an alternative strategy was explored to further enhance strength and stiffness. Dicyclopentadiene (DCPD) was introduced as a comonomer to increase the crosslink density of the polymer network, as previous studies have demonstrated DCPD can significantly improve the mechanical properties of sulfur-based polymers derived from vegetable oils.⁴²

Polymer systems containing 5 wt%, 10 wt% and 20 wt% DCPD were synthesised by partially replacing the waste cooking oil component while maintaining a constant sulfur content of 50 wt%. FT-IR spectra of the DCPD-containing polymers closely resemble that of the reference polymer, with the absence of characteristic alkene absorption bands confirming the consumption of double bonds from both the waste cooking oil and DCPD during polymerisation. No significant changes are observed in the fingerprint region, indicating that the overall chemical structure of the polymer backbone remains largely unchanged at the investigated DCPD contents. DSC analysis reveals a decreasing presence of residual sulfur with increasing DCPD content. A melting peak at approximately 120 °C is observed for poly(S_{50} -WCO $_{45}$ -DCPD $_5$), indicating incomplete sulfur incorporation, while no residual sulfur peaks are detected for polymers containing 10 wt% and 20 wt% DCPD, suggesting improved sulfur stabilisation at higher comonomer content. In addition, the glass transition temperature increases systematically with increasing DCPD fraction, with T_g values of approximately -33 °C for the reference polymer and -30 °C, -20 °C, and -15 °C for systems containing 5 wt%, 10 wt%, and 20 wt% DCPD, respectively. This trend is consistent with literature reports for comparable inverse vulcanised systems based on vegetable oils and DCPD, where increased crosslink density leads to elevated T_g values.⁴² TGA measurements show comparable degradation behaviour for all samples, exhibiting the characteristic two-step mass loss. No significant changes in the degradation profile are observed upon incorporation of DCPD. DSC and TGA thermograms are provided in SI.

The mechanical properties of the DCPD-modified polymers were evaluated by tensile testing and compared with those of the reference polymer and the textile fibre-reinforced systems. The formulation containing 20 wt% DCPD exhibited pronounced brittleness and fractured during sample preparation, preventing the fabrication of standardised dog bone specimens. Consequently, mechanical characterisation was restricted to polymers containing 5 wt% and 10 wt% DCPD.

The incorporation of DCPD leads to a pronounced increase in tensile strength. While the reference polymer exhibits a tensile strength of approximately 0.09 MPa, values of 0.16 MPa and 0.31 MPa were obtained for polymers containing 5 wt% and 10 wt% DCPD, respectively, corresponding to an increase of up to 240%. A similar trend is observed for Young's modulus, which increases from 1 MPa for the unmodified polymer to 5.8 MPa at 10 wt% DCPD, indicating substantial stiffening due

to increased crosslink density. The stiffness achieved at 10 wt% DCPD is comparable to that of composites reinforced with 10 wt% textile fibres. In contrast, ductility generally decreases with increasing DCPD content, with the strain at break reaching 6.7% at 10 wt% DCPD.

To evaluate whether the reinforcing effect of textile fibres can be transferred to a chemically crosslinked system, fibre reinforcement was investigated for poly(S_{50} -WCO $_{40}$ -DCPD $_{10}$). This composition was selected as it provides a balance between enhanced mechanical performance and sustainability while still allowing reliable specimen preparation and mechanical testing.

The incorporation of textile fibres leads to a pronounced increase in tensile strength at low to intermediate fibre loadings. At a fibre content of 2.5 wt%, the tensile strength increases from approximately 0.3 MPa to 0.5 MPa, indicating a moderate reinforcement effect. A maximum tensile strength of approximately 0.9 MPa is observed at 5 wt% fibre content, representing the highest tensile strength achieved within this study. However, statistical analysis revealed no significant difference between the 2.5 wt% and 5 wt% formulations. Consequently, while the results suggest that the optimum fibre loading lies within this intermediate loading range, the available data do not allow the exact optimum fibre content to be identified.

A further increase in fibre loading to 10 wt% results in a substantial decrease in tensile strength to 0.3 MPa, indicating that reinforcement efficiency is reduced at higher fibre contents. This behaviour suggests the presence of an optimal fibre loading. While lower fibre contents limit stress transfer due to insufficient reinforcement, excessive fibre incorporation promotes fibre-fibre interactions and agglomeration, leading to structural inhomogeneities and impaired load transfer within the composite.

