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Deep eutectic solvent as a solution for polyester/cotton textile recycling☆

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ABSTRACT

Global fibre production has expanded rapidly, with polyester and cotton dominating, significantly contributing to textile waste and increasing demand for sustainable solutions. This study presents innovative method to recycle polyester/cotton (PET/CO) blends using hydrophobic deep eutectic solvents (DESs), eliminating the need for toxic chemicals while achieving high dissolution yields. PET was completely dissolved within 5 min, substantially outperforming state-of-the-art methods and facilitating the efficient and selective recovery of both components, PET (97%) and CO (100%). SEM imaging confirmed no morphological changes in cotton fibres after treatment. The thermal stability of the recovered materials was validated using DSC and TGA analyses, while ATR-FTIR spectroscopy indicated no chemical changes. Mechanical testing confirmed recovered cotton's tenacity and elongation are within expected ranges despite showing a decrease of 28% in tenacity and 34% in elongation. Hence, the proposed process provides an efficient and sustainable recycling solution for PET/CO blends, retaining both polymers in a condition similar to virgin materials used in textile manufacturing with minimal processing time.

1. Introduction

Fibre production worldwide has grown rapidly in the last two decades, from 58 million tonnes in 2000 to 124 million tonnes in 2023. If current trends continue, it is projected to reach 160 million tonnes by 2030 (Textile Exchange, 2024). Identifying man-made fibres as exclusive drivers of this growth, polyester (PET) fibre production governs the market with about 57 %. This is followed by cotton (CO) at 20 % (Textile Exchange, 2024), albeit stagnating. Simultaneously, recycled shares are as low as 1 % for CO, and while 12.5 % recycled material in the PET fibre market may seem higher, only 2 % thereof stem from textile recycling, opposed to 98 % coming from plastic bottles (Textile Exchange, 2024). Hence, this unbalanced expansion severely affects both humans and the environment (Textile Exchange, 2022). In response, the concept of sustainable fashion and the circular economy emerged, proclaiming to replace traditional linear industry practices (manufacture, use, and disposal) by reuse and recycling strategies, and concurrently extending fashion textiles' service life (Chen et al., 2021; EC, 2022, 2023; EEA, 2019).

Textiles are typically composed of fibre blends because they tend to perform better than textiles made from a single fibre type (De Silva et al., 2014; Jeihanipour et al., 2010; Zou et al., 2011). Within this category, polyester/cotton (PET/CO) blends are the most common (De Silva et al., 2014; Rosson et al., 2024; Zou et al., 2011), contributing to the complex composition of textile waste (Kählig et al., 2025), as it naturally reflects the diversity of fibre blends used in production (De Silva et al., 2014;

Abbreviations: ATR-FTIR, Attenuated total reflection Fourier transform infrared spectroscopy; CO, Cotton; DES, Deep eutectic solvent; DSC, Differential scanning calorimetry; LOM, Light optical microscopy; MCC, Microcrystalline cellulose; p, Statistical p-value; PET, Polyethylene terephthalate; SEM, Scanning electron microscopy; T_{deg}, Degradation temperature (°C); TGA, Thermal gravimetric analysis; DTG, Derivative thermogravimetry; Tm, Melting temperature (°C); $T_{max.deg}$, Maximum degradation peaks temperatures (°C); TPA, Terephthalic acid; X_c, Percent crystallinity (%); ΔHm, Melting enthalpy (J/g); ΔHm°, Theoretical melting enthalpy (J/g.

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Harmsen et al., 2021; Ling et al., 2019). Although PET and CO can be recycled individually, complex intermingling of PET and CO fibres makes recycling PET/CO blends really challenging (EC., 2022; Textile Exchange, 2022; Zou et al., 2011). Mechanical separation is ineffective for PET/CO blends (Harlin et al., 2024), and although chemical separation has been explored, it relies on harsh traditional solvents or concentrated acids that are environmentally unsustainable (Enking et al., 2025).

In literature, enzymatic processes are also described. These methods avoid using toxic solvents and require only moderate reaction conditions (approximately 50 °C, ambient pressure). The drawback is that one fibre polymer is hydrolysed into its constituent monomers, meaning only a fraction of the textiles can be directly re-incorporated into the production process as polymers (Piribauer et al., 2021).

Recently, ionic liquids (ILs), have drawn attention due to their low volatility and flammability. They have been demonstrated a potential replacement for harsh organic solvents in textile recycling (Tripathi et al., 2024). However, their high stability in water raises the question about long-term environmental impacts (Khandelwal et al., 2016). Deep eutectic solvents (DESs) were first introduced by Abbot in 2003 as analogues of and promising alternative to ILs (Abbott et al., 2002) as they offer reduced toxicity, good biodegradability (Clarke et al., 2018; Zhang et al., 2012), and easy preparation from readily available materials (Ijardar et al., 2022; Khandelwal et al., 2016). Their low ecological footprint, non-flammability, affordability, and minimal purification requirements have made DESs increasingly attractive for large-scale applications in both scientific and industrial sectors (Florindo et al., 2019a; Florindo et al., 2019b; Ijardar et al., 2022; Jin et al., 2020; Zhang et al., 2012). They have been applied widely as solvents, reaction media, catalysts, additives, lubricants, and materials across diverse fields ranging from pharmaceuticals to energy processes (Florindo et al., 2019a).

DESs are typically binary or ternary mixtures comprising at least one hydrogen bond donor and one acceptor, forming a eutectic with a melting point lower than that of individual components due to strong hydrogen bonding (Cherniakova et al., 2023; Clarke et al., 2018; Craveiro et al., 2016; Li and Row, 2016; Perna et al., 2020; Smith et al., 2014; Wang et al., 2022c). They are commonly prepared by heating and stirring components under an inert atmosphere until a homogeneous liquid forms (Hansen et al., 2021).

It was shown that DESs have great potential for use as a solvent for cellulose dissolution (Chen et al., 2019; Wang et al., 2022b; Zhang et al., 2020) and as catalysts for PET depolymerization, though their application in textile recycling remains underexplored.

