

Research on the recycling of sulfuric acid waste liquid in textile composition detection based on the ISO 1833-11 standard

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ABSTRACT – REZUMAT

Research on the recycling of sulfuric acid waste liquid in textile composition detection based on the ISO 1833-11 standard

In the field of textile quantitative component analysis, the 75 wt% sulfuric acid solution dissolution method, a globally adopted standard, generates substantial acidic waste solution, imposing both environmental burden and economic costs. To address this, this study selected white knitted fabric (cotton/polyester blended fabric) and red knitted fabric (viscose/polyester blended fabric) as research subjects based on ISO 1833-11, conducting experiments with 0–8 cycles of waste liquid recycling to compare dissolution efficiency, residual fibre morphology, and quantitative determination results between fresh and recycled dissolution agents. Results indicated that within eight reuse cycles, the measurement deviation between fresh and recycled acid remained minimal, with relative deviation generally below 2%, while microscopic observation confirmed structurally intact polyester fibres in residues across all cycles, though minor surface deposits emerged only after higher cycle counts without significantly affecting quantitative outcomes. Further assessment revealed that this recycling strategy could reduce acid consumption and waste discharge by approximately 80%, offering both environmental benefit and economic benefit, thereby validating the feasibility of multi-cycle waste liquid recycling of concentrated sulfuric acid in textile detection and providing a practical foundation for green testing laboratories.

Keywords: textile detection, quantitative chemical analysis, sulfuric acid method, waste liquid recycling, sustainability

Cercetare privind reciclarea deșeurilor lichide de acid sulfuric în cadrul procesului de detectare a compoziției materialelor textile, în conformitate cu standardul ISO 1833-11

În domeniul analizei cantitative a componentelor textile, metoda de dizolvare cu soluție de acid sulfuric 75% în greutate, printr-un standard adoptat la nivel mondial, generează o cantitate considerabilă de deșeuri acide, ceea ce implică atât un impact asupra mediului, cât și costuri economice. Pentru a aborda această problemă, studiul de față a selectat un tricot alb (tricot din amestec de bumbac/poliester) și un tricot roșu (tricot din amestec de viscoză/poliester) ca subiecte de cercetare, în conformitate cu ISO 1833-11, efectuând experimente cu 0–8 cicluri de reciclare a lichidului rezidual pentru a compara eficiența dizolvării, morfologia fibrelor reziduale și rezultatele determinării cantitative între agenții de dizolvare proaspeți și cei reciclați. Rezultatele au indicat că, în cadrul a opt cicluri de reutilizare, abaterea de măsurare între acidul proaspăt și cel reciclat a rămas minimă, cu o abatere relativă în general sub 2%, în timp ce evaluarea microscopică a confirmat fibrele de poliester intacte din punct de vedere structural în reziduuri în toate ciclurile, deși au apărut depozite superficiale minore doar după un număr mai mare de cicluri, fără a afecta în mod semnificativ rezultatele cantitative. O evaluare suplimentară a relevat că această strategie de reciclare ar putea reduce consumul de acid și deversarea deșeurilor cu aproximativ 80%, oferind atât beneficii de mediu, cât și beneficii economice, validând astfel fezabilitatea reciclării în mai multe cicluri a deșeurilor lichide de acid sulfuric concentrat în detectarea textilelor și oferind o bază practică pentru laboratoarele de testare ecologice.

Cuvinte-cheie: detectarea materialelor textile, analiza chimică cantitativă, metoda cu acid sulfuric, reciclarea deșeurilor lichide, sustenabilitate

INTRODUCTION

The rapid expansion of the global textile industry, combined with the prevailing fast fashion model, has led to a continuous surge in textile waste. Each year, over 92 million tons of discarded textiles are generated, with a substantial portion ultimately ending up in landfills or incineration, imposing a severe environmental burden [1]. Among these wastes, cotton/polyester blended fabrics (polycotton) are predominant, accounting for nearly 50% of disposed textiles

due to their combined advantages of cotton fibre comfort and polyester fibre durability, making them widely used in both apparel and home textiles [2]. However, the intimate binding between cotton and polyester presents a significant challenge to separation and recycling, resulting in over 80% of textile waste being excluded from high-value recovery pathways [3, 4]. Thus, developing efficient and eco-friendly separation and recycling use methods is vital for promoting the circular economy and advancing the sustainability of the textile sector.

At present, the primary approach to recycling polycotton fabrics involves chemical separation, particularly acid hydrolysis, which selectively dissolves cellulose-based fibres while preserving polyester fibres. This technique has been widely studied and applied in both laboratory research and industrial pilot processes [5–7]. Leenders et al. [8], for instance, employed a 43 wt% hydrochloric acid solution to achieve effective hydrolysis of the cotton component, yielding up to 75% glucose, while the retained polyester was further depolymerised to produce high-purity bis(2-hydroxyethyl) terephthalate (BHET), enabling cascade recycling of the blended textile/fabric. Similarly, Costa et al. [5] utilised the sulfuric acid method to recover cellulose powder from blended textile waste with yields ranging from 65–88%, which was subsequently converted into carboxymethyl cellulose, cellulose acetate, and other derivatives to serve as regenerated raw materials for the textile industry. Emerging alternatives such as hydrothermal treatment [9], high-ethanol alkaline hydrolysis [4], and switchable solvent systems [10] also demonstrate promising separation capabilities, yet still face limitations including high energy demands, challenges in solvent recovery, and inadequate scalability. Beyond material recovery, increasing attention has been drawn to the release of microplastics from textile waste during usage and processing. Studies have shown that polyester fibres embedded within blended fabrics release more microplastic particles compared to those in pure polyester textiles, posing substantial threats to aquatic systems and ecological health [11, 12]. Therefore, the development of separation techniques that are not only efficient and value-added but also environmentally benign is becoming a central focus in textile waste management research [13–15].

In the field of quantitative component analysis for textiles, the ISO 1833-11 and its Chinese National Standard GB/T 2910.11 equivalent recommend the use of the 75 wt% sulfuric acid solution method for determining the composition ratio in polycotton blends [16]. This approach enables rapid and accurate dissolution of cotton fibres, preserving the polyester component for quantitative determination results. However, the process generates large volumes of concentrated acidic waste solution, increasing both treatment costs and environmental risks [17, 18]. While current research often focuses on novel separation technologies, studies on waste liquid recycling within standardised testing processes remain limited. Notably, industries have already adopted various recycling use strategies for acidic solutions, such as membrane separation technology, electro dialysis, and diffusion dialysis, with encouraging results [19–23]. Translating such circular utilisation strategies to textile testing scenarios offers significant environmental and economic benefits.

Against this backdrop, the present study, grounded in the ISO 1833-11 [16], investigates the waste liquid recycling potential of 75 wt% sulfuric acid solution in the context of quantitative component analysis. Two

representative fabrics were selected: a white knitted fabric composed of cotton/polyester blended fabric, and a red knitted fabric composed of viscose/polyester blended fabric. A series of controlled experiments was conducted across 0–8 reuse cycles, assessing variations in dissolution efficiency, residual fibre morphology, and quantitative determination results. Through combined microscopic observation and analytical evaluation, the study aims to validate the feasibility and reliability of acid recycling use, while also evaluating the sustainability and environmental benefit of such an approach. The findings are expected to contribute practical methodologies for green testing laboratory development and provide theoretical and empirical support for the resource-saving and waste acid reduction in the reuse of blended textile materials.

MATERIALS AND METHODS

Textile samples

Two types of textile specimens were selected in this study: a white knitted fabric composed of a typical cotton/polyester blended fabric (polycotton), and a red knitted fabric composed of a viscose/polyester blended fabric. The former, being the most commonly discarded textile type, was used to systematically evaluate the dissolution efficiency and quantitative determination results under different waste liquid recycling conditions. The latter served to verify the applicability of the method to viscose-based blended textile/fabric systems, assessing its performance in terms of fibre separation and analytical accuracy.

To ensure result comparability, both fabric types were cut into samples weighing 0.8–1.0 g. For each type, experiments were grouped into a fresh acid group (0 cycles of reuse) and waste liquid recycling groups spanning 1 to 8 reuse cycles. This design enabled systematic analysis of the effect of recycling frequency on dissolution performance and fibre composition measurements.

Dye/finish specification of the T/R red fabric. The viscose/polyester (T/R) red knitted fabric used in this study is a commercially dyed jersey. Consistent with standard mill practice for these fibres, the polyester component was dyed with disperse dyes, and the viscose component with reactive dyes (medium red shade, approximately standard depth; mill label: Disperse Red 60 / Reactive Red 195, shade code TR-Red-3, nominal 2.0% o.w.f.). According to the supplier's note, the fabric carried a conventional silicone softener (nonionic amino-modified silicone softener, nominal 1.0% o.w.f.) and no functional/ resin/ fluorochemical finishing was declared. These specifications are provided to contextualise the “dyes/finishes” limitation noted later in the 4th section.