In contrast to the non-reinforced poly(S_{50} -WCO $_{40}$ -DCPD $_{10}$) system, the strain at break decreases markedly with increasing fibre content. While the DCPD-containing matrix without fibres retains moderate ductility, fibre-reinforced composites exhibit significantly reduced elongation at break, with values of approximately 1–2.5%. This pronounced reduction indicates a transition towards brittleness upon fibre incorporation.

The loss of ductility can be attributed to the combined effects of chemical crosslinking and mechanical constraint. The incorporation of DCPD introduces a chemically crosslinked polymer network with reduced chain mobility, while the embedded textile fibres act as rigid inclusions that further restrict matrix deformation. The simultaneous presence of these two reinforcing mechanisms therefore limits the ability of the material to undergo plastic deformation prior to failure.

Young's modulus increases substantially with fibre loading, reflecting a significant enhancement in stiffness. The modulus rises sharply with increasing fibre content and reaches values of up to 70 MPa at a fibre loading of 5 wt%. This pronounced increase indicates efficient load transfer between the polymer matrix and the reinforcing fibres at intermediate loadings. Although the highest modulus values were observed at 5 wt% fibre content, no statistically significant difference was detected

between the 2.5 wt% and 5 wt% formulations, indicating comparable reinforcement efficiency within this loading range.

At 10 wt% fibre content, the modulus decreases relative to the maximum value, further supporting the conclusion that excessive fibre incorporation reduces reinforcement efficiency due to diminished dispersion and increased microstructural heterogeneity.

Additionally, dynamic mechanical analysis (DMA) was performed on selected polymers. However, all DCPD-containing polymer samples exhibited a strain at break that was too low to withstand the testing conditions. Consequently, the specimens fractured before oscillatory loading could be applied, preventing DMA measurements. Therefore, DMA measurements were only conducted on the unreinforced polymer and the polymer containing 10 wt% TF without DCPD.

Measurements were performed in triplicate and showed good reproducibility. During oscillatory loading, both materials exhibited stable viscoelastic behaviour with only minor changes in storage modulus and damping factor over time. The polymer containing 10 wt% TF showed a consistently higher storage modulus of approximately 7 MPa compared to the unreinforced polymer (approximately 2 MPa), indicating a substantial increase in stiffness upon fibre addition. In contrast, $\tan \delta$ values remained close to 0.1 for both materials, suggesting that fibre incorporation had little effect on the damping behaviour and that both materials retained a predominantly elastic response under cyclic loading.

Since all specimens fractured at the clamping region, the measurements mainly provide information on the viscoelastic response rather than fatigue performance. Representative DMA curves are shown in the SI.

Although fibre reinforcement significantly improved the tensile properties of the inverse vulcanised polymers, the achieved tensile strengths remain substantially below those reported for other fibre-reinforced vitrimer systems. For example, aligned flax fibre-reinforced composites based on the commercial imine-linked vitrimer Vitrimax T100™ have been reported to exhibit tensile strengths of approximately 121 MPa at a fibre volume fraction of 17.9%.⁴³ Likewise, carbon fibre-reinforced composites based on an aromatic disulfide-containing epoxy vitrimer have been reported to achieve tensile strengths of up to 334.5 MPa.⁴⁴ However, the substantially higher tensile strengths reported for these systems are likely a consequence of the use of high-performance reinforcements, high fibre loadings, and mechanically robust vitrimer

matrices specifically developed for structural composite applications. In contrast, the composites presented in this work were designed primarily to valorise post-consumer textile waste and other waste-derived feedstocks, while maintaining reprocessability through a sulfur-rich inverse vulcanised network. Consequently, although the achieved tensile strengths are substantially lower than those of high-performance vitrimer composites, they are comparable to those of commercially used rubberised cork sheet made from nitrile rubber (0.9–1.5 MPa), used for sealing, insulation, isolation, and vibration damping applications.⁴⁵ Therefore, the present materials may be more suitable for similar non-structural applications, whereas their current mechanical performance limits their applicability in load-bearing or structural components.