Polymer recycling typically involves full or partial depolymerization or polymer dissolution. Full depolymerization breaks polymers into monomers requiring purification and repolymerization; partial depolymerization lowers molar mass. Dissolution, by preserving polymer chains, simplifies separation and reduces the need for repolymerization (Harlin et al., 2024). Efficient PET/CO separation without degrading either component remains a major challenge, with many approaches still under development (Rosson et al., 2024).

Several studies have explored DESs for PET/CO recycling. Liu et al. (2022) used a betaine-based DES to fully degrade PET into BHET (85 % yield) while recovering 95 % of CO intact. Choi & Choi (2019) applied a bio-based DES (ethylene glycol–choline chloride with 5 % NaOH) and microwaves to depolymerize PET in under 140 s. Wang et al. (2022a) used choline chloride p-toluene sulfonic acid DES to extract PET, microcrystalline cellulose and glucose from waste PET/CO fabrics. The study identified optimal separation conditions (110 °C, 10 min) that facilitated PET's efficient separation after the degraded CO component (Wang et al., 2022a).

Yang et al. (2024) designed a metal salt hydrate-based DES that selectively dissolved CO while preserving PET fibres.

Even though the existing research on the application of DESs in the recycling of PET/CO blends has demonstrated their efficiency, they were either dissolving or degrading cellulose or degrading PET, and no study has demonstrated the dissolution of PET from the blend. Most rely on hydrophilic DESs, though the 2015 introduction of hydrophobic DESs which show water-immiscibility, improved performance, and lower environmental impact marked a key advance compared to traditional organic solvents (Jin et al., 2020; Ribeiro et al., 2015).

This study examines the potential of hydrophobic DES composed of menthol and benzoic acid as a new and an environmentally friendly solvent. It investigates efficient separation of dissolving PET from PET/CO blends, preserving polymer integrity to advance sustainable textile recycling technologies. The recovered cotton can be directly used as high-quality secondary feedstock for yarn production, while the PET fraction is suitable for regranulation, fibre re-spinning, and various thermoplastic applications. This approach surpasses current state-of-the-art methods and opens pathways for diverse applications, ranging from apparel and nonwovens to filter media, insulation materials, packaging, and biorefinery processes.

2. Materials and methods

2.1. Materials

For this research, PET/CO post-business textile waste bedsheets were used. The material composition was labelled as 50 % CO and 50 % PET, but a test with m-cresol according to AATCC Test Method 20–2021 (AATCC, 2021) revealed a composition of 47 % CO and 53 % PET. Pure CO and PET fabrics were purchased to serve as reference materials. A commercial CO ring-spun yarn (Ne 26) was purchased for the tensile strength testing. DL-Menthol (≥ 95 % purity, food grade) and benzoic acid (≥ 99 % purity, synthesis grade), were provided by Sigma-Aldrich and used as received. Ethanol (96 % purity, reagent grade) was also supplied by Sigma-Aldrich and used without further purification.

2.2. Procedure for separation of PET and CO in textile using DES

PET/CO samples were cut into 5 x 5 cm2 (≈0.4 g) sections and treated without any further preparation. The menthol and benzoic acid DES at a molar ratio of 3:1 (Fig. 1a) was selected based on literature evidence indicating its low viscosity and hydrophobic properties (Aroso et al., 2016; Duarte et al., 2017; Florindo et al., 2019b). Supplementary Note 1 provides a comprehensive explanation for the choice of menthol and benzoic acid as the components of the solvent system. The mixture was heated to 50 °C with continuous stirring at a rate of 200 min^{-1} until a homogeneous, transparent liquid was obtained. The DES was stored in a desiccator at room temperature under ambient pressure to facilitate gradual cooling while preventing moisture absorption.

Two beakers of pure DES preheated to the desired reaction temperature were prepared for the dissolution experiments. Original PET/CO textile blend (Fig. 1b) was placed in the first beaker (treatment stage 1) and subsequently transferred to the second (treatment stage 2). The experimental conditions, including dwell time and temperature, were adjusted for each beaker, while a low stirring rate (80 min^{-1}) was maintained throughout. The optimal experimental parameters to achieve complete CO fibre release without any PET residues were determined. After the dissolution of the PET share, the undissolved CO fabrics were removed, rinsed with tap water and a 96 % ethanol solution, and then oven-dried as shown in Fig. 1c. The dissolved PET was allowed to cool, leading to precipitation, and was subsequently vacuum-filtered. The precipitate was then washed with 96 % ethanol to remove excess solvent and dried in an oven at 80 °C for 24 h.

2.3. Analytical methods

2.3.1. Scanning electron microscopy (SEM)

With the implementation of SEM, the microscale characteristics were analysed. To provide adequate electrical conductivity, an ultra-thin gold

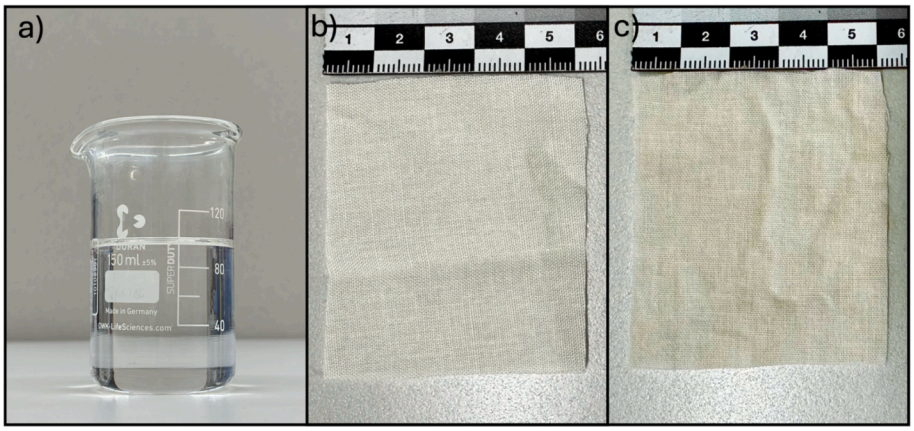

Fig. 1. a) DES composed of menthol and benzoic acid in molar ratio3:1 used in the process as a selective solvent. b) Untreated PET/CO textile blend (measuring scale in cm). c) PET-free CO textile after separation with DES (measuring scale in cm).

layer was applied twice to the textile samples prior to analysis using a COXEM SPT-20 (COXEM, South Korea) ion coater. Measurements were performed using a COXEM EM-30-plus (COXEM, South Korea) tabletop microscope in secondary electron detection mode, applying an acceleration voltage of 15–20 kV under high vacuum at room temperature on all coated samples.

