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Accurate quantification of polyester in textiles through complete depolymerisation and HPLC

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The bulk of textile waste is still landfilled or incinerated, despite its components being potentially recyclable. Textile waste, however, requires prior characterisation to produce homogenous feedstocks that can be recycled with current technologies. Available analytical pipelines are not accurate enough to meet the maximum threshold of contaminants tolerated by recycling processes. Complete depolymerisation of several synthetic fibres into their constituting monomers has already been achieved: the resulting mixtures could be characterised through chromatography, providing an accurate fingerprint of the composition of the fibres. The present work describes an analytical method based on this approach to selectively quantify poly(ethylene terephthalate), the most widely used synthetic fibre, through the quantification of its depolymerisation product, terephthalic acid, using high-performance liquid chromatography.

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Introduction

Textile waste is still predominantly landfilled or incinerated,[1] despite its components (*e.g.*, cotton, polyesters, polyamides)[2–5] being resource-intensive to manufacture. Coupled to the steadily increasing production of new textiles[6] and their decreasing life-time,[1,7,8] this has led to dramatic environmental[9–11] and social consequences,[12–15] positioning the textile and fashion industry as one of the most polluting industries in the world.[10–12,16–19] Textile recycling is thus not just a business opportunity, but also a global environmental and ethical necessity. Polyester fabrics (namely, poly(ethylene terephthalate), PET), in particular, should be considered as prime target for recycling, as they have the largest share on the textile market:[6,20] in 2024, the global production of polyester fibres and filaments surpassed 77 million tonnes.[21]

Although different approaches for the recycling of post-consumer PET waste have been proposed (*e.g.*, mechanical recycling,[22,23] chemical/solvolytic recycling,[24–26] and enzymatic recycling[27–31]), deployment to scale has been dominated by mechanical recycling of selected waste streams (*e.g.* PET bottles),[22,24,26,32–34] and applications to textile waste have been limited.[35–38] Textile waste is highly heterogeneous in terms of composition, processing history, and quality: this heterogeneity requires effective analysis, fractionation, and purification strategies to obtain a homogenous feedstock that can actually be recycled.[39–45] Mimicking plastic waste recycling,[46–48] automated techniques based on optical spectroscopy have been applied to the initial sorting and categorising of textiles,[49–53] but only probe their surface, leading to misclassification of thicker, surface-treated, dyed, or extensively weathered items,[50,53–57] and cannot provide information about the bulk composition of the items. While valuable for initial pre-processing, these methods can thus not be employed as stand-alone quantification strategies.

The current standard for the quantification of the bulk composition of textile fibres is the ISO 1833 series. This comprises methods for the quantification of different fibres (*e.g.*, cellulosics,[58] nylon,[59] and elastane[60]) in fibre mixtures that rely on the selective dissolution and/or degradation of one component, and the recovery and gravimetric quantification of the other, from which a mass balance is then established. Outside of binary mixtures, quantification further requires sequential application of the standards in the correct order, possibly resorting to parallel processing of multiple aliquots,[61] as not all procedures are mutually compatible. Critically, this also requires *a priori* knowledge of the components in the textiles being analysed, which may be possible in the case of, *e.g.*, label compliance determination (one of the key applications of the ISO 1833 series, *e.g.*, within the application of the European regulation No 1007/2011),[62] but is rarely the case for textile waste. Additionally, although direct quantification of PET in textiles is covered in some of the standards, available methods are restricted to highly niche textile compositions (*e.g.*, PET/aramid textiles,[63,64] used primarily in high-performance personal protective equipment). Even in the case of exceedingly common blends (*e.g.*, the cotton/PET/elastane of a basic T-shirt), PET can thus only be quantified as the leftover fraction at the end of the application of an *ad hoc* sequence of standards. In the context of textile recycling, this approach does not provide

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sufficient accuracy, as even moderate fractions of contaminants and/or misclassified fibres (*e.g.*, leftover elastane in a PET recycling process) can compromise both mechanical and chemical recycling.[65–73] Additionally, the time required to complete such analyses, even at laboratory scale, is substantial (*e.g.*, over 5 h[58,74]), making them unsuitable for at line implementation.

Successful fibre-to-fibre recycling realities have therefore been limited to processing carefully selected, one-material streams and/or closed-loop pre-consumer waste, for which the components of the waste stream are already known, often at low throughput and/or while downcycling all but one fraction of the waste to lower value, non-textile products (*e.g.*, non-woven material, insulation, packaging…).