To further evaluate the composite properties for applications in *e.g.* constructions, their flammability was investigated as flame-retardant requirements are mandatory for insulation and damping materials. Sulfur and disulfide moieties have been reported as flame-retardant active elements in polymeric materials by both, gas- and condensed-phase flame retardant mechanisms.^{46–50} In the gas phase, sulfur-containing thermal decomposition products show flame inhibition caused by radical scavenging due to thiyl radicals, interrupting the combustion chain reactions in the flame by the conversion of $H^\bullet$ and $OH^\bullet$ radicals to the less reactive species H_2 and H_2O .^{47,48} In the condensed phase, the comparatively low bond dissociation energy of disulfide bonds promotes radical chain scission even at low fire temperatures, reducing the polymer's molecular weight and facilitating liquefaction and rapid dripping of the molten material away from the flame source, thereby removing heat and burnable material from the combustion zone.^{51,52}

To study the flammability, tests based on the UL94 vertical burning standard were performed on three specimen each of poly(S_{50} -WCO₅₀), poly(S_{50} -WCO₅)_TF₁₀, poly(S_{50} -WCO₄₀-DCPD₁₀)_TF₅, poly(S_{50} -WCO₄₀-DCPD₁₀)_TF₁₀ and on one specimen of poly(S_{50} -WCO₄₀-DCPD₁₀). The testing length from the lower sample edge to the mounting clamp was set to 60 mm. All specimens burned completely up to the clamp after the first flame treatment of 10 s – therefore no UL94 classification is reached. However, differences in fire behaviour can be observed. For a more detailed analysis, the average afterflame times following the 10 s flame treatment, measured until the flame self-extinguished as a result of complete combustion of the specimen, together with the average onset time of dripping and the average time at which the flame reached the clamp, are

Table 1 Summarised UL94 test results of representative sulfur composite samples. The tests were performed using a UL94 vertical burning test device

Sample	Av. after flame time [s]	Av. start of dripping [s]	Av. time until flame reached the clamp
poly(S_{50} -WCO ₅₀)	12 ± 2.1	5 ± 0.6	7 ± 1.0
poly(S_{50} -WCO ₄₀ -DCPD ₁₀)	9	11	4
poly(S_{50} -WCO ₅₀)_TF ₁₀	13 ± 3.5	10 ± 2.8	5 ± 0.6
poly(S_{50} -WCO ₄₀ -DCPD ₁₀)_TF ₅	15 ± 3.2	13 ± 1.2	5 ± 0.6
poly(S_{50} -WCO ₄₀ -DCPD ₁₀)_TF ₁₀	38 ± 4.2	19 ± 0.6	5 ± 0.0

summarised in Table 1. For more visual details refer to the SI and SI videos found in the data repository.

The neat poly(S_{50} -WCO $_{50}$) specimen without DCPD and without textile fibres burned very rapidly and exhibited pronounced dripping, which started 5 s after the start of the flame treatment. We assume this is caused by its high sulfur content, likely promoting rapid chain scission leading to low melt viscosity.

In contrast, both DCPD-containing as well as the fibre-reinforced specimens showed delayed dripping behaviour. For DCPD-modified specimens without fibres, a shorter afterflame time of 9 s was observed. We assume the reduced dripping caused by crosslinking allowed the flame to propagate more directly up the specimen toward the clamp, while the onset of dripping itself was delayed to 11 s due to the increased melt viscosity resulting from crosslinking. Fibre-reinforced specimens without DCPD exhibited faster initial flame propagation toward the clamp, presumably due to a wicking effect of the fibres. However, overall longer afterflame times and a later onset time of dripping of 10 s is observed, suggesting that the fibres reinforce the melt and restrict flow rather than exerting a chemical flame-retardant effect. Specimens combining both DCPD and 10 wt% fibres showed significantly longer afterflame times of 38 s and a substantially delayed onset of dripping of 19 s, indicating a synergistic effect in faster disruption of polymer degradation and restricting melt flow and dripping. Furthermore, it should be noted that the observed dripping did not correspond to the release of molten polymer droplets but rather to the detachment of approximately half of the specimen. Furthermore, char formation was visually observed on the detached, still-burning fragment, in contrast to the poly(S_{50} -WCO $_{50}$) specimens, where no comparable char layer was detected.