2.3.2. Differential scanning calorimetry (DSC)

The textile waste samples were analysed using a TA Q2000 DSC instrument equipped with an autosampler (TA Instruments, USA). Both treated and untreated samples, along with reference samples, were placed in aluminium pans (4–5 mg). Characterisation was conducted during the initial heating cycle covering a temperature range of 30 °C to 500 °C under a nitrogen atmosphere (50 mL/min N_2) with a heating rate of 10 K/min. For PET samples, measurements included an additional cooling phase followed by a second heating cycle, covering a temperature range of 0 °C to 280 °C, while maintaining a consistent heating rate and nitrogen atmosphere. Specific points of interest were determined using the TA Universal Analysis Software.

The melting temperature (Tm) and melting enthalpy (ΔHm) were determined from the second heating cycle during thermal analysis while the degradation temperature (T_{deg}) was obtained from the first heating cycle. The percentage crystallinity (X_c) was determined from the second heating cycle using the formula in Eq. (1) (Tang et al., 2020):

$$Xc(\%) = 100 \cdot \frac{\Delta Hm}{\Delta H_m^{\circ}} \quad (1)$$

In this equation, ΔHm° represents the theoretical melting enthalpy for fully crystalline PET, which is taken conventionally as 140 J/g, based on values reported in the literature (Sichina, 2000; Tokuda et al., 2020; Van Kets et al., 2019; Viora et al., 2023).

2.3.3. Thermogravimetric analysis (TGA)

The degradation behaviour and potential polymer changes were assessed using a TGA Q500 instrument (TA Instruments, USA). Textile samples (5–6 mg) were heated from 35 °C to 500 °C in a nitrogen atmosphere (90 mL/min N_2) with a temperature ramp of 10 K/min and placed in a ceramic crucible. The decomposition behaviour was analysed using TA Universal analysis software. The temperatures at maximum degradation peaks ($T_{max.deg}$), and the percentages of residual mass were determined.

2.3.4. Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR)

Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) was used to analyse molecular bond changes and similarities in the samples, allowing the detection of degradation in the recovered materials. The analysis was performed using a Bruker Vertex 70 FTIR spectrometer with a scan resolution of 4 cm^{-1}, covering the spectral range of 600–4000 cm^{-1}. Each sample was scanned 64 times, and the resulting data were normalised for consistency.

2.3.5. Mechanical properties via single fibre tearing tests

Fibre tenacity and elongation at break were assessed as key parameters reflecting mechanical stability. For this purpose, stress–strain curves were recorded in a single-fibre tearing test setup employing a Vibrodyn 400 and Vibroskop 400 (Lenzing Instruments, Gampern, Austria) where the latter was used for determining the fibre titre as the standard normalisation parameter for the recorded tensile force. Using the associated VPNX software during measurement, parameter values were recorded and normalised automatically. This integration enables the inclusion of the titre in the evaluation process, in accordance with ISO 5079:2020 standards.

A commercial CO ring-spun yarn (Ne 26) was used as standard test material to evaluate CO fibre properties in the virgin material compared to the material exposed to the conditions of the separation process. For the test, fibres were individualised from the untreated reference and from the treated yarn specimen and the test was repeated 30 times for each sample to obtain mean values. The tests were performed under controlled ambient conditions, with a testing speed of 20 mm/min and using a gauge length of 10 mm. The measured properties included linear density also known as titre (dtex), elongation at break (%), elastic modulus (cN/tex), and tensile strength expressed as tenacity for fibres (cN/tex).

3. Results and discussion

3.1. Effectiveness of polyester dissolution

To achieve efficient separation of the PET/CO blend using menthol and benzoic acid DES, various process parameters were evaluated, including three different temperatures and five treatment durations. The effectiveness of PET separation from CO was verified by calculating the yield of PET dissolution. This calculation as shown in Equation (2) was based on the difference in the PET/CO sample's dry mass before and after treatment, with consideration given to the determined PET content in the PET/CO textile. The PET content in the PET/CO blend was determined through the m-cresol test, following AATCC Test Method 20–2021 (AATCC, 2021), and was calculated to be 53.38 %.

$$\text{Yield}(\%) = \frac{100}{0.5338} \cdot \left(1 - \frac{\text{mass after treatment}}{\text{mass before treatment}}\right) \quad (2)$$

The experimental design began with an investigation of the temperature required for PET dissolution. After observing that the reaction was initiated at 200 °C, subsequent measurements were performed at temperatures above this threshold. The results indicate reliable conditions for separating PET/CO blends, where the dissolution yield of PET reaches 100 % at 216 °C with a treatment duration of 3 min in treatment stage 1 and an additional 2 min in stage 2. At temperatures below 216 °C, dissolution was found incomplete, with a maximum yield of just above 99 % observed after 15 min of treatment at 212 °C (Fig. 2a). The exact mass that was recovered after full separation was found to be reaching 100 % for CO and 97 % of the PET polymer could be recovered after filtration. The minor losses of PET material were likely due to imperfections occurring during PET post-processing.

Fig. 2b illustrates the effect of the solid–liquid ratio on the separation of PET/CO blends using DES. The data demonstrate that as the solid–liquid ratio increases, the PET dissolution yield also increases. This trend is logical because a higher solvent volume facilitates greater interaction with the fabric. The results indicate that the minimum solid–liquid ratio at which a 100 % PET dissolution yield is achieved, is around 1:30. However, all tested solid–liquid ratios resulted in high PET dissolution yields, exceeding 96 %.