Solvolytic depolymerisation has been shown to achieve complete conversion of different synthetic polymers.[39,75–83] The same process could be used as part of an analytical pipeline, as most polymers used in synthetic fibres contain at least one unique monomer: the resulting (mixture of) reaction products could be accurately quantified through chromatographic separation, providing a chemical fingerprint of the textiles.

This work presents an analytical method to directly characterise the PET content of textile samples based on the quantification of their depolymerisation products. Quantitative and efficient depolymerisation is achieved through microwave assisted heating,[84] allowing the complete conversion of PET to terephthalic acid (and ethylene glycol) within minutes. Selective quantification is then performed using high-performance liquid chromatographic (HPLC). The method is validated with pure PET fibres, *ad hoc* mixtures of PET and cotton fibres, of PET and other common types of fibres, as well as commercial fabrics with different PET content. Accurate quantification is obtained for all cases, enabling the direct determination of the PET content of textiles and textile waste.

Experimental

Chemicals

Potassium hydroxide (hereafter, KOH, 10448990, Thermo Fisher Scientific, Finland), methanol (8.22283, Merck, Finland, for sample preparation; K977, Avantor, Finland, for chromatography) and terephthalic acid (hereafter, TA, 99+%, 10654682, Thermo Fisher Scientific, Finland) were purchased and used as received. Ammonia (25% w/v, 10642251, Thermo Fisher Scientific, Finland) was diluted to 0.25% w/v using Milli-Q® grade water before use.

Textiles

Single-component fibres were obtained from Indorama, Brazil (polyester), Hyosung, South Korea (creora™ elastane), Mirka Oy, Finland (Nylon 6,6 pre-consumer waste), Farmers' Cooperative, USA (cotton), Jon Vanneste, Belgium (linen), and the ITFC 2023 exposition, Turkey (wool, cashmere and mohair). Fabrics with known polyester content (different blends, colours and thicknesses) were purchased from the Finnish commercial suppliers Eurokangas, Tekstiilipalvelu, and Finlayson. Their composition is listed in Table 1. The chemical structures of PET and TA are shown in Fig. 1. All fibres and fabrics were used as received, without any prior washing or pre-treatment.

Table 1 Composition of mixed poly(ethylene terephthalate) (PET) fabrics. Values are reported as mass percentages, as indicated on the product label

Fabric	Composition	Fabric	Composition
	100% PET		67% PET 33% cotton
	100% PET		65% PET 35% cotton
	95% PET 5% elastane		40% PET 60% cotton
	84% PET 16% elastane		28% PET 70% cotton 2% elastane

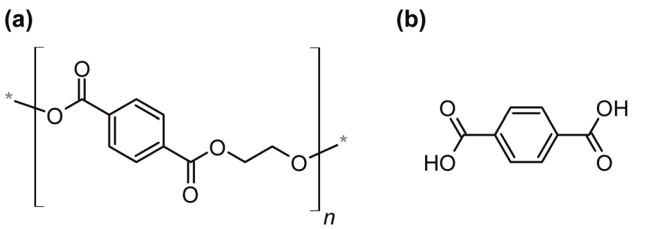

Fig. 1 Chemical structure of (a), the repeat unit of poly(ethylene terephthalate); (b), terephthalic acid. The figure was generated using ChemSketch v.2024.2.3.

Depolymerisation of PET and downstream sample processing

Depolymerisation was performed in a Monowave 300 reactor (Anton Paar, Austria). Aliquots (≈10–40 mg) of fibres or fabrics were weighed, then transferred to a G30 reaction vial (82 723, Anton Paar, Austria) with a Teflon stir-bar. 10 ml of a room temperature 1.25 M solution of KOH in methanol was added to each vial. The vial was sealed with a Teflon septum, placed in the reactor, then the following programme was executed: reaction temperature, 95 °C; heating power, "as fast as possible"; stirring speed, 600 rpm; hold time, 12 min; cool-down

temperature, 50 °C. After the programme was concluded, the sample was removed, diluted 1 : 1 v/v with deionised water and thoroughly mixed, then sonicated at room temperature for 2 minutes to ensure all TA was released from leftover solids (*e.g.*, cotton fibres) and dissolved. Three replicates were then prepared for each sample by diluting it 1 : 10 v/v in 0.25% w/v ammonia (final pH ≈ 13), filtered through a 0.2 μm pore size polytetrafluoroethylene membrane filter, and transferred to quartz cuvette (spectrophotometric quantification) or a polypropylene HPLC vial (HPLC quantification) for analysis.