Char formation is usually beneficial for the flame retardancy of composites, as it protects the underlying material layers and limits heat transfer. Higher DCPD and fibre contents may thus improve UL94 classification if a stable char layer forms quickly enough to limit pyrolysis. However, it is known that sulfur-containing polymers on their own often do not exhibit sufficient flame-retardant properties and the tests are only passed by adding synergistic flame-retardant additives, such as phosphorus compounds – what we recommended for this type of materials.^{46,49}

2.3 Reprocessing of polymer composites

Inverse vulcanised polymers exhibit vitrimer-like behaviour, enabling reprocessing through dynamic sulfur–sulfur bond exchange. This dynamic network can be activated either thermally under elevated temperature and pressure or chemically *via* nucleophile-induced S–S exchange, for example using amines or phosphines to facilitate bond rearrangement under milder conditions.⁵³ However, the incorporation of reinforcing fibres may restrict this property. It is therefore essential to assess whether the reprocessability observed in the neat polymer is retained in fibre-reinforced composites and to what

extent the mechanical properties can be recovered upon repeated reprocessing.

Two representative systems were selected for this study: poly(S_{50} -WCO $_{50}$)_TF $_5$ and poly(S_{50} -WCO $_{40}$ -DCPD $_{10}$)_TF $_{10}$. Each material was subjected to five consecutive hot-pressing cycles. After each reprocessing step, tensile tests were performed to evaluate the evolution of mechanical properties. The tested specimens were subsequently manually fragmented into larger pieces and reused for the next reprocessing cycle (Fig. 5(A)). All hot-pressing and tensile testing steps within a given cycle were conducted on the same day, whereas subsequent cycles were carried out on consecutive days to minimise the influence of degradation.

The evolution of the mechanical properties of poly(S_{50} -WCO $_{50}$)_TF $_5$ over five reprocessing cycles is shown in Fig. 5. The tensile strength decreases gradually from approximately 0.20 MPa in the first cycle to around 0.13 MPa in the fifth cycle, indicating a moderate but continuous loss of load-bearing capacity. This decrease correlates with the slight increase in the crystalline sulfur peak observed in DSC thermograms (SI), suggesting partial sulfur recrystallisation and a reduction in effective crosslink density.

The strain at break exhibits a slight increase during intermediate reprocessing cycles, followed by a decrease in the final cycle. However, all values lie within relatively large error bars, indicating substantial variability. The initial increase in strain may be associated with network rearrangement and improved homogenisation during early reprocessing steps, whereas the subsequent decrease is likely related to the formation of more rigid sulfur-rich domains. Similarly, Young's modulus decreases after the first reprocessing cycle and remains at reduced values during intermediate cycles before increasing slightly in the final cycle. This behaviour reflects competing effects of network relaxation and gradual sulfur crystallisation during repeated thermal treatment.

In contrast, the DCPD-containing composite poly(S_{50} -WCO $_{40}$ -DCPD $_{10}$)_TF $_{10}$ exhibits a markedly different reprocessing behaviour. DSC measurements show no detectable sulfur melting peak even after multiple reprocessing cycles, indicating more stable sulfur incorporation within the network. TGA analysis reveals only minor variations, with a slight decrease in the relative contribution of the first degradation step, suggesting subtle structural rearrangements or post-curing effects during repeated processing.

These structural differences are reflected in the mechanical properties. The tensile strength increases continuously from approximately 0.33 MPa in the first cycle to 0.85 MPa after the fifth cycle, indicating a significant improvement in load-bearing capacity. A similar trend is observed for the strain at break, which increases from approximately 2.5% to around 5.2% across successive cycles, while Young's modulus remains approximately constant within experimental error. This combination of increased ductility and preserved stiffness suggests improved structural homogeneity rather than additional stiffening. The observed enhancement in mechanical performance is therefore attributed primarily to improved fibre dispersion, reduced structural inhomogeneities, and continued