Applying the optimized parameter set, PET was effectively separated from the CO fabric using a DES. Following separation, the PET was left to precipitate before being vacuum-filtered and washed with ethanol and water to produce recovered PET. The morphology, chemical structure, and thermal properties of the separated CO fabric and recovered PET were examined using SEM, TGA, DSC, and ATR-FTIR. Additionally, the mechanical properties of the fibres within the recovered CO fabric were assessed via single-fibre tearing tests. Furthermore, a preliminary study was conducted regarding the reuse of the solvent and is presented in Supplementary Note 4.

3.2. Effects of separation treatment on polyester and cotton

3.2.1. Textile analysis using scanning electron microscopy (SEM)

To gain insight into the success of the separation of PET from PET/CO blends, both the original and treated fabrics were analysed by SEM. As shown in Fig. 3a, the two different fibre types are clearly distinct. Due to their formation by melt-extrusion, standard PET fibres typically exhibit smooth and uniform surfaces. As shown, PET fibres appear as straight, cylindrical fibres without natural twists, unlike CO fibres (Choi & Choi, 2019; Liu et al., 2022; Wang et al., 2022a; Yang et al., 2024). CO fibres typically display a rough, irregular surface with an inherent ribbon-like twist, characteristic of their natural cellulose superstructure (Liu et al., 2022; Yu, 2015). Fig. 3b confirms the importance of SEM analysis as it illustrates incomplete dissolution of PET when using an inadequate parameter set. In contrast, Fig. 3c shows that upon completion of the process after parameter optimization, only the CO fibres remain in the fabric. Naturally, SEM does not allow for a quantitative assessment of the respective fibre material and will only provide for qualitative distinction of the uppermost layer of fibres. However, the represented images (Fig. 3) offer compelling evidence of the effectiveness of the separation process facilitated by DES, resulting in the recovery of CO fibres that maintain structural integrity and exhibit no observable surface damage.

One other interesting observation is the fact that after the removal of the PET share the yarn structure is loosened compared to the non-treated material, which is presented in Supplementary Note 5. This can be considered beneficial when applying mechanical tearing lines to retrieve individual fibres.

3.2.2. Investigation of thermal behaviour by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA)

The thermal stability of all samples was examined through duplicate DSC and TGA measurements and compared to the reference samples. These methods enable the identification of thermal degradation phenomena such as polymer chain scission, or morphological changes potentially induced during the dissolution process.

The DSC thermograms of the treated PET/CO fabric, recovered CO after PET/CO treatment as well as the reference materials are presented in Fig. 4a. A clear distinction between treated and untreated PET/CO samples provides evidence of successful separation due to the lack of the PET melt peak. The untreated PET/CO sample exhibited four peaks, two of which correspond to CO fibres and two to PET (Manich et al., 2020). Comparison with the untreated CO sample reveals that the peaks of the untreated PET/CO fabric at 87 °C and 351 °C are associated with the CO fibres (Manich et al., 2020; Wu et al., 2023). Specifically, the endothermic peak at 87 °C is attributed to the elimination of bound water, whereas the peak at 351 °C represents the onset thermal decomposition of CO (Trivedi et al., 2015). It is evident that the recovered CO from the treated PET/CO blend, after DES treatment, no longer exhibits the peaks characteristic of PET, confirming the successful separation of PET and CO fibres. Additionally, Fig. S1 shows that treatment below 216 °C results in incomplete separation, as indicated by the presence of the PET

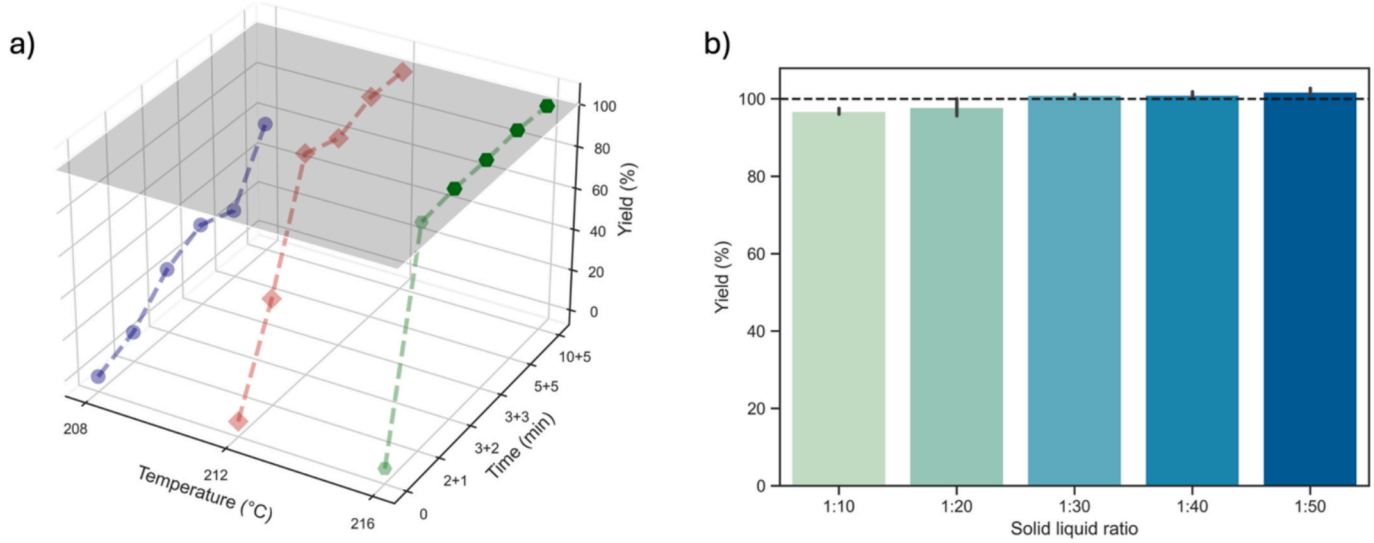

Fig. 2. a) PET dissolution results. The experimental results indicate that a total treatment duration of 5 min is sufficient for the effective separation of PET/CO blends using DES, with the grey plane at 100 % yield serving as the reference level. b) Effect of the solid–liquid ratio on the yield of dissolution of PET from PET/CO blends.