Sample scope and representativeness. This study evaluates two representative blended fabrics under ISO 1833-11 conditions: a white cotton/polyester (C/T) knitted fabric and a red viscose/polyester (T/R) knitted fabric. Across 0–8 reuse cycles, both systems maintained quantitative stability within $\pm 2\%$, with T/R

showing a lower inter-cycle RSD (~0.13%). The moisture-corrected compositions are approximately C/T: cotton ~61% / polyester ~39% and T/R: polyester ~64% / viscose ~36%, providing two distinct cellulose polyester ratios and one dyed system for stability comparison. Future extensions will include additional fibre ratios and construction styles. Scope extension and challenging-case triage. Beyond the two representative blends evaluated here (C/T white; T/R red), the protocol is designed to handle challenging cases often encountered in practice, such as elastane-containing blends, resin-finished fabrics, and dark, reactive-dyed cellulose, without altering the core dissolution and QC steps.

Reagents and instruments

The dissolution agent used in the experiments was a 75 wt% sulfuric acid solution, which is prescribed by the ISO 1833-11 for the selective dissolution of cellulose-based fibres [16], allowing for reliable quantitative component analysis of blended textiles. All sulfuric acid used was of analytical grade, freshly prepared, and stored in airtight containers to prevent concentration fluctuation.

Perform all acid work in a fume hood and use appropriate personal protective equipment. Required PPE includes chemical splash goggles, a face shield for pouring, acid-resistant gloves, a lab coat paired with an apron, and closed-toe shoes. For any dilution, add the acid to water slowly. Use external cooling and gentle stirring during this process. Prepare in advance a labelled container for acidic rinsates and a base solution. Suitable base solutions include 5–10 wt% sodium carbonate or dilute ammonia. After use, collect and cool the acidic rinsates. Neutralise them gradually under stirring while monitoring pH, allowing pauses to accommodate effervescence and heat. Stop neutralisation when the pH reaches 6.5–8.5 and manage the neutralised waste in accordance with institutional standard operating procedures and local regulations. This quick note is intended to facilitate direct adoption. It aligns with the process rinse endpoint specified in the 2nd section, which requires a filtrate pH of ≥ 6.8 .

The key laboratory equipment included:

- constant temperature water bath (20–100 °C), set to 130 rpm, for maintaining the experimental temperature at 50 ± 5 °C;
- analytical balance (accuracy: 0.1 mg) for determining the dry mass of samples and residues;
- optical microscope equipped with a digital imaging system for conducting microscopic observation of residual polyester fibre morphology and potential surface deposits;
- standard glassware and consumables, including sealed Erlenmeyer flasks, glass rods, filter papers, and a vacuum filtration device;
- acid reuse system consisting of an acid storage container and a liquid separation module, allowing controlled waste liquid recycling of the 75 wt% sulfuric acid solution;

- digital densimeter or 20 ml Gay-Lussac pycnometer, operated at 20.0 ± 0.1 °C, for per-cycle density measurement of the recycled acid;
- acid-base titration setup (class-A burette; standardised NaOH; phenolphthalein or potentiometric endpoint) for titrated acidity checks on a defined dilution.

Uncertainty alignment

The narrative budget indicates that filtration/drying repeatability is the dominant contributor, whereas balance precision and moisture correction are minor-to-moderate; this is consistent with the small inter-cycle RSDs (C/T $\approx 0.55\%$; T/R $\approx 0.13\%$) and the $\leq \pm 2\%$ deviations already observed across 0–8 cycles.

Experimental basis and principles

All experimental procedures in this study were based on the ISO 1833-11:2017 Standard [16], which specifies a method for quantitative component analysis of blended textile/fabric by selectively dissolving cellulose-based fibres using a 75 wt% sulfuric acid solution. The underlying principle lies in the ability of concentrated sulfuric acid to rapidly disrupt the β -1,4-glycosidic bonds within cellulose, hydrolysing and dissolving the cellulose into soluble sugars and oligosaccharides. Under such strong-acid conditions, partial dehydration by-products may also form. Meanwhile, polyester fibres and other insoluble components remain structurally intact under these conditions [6, 8, 9, 24]. By measuring the dry weight of the remaining residue after dissolution, the percentage content of polyester fibre can be accurately quantified, and the proportion of cellulose-based fibres can be calculated accordingly [25].

In the experimental setup concerning waste liquid recycling, this study assumed that the dissolution agent would exhibit minimal variation in performance over multiple cycles of reuse. Previous research has demonstrated that although the accumulation of by-products, such as soluble sugars and dye-related/dehydration products, during acidic waste-solution recycling is inevitable, their influence on the cellulose-hydrolysis/dissolution rate and on the structural integrity of polyester fibres remains relatively minor [8, 12]. Based on this premise, comparative experiments using both fresh acid and recycled acid over 1–8 reuse cycles were conducted to evaluate the feasibility and stability of waste liquid recycling in this context. The ultimate goal was to provide experimental evidence supporting the advancement of a green testing laboratory and promote low-carbon, sustainable practices within the field of textile quantitative component analysis.

Experimental procedure

To ensure operational consistency and experimental reproducibility, this study strictly followed the standard protocol outlined in the ISO 1833-11:2017 Standard [16], while incorporating an additional investigation into waste liquid recycling. The overall experimental procedure is illustrated in figure 1.

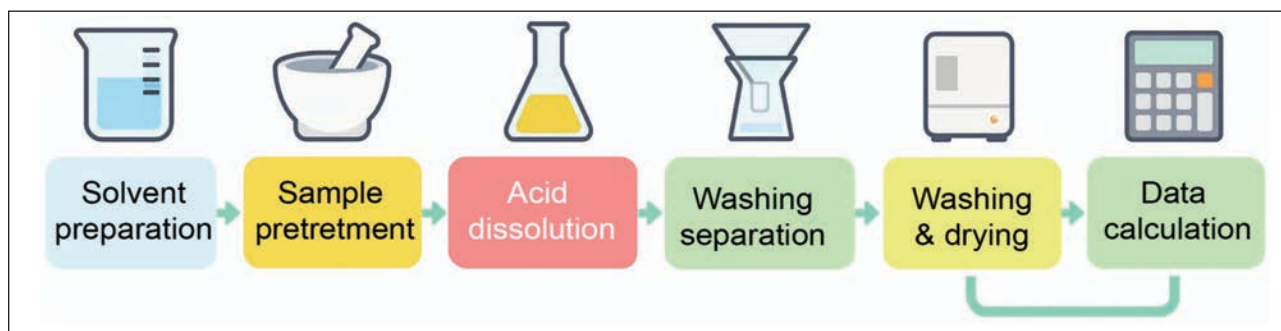


Fig. 1. Overview of the experimental procedure

As depicted in figure 1, the process begins with sample pre-treatment and solution preparation, followed by acid dissolution, residue separation, and residue weighing, ultimately leading to the calculation of the percentage content of each fibre component. While aligned with the ISO standard methodology, this procedure introduces a dedicated branch for waste liquid recycling, assessing the performance of acidic waste solutions reused from 0 to 8 cycles. This modification ensures compliance with international standards while advancing the practical implementation of green testing laboratory practices.

To disentangle the independent effects of dyes and finishing auxiliaries on experimental stability without altering the core workflow, this study adopts a paired-control approach for future datasets:

Test undyed (greige) and piece-dyed fabrics of identical construction and fibre ratio to isolate dye class/level effects from matrix effects.

Compare mass-coloured (dope-dyed) yarns against disperse-dyed yarns of matched linear density.

Spike the recycled solution with representative levels of softener/resin used on the fabric to screen whether deposits/variance increase relative to the greige baseline.

Include higher-cellulose and higher-polyester blends to probe composition-dependent variance.

Elastane-blended textiles will be handled following the relevant ISO 1833 series guidance for such systems and then evaluated under the same recycling QC, recognising this class as outside the present C/T or T/R scope.

Sample pre-treatment

Selected samples of cotton/polyester blended fabric (polycotton) and viscose/polyester blended fabric, referred to as white knitted fabric (C/T) and red knitted fabric (T/R), respectively, were cut into test pieces weighing 0.8–1.0 g, ensuring consistent mass across both sample types. The specimens were dried in a drying oven at 105°C to constant weight (validated such that the C/T white knitted fabric showed a mass change < 0.05% within 60 min after 5 h of drying, and the T/R red knitted fabric showed a mass change < 0.05% within 60 min after 6 h of drying) to remove moisture, then cooled to room temperature and stored in a desiccator before use. This step min-

imised the influence of initial moisture content differences on subsequent dissolution outcomes.

Solution preparation and experimental grouping

A 75 wt% sulfuric acid solution was employed as the dissolution agent, and samples were divided into three experimental groups:

- fresh acid group (0 cycles): utilising newly prepared concentrated sulfuric acid;
- recycling group (1–8 cycles): using acidic waste solution recovered from the previous cycle.

Each experimental condition was conducted in duplicate to ensure the reliability and reproducibility of the data.

Dissolution and residue separation

Following pre-treatment, each sample was placed into a stoppered Erlenmeyer flask; 200 ml of the acid solution per gram of specimen was added, and the mixture was maintained in a constant-temperature water bath at $50 \pm 0.5^\circ\text{C}$ for approximately 1 h with mechanical shaking. After completion of the hydrolytic dissolution of the cellulose-based fibres, the undissolved polyester fibre residue was separated using a vacuum filtration device.

The collected residue was thoroughly rinsed with distilled water until the filtrate pH was ≥ 6.8 , and then dried in a drying oven at 105°C to constant weight. The collected residue was first drained by vacuum suction, then the remaining solids were washed several times with cold water, washed twice with dilute ammonia solution, and finally thoroughly rinsed with cold water.