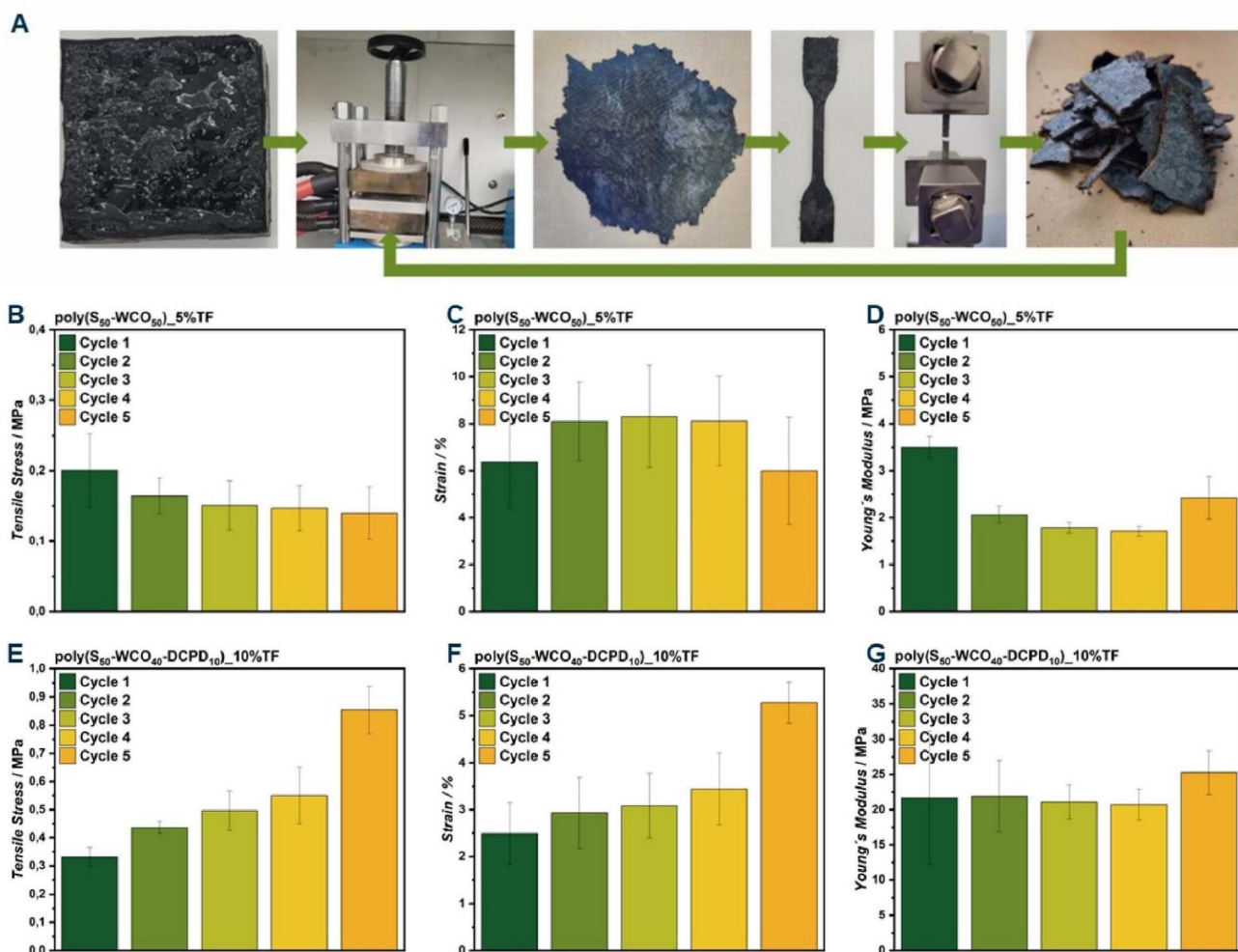


Fig. 5 (A) Reprocessing flowchart of fibre-reinforced inverse vulcanised polymers, including hot pressing, repeated tensile testing and fragmentation over five cycles. Evolution of mechanical properties upon repeated reprocessing cycles: tensile test results for poly(S₅₀-WCO₅₀)_TF₅: tensile strength (B), strain at break (C), Young's modulus (D), and for poly(S₅₀-WCO₄₀-DCPD₁₀)_TF₁₀: tensile strength (E), strain at break (F), and Young's modulus (G), measured over five consecutive reprocessing cycles. Each bar shows the average and standard deviation of 3–4 tensile test conducted for each polymer.

network rearrangement during repeated hot-pressing, rather than changes in the intrinsic stiffness of the polymer matrix.