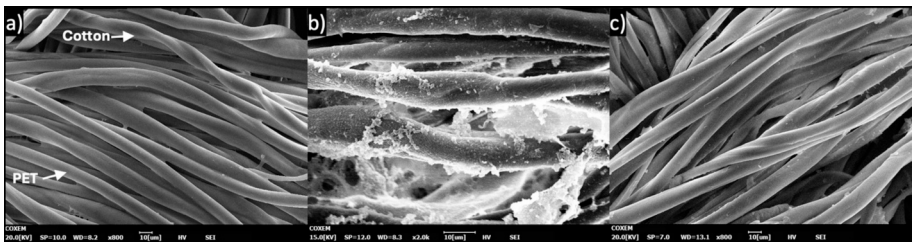

Fig. 3. SEM images of a) the untreated PET/CO fabric, b) incomplete dissolution of PET from the PET/CO fabric, c) recovered CO fabric share with no visible PET residues after DES treatment.

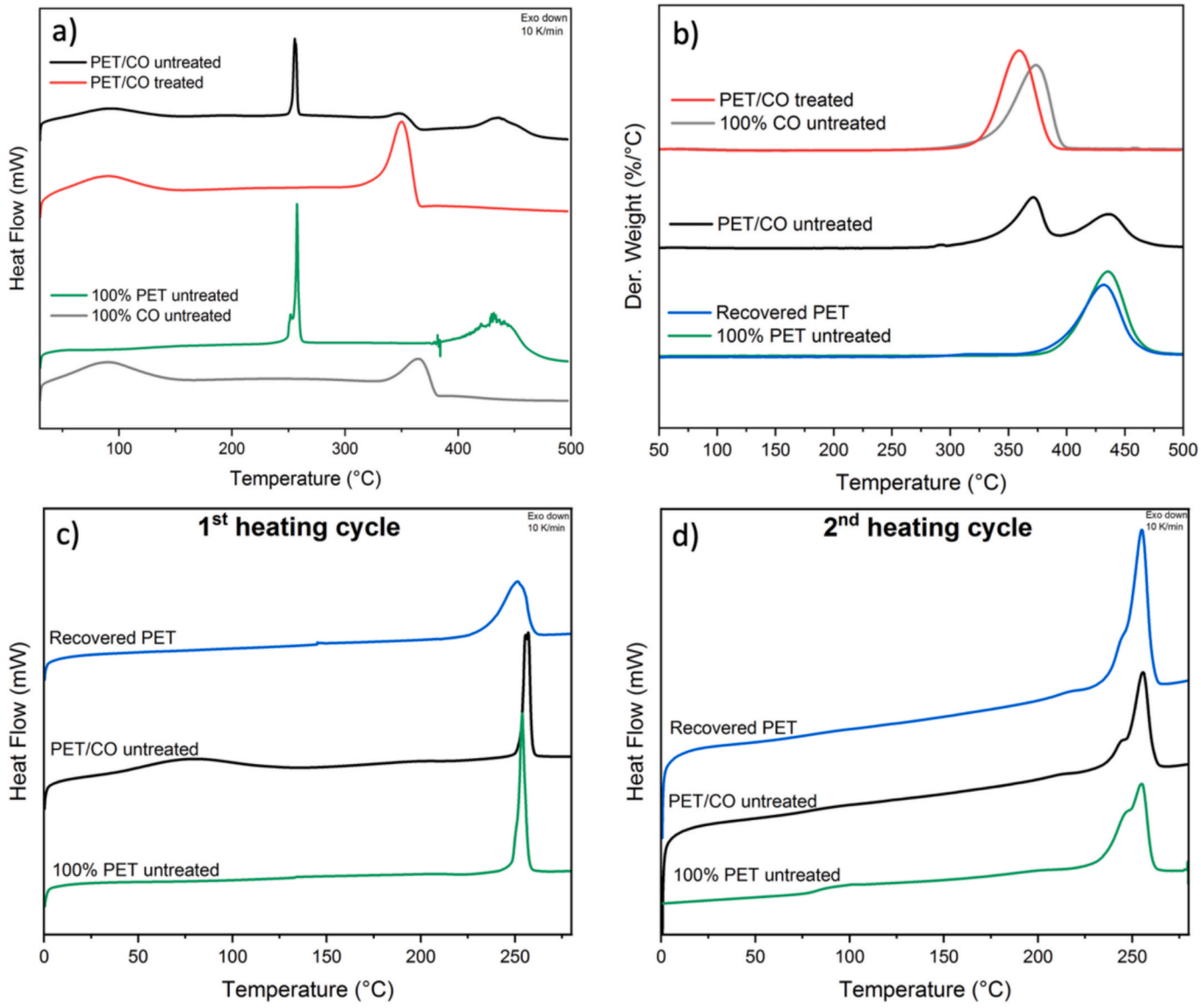

Fig. 4. Thermal analysis. a) DSC curves of first heating cycle of untreated PET/CO fabric and recovered CO fabrics after DES treatment (PET/CO treated) and of untreated reference PET and CO. b) DTG curves of the untreated PET/CO fabric, recovered CO and PET, as well as their respective reference materials. c) DSC curves of first heating cycle of recovered PET, untreated PET, and untreated PET/CO fabric. d) DSC curves of second heating cycle of recovered PET, untreated PET, and untreated PET/CO fabric.

related melting peak. The recovered CO exhibited no significant change in thermal stability, as indicated by the degradation peak appearing at the same temperature as that observed in the untreated PET/CO blend (Isola et al., 2024; Trivedi et al., 2015).