To visualise the transformation in fibre morphology before and after dissolution, tables 1 and 2 present direct comparisons of C/T white knitted fabric and T/R red knitted fabric, respectively, across the three key stages: original state, post-dissolution, and microscopic observation using an optical microscope equipped with a digital imaging system. For trace-colour carryover checks specific to dyed substrates, laboratories may optionally record the visual colour of the recycled acid and keep a brief note; any progressive reddening across cycles can be used as a practical trigger for make-up/replacement, alongside the density checks already reported. This note is intended for dyed systems such as the T/R red fabric.

Filtration was performed with acid-resistant hardware and a staged capture strategy to minimize blinding

while preserving mass balance: glass-fibre filter, 1.6 μm (47 mm, binder-free) in a PTFE holder; pre-exposed to working acid before use; 100–150 μm stainless-mesh basket at the funnel entrance to retain lint clumps/large fragments; upstream 2.7 μm glass-fibre disc when heavy finish or dark-dye particulates are present; the final 1.6 μm layer remains the quantitative collection stage; where crucibles are preferred, a borosilicate sintered-glass support, porosity P3–P4 ($\sim 40\text{--}10\ \mu\text{m}$) may be used beneath the 1.6 μm disc to spread flow on high-load samples. Maintain $50 \pm 0.5^\circ\text{C}$ during transfer to reduce viscosity; use wide-bore funnels, short pulses of moderate vacuum ($-20 \rightarrow -60\ \text{kPa}$) to avoid cake compaction; gently re-suspend the residue with small aliquots of working acid if local sealing occurs; enlarge filter area ($25 \rightarrow 47\ \text{mm}$) when needed.

Triage notes

Elastane-containing blends: process as usual if elastane is a minor fraction and does not form persistent gels under these study conditions; otherwise, route via the relevant ISO-1833 elastane procedure first, then apply this acid-reuse workflow to the non-elastomeric fraction under the same QC gates.

Resin-finished fabrics: do not pre-extract finishes; rely on the staged filtration above. If rinsing to $\text{pH} \geq 6.8$ requires non-routine volumes or if repeated blinding occurs, treat as an early replacement/top-up trigger.

Dark, reactive-dyed cellulosics: use the optional pre-filter and pulsed-vacuum mode to limit compacting; monitor the diluted-conductivity/visual-colour cue as an early-warning for replacement if ionic/organic build-up accelerates.

Table 1

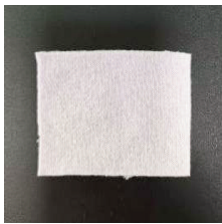
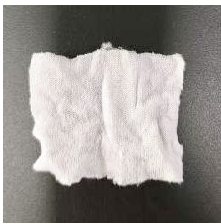

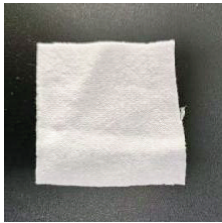

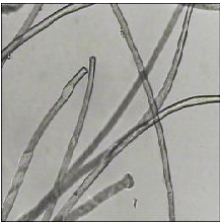
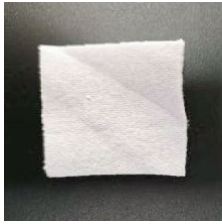

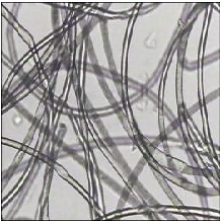


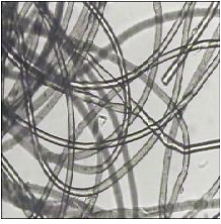


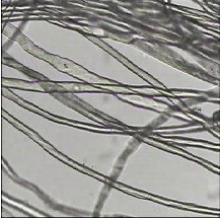
C/T WHITE KNITTED FABRIC BEFORE AND AFTER DISSOLUTION UNDER DIFFERENT ACID RECYCLING CONDITIONS			
Dissolution agent	Original sample	Residue after dissolution	Microscopic image 2 (500X)
75 wt% H_2SO_4 (0-cycle, fresh) $\rho(20^\circ\text{C})\ 1.682\ \text{g/ml}$			
1-cycle $\rho(20^\circ\text{C})\ 1.678\ \text{g/ml}$			
2-cycle $\rho(20^\circ\text{C})\ 1.676\ \text{g/ml}$			
3-cycle $\rho(20^\circ\text{C})\ 1.676\ \text{g/ml}$			
4-cycle $\rho(20^\circ\text{C})\ 1.668\ \text{g/ml}$			

Table 1 (continuation)



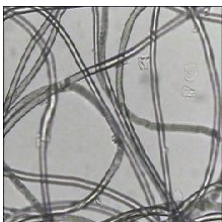
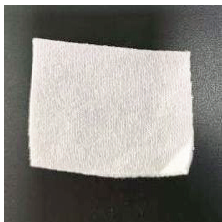

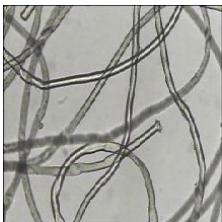
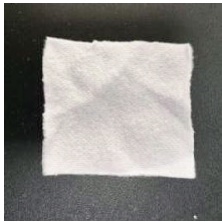





Dissolution agent	Original sample	Residue after dissolution	Microscopic image 2 (500X)
5-cycle $\rho(20^{\circ}\text{C})$ 1.668 g/ml			
6-cycle $\rho(20^{\circ}\text{C})$ 1.662 g/ml			
7-cycle $\rho(20^{\circ}\text{C})$ 1.652 g/ml			
8-cycle $\rho(20^{\circ}\text{C})$ 1.652 g/ml			

Table 2




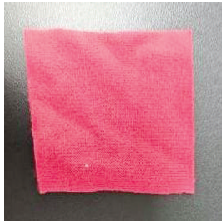

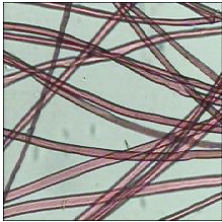


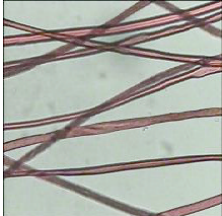

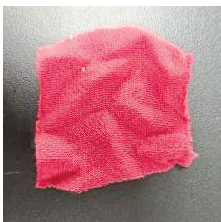
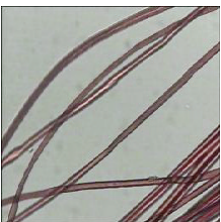


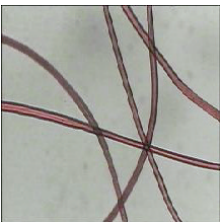



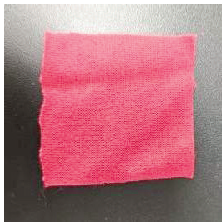

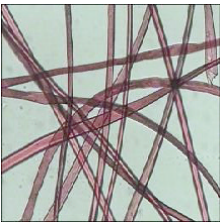
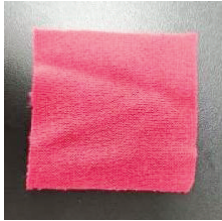

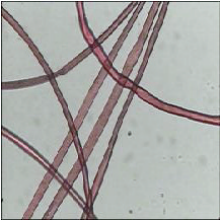


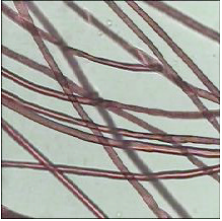
T/R RED KNITTED FABRIC BEFORE AND AFTER DISSOLUTION UNDER DIFFERENT ACID RECYCLING CONDITIONS			
Dissolution agent	Original sample	Residue after dissolution	Microscopic image 2 (500X)
75 wt% H_2SO_4 (0-cycle, fresh) $\rho(20^{\circ}\text{C})$ 1.686 g/ml			
1-cycle $\rho(20^{\circ}\text{C})$ 1.684 g/ml			
2-cycle $\rho(20^{\circ}\text{C})$ 1.682 g/ml			

Table 2 (continuation)

Dissolution agent	Original sample	Residue after dissolution	Microscopic image 2 (500X)
3-cycle $\rho(20^{\circ}\text{C})$ 1.674 g/ml			
4-cycle $\rho(20^{\circ}\text{C})$ 1.674 g/ml			
5-cycle $\rho(20^{\circ}\text{C})$ 1.670 g/ml			
6-cycle $\rho(20^{\circ}\text{C})$ 1.668 g/ml			
7-cycle $\rho(20^{\circ}\text{C})$ 1.662 g/ml			
8-cycle $\rho(20^{\circ}\text{C})$ 1.660 g/ml			

As illustrated in table 1, the residues of the C/T white knitted fabric after dissolution appear as smooth and structurally intact polyester fibres with uniform diameter, indicating that the 75 wt% sulfuric acid solution offers strong selectivity towards cellulose fibres while exerting minimal effect on the polyester fibre structure. In table 2, partial surface roughness is observed on the residue fibres of the T/R red knitted fabric following dissolution, yet the overall fibre morphology remains intact. This suggests that the use of acidic waste solution, particularly under extended waste liquid recycling conditions, may result in minor surface

deposits on polyester fibres, but does not significantly affect the quantitative determination results.

For each reuse cycle, the working acid was routinely checked to contextualise minor deposits and variance:

- density at 20°C was recorded to confirm restoration to the 75 wt% target;
- conductivity was measured on a defined dilution using a conductivity meter as a qualitative indicator of ionic build-up.