Spectrophotometric quantification of terephthalic acid

Spectrophotometric quantification was performed using a Shimadzu UV-2550 spectrophotometer. UV absorbance was measured in the 285–305 nm range with a sampling step of 0.2 nm and three consecutive scans per each sample.

As the absorbance spectrum for TA in the solvent system and wavelength range selected here shows no distinct characteristic peak but does show an inflection point, the first derivative of the spectra with respect to the wavelength was also computed[85] by smoothing individual spectra with shrinkage-penalised cubic regression splines,[86] then approximating the derivative with the incremental ratio.

The average absorbance (at 286 nm) or average derivative (at 294.8 nm) of technical replicates (replicate scans, $n = 3$, replicate dilutions of individual samples, $n = 3$) was used in all further calculations. Examples of spectra of the TA standards and of the reaction products are included in Section 1 of the (SI).

The concentration of TA in each sample was estimated based on a standard calibration curve (7 equispaced concentrations in the 0–60 mM range). Standard dilutions were prepared in a solvent mix that matched the concentrations of KOH, methanol and ammonia resulting from the sample dilutions steps. Spectrophotometric quantification was only used during early optimisation, to confirm the complete depolymerisation of pure PET fibres under the chosen reaction conditions.

HPLC quantification of terephthalic acid

HPLC quantification was performed using a Nexera Lite CBM-40 HPLC system (Shimadzu, Japan) equipped with a LC-40D quaternary pump, SIL-40C autosampler maintained at 15 °C, CTO-40 C column oven and SPD-40 UV-detector. Separation was achieved using a XBridge C18 column (3.5 μm particle size, 4.6 × 50 mm, 186003031, Waters, USA) maintained at 50 °C, and 0.25% w/v ammonia at a flow rate of 0.8 ml min^{-1} as mobile phase. The pump was operated at constant flow for 4 minutes, during which terephthalic acid eluted (average elution time over 740 injections: 35 s, RSD: 3.6%). The mobile phase was switched to methanol over the course of 1 minute, then, after 3 minutes, switched back to 0.25% w/v ammonia over the course of 1 minute again. The system was then re-equilibrated for 3 minutes before the next injection. Each sample was injected 3 times, with an injection volume of 10 μl each. Absorbance was monitored at 245 nm and signal intensity quantified as peak area. Both TA standards and samples were confirmed to be stable at 15 °C in the dark over the course of several days, see also Section 2, SI.

The baseline of each chromatogram was corrected using asymmetric least square smoothing,[87] then the area of the TA peak was calculated using the trapezoidal rule. The median area of technical replicates (replicate injections, $n = 3$, replicate dilutions of individual samples, $n = 3$) was used in all further calculations. The concentration of TA in each sample was estimated based on a standard calibration curve (5 equispaced concentrations in the 0–60 mg l^{-1} range. Standard dilutions were prepared in a solvent mix that matched the concentrations of KOH, methanol and ammonia resulting from the sample dilutions steps). We note that other common solvents for TA (*e.g.*, dimethylsulfoxide, dimethylacetamide[88]) are not compatible with this protocol, as they also dissolve (polymeric) elastane in textiles.

Data analysis

All data analysis was performed in R 4.5.3[89](IDE RStudio 2026.1.1.403,[90] Quarto 1.9.31).[91] The complete list of packages used for the analysis is included in Section 3, SI.

Bland–Altman analysis

Bland–Altman analysis[92] was performed using the PET masses of the samples as reference, and their corresponding estimates from our new method as test values. Linearity of the method's response was established using ordinary least squares regression between measured and reference values, average bias as their average difference, proportional bias using ordinary least squares regression between their difference and their average. The region of agreement is reported as the limits of the 95% tolerance interval (TI).[93]

Lin's concordance correlation coefficient (CCC)[94–96] was estimated as:

$$-1 \leq \mathrm{CCC} = \frac{2rs_x s_y}{s_x^2 + s_y^2 + (\bar{x} - \bar{y})^2} \leq 1 \quad (1)$$

where r is Pearson's sample correction coefficient, s_x, s_x^2 and $\bar{x}$ are the sample standard deviation (mg), sample variance (mg^2), and sample average (mg) of the known PET masses of the samples, respectively, and s_y, s_y^2 and $\bar{y}$ are the corresponding values for their estimates.