2.4 Environmental metrics

Beyond the technical feasibility of PET depolymerisation and subsequent fibre upcycling, the sustainability of the overall process was evaluated using established green chemistry metrics. To this end, the environmental factor (E-factor) according to Sheldon⁶⁰ as well as the modified E-factor proposed by Thielemans *et al.*⁶¹ were determined for the depolymerisation step and the integrated textile-to-composite valorisation route. The corresponding calculations are provided in the SI, while an overview of the results is summarised in Table 2 and Fig. 6. The E-factor, which simply corresponds to the total amount of waste generated for a certain amount of product, remains one of the most widely used indicators for assessing material efficiency and quantifies the mass of waste generated per unit mass of product. In the context of PET

depolymerisation, Thielemans and coworkers further adapted this concept by accounting for industrial solvent recovery and by focusing on the material inputs that directly contribute to waste generation during chemical recycling. The depolymerisation process exhibited a Sheldon E-factor of 221 when all input streams were considered, and no solvent recovery was assumed. This value is substantially higher than E-factors typically reported for PET depolymerisation processes in the literature and is primarily caused by the large quantities of washing media, solvents and process water required for purification of the heterogeneous post-consumer textile waste stream. Similar observations have been reported for textile recycling processes, where downstream purification and separation operations rather than the depolymerisation reaction itself dominate the material footprint.⁵⁹ However, E-factors calculated without considering solvent recovery often provide a conservative and unrealistic representation of industrial operation. Assuming a solvent recovery rate of 90%, as

Table 2 Summary of comparable PET depolymerisation strategies with respective reaction parameters, E-factors calculated according to Thielemans and placement of the E-factor calculated for the present work

Catalyst	Temperature [°C]	Reaction time [min]	Yield [%]	Solvent/PET [w/w]	Cat/PET [w/w]	E-factor (Thielemans)	Ref.
TBD:MSA	180	120	91	6.46	0.61	0.95	54
CuNa/SiO ₂	210	360	100	65.83	0.83	13.41	55
PTSA	150	90	96.2	11.1	8.9	12.03	56
NaOH	80	20	95	50	2.5	9.13	57
ZSM-5	230	30	97.5	120	0.5	14.83	58
KOH	35	30	98.7	31.2	1.5	5.41	59
Zinc acetate	180	360	99.7	30	0.05	5.69	This work

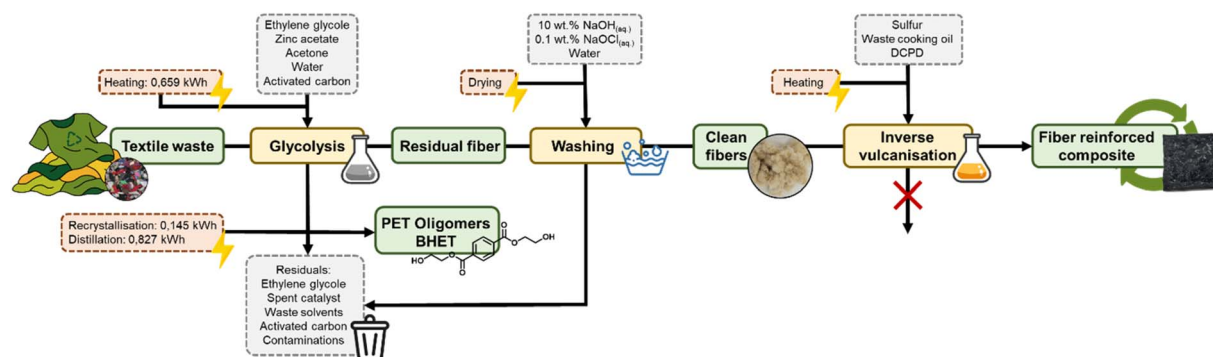


Fig. 6 Schematic representation of the material and process input and output streams considered for the E-factor calculation of the textile waste upcycling route. Inputs include textile waste, chemicals, solvents, catalysts, sulfur, comonomers, water, and energy, while outputs comprise the target products (clean fibres, BHET, and fibre-reinforced composites) as well as waste streams such as contaminated solvents, spent catalysts, activated carbon, process residues, and emissions. Energy consumption of some processes were recorded but are not further considered for the calculation of the E-factor of this process.