Dissolved organics were assessed on a neutralised, diluted aliquot by simple qualitative checks suitable for routine labs, e.g., a reducing-sugar colour check

and a quick UV vis scan including the HMF region, to flag any progressive presence of hydrolysis-related species without adding new figures/tables. These checks are intended as practice-oriented flags; numerical speciation is beyond the scope of this study.

QC thresholds and replacement criteria

To enable conditional extension beyond eight reuse cycles while maintaining data quality, this study defines the following routine QC checks and decision rules:

- Primary control – 20°C density window; after make-up, the working acid must fall within 1.66–1.69 g ml⁻¹ at 20°C. If the density cannot be restored to this window, replace the solution.
- Diluted conductivity (25°C, defined dilution) is used as a qualitative indicator of ionic build-up. Warning if conductivity rises by >30% vs. fresh; action/replace if >60% or if warnings persist for two consecutive cycles.
- Quick screen of dissolved organics: reducing-sugar colour check and a UV-Vis scan including the HMF region. Warning if the 280–290 nm absorbance increases by ≈0.10 (1 cm path) vs. fresh; action/replace if ≈0.20 or if a monotonic rise is seen across two cycles.
- Process performance checks: the standard rinse sequence should still achieve filtrate pH ≥ 6.8 without exceeding the routine volumes; if not, replace. Marked process slowdowns also trigger replacement.
- Top-up vs. replace: a single warning allows top-up and continue; any action criterion, or ≥2 warnings in the same cycle, triggers replacement.
- Operational cap: unless an action criterion is reached earlier, this study set a conservative pilot cap of 12 cycles for routine practice, after which replacement is recommended.

Full raw weighing entries for every cycle (0–8) and for all replicates (parallel sample 1–3) are provided in table 3.

For microscopic observations, this study additionally acquired higher-magnification micrographs and performed a minimal image-analysis routine to quantify deposit area-fraction: (i) calibrate pixel size from the scale bar; (ii) select a fixed ROI (≥0.04 mm²) avoiding edges; (iii) convert to grayscale and apply Otsu global threshold; (iv) apply a 3×3 opening to suppress isolated pixels; (v) compute area-fraction (%) = 100 × (foreground pixels / ROI pixels). The procedure was repeated on three replicate images (n = 3) per condition. Raw masks and a CSV of area fractions are provided in the Supplementary Dataset; higher-magnification images are provided in the Supplementary Image Set.

Safety and waste neutralisation

All work with concentrated sulfuric acid was performed in a fume hood with appropriate personal protective equipment: chemical splash goggles, face shield during pouring, acid-resistant gloves, lab coat with acid-resistant apron, long pants, and closed-toe

shoes. Secondary containment and spill kits were kept at hand. For any dilution, acid was added into water slowly with cooling and gentle stirring to control heat release. Acidic rinsates and used solutions were collected separately, allowed to cool to room temperature, and neutralised gradually with an aqueous base under stirring while monitoring pH; additions were paced to control exotherm and avoid localised boiling or splashing. Neutralisation was stopped once the bulk pH reached approximately 6.5–8.5. Resulting neutral salts and any precipitates were handled and disposed of as hazardous waste in accordance with institutional SOPs and local regulations; no neutralised liquids were discharged without authorisation. Waste streams were segregated and kept away from incompatible reagents.

Per-sample “green lab” metrics and sensitivity

The detailed derivation of the “~80% saving at 8 reuse cycles” and the definitions of all loss/Make-up terms have been moved to the Supplementary Note. Calculations are normalised to one specimen (≈1.0 g) dissolved at 200 ml·g⁻¹ of 75 wt% H₂SO₄ (20°C density target), with the batch restored to the 75 wt% density after each cycle. Loss terms include evaporation, entrainment/carryover moisture on glassware/filter and wet residue before rinsing, and neutralisation equivalents required to reach filtrate pH ≥ 6.8; measured rinse volumes and titration-derived neutralisation equivalents per cycle are summarised in the SI table. The nominal outcome is a ≈80% reduction of fresh-acid consumption and acid-equivalent discharge at N=8; a one-page sensitivity shows this outcome is robust within a ~70–85% band over the measured loss ranges.

Residue weighing and data calculation

The dried residues were cooled to room temperature and weighed using an analytical balance with a precision of 0.1 mg. According to the ISO 1833-11 [16], the content of insoluble fibres (i.e., polyester fibres) in the fabric was calculated, and the proportion of dissolved fibres (i.e., cellulosic components) was deduced by difference.

The quantitative determination results were recorded in tabular form. The residue mass and component content of the C/T white knitted fabric and the T/R red knitted fabric under 0–8 cycles of waste acid reuse are presented in table 3.

Mass-balance basis

Calculations are normalised to a 1.0-g specimen dissolved at a fixed acid-to-fibre ratio of 200 ml per gram using 75 wt% H₂SO₄. A single 200 ml charge is reused for eight consecutive cycles. Between cycles, the working solution is restored to the 75 wt% target based on its density at 20°C. The small per-cycle make-up accounts for three routine losses observed in the workflow: minor evaporative loss of about 1 ml per cycle with stoppered flasks, entrainment and carryover on glassware and in the wet cake before rinsing of about 12 ml per cycle, and the acid consumed during the ammonia rinse to reach a filtrate pH ≥ 6.8,

RESIDUE MASS AND QUANTITATIVE DETERMINATION RESULTS OF C/T WHITE KNITTED FABRIC AND T/R RED KNITTED FABRIC UNDER DIFFERENT RECYCLING CYCLES						
Fabric type	Reagent	Sample parameters	Parallel sample 1	Parallel sample 2	Parallel sample 3	
A piece of white fabric (C/T)	75 wt% H ₂ SO ₄ (0-cycle, fresh)	Net dry total weight (g)		0.9914	0.8852	1.0226
		Net dry weight of insoluble fibres (g)		0.3981	0.3553	0.4106
		Net dry content percentage (%)	Cotton	59.84	59.86	59.85
			Polyester fibres	40.16	40.14	40.15
	Content adjusted by standard moisture regain (%)		Cotton: 61.4%; Polyester fibres: 38.6%			
	1-cycle	Net dry total weight (g)		0.9411	0.9522	0.9761
		Net dry weight of insoluble fibres (g)		0.3779	0.3814	0.3920
		Net dry content percentage (%)	Cotton	59.84	59.95	59.84
			Polyester fibres	40.16	40.05	40.16
	Content adjusted by standard moisture regain (%)		Cotton: 61.4%; Polyester fibres: 38.6%			
	2-cycle	Net dry total weight (g)		1.0182	0.9286	0.8156
		Net dry weight of insoluble fibres (g)		0.4089	0.3709	0.3275
		Net dry content percentage (%)	Cotton	59.84	60.06	59.85
			Polyester fibres	40.16	39.94	40.15
	Content adjusted by standard moisture regain (%)		Cotton: 61.5%; Polyester fibres: 38.5%			
	3-cycle	Net dry total weight (g)		0.9131	1.0074	0.9258
		Net dry weight of insoluble fibres (g)		0.3678	0.4044	0.3718
		Net dry content percentage (%)	Cotton	59.72	59.86	59.84
			Polyester fibres	40.28	40.14	40.16
	Content adjusted by standard moisture regain (%)		Cotton: 61.4% Polyester fibres: 38.6%			
	4-cycle	Net dry total weight (g)		0.8928	0.9151	0.8763
		Net dry weight of insoluble fibres (g)		0.3585	0.3661	0.3519
		Net dry content percentage (%)	Cotton	59.85	59.99	59.84
			Polyester fibres	40.15	40.01	40.16
	Content adjusted by standard moisture regain (%)		Cotton: 61.4%; Polyester fibres: 38.6%			
	5-cycle	Net dry total weight (g)		0.8692	0.8645	0.9522
		Net dry weight of insoluble fibres (g)		0.3453	0.3440	0.3824
		Net dry content percentage (%)	Cotton	60.27	60.21	59.84
			Polyester fibres	39.73	39.79	40.16
	Content adjusted by standard moisture regain (%)		Cotton: 61.7%; Polyester fibres: 38.3%			
	6-cycle	Net dry total weight (g)		0.8611	0.8704	0.9874
		Net dry weight of insoluble fibres (g)		0.3455	0.3484	0.3965
Net dry content percentage (%)		Cotton	59.88	59.97	59.84	
		Polyester fibres	40.12	40.03	40.16	
Content adjusted by standard moisture regain (%)		Cotton: 61.4%; Polyester fibres: 38.6%				
7-cycle	Net dry total weight (g)		0.8459	0.7879	0.8914	
	Net dry weight of insoluble fibres (g)		0.3400	0.3160	0.3579	
	Net dry content percentage (%)	Cotton	59.81	59.89	59.85	
		Polyester fibres	40.19	40.11	40.15	
Content adjusted by standard moisture regain (%)		Cotton: 61.4%; Polyester fibres: 38.6%				
8-cycle	Net dry total weight (g)		0.8092	0.8141	0.8674	
	Net dry weight of insoluble fibres (g)		0.3193	0.3225	0.3497	
	Net dry content percentage (%)	Cotton	60.54	60.39	59.68	
		Polyester fibres	39.46	39.61	40.32	
Content adjusted by standard moisture regain (%)		Cotton: 61.8%; Polyester fibres: 38.2%				