LOD and LOQ

The limit of detection (LOD) and limit of quantification (LOQ) of the method were estimated using a linear response model[97] for a symmetric type I and type II error probability $\alpha = \beta = 0.05$. The LOD was estimated as:

$$\mathrm{LOD} = 2\frac{s_{y,x}}{b} t(\alpha, N - 2)\sqrt{1 + h_0} \quad (2)$$

where $s_{y,x}$ (mg) is the standard deviation of the residuals of the linear response model, b is the slope of the linear response model, $t(\alpha, N - 2)$ is the value of the Student-t distribution for a type I error probability α and N degrees of freedom, N is the number of independent samples included in the model, and h_0

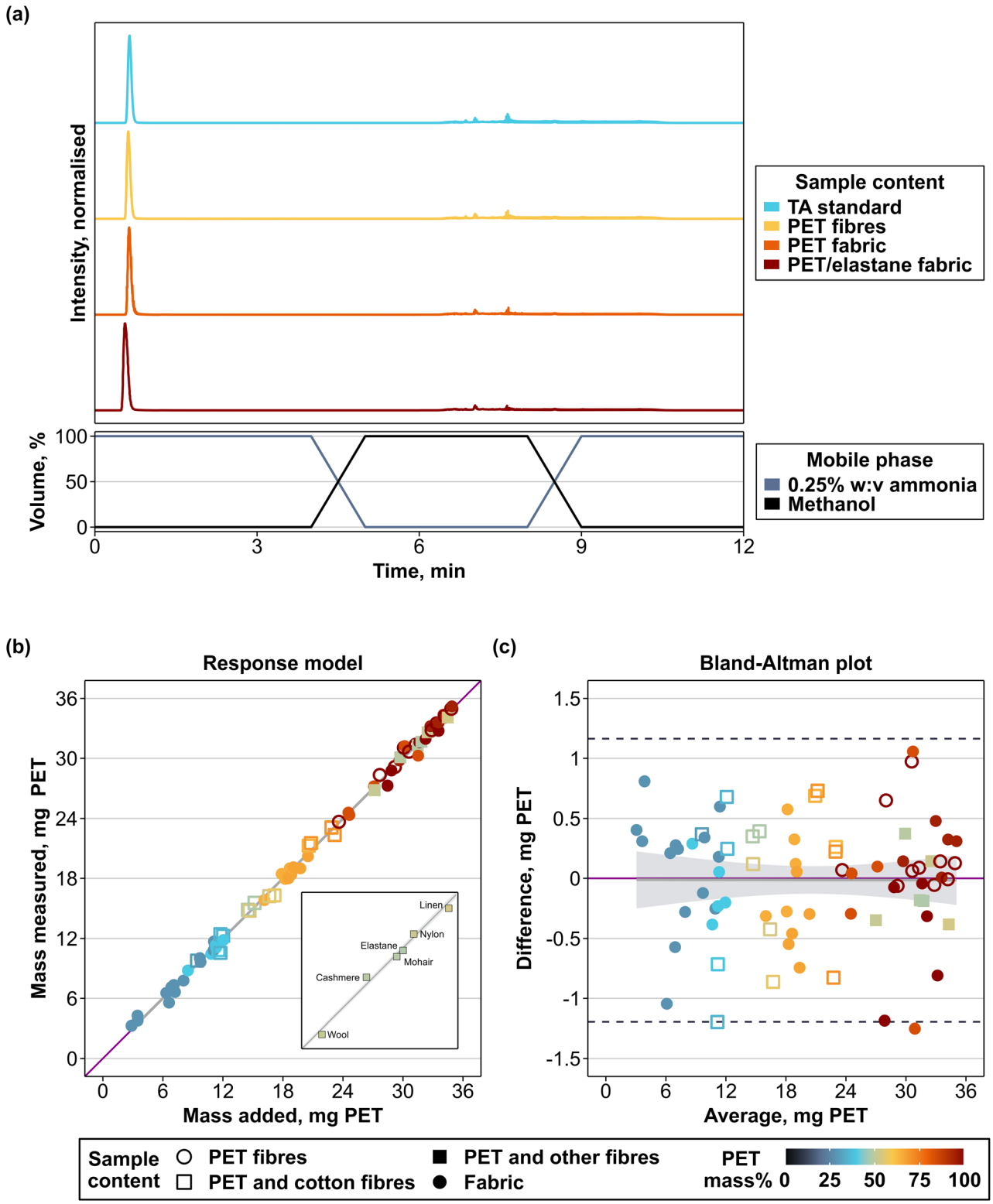

Fig. 2 (a) Top, chromatograms obtained for a terephthalic acid (TA) standard and the reaction products of poly(ethylene terephthalate) (PET) fibres, a PET fabric and a PET/elastane fabric (chosen as example of a blend); bottom, mobile phase settings during the run; (b) masses of PET as estimated through depolymerisation as a function of the masses added to the samples. The grey line and grey shaded area represent the best fit obtained by ordinary least squares regression and its 95% confidence interval, respectively. The purple line represents the ideal relation (intercept = 0, slope = 1). The estimates in the group "PET and other fibres" have additionally been reproduced in the inset; (c) values in (b) after transformation for Bland–Altman analysis (the differences on the *y* axis are reported as mass measured − mass added). The grey line and grey shaded area represent the best fit obtained by ordinary least squares regression and its 95% confidence interval, respectively. The purple line represents the ideal relation (intercept = 0, slope = 0). The dashed black lines represent the limits of the 95% tolerance interval for the difference.

Table 2 Figures of merit. All estimates are reported with their 95% confidence interval ($n = 75$)

Figures of merit		
Response model	Slope	0.999 [0.987, 1.011] $p = 0.808$ ($H_0 = 1$)
	Intercept	0.015 [−0.254, 0.283] $p = 0.912$ ($H_0 = 0$)
	R^2	0.997 [0.995, 0.998]
	CCC	0.998 [0.997, 0.999]
Bland–Altman analysis	Average difference (average bias)	−0.015 [−0.129, 0.099] mg PET $p = 0.797$ ($H_0 = 0$)
	TI	[−1.194, 1.161] mg PET