commonly applied in green chemistry assessments, the E-factor of the depolymerisation step decreases significantly to 26.⁶¹ While this value remains above the best-performing PET recycling systems reported in literature, it should be noted that most published studies evaluate depolymerisation of comparatively clean PET feedstocks with high fractions of polyesters and typically neglect extensive textile washing and purification steps. The modified E-factors suggested by Thielemans *et al.*⁶¹ for PET depolymerisation, typically range from approximately 0.95 to 14.8 (Table 2) for optimised PET depolymerisation systems as reported in literature, while the value calculated in this work of 5.69 is situated in between. In addition to its competitive E-factor, the present process achieved a PET conversion of 99.7%, which is among the highest values reported, while requiring the lowest catalyst loading of all compared systems. Furthermore, the solvent-to-PET ratio of 30 is lower than that reported for several literature methods and considerably lower than values of up to 120 reported for ZSM-5-catalysed depolymerisation. Although some literature procedures operate at lower temperatures or shorter reaction times, these conditions are frequently associated with higher catalyst loadings, larger solvent requirements, or more homogeneous PET feedstocks. Considering, that the present work addresses a more complex post-consumer blended textile waste stream and simultaneously preserves the non-PET fraction for further

utilisation, these values suggest a promising approach for further optimisation and adaption for pilot scale application. A more representative sustainability assessment is obtained when the complete valorisation concept is considered. In the present process, the residual fibre fraction generated after PET removal is not treated as waste but is instead incorporated into sulfur-based composites. When the recovered fibres are fully utilised in the composite manufacturing step, the overall E-factor decreases to 25 according to the classical Sheldon approach and to only 3.14 when solvent recovery is assumed (Table 3). Since the E-factor of inverse vulcanised polymers is very low (typically <1),^{20,62} these values show that the majority of the apparent waste generation originates from process auxiliaries rather than from losses of the textile feedstock itself. This observation highlights an important limitation of applying E-factor analysis solely to intermediate process steps. Considered on its own, the glycolysis process appears comparatively material intensive. However, when evaluated across the entire cascade utilisation concept, a substantial fraction of the initially generated “waste” is converted into useful products. Overall, the sustainability metrics underline the challenges and opportunities associated with chemical recycling of blended textile waste. The comparatively high E-factor of the current laboratory-scale process indicates significant potential for further optimisation, particularly through reduced water consumption, improved

Table 3 Summary of E-factors calculated according to Sheldon. Values were calculated solvent adjusted to represent a more industrial relevant process. Comparative values of other industrial processes are provided for better classification

	Total mass of waste [g]	Total mass of product [g]	E-factor (Sheldon)
Depolymerisation	8214.6	38.9	218
Depolymerisation (solvent adjusted)	932.5		25
Sulfur composite manufacturing	8214.6	297	28
Sulfur composite manufacturing (solvent adjusted)	932.5		3.14
Bulk chemicals			<1–5 (ref. 60)
Inverse vulcanisates			<1 (ref. 62)

washing procedures and more efficient solvent recycling. At the same time, the low integrated E-factor achieved for the final composite material demonstrates the benefit of combining chemical recycling with material upcycling. By valorising the PET and non-PET fractions of post-consumer textile waste, the presented approach increases overall resource efficiency and contributes towards a more circular utilisation of complex textile waste streams.

Even though the E-factor is a valuable and easily applicable metric for assessing the waste efficiency of chemical processes, particularly at the laboratory scale, it has several important limitations when used as a sustainability indicator. Its primary weakness is that it only quantifies the amount of waste generated per mass of product and therefore does not directly measure environmental impacts. While a lower E-factor generally indicates reduced waste generation, it does not provide evidence that the overall environmental footprint of a process is lower. For example, factors such as toxicity, resource depletion, greenhouse gas and other emissions (*e.g.* H₂S or SO₂), and ecological impacts are not considered. In addition, the E-factor does not account for energy consumption, which can represent a significant environmental burden, especially for energy-intensive chemical processes. Consequently, the E-factor is a useful screening tool for identifying waste-efficient synthetic routes and can encourage more resource-efficient practices, yet cannot provide a comprehensive assessment of sustainability and should be complemented by broader methodologies such as Life Cycle Assessment (LCA).

3 Conclusion

This work demonstrates an integrated valorisation approach for post-consumer textile waste by combining selective chemical recycling with subsequent material upcycling. PET was completely depolymerised *via* glycolysis within moderate reaction times of 4 hours, yielding up to 36% of BHET with high purity up to 99%. The BHET obtained shows a high quality and is suitable for repolymerisation and can therefore be reprocessed into recycled PET fibres to close the loop for PET-containing textile waste. However, the yield of the reaction remains low and needs to be optimised to enable an economically viable recycling process ideally while simultaneously reducing reaction time and temperature. Future work will therefore focus on textile waste streams with higher PET

content, enhanced catalyst development and improved process design. Despite the moderate BHET yield, the glycolysis process achieved a PET conversion of 99.7% and a modified E-factor of 5.69, placing it within the range reported for established PET depolymerisation processes. Furthermore, E-factor analysis demonstrated that utilising the recovered fibre fraction in sulfur-based composites substantially improved the overall material efficiency of the process, highlighting the benefits of the integrated valorisation concept.