Figs. 4c and 4d display the DSC thermograms of PET samples. The thermograms from the first heating run are presented in Fig. 4c, providing clear evidence of the successful separation of PET and CO. In particular, the water evaporation peak associated with CO is absent in the recovered PET, in contrast to the untreated PET/CO blend.

Fig. 4d presents the DSC thermograms showing the melting peaks obtained from the second heating run. This phase of analysis was chosen because it reflects the thermal properties of PET after eliminating its

prior thermal history (Šudomová et al., 2023). The thermograms provide a clear comparison between the untreated PET/CO blend, the reference untreated PET, and the recovered PET. The recovered PET has a melting peak at 255 °C, that is identical to the untreated PET/CO sample, indicating that the dissolution and precipitation procedure had no significant impact on its thermal properties. This observation corresponds with findings reported in the literature (Boschmeier et al., 2023; Celik et al., 2022). The crystallinity of the recovered PET, calculated from the second heating run, was determined to be 40 %, which falls within the reported literature range for PET (25 – 40 %) (Celik et al., 2022). Furthermore, the recycled PET demonstrates higher crystallinity compared to the original PET, which has a crystallinity of 35 %. This increase in crystallinity may be attributed to structural reorganization during the dissolution and precipitation process, which allows polymer chains to adopt more ordered conformations due to reduced entanglement and increased mobility (Viora et al., 2023).

The TGA results, presented as DTG (derivative thermogravimetric) curves, provide a clear comparison of the thermal behaviour of recovered PET and CO, untreated PET/CO blend, and reference materials (Fig. 4b). The PET/CO fabric exhibits two distinct weight loss events between 300 °C and 500 °C, corresponding to the degradation of its PET and CO components. The initial weight loss, associated with the cellulosic content, occurs at a maximum degradation peak temperature of 371 °C, slightly higher than that of recovered CO (cf. PET/CO treated in Fig. 4b) at 365 °C (Manich et al., 2020). Although the reference CO material (cf. 100 % CO untreated) has a degradation peak at 374 °C, differences in material origin limit direct comparability (Table S1).

Consequently, it is evident that DES separation process had minimal impact on the thermal stability of the CO. These results align with values reported in the literature, confirming that the thermal stability of the recovered CO falls within the expected range for CO materials (Choi and Choi, 2019; Isola et al., 2024; Trivedi et al., 2015; Yousef et al., 2020). The findings highlight the effectiveness of the DES treatment in preserving the thermal properties of the recovered CO while achieving successful separation.

The DTG analyses furthermore demonstrate that the thermal properties of recovered PET are effectively maintained following DES treatment, showing close alignment with those of untreated reference PET and the untreated PET/CO blend (Fig. 4b). The degradation peak for all PET samples was consistently recorded at around 438 °C. The residual mass after decomposition, approximately 13.0 % for all PET samples, is consistent with values reported in the literature, further supporting the preservation of PET's thermal characteristics (Alhulaybi & Dubdub, 2023; Wang et al., 2022a).

Moreover, the absence of CO degradation peaks in the PET thermogram and PET degradation peaks in the CO thermogram confirms the efficiency of the separation process. This means that not only thermal stability of recovered materials is maintained but also precise and efficient separation of the polymer components is achieved. The findings from DSC and TGA of samples tested in this work are summarized and presented in Table S1.

3.2.3. Spectral investigation by attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR)

ATR-FTIR spectroscopy was employed to characterise the untreated PET/CO blend, recovered PET and CO and their reference materials regarding any possible changes in (macro-)molecular structure and as a complementary method to evaluate the quality of recovered PET and CO against their respective reference materials. Fig. 5a clearly demonstrates the successful separation of the PET/CO blend using DES. The untreated and treated PET/CO blends exhibit differences in PET-specific bonds, which corresponds to those observed in the reference PET. The spectrum of the treated PET/CO blend exclusively exhibits CO-specific peaks, with all PET related peaks absent. This confirms that the dissolution process successfully separated PET, leaving behind only CO.