	Slope (proportional bias)	0.001 [−0.012, 0.012] $p = 0.979$ ($H_0 = 0$)
	Intercept	−0.011 [−0.281, 0.257] mg PET $p = 0.932$ ($H_0 = 0$)
Analytical limits (for $\alpha = \beta = 0.05$)	LOD	1.687 [1.678, 1.696] mg PET
	LOQ	5.568 [5.539, 5.597] mg PET

is the estimated leverage of the origin. The LOQ was then estimated as:

$$\mathrm{LOQ} = 3.3\ \mathrm{LOD} \tag{3}$$

Confidence intervals for the LOD and LOQ were obtained using balanced non-parametric bootstrap with replacement.

Results and discussion

Initial mass balance and product confirmation

The methanolysis of PET theoretically yields equimolar amounts of dimethyl terephthalate and ethylene glycol.

In practice, however, it has been previously shown that even trace amounts of water (*e.g.*, in the solvent) are sufficient to shift the reaction equilibrium towards hydrolysis, resulting in the exclusive production of TA (and ethylene glycol) instead.[84] Additionally, dimethyl terephthalate is not stable in aqueous solutions at high pH, and is itself converted to TA.[84] Initial optimisation of the reaction conditions was therefore performed using spectrophotometric quantification of the products from pure PET fibres relative to TA standards. Ethylene glycol was not considered for quantification, as its UV detection would have required further derivatisation[98–100] to be compatible with the operational range of the detector used herein. Once spectrophotometric analysis indicated quantitative TA release (apparent TA yield: 100%, 95% CI [99, 101]), the reaction products were further analysed *via* HPLC.

Example chromatograms for the TA standard, and for reaction products from PET fibres and fabric samples are shown in Fig. 2(a), confirming TA was produced during depolymerisation.

Method validation

The method was validated by processing samples containing pure PET fibres, ad hoc mixtures of PET and cotton fibres (30 to 70% PET by mass), *ad hoc* mixtures of PET and other fibres (50% PET by mass), and fabric samples (28 to 100% PET by mass, see Table 1). Data were pooled for analysis ($n = 75$, ranging from approximately 3 mg to 35 mg PET). The results are summarised in Fig. 2(b) (estimates obtained *via* our new method as a function of the mass of PET in the samples) and (c) (Bland–Altman analysis). Figures of merits have been compiled in Table 2.