As a result, the cellulose-rich residual textile fraction was not treated as waste but was instead incorporated into inverse vulcanised polymers derived almost entirely from waste-based feedstocks. The mechanical properties of these materials were systematically tailored through a combination of fibre reinforcement and chemical crosslinking.

The incorporation of textile waste fibres enables effective stiffening and strengthening of sulfur-rich polymers, while the addition of dicyclopentadiene increases crosslink density and enhances load-bearing capacity. The highest overall mechanical performance is achieved by combining both approaches. In poly(S₅₀-WCO₄₀-DCPD₁₀) reinforced with textile fibres, the complementary effects of chemical crosslinking and fibre reinforcement result in tensile strengths of up to approximately 0.9 MPa. These results highlight the potential of integrating multiple reinforcement mechanisms to optimise the performance of inverse vulcanised polymers while maintaining a high content of waste-derived components. The obtained mechanical strength is comparable to commercially used rubberised cork sheet, which is used for sealing, insulation, isolation and vibration dampening. The synthesised composite materials could be used in similar applications. However, the characteristic odor and high flammability must be considered.

Reprocessing experiments further demonstrate that the investigated materials retain structural integrity over multiple hot-pressing cycles. While fibre-reinforced polymers without DCPD show a moderate decrease in tensile strength upon repeated reprocessing, DCPD-containing composites even improve their mechanical performance. This behaviour is attributed to enhanced homogenisation, improved fibre dispersion and ongoing network rearrangement during repeated thermal processing, confirming the vitrimer-like reprocessability of these systems. This combination of fibre reinforcement and reprocessability is particularly noteworthy,

as conventional fibre-reinforced composites are typically based on thermoset matrices that are inherently difficult to recycle.

Despite these promising results, limitations related to fibre dispersion and mixing efficiency during synthesis affect reproducibility and reinforcement efficiency, particularly at higher fibre loadings. Future work should therefore focus on improved fibre size reduction and the implementation of advanced processing techniques, such as high-shear mixing, milling, calendaring, or screw extrusion, to improve fibre dispersion and achieve more homogeneous composites. The system could be further improved by compatibilising fibres with the polymer matrix, for example by partially functionalising the cellulose fraction. Addressing these challenges is expected to enable higher fibre contents and further improvements in mechanical performance, strengthening the potential of inverse vulcanised polymers as sustainable, reprocessable materials derived from industrial and consumer waste streams.

Author contributions

Z. S.: investigation, formal analysis, data curation, visualisation writing – original draft (polymer part), L. K.: investigation, formal analysis, data curation, writing – original draft (depolymerisation part), V. B.: formal analysis, data curation, visualisation writing – original draft (polymer part), D. V.: formal analysis, data curation, T. H.: supervision, resources, writing – review & editing, A. S. K. H.: supervision, resources, R. D.: supervision, resources, C.-C. H.: supervision, writing – review & editing, P. T.: supervision, writing – review & editing, C. W. S.: conceptualisation, supervision, data curation, visualisation, writing – review & editing.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Original data for this article, including SEM, ATR-FTIR, DSC, NMR, HPLC, TGA, rheological data and videos of the burning tests are available at Radar4Chem at DOI: <https://doi.org/10.22000/6b48x93fuz0tuw8d>. Supplementary information is available. See DOI: <https://doi.org/10.1039/d6su00426a>.

Acknowledgements

The work presented in this paper stems from the research conducted between 2022 and 2025 for an ongoing project titled “Fraunhofer Cluster Circular Plastics Economy CCPE”, funded by the “Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e. V.”. The authors would like to acknowledge Wenka Schweikert and Stefan Müller (both Fraunhofer ICT) for the measurements of the presented FT-IR data as well as Yvonne Kasimier (Fraunhofer ICT) for the reported HPLC measurements. The authors thank D. Zimmermann for the

support with rheological measurements. Support by the Helmholtz Association (43.33.11) is gratefully acknowledged.

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