Fig. 5b shows the chemical similarities between the recovered PET

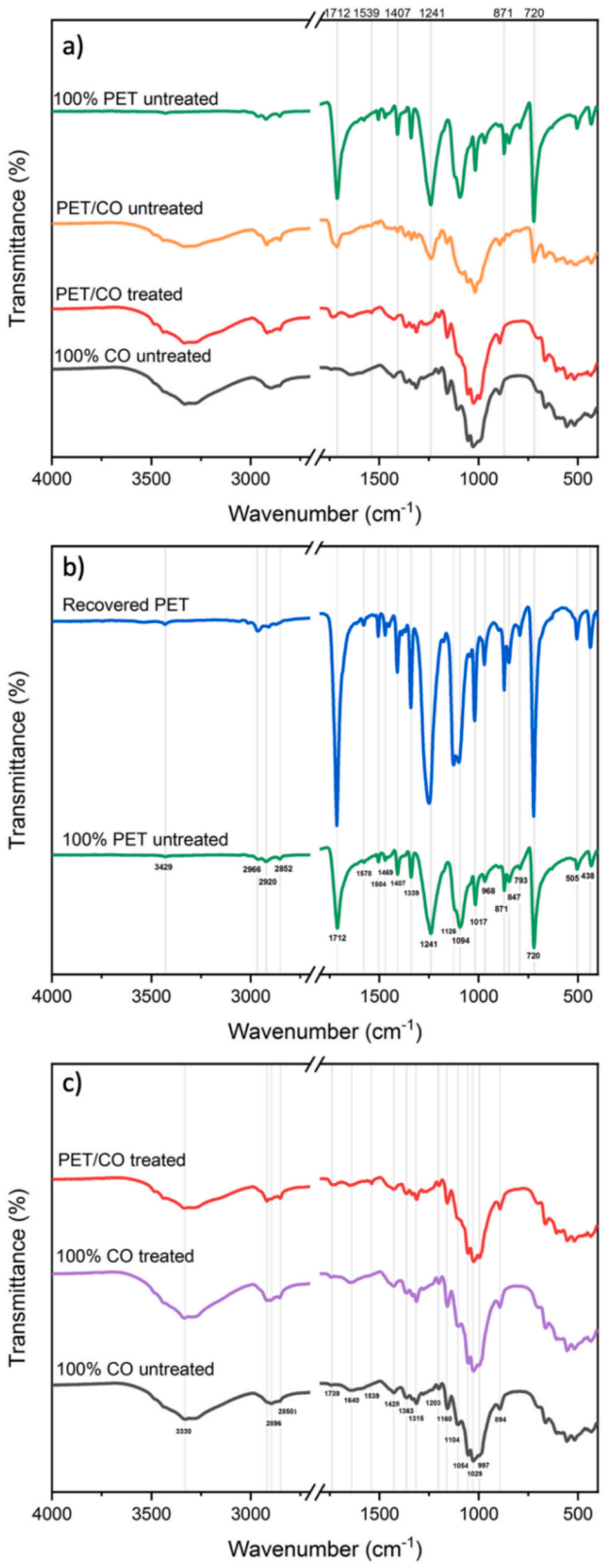

Fig. 5. FTIR spectra of a) untreated and DES-treated PET/CO blend and untreated materials of PET and CO as a reference, b) recovered PET after DES treatment of PET/CO blend and reference PET untreated material, c) untreated CO reference material, DES-treated CO reference material, and recovered CO from PET/CO blend treated.

and untreated PET. The prominent peaks between 1700 cm^{-1} and 600 cm^{-1} represent the original signals of PET, including the absorption peak at 1712 cm^{-1} which corresponds to characteristic stretching vibrations of the C=O bond (Maradini et al., 2021; Pitchai et al., 2014). The peaks at 1094 cm^{-1} and 1241 cm^{-1} are attributed to the stretching vibrations of the C-O-C bond (Wang et al., 2022a). Peaks observed at 871 cm^{-1} and 720 cm^{-1} are related to the polar ester groups and the vibration of C–H in benzene (Wang et al., 2022a). Furthermore, peaks in the 2850–3000 cm^{-1} range are attributed to C–H bond stretching (Badia et al., 2012; Maradini et al., 2021). The two spectra (Fig. 5b) exhibit a high degree of similarity, with no additional peaks in the degradation product spectral region (further justification concerning the regions of degradation products in Supplementary Note 2), suggesting the preservation of the original chemical structure. This indicates that the DES dissolution process is suitable for treating PET/CO blends, especially given that the observed peaks closely align with those of untreated PET and the literature (Peets et al., 2019; Valh et al., 2020).

As shown in Fig. 5c, untreated pure CO, pure CO treated in DES, and PET/CO sample treated in DES all exhibit similar spectral characteristics of CO. A broad, intense group of O–H stretching bands, spanning 3660 to 3000 cm^{-1}, is observed in all samples. Upon closer examination, this grouping consists of a shoulder band near 3437 cm^{-1}, along with two more dominant bands centred at approximately 3330 and 3288 cm^{-1} (Cintrón & Hinchliffe, 2015). Peaks between 2800 and 2980 cm^{-1} are associated with C–H stretching vibrations in cellulose and hemicellulose; typical of CO fabric (Portella et al., 2016; Wang et al., 2022a). In the spectra of treated pure CO and treated PET/CO blend, stronger peaks at 2918 and 2849 cm^{-1} are observed, but no shift in the peaks occurred. Earlier research has identified strong peaks in the 1700 to 1580 cm^{-1} spectral region as markers of water absorption in CO fibres. In all our samples, a broad, moderately intense band was detected at 1640 cm^{-1}. The DES-treated PET/CO sample exhibits a new peak at 1539 cm^{-1}, which is absent in both the DES-treated and untreated pure CO samples. Particularly, this peak is also present in the untreated PET/CO blend (Fig. 5a), suggesting its association with the PET/CO composite structure. However, as the peak is not observed in the untreated PET or the recovered PET samples, it appears to be specific to the PET/CO blend rather than PET alone. Peaks between 1500 and 1200 cm^{-1} correspond to the fingerprint region associated with C–H, O–H, C-O, and C-O-C vibrations, which are characteristic of cellulose and consistent with the literature (Abd El-Hady et al., 2022; Chung et al., 2004; Nandiyanto et al., 2022; Portella et al., 2016).

The consistent presence of these characteristic peaks across all CO samples indicates that the chemical structure of the CO material remained intact throughout the DES treatment.

3.2.4. *Mechanical properties of recovered cotton fibre*

To determine the suitability of recovered CO fibres for reuse, it is essential to evaluate their mechanical properties, particularly the strength of the fibres, after the treatment process (Hebert et al., 1995). According to the literature, CO fibres exhibit a wide range of mechanical properties depending on the type and source of the fibre. The tenacity of untreated CO fibres typically ranges from 15 to 43 cN/tex (Elmogahzy & Farag, 2018; Frydrych, 1995; Vigotex, 2023), while elongation varies between 3 and 10 % (Elmogahzy & Farag, 2018; Frydrych, 1995; Pennas et al., 2019). Supplementary Note 3 discusses the practical methodology using a suitable reference material. Measurements revealed a reduction in both tenacity and elongation for treated CO compared to untreated CO, as shown in Fig. 6. This decline aligns with expectations, as recycled CO generally exhibits lower tenacity and elongation due to the processing and degradation of fibres to a certain extent (Yuksekkaya et al., 2016).