Measurements obtained *via* our new method were found to be in excellent agreement with their reference values: in fact, neither parameter of the response model differed significantly from the ideal identity case (slope = 1, intercept = 0 mg). Consistently, the mass balance was found to be complete (TA yield: 100%, 95% CI [99, 101]).

The high CCC (0.998) further suggests quantification is both accurate and precise. Bland–Altman analysis found no evidence of systematic bias (average or proportional), with tolerance limits extending from −1.194 to 1.161 mg only.

LOD/LOQ

The LOD and LOQ of the overall quantification method (depolymerisation, sample downstream processing, and HPLC quantification) were estimated to be 1.687 and 5.568 mg PET, respectively. Notably, despite the non-PET components in the samples being affected very differently by the depolymerisation process (*e.g.,* nylon and elastane were only faintly discoloured, while no solid residues were recovered for animal fibres; some dyed fabrics retained their colour, whereas others leached dyes into the solvent before or after depolymerisation…), no interference with PET quantification was encountered for any of the blends or fabrics tested herein, showing that the method can be applied to generic textiles. Example chromatograms of non-PET fibres are included in Section 4, SI.

Conclusions

This work aimed at developing and evaluating a method for quantifying PET in textiles, based on their depolymerisation followed by HPLC analysis. The results presented herein successfully demonstrate that accurate and precise quantification can be achieved for a variety of fibre blends, with no interference, and with a LOD as low as 1.728 mg PET.

While this study focused on the technical development and analytical performance of the method, the results already highlight its potential to be applied at line for the characterisation of pre- and post-consumer textile waste. Critically, the infrastructure that would be required for porting the method at scale is in many cases already integrated into existing (textile) recycling pipelines. For example, as mentioned in the introduction, solvolytic depolymerisation (used herein to release TA for quantification) can be employed for the recycling process of PET.[39,76,81,101–106] Extensive screening and selection (*e.g.*, to sort and recover reusable items, remove non-textiles components like zippers, buttons…), cleaning, shredding, grinding, and homogenisation are also already part of the pipeline of (textile) recycling at scale, both as part of the quality control/quantification steps, as well as of the processing of the feedstock itself: representative sub-sampling is, therefore, already feasible at line.

Microwave-assisted heating provides clear advantages in terms of reaction time and energy-efficiency; however, larger sample volumes can be easily processed in common high-pressure industrial reactors and still be competitive compared to current approaches (*e.g.*, ISO 1833–11) in terms of total analysis time.[58] In contrast, the method proposed herein enables the direct quantification of PET in textiles and textile waste with unprecedented accuracy.

Thus, our method addresses a critical bottleneck in the mechanical and chemical recycling of PET and synthetic fibres. Additionally, it supports the chemical recycling of (man-made) cellulosic fibres, for which PET represents, in turn, one of the undesired fractions that need to be quantified and removed for processing.[53,107,108] Finally, the PET content of pristine textiles can also be quantified using the same approach (*e.g.*, for quality control or label compliance and consumer protection).

Author contributions

Mattia Cerri designed the experiments. Mattia Cerri, Leena Pitkänen, and Corné Schutgens performed the experiments. Mattia Cerri performed all data analysis. Mattia Cerri and Michael Hummel wrote the manuscript. All authors have given approval to the final version.

Conflicts of interest

The authors declare no competing financial interest.

Abbreviations

CCC	Concordance correlation coefficient
CI	Confidence interval
HPLC	High-performance liquid chromatography
KOH	Potassium hydroxide
LOD	Limit of detection
LOQ	Limit of quantification
PET	Poly(ethylene terephthalate)
TA	Terephthalic acid
SI	Supplementary information
TI	Tolerance interval

Data availability

Data are available at the zenodo repository associated with this article upon request. See: DOI: (https://doi.org/10.5281/zenodo.19031533).

Supplementary information (SI): UV-vis spectra of terephthalic acid in solution, stability assessment for TA in solution, a list of the R packages used for data analysis, and additional chromatograms of fibre samples. See DOI: https://doi.org/10.1039/d6ay00086j.

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