Table 3 (continuation)

Fabric type	Reagent	Sample parameters	Parallel sample 1	Parallel sample 2	Parallel sample 3	
A piece of white fabric (C/T)	75 wt% H ₂ SO ₄ (0-cycle, fresh)	Net dry total weight (g)	0.9805	0.9838	0.9981	
		Net dry weight of insoluble fibres (g)	0.6493	0.6507	0.6609	
		Net dry content percentage (%)	Polyester fibres	66.22	66.14	66.22
			Viscose fibres	33.78	33.86	33.78
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.8%; Viscose fibres: 36.2%			
	1-cycle	Net dry total weight (g)	0.9742	0.8966	0.8969	
		Net dry weight of insoluble fibres (g)	0.6461	0.5927	0.5939	
		Net dry content percentage (%)	Polyester fibres	66.32	66.11	66.22
			Viscose fibres	33.68	33.89	33.78
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.8%; Viscose fibres: 36.2%			
	2-cycle	Net dry total weight (g)	0.9431	0.8728	0.9731	
		Net dry weight of insoluble fibres (g)	0.6226	0.5780	0.6444	
		Net dry content percentage (%)	Polyester fibres	66.02	66.22	66.22
			Viscose fibres	33.98	33.78	33.78
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.8%; Viscose fibres: 36.2%			
	3-cycle	Net dry total weight (g)	0.9928	0.9145	1.0009	
		Net dry weight of insoluble fibres (g)	0.6578	0.6036	0.6662	
		Net dry content percentage (%)	Polyester fibres	66.23	66.00	66.56
			Viscose fibres	33.77	34.00	33.44
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.8%; Viscose fibres: 36.2%			
	4-cycle	Net dry total weight (g)	0.9672	1.0351	0.9188	
		Net dry weight of insoluble fibres (g)	0.6402	0.6839	0.6074	
		Net dry content percentage (%)	Polyester fibres	66.19	66.07	66.11
			Viscose fibres	33.81	33.93	33.89
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.7%; Viscose fibres: 36.3%			
	5-cycle	Net dry total weight (g)	0.9033	0.9567	0.9763	
		Net dry weight of insoluble fibres (g)	0.5962	0.6307	0.6442	
		Net dry content percentage (%)	Polyester fibres	66.00	65.92	65.98
			Viscose fibres	34.00	34.08	34.02
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.6%; Viscose fibres: 36.4%			
	6-cycle	Net dry total weight (g)	0.9532	1.0068	1.0084	
		Net dry weight of insoluble fibres (g)	0.6286	0.6648	0.6658	
		Net dry content percentage (%)	Polyester fibres	65.95	66.03	66.03
			Viscose fibres	34.05	33.97	33.97
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.6%; Viscose fibres: 36.4%			
	7-cycle	Net dry total weight (g)	1.0648	1.0012	0.8364	
		Net dry weight of insoluble fibres (g)	0.7032	0.6611	0.5529	
		Net dry content percentage (%)	Polyester fibres	66.04	66.03	66.10
			Viscose fibres	33.96	33.97	33.90
		Content adjusted by standard moisture regain (%)	Polyester fibres: 63.7%; Viscose fibres: 36.3%			
8-cycle	Net dry total weight (g)	0.8551	0.8604	1.0036		
	Net dry weight of insoluble fibres (g)	0.5639	0.5690	0.6613		
	Net dry content percentage (%)	Polyester fibres	65.95	66.13	65.89	
		Viscose fibres	34.05	33.87	34.11	
	Content adjusted by standard moisture regain (%)	Polyester fibres: 63.6%; Viscose fibres: 36.4%				

this consumption is reported as the equivalent volume of the working acid, about 3 ml per cycle. The standard rinsing sequence per cycle includes two cold water rinses of ~100 ml each, two dilute ammonia rinses of ~50 ml each, and two final cold-water rinses of ~100 ml each, continuing until the filtrate reaches pH \geq 6.8. With these inputs, the cumulative fresh-acid requirement after eight cycles is about one-fifth of the no-reuse baseline, which aligns with the ~80% saving, and the acid-equivalent discharge is of the same order.

Based on table 3, it is evident that both fabric types exhibited minimal variation in residue mass and fibre content across 0–8 cycles of acid reuse. The test results remained highly consistent with those of the fresh acid group, with relative deviations within $\pm 2\%$. These findings demonstrate that even after multiple reuse cycles, the 75 wt% sulfuric acid solution maintained reliable dissolution efficiency and analytical accuracy. This outcome validates the feasibility and environmental value of waste liquid recycling in quantitative fibre analysis.

Safety, corrosion, and materials of construction

All handling was performed in a fume hood with appropriate PPE (goggles/face shield during pouring,

acid-resistant gloves, lab coat with apron). For any dilution, acid was added to water slowly with cooling and gentle stirring. Corrosion-related notes for 75 wt% H₂SO₄ at 50°C: (i) prefer glass/borosilicate vessels and PTFE/PVDF wetted parts; (ii) avoid unlined carbon steel and aluminum; (iii) 316L stainless steel hardware is not recommended under these conditions; Hastelloy-class alloys may be acceptable for short-contact fittings if lined parts are not feasible; (iv) choose elastomers cautiously, PTFE/FFKM preferred, EPDM/NR unsuitable; (v) minimize chloride ingress to avoid pitting, and rinse/neutralize contact surfaces promptly after use.

EXPERIMENTAL RESULTS

Microscopic observation

To visually assess the effect of waste liquid recycling on fibre morphology, this study conducted a comparative microscopic observation of white knitted fabric (cotton/polyester blended fabric) and red knitted fabric (viscose/polyester blended fabric) under different recycling conditions. Tables 4 and 5 present representative images of both fabric types under the original condition (0 cycles) and after eight reuse cycles.

Table 4

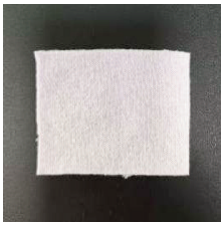
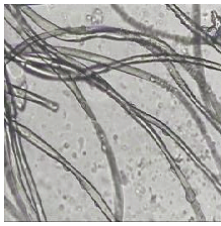

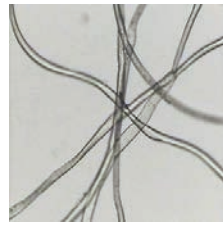
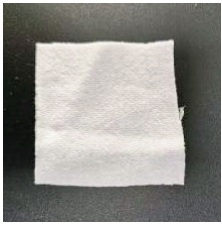
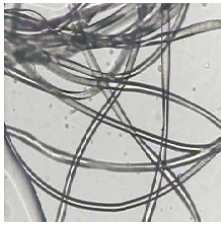
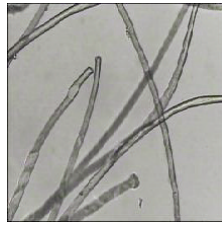
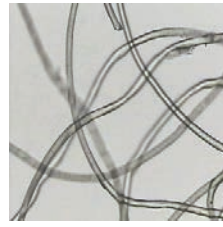

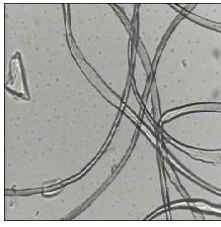
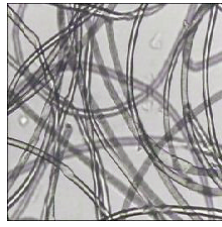
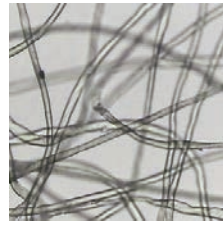


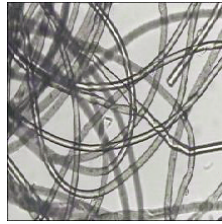



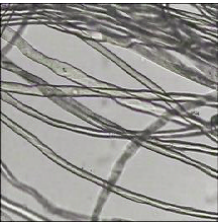
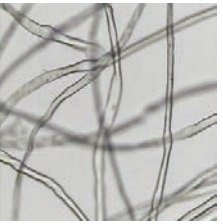


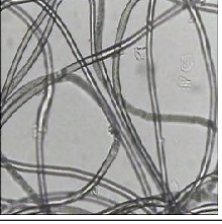
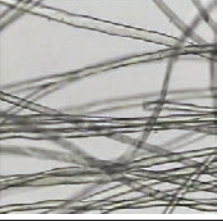
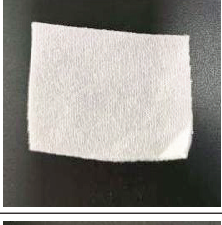

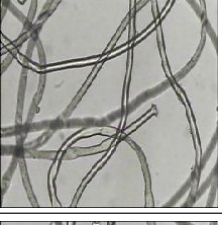

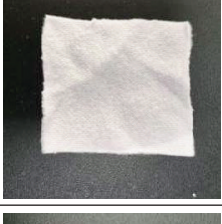
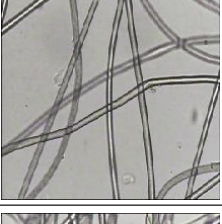
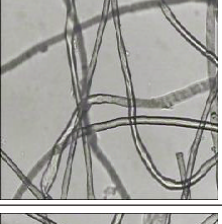