A statistical comparison with the tenacity and elongation ranges of untreated CO reveals that the mean tenacity of the DES-treated CO, calculated as 17.98 cN/tex from measurement series, passes one-sample t-tests for a mean tenacity of at least 15 cN/tex ($p = 0.99689$) at a

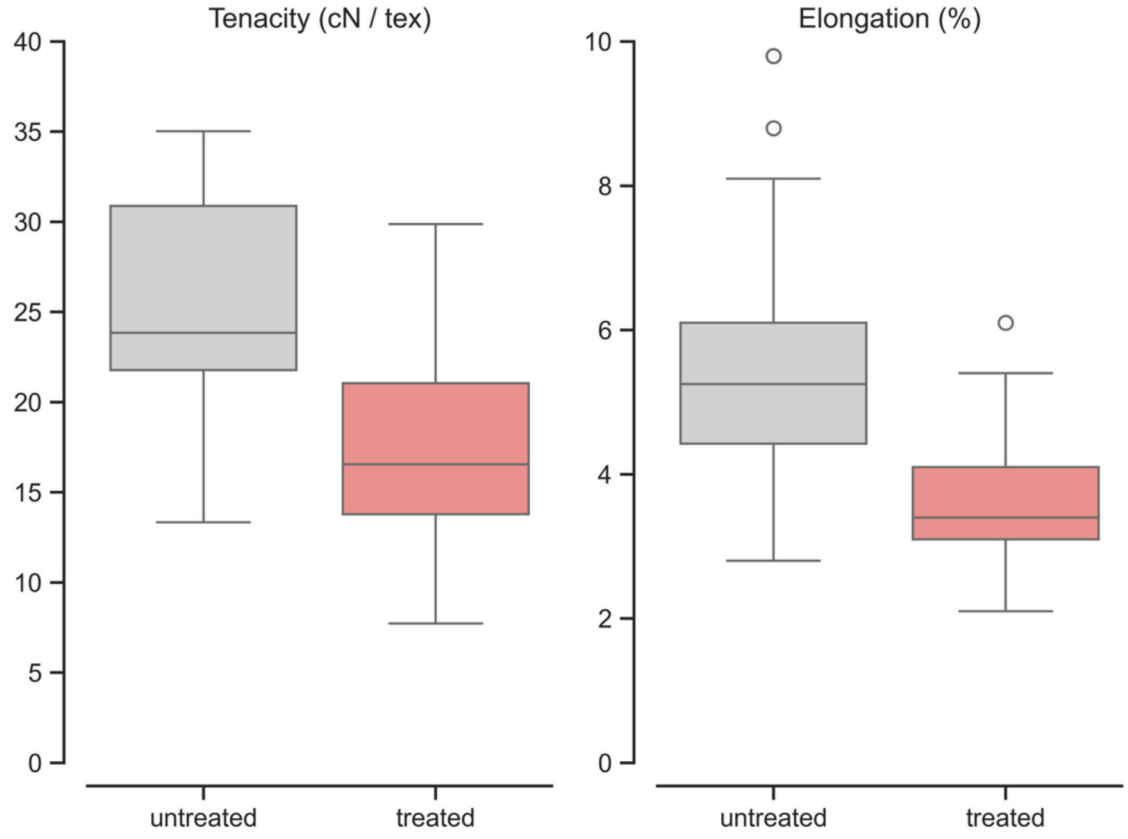

Fig. 6. Comparison of the mechanical properties gathered in single fibre tearing tests of untreated CO fibres and treated CO fibres.

significance level of 5 %, suggesting that the recycling process has effectively preserved the fibre's strength. Additionally, under the same significance level, the mean elongation calculated from the measurement series of 3.65 % passes one-sample t-tests for an elongation mean of at least 3 % ($p = 0.99979$) further confirming the high quality of the recovered CO fibres for reuse. The mechanical properties data, along with the results of the statistical analysis, are provided in Supplementary Note 3.

4. Conclusion

This study demonstrates the successful development of an environmentally friendly process for recycling PET/CO textile blends. A deep eutectic solvent (DES) consisting of menthol and benzoic acid was used as a sustainable solution for textile recycling. PET/CO textiles were treated under optimized conditions (216 °C, 5 min), achieving complete PET dissolution, CO recovery, and PET precipitation via cooling and vacuum filtration.

Diverse analyses were conducted to demonstrate the effective development of separation technology. SEM imaging showed no visible changes in CO fibre morphology before and after treatment, confirming the effective separation of PET from CO. Complementary DSC and TGA analyses validate the separation process's success. Both CO and PET exhibited very good thermal stability and within the expected ranges, affirming the successful separation. Spectroscopic analysis with ATR-FTIR revealed no chemical alterations in the recovered CO and PET, further confirming the successful dissolution of PET. Mechanical testing of single fibres was conducted to assess the potential for the reuse of recovered CO in textile manufacturing. Results indicated that recovered CO showed a somewhat decreased tenacity and elongation compared to the original fibre but remained well within a suitable range.

This process offers an efficient solution for PET/CO recycling by preserving both components in their polymeric form, where for both components high recovery yields can be achieved. Unlike state-of-the-art methods, it enables reintegration into the textile processing chain at a higher level with significantly shorter treatment times. The recovered PET-free CO retains its fibrous structure. Hence, after passing a conventional tearing line, this fibre material can serve as a high-quality secondary feedstock for CO yarn production. Simultaneously, the separated PET represents a clean feedstock for regranulation and fibre respinning and subsequent yarn manufacture. This recycling approach of PET/CO textiles is reaching beyond the state of the art. Applications, however, are not limited to apparel and extend to, e.g., production of cellulosic filter media, nonwoven textiles / insulation materials or cellulose-based biorefinery for the CO fraction, and many other melt-extrusion / melt-blowing / thermoplast applications for the PET fraction, including packaging materials. While the DES demonstrates effective performance in the treatment process and shows potential for reuse, its requirement for relatively high processing temperatures remains a limitation. This high-temperature condition may impact energy efficiency and could contribute to some degradation of the cellulose fibers. Future work should focus on optimizing the process to lower the temperature while maintaining efficacy and solvent recyclability. While currently demonstrated at the laboratory scale, the process shows strong potential for scalable industrial implementation.

CRediT authorship contribution statement

Nika Depope: Writing – original draft, Methodology, Data curation, Conceptualization. **Al Depope:** Writing – review & editing, Data curation. **Vasiliki-Maria Archodoulaki:** Writing – review & editing, Resources, Investigation. **Wolfgang Ipsmiller:** Writing – review & editing, Methodology. **Andreas Bartl:** 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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.wasman.2025.115177.

Data availability

Data will be made available on request.

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