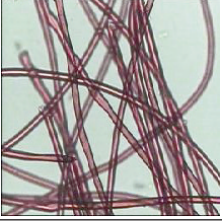



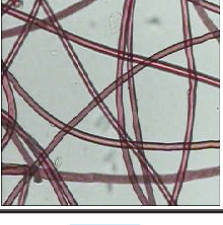
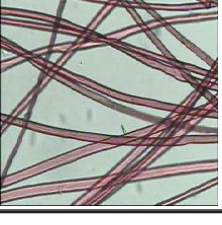

MICROSCOPIC IMAGES OF WHITE KNITTED FABRIC BEFORE AND AFTER DISSOLUTION				
Dissolution agent	Original sample	Microscopic image 1 (500X)	Microscopic image 2 (500X)	Microscopic image 3 (500X)
75 wt% H ₂ SO ₄ (0-cycle, fresh)				
1-cycle				
2-cycle				
3-cycle				



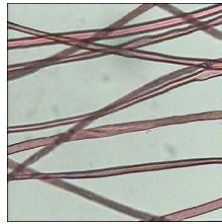
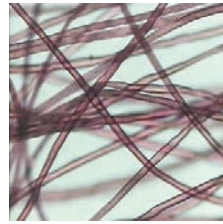


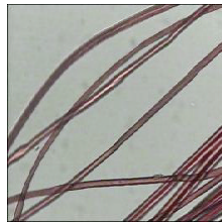
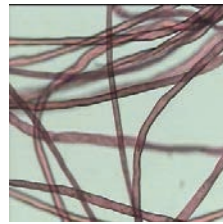


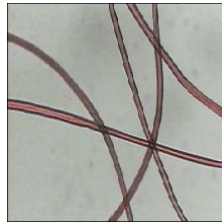
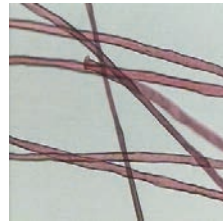



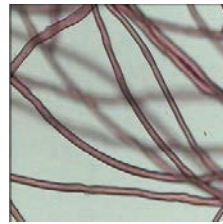

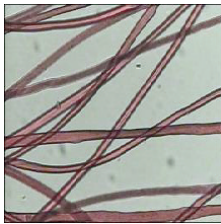
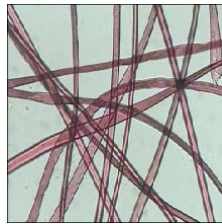
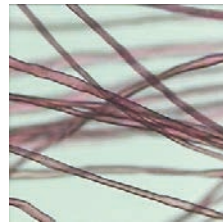

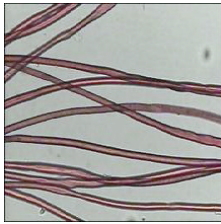
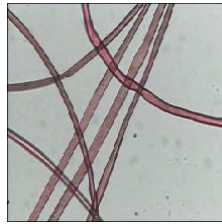
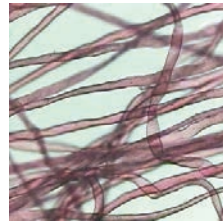

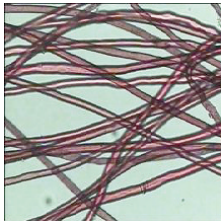
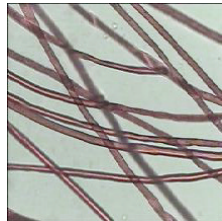
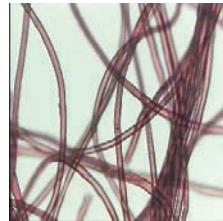
Table 4 (continuation)

Dissolution agent	Original sample	Microscopic image 1 (500X)	Microscopic image 2 (500X)	Microscopic image 3 (500X)
4-cycle				
5-cycle				
6-cycle				
7-cycle				
8-cycle				

Note: Left: Original sample; Centre: Residue after 0-cycle dissolution; Right: Residue after 8-cycle dissolution.

Table 5

MICROSCOPIC IMAGES OF RED KNITTED FABRIC BEFORE AND AFTER DISSOLUTION				
Dissolution agent	Original sample	Microscopic image 1 (500X)	Microscopic image 2 (500X)	Microscopic image 3 (500X)
75 wt% H ₂ SO ₄ (0-cycle, fresh)				
1-cycle				

Dissolution agent	Original sample	Microscopic image 1 (500X)	Microscopic image 2 (500X)	Microscopic image 3 (500X)
2-cycle				
3-cycle				
4-cycle				
5-cycle				
6-cycle				
7-cycle				
8-cycle				

Note: Left – Original Sample; Centre – Residue after 0-cycle Dissolution; Right – Residue after 8-cycle Dissolution.

As illustrated in table 4, the post-dissolution residues of white knitted fabric mainly consisted of uniformly sized, smooth-surfaced polyester fibre, with negligible differences in morphology between 0 and 8 reuse cycles. No evident fibre breakage or surface damage

was observed, indicating that both fresh acid and recycled acidic waste solution retained excellent cellulose-selective dissolution agent performance. In contrast, table 5 shows that the red knitted fabric exhibited minor surface deposits and surface roughness

on some polyester fibre strands after eight cycles. These features are likely attributable to the accumulation of byproducts or viscose fibre residues generated during acid hydrolysis. Nevertheless, the fibres largely maintained their structural continuity and integrity, with no signs of extensive degradation, confirming that waste liquid recycling does not compromise the accuracy of quantitative component analysis.

Quantitative determination results

To further verify the quantitative accuracy of waste liquid recycling, this study measured the residue mass and Percentage Content of components in the two fabric types under 0–8 cycles of reuse of the 75 wt% sulfuric acid solution. The quantitative determination results are presented in table 3.

To facilitate a more intuitive Cross-comparison of the variation under different recycling conditions, figures

2 and 3 illustrate the changes in polyester fibre content with increasing recycling cycles for the white knitted fabric and red knitted fabric, respectively.

Points whose 95% CIs lie entirely within ± 2 percentage points are described as equivalent within margin; reported effect sizes include Δ_{pp} and the tolerance-scaled effect $e = |\Delta_{pp}|/2$.

Impact of waste liquid recycling on analytical accuracy

As shown in figures 2 and 3, the polyester contents of both textile types under 0–8 cycles of waste liquid recycling remain highly consistent with the results obtained using the fresh 75 wt% sulfuric acid solution. The relative deviation is controlled within $\pm 2\%$, demonstrating excellent experimental reproducibility. The polyester content in the cotton/polyester blended fabric (C/T white knitted fabric) remains approximately 39–40% (moisture-corrected value: 38.6%), with

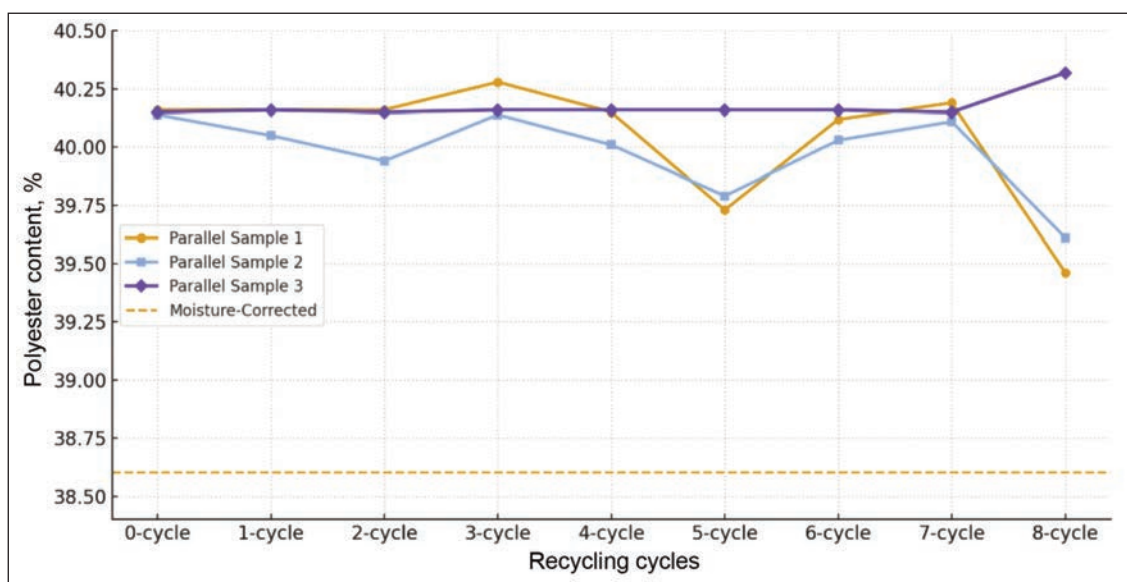


Fig. 2. Variation in polyester content of C/T white knitted fabric under different recycling cycles

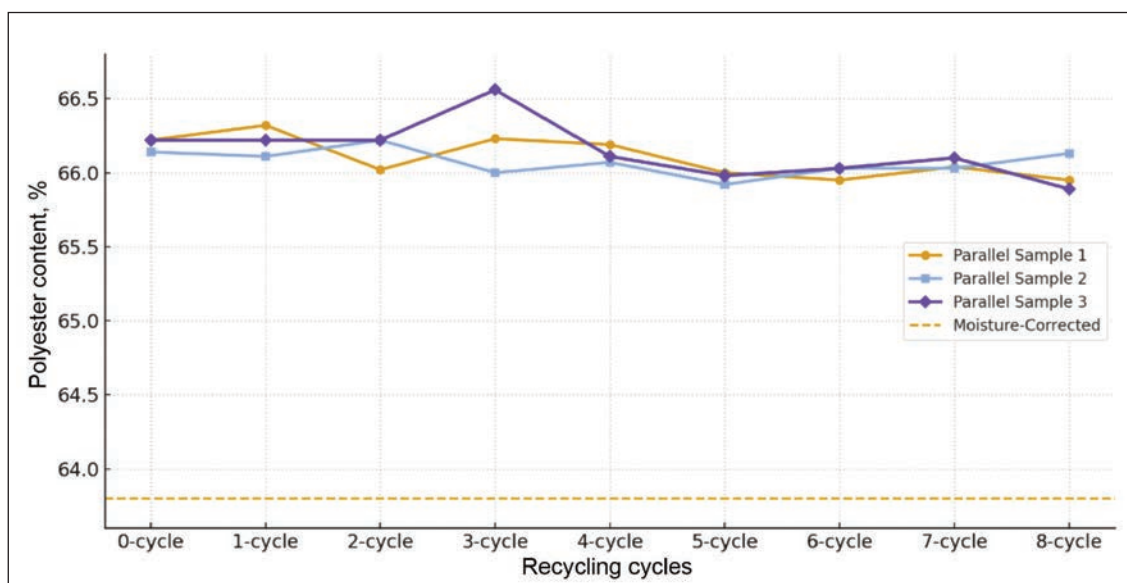


Fig. 3. Variation in polyester content of T/R red knitted fabric under different recycling cycles

minimal data fluctuation across cycles. Similarly, the viscose/polyester blended fabric (T/R red knitted fabric) maintains a stable polyester content around 66% (moisture-corrected value: 63.8%), even after eight consecutive cycles, which matches the quantitative determination results of the fresh solution group.

These findings indicate that even at high recycling frequencies, the accumulation of impurities in the acidic waste solution has a limited impact on the Dissolution efficiency of cellulose and the preservation of polyester residues. When combined with the microscopic observation data, it can be concluded that the 75 wt% sulfuric acid solution maintains excellent selectivity and analytical precision within eight cycles. This ensures reliable quantitative component analysis while significantly reducing both fresh acid consumption and waste acid reduction.

Cross-comparison and deviation analysis

To comprehensively evaluate the influence of waste liquid recycling on analytical accuracy, a Cross-comparison was conducted between the fresh solution group and various recycling groups. Corresponding relative deviations were calculated to assess experimental variability. This study now reports $n=3$ per cycle with mean \pm 95% CI and per-cycle RSD in figures 2 and 3 (table 3). Across 0–8 cycles, all results remained within $\pm 2\%$, with inter-cycle RSDs of $\approx 0.55\%$ (C/T) and $\approx 0.13\%$ (T/R), supporting high repeatability under the disclosed QC. For transparency and independent verification, the raw replicate weighings and calculated contents for each cycle are compiled in table 3.

Cotton/polyester blended fabric (C/T white knitted fabric)

As shown in table 3 and supported by the experimental data, the polyester content of the C/T white knitted fabric remains consistently around 39–40% across 0–8 recycling cycles (moisture-corrected value: 38.6%), with relative deviation typically within $\pm 0.5\%$ to $\pm 1.5\%$ (and within $\pm 2\%$ overall). The content curve in figure 2 is nearly horizontal, confirming that the residue-weighing results after repeated recycling are highly consistent with those obtained using the fresh acid.

Microscopic observations for the C/T fabric are presented in table 1. The polyester fibre surfaces remain smooth and intact throughout the recycling cycles. Any minor surface deposits observed under extended recycling do not compromise the quantitative outcomes, indicating that impurity accumulation has a negligible impact on cellulose-dissolution selectivity or on the integrity of polyester residues.

Viscose/polyester blended fabric (T/R red knitted fabric)

For the T/R red knitted fabric, the polyester content remains essentially stable at $\approx 66\%$ across 0–8 recycling cycles (moisture-corrected value: 63.8%), with relative deviations controlled within $\pm 2\%$. Compared with C/T, T/R shows slightly lower inter-cycle variability (RSD $\approx 0.13\%$ for T/R vs. $\approx 0.55\%$ for C/T; table 3). The slight variability observed is plausibly

linked to by-products from viscose acid hydrolysis at higher recycling cycles, which can lead to minor surface deposits on polyester fibres.

This interpretation is supported by the microscopy images in table 5. After the eighth cycle, the polyester fibres exhibit slight surface roughness/deposition while the overall morphology remains intact, no rupture or significant degradation, so the deviations stay well within acceptable limits for quantitative component analysis.

Consistent with these observations, the content-versus-cycle curve for T/R in figure 3 is nearly horizontal, in line with the ISO-compatible $\pm 2\%$ precision demonstrated across cycles.

Despite the dyed nature of T/R, no monotonic drift was observed in the polyester content across 0–8 cycles; relative deviations remained within $\pm 2\%$, and the inter-cycle RSD for T/R was approximately 0.13%, arguing against a progressive build-up that would bias quantification. Microscopy after eight cycles shows minor surface deposits on polyester residues, plausibly linked to dye-related fragments or cellulose hydrolysis products, yet these did not compromise the analytical outcome within eight cycles. About the link to deposits and variance, consistent with tables 1 and 2, the slight drift in 20°C density across cycles and the qualitative conductivity/organic-screening flags are in line with minor ionic/organic build-up expected under reuse and with the occasional light deposits observed for the dyed T/R fabric at later cycles. Importantly, polyester contents remained within $\pm 2\%$, and inter-cycle RSD for T/R was $\approx 0.13\%$, indicating that such build-up did not translate into systematic bias within eight cycles.

Cross-comparison between textile types

When comparing both fabric types, it is evident that despite repeated recycling, both maintain high consistency in quantitative determination results.

Nevertheless, the T/R red knitted fabric demonstrates marginally lower inter-cycle variability than the C/T white knitted fabric (inter-cycle RSD: C/T $\approx 0.55\%$ vs. T/R $\approx 0.13\%$; table 3), leading to the following insights:

The viscose/polyester (T/R) blended fabric exhibits slightly superior stability under waste-liquid recycling (lower inter-cycle RSD), while both fabric types remain within $\pm 2\%$.

The viscose/polyester blended fabric is more susceptible to interference from by-products generated during cellulose dissolution, but still maintains relative deviations within $\pm 2\%$, aligning with analytical precision standards.

Comprehensive deviation analysis

Integrating results from both the C/T white knitted fabric and the T/R red knitted fabric, it is evident that all polyester content measurements across eight recycling cycles remain within $\pm 2\%$ relative deviation. This fully satisfies the accuracy requirements of quantitative component analysis as defined in the ISO 1833-11 [16].

The primary sources of deviation are identified as follows:

- 1) Accumulation of impurities in the acidic waste solution, leading to localised surface deposits.
- 2) Minor inconsistencies in Residue Weighing due to experimental handling.
- 3) Variations in fabric structure, such as dye composition or viscose fibre content, which affect Dissolution byproducts.

The results demonstrate that the impact of waste liquid recycling on analytical precision is minimal and well-controlled, ensuring methodological reliability and reproducibility across different textile types [15]. For both C/T and T/R, the 95% CIs of cycle-to-cycle differences and of the fresh-vs-cycle-8 contrast lie within the ± 2 pp acceptance band; the estimates are equivalent within a margin. The corresponding tolerance-scaled effects ($e = |\Delta_{pp}|/2$) are < 1 , and standardised effects (Hedges' g) fall in the small range, in line with the observed inter-cycle RSDs ($\approx 0.55\%$ for C/T; $\approx 0.13\%$ for T/R) and the $\pm 2\%$ stability already documented (figures 2 and 3; table 3).

DISCUSSION

Impact of waste liquid recycling on analytical accuracy

Per-sample perspective. Interpreted per specimen, the eight-cycle scheme reduces fresh-acid use and acid-equivalent discharge to $\sim 20\%$ of the no-reuse baseline, while dissolution and drying energy per specimen remain essentially unchanged; the time spent on neutralisation and hazardous-waste handling decreases in proportion to the reduced residual stream. A short sensitivity check indicates that these conclusions are robust within a ~ 70 – 85% saving window.

The results of this study demonstrate that within 0–8 cycles of reuse, the quantitative determination results for different blended textile/fabric types remained highly consistent with those obtained using fresh solution, with the relative deviation maintained within $\pm 2\%$ (figure 2 for C/T and figure 3 for T/R; data summarized in table 3). This level of precision not only falls well within the allowable range for experimental reproducibility defined by the ISO 1833-11 [16], but also aligns with precision thresholds reported in both domestic and international literature on quantitative component analysis [18, 26]. Together with the microscopy images (table 4 for C/T; table 5 for T/R), these findings confirm that recycling the waste acid does not significantly compromise cellulose dissolution or the retention of polyester residues, thereby verifying its feasibility for routine fibre analysis.

Additionally, the reliability of the quantification was corroborated through microscopic observation. The surface morphology of polyester fibre in cotton/polyester blended fabric (polycotton) remained smooth and intact under all reuse conditions. Although minor surface deposits were observed on viscose/polyester blended fabric after multiple recycling cycles, no evidence of fibre breakage or degradation was found. This is consistent with previous findings that concentrated sulfuric acid selectively

dissolves cellulose fibre while leaving polyester fibre largely unaffected [5, 9]. Hence, it can be inferred that the gradual accumulation of by-products in the acidic waste solution exerts only superficial effects on fibre morphology without altering the fundamental quantification outcome.

Variability among different fibre systems

Notably, the quantification of white knitted fabric (C/T) showed slightly greater data fluctuation under recycling conditions compared to the more stable results observed for red knitted fabric (T/R). This discrepancy is likely due to differences in the proportion of cellulose fibre content. In the polycotton fabric, a higher percentage of cotton fibre leads to increased by-product accumulation during dissolution, thereby causing greater variability in the measurements. Conversely, the lower and more homogeneously dissolving viscose fibre content in the T/R samples likely contributes to more stable data. This is consistent with the lower inter-cycle RSD observed for T/R ($\approx 0.13\%$) relative to C/T ($\approx 0.55\%$).

Furthermore, dyeing processes may also play a role in the observed differences. To operationalise this observation, this study's expansion plan includes greige vs dyed and dope-dyed vs piece-dyed controls, plus auxiliary add-back screens, so that any dye/auxiliary-related contribution to minor deposits can be separated from fibre-ratio effects. The red dye components in T/R fabrics could interact with 75 wt% sulfuric acid solution or its hydrolysis products, leading to increased formation of surface deposits on residual fibres [27]. These changes, reflected as surface roughness under microscopy and minor variations in mass during Residue Weighing, suggest a degree of acid hydrolysis-induced structural transformation in cellulose fibre. Nevertheless, since all variations remain within the acceptable threshold of experimental reproducibility, the findings affirm the applicability of waste liquid recycling even for viscose/polyester blended fabric, though some caution is warranted for long-term operational deployment.

Environmental and economic benefits

Conventional quantitative component analysis requires extensive use of fresh 75 wt% sulfuric acid solution, generating substantial quantities of acidic waste solution that pose a serious environmental burden and increase laboratory operational costs [17]. The results of this study reveal that reliable analytical outcomes can be maintained over eight cycles of reuse/recycling, potentially reducing acid consumption and waste generation by approximately 80%.

This achievement holds significant implications for the development of a Green Testing Laboratory:

- Environmental benefits: reduction in high-concentration acidic waste solution discharge lowers the risks of water and soil acidification;
- Economic benefits: significant savings in reagent procurement and waste treatment costs, especially valuable for high-throughput testing scenarios;

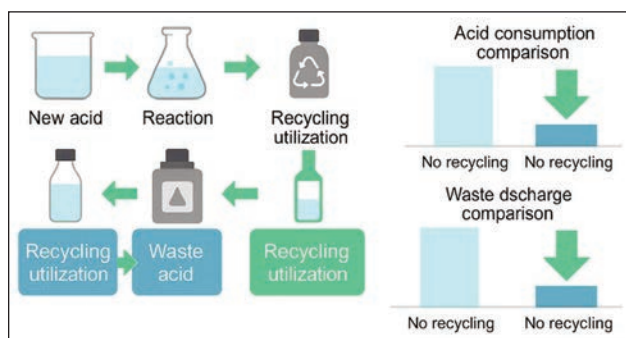


Fig. 4. Illustrates the projected resource conservation and waste acid reduction potential brought about by the proposed waste liquid recycling protocol

- Sustainability impacts: demonstrates a model for sustainable practices in analytical laboratories, in alignment with the national Dual Carbon Strategy [19–21]. Under the disclosed mass-balance basis, the eight-cycle workflow uses ~20% of the fresh acid and leads to an acid-equivalent discharge of the same order relative to a no-reuse baseline, aligning with the ~80% saving shown in figure 4.

For each cycle, the recycled acid met the 20°C density window (1.65–1.69 g ml⁻¹), and the titration-equivalent 75 ± 0.5 wt% band before use; filtrate pH ≥ 6.8 was confirmed.

From figure 4, it is evident that when the number of reuse/recycling cycles reaches eight, both 75 wt% sulfuric acid solution consumption and acidic waste solution discharge decrease to approximately 20% of the original volume, reflecting substantial gains in resource saving and waste acid reduction. Combined with the stability of quantitative determination results across cycles, this trend highlights the strong potential of the proposed method for practical implementation in quantitative component analysis of blended textile/fabric.

Application prospects and limitations

While the results of this study are consistent across 0–8 cycles (±2%; T/R inter-cycle RSD ~0.13%), this study now provides explicit QC thresholds and stopping rules so that reuse can be extended conditionally. In practice, continuation beyond eight cycles is permitted only if the QC window is met; otherwise, early top-up or replacement is performed.

A concise practitioner safety note summarises fume-hood/PPE practice, acid-into-water dilution, segregated collection, and gradual neutralisation to pH ≈ 6.5–8.5, aligning with this study rinse endpoint (filtrate pH ≥ 6.8).

From an application perspective, the method proposed in this study is not only well-suited for use in Green Testing Laboratory environments but may also serve as a reference framework for the separation and recovery of textile waste. However, several limitations must be acknowledged:

1) The current study validates up to eight cycles of waste liquid recycling. Whether further cycling will lead to increased data fluctuation due to by-product accumulation remains to be explored.

2) Beyond the two systems tested here (C/T and dyed T/R), additional fibre ratios, dye/finish systems, and elastane-containing textiles should be examined. This study, therefore specify a paired-control and add-back design to isolate dye/auxiliary effects from substrate effects. These studies will determine whether the ±2% stability observed over 0–8 cycles generalises across challenging cases and will refine actions if deposits/variance become material.

3) As the current analysis is based on laboratory-scale testing, extending the method to large-scale or industrial applications will necessitate additional consideration of acid management systems, recycling use technology, and process safety.

Addressing these issues will be crucial in advancing the practical value of concentrated sulfuric acid recycling within the textile industry, thereby promoting both sustainability and alignment with the broader goals of the national Dual Carbon Strategy.

This study now discloses the dye/finish information of the T/R red fabric to ground this limitation. Within eight cycles, this study did not find evidence of dye-hydrolysate accumulation at a level that alters analytical stability: contents stayed within ±2%, and T/R exhibited a low RSD (~0.13%). Minor deposits seen under microscopy likely reflect trace by-products but do not translate into systematic bias.

For practice, a simple visual colour check of the recycled acid can serve as an early-warning flag in dyed systems.

In routine use, laboratories may keep the density-to-target control as primary QC and add the diluted-conductivity and quick organic-screening notes above as qualitative early-warning cues for replacement/top-up decisions in dyed or heavily finished systems.

About regeneration options vs direct neutralisation, qualitative cost-benefit framing. With eight-cycle reuse, fresh-acid demand and acid-equivalent discharge fall to ~20% of the no-reuse baseline, leaving a smaller residual stream to manage. Direct neutralisation/disposal involves no CAPEX and is simple, but incurs ongoing base/neutralisation and hazardous-waste fees, well-suited to low-throughput labs. Diffusion dialysis (DD) is energy-lean, recovering free H₂SO₄ using anion-exchange membranes; CAPEX is moderate, and OPEX low, and acid purity can suffice for make-up after density restoration. Electrodialysis (ED/BMED) has higher power/control needs but can co-recover acid/base, cutting neutral-salt waste. Membrane-based impurity removal can extend reuse lifetime by removing dyes/particulates or polishing streams upstream of DD/ED; it is less about free-acid recovery per se but valuable as a life-extension step. References on DD/ED/hybrid economics and performance are listed in this study's bibliography.

CONCLUSION

This study, based on the ISO 1833-11 [16], investigated the waste liquid recycling of concentrated sulfuric acid in the quantitative component analysis of cotton/polyester blended fabric (polycotton) and viscose/

polyester blended fabric, specifically targeting white knitted fabric and red knitted fabric, respectively. Through a combination of microscopic observation and quantitative assessment across 0–8 recycling cycles, the following conclusions were drawn:

- 1) Stable analytical accuracy: within eight cycles, the Percentage Content of polyester fibre in both fabric types remained highly consistent with results from fresh acid usage, with relative deviation controlled within $\pm 2\%$, thereby fulfilling the precision requirements of textile quantitative component analysis.
- 2) Intact fibre morphology: polyester fibre in polycotton retained its structural integrity throughout all cycles, while viscose/polyester blended fabric exhibited minor surface deposits at higher cycles, without compromising quantitative determination results.
- 3) Significant environmental and economic benefits: acid reuse resulted in a reduction of approximately 80% in both reagent consumption and acidic waste solution generation, effectively alleviating both the

environmental burden and associated laboratory costs.

4) Broad applicability: the proposed method proved effective not only for cotton/polyester blended fabric but also for viscose/polyester blended fabric, indicating promising generalizability across multiple blended textile/fabric systems.

Collectively, these findings confirm the feasibility and efficiency of multi-cycle reuse of 75 wt% sulfuric acid solution in textile component testing. The method balances analytical rigour with sustainability, providing a scientific foundation for the development of green testing laboratories and offering practical insights for the textile industry's transition toward waste liquid recycling and low-carbon operations.

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