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Accuracy assessment of a micro-Raman spectroscopy method for small microplastic particles in infant milk formula

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ABSTRACT

The presence of microplastics (MPs) in the food chain is increasingly documented, raising concerns over potential risks to human health. Despite growing efforts, standardized methods for MPs detection in food matrices remain limited. This study presents an interlaboratory comparison (ILC) aimed at assessing the accuracy and comparability of an analytical approach for the identification and quantification of small MPs (5–100 μm) in infant milk powder using μ -Raman spectroscopy and a representative polyethylene terephthalate (PET) reference material (RM). The RM, formulated as water-soluble tablets, was designed to replicate the morphology, size distribution, and polymer composition of environmentally relevant MPs, and was previously assessed for homogeneity and stability for mass fraction and particle numbers.

The approach was assessed using two PET RM batches with different MPs particle numbers (high load batch: 1759 ± 141 MPs; low load batch: 160 ± 22 MPs), subjected to an enzymatic–chemical digestion, followed by μ -Raman analysis performed independently in two laboratories with different instruments and operators. Results are reported as absolute particle counts per analyzed sample and demonstrated excellent recovery across all size classes, including the smallest particles (down to 5 μm), with recovery rates ranging from 82 % to 88 %, in good agreement with the RM reference values.

The analytical approach proved to be robust, reproducible, and suitable for low-level MPs quantification in complex food matrices, supporting ongoing efforts toward method harmonization and standardization for reliable MPs monitoring in the food sector.

1. Introduction

Plastic with its chemical and physical resistance, low cost and ease of production, has become widely used across various applications, with global production increasing steadily over the years and reaching 413.8 Mt in 2023 [1]. However, this revolutionary material has slowly become a global environmental threat, with large quantities of used plastics either landfilled or end up in aquatic and terrestrial ecosystems through improper disposal. Plastic items exposed to environmental factors such as ocean current dynamics, solar radiation, mechanical abrasion, and

interactions with organisms, slowly degrade and fragment into smaller plastic particles, known as microplastics (MPs) [2–5]. So far, there is no harmonization regarding the size thresholds used to classify MPs. Reported values range from 1 - 20 μm at the lower size [6] and from 500 μm up to 1 mm, or even 5 mm, at the upper size [7]. According to ISO 24187:2023 [8], MPs are defined as solid plastic particle insoluble in water in the size range of 1 μm – 1000 μm . These small particles are of particular concern due to their ability to be ingested by organisms and transferred across trophic levels, potentially posing risks to human health [9,10]. They have been identified in several food products,

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including fish [11], fruits and vegetables [12], meat [13], drinking water [14], beverages (energy and soft drinks) [15] and dairy products, like milk [16–18], although the sources of contamination remain unclear (e.g. environmental sources or food processing, storage, transportation, and packaging) [19].

Milk and its derivatives are an essential component of healthy human nutrition, with global consumption steadily rising over the past decades to nearly 981 MT in 2024 [20]. As demand continues to grow, ensuring the safety of milk has become more critical than ever. All activities developed during the production process of milk, from farms to the dairy industry, could be a risk of contamination by MPs (e.g. poor cleanness procedure equipment, surrounding environment, water supply conditions, inadequate handling of milk and packaging) [21]. Despite growing concern, a limited number of studies have investigated MPs contamination in milk, particularly in infant milk powder [22,23], focusing on the detection of MPs in different commercially available liquid milk brands to assess human exposure through oral ingestion [16, 24–29]. The majority of these works used visual identification and counting of MPs under a microscope followed by chemical characterization using Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (ATR-FTIR) [24–28] or, in few cases, Raman spectroscopy [16,23,30,31] or a combination of both techniques [29] for a comparative qualitative study [32]. Due to the size limitations of optical microscopy, the reported MPs are generally in the millimeter range, thus excluding smaller MPs (< 100 μm), which are more relevant from a biological and toxicological perspective. Only three studies focused on milk powder [17,27,23], two of which examined infant formula [22,23]. Zhang et al. [22] detected MPs in milk using ATR-FTIR in a study aimed at investigating for the first time MPs pollution in infant formula (MPs abundance from 1 ± 1 to 11 ± 1 units/100 g, with average sizes from $139 \pm 343 \mu\text{m}$ to $193 \pm 415 \mu\text{m}$). Kadam-Czapska et al. [23] introduced method validation using commercial plastic standard particles that differ in terms of polymer type, size, shape and color, including polyamide (PA: irregular shaped fragments, 1 mm), polyethylene (PE: spheres, 30 - 50 μm), polypropylene (PP: granules, 2 mm), polyethylene terephthalate (PET: granules, 2 mm). It should be noted that MPs do not adequately represent the complexity of real MPs found in matrices in terms of shape and size. In contrast, Da Costa Filho et al. [17] used more representative size standards (5 - 40 μm) for several polymers (e.g. PP, PE, PS, PA, PMMA) although PET, one of the most widespread environmental polymers, was not included. This latter study also addresses contamination more thoroughly by analyzing blank samples over multiple days and reporting an average background value, which was then subtracted from the results of real sample analysis. However, it is worth noting that background subtraction in MPs analysis is questionable, given the high variability and pervasive MPs contamination across laboratory environments [27,28]. Furthermore, no statistically defined reporting limit (RL) or equivalent limit of quantification (LOQ) was established – an issue common to all cited studies, where MPs in blanks are simply identified and excluded from the sample results without applying any quantitative threshold. Therefore, critical methodological limitations are widespread throughout the existing literature. In this context, the development of efficient and reliable standardized methods remains a critical challenge for MPs monitoring and is urgently required to support EU legislation (e.g. Drinking Water Directive) and ensure reliability and comparability of data for health and environmental risk assessments. This need has already been partially addressed in the water quality evaluation: the International Organization for Standardization (ISO) body ISO/TC 147/SC 2/JWG1 developed international standards for the identification and quantification of MPs in water by number-based spectroscopy (ISO 16094–2:2025) [35] and mass-based thermo-analytical detection methods (ISO/DIS 16094–3) [36]. Reference materials (RMs) are necessary to validate each analytical method and to ensure comparability and harmonization across MPs measurements, but they are currently lacking. The main challenge is producing RMs that realistically mimic MPs found in environmental and food

samples in terms of irregular shape, surface characteristics and size distribution [37]. ISO 17034:2016 [38] specifies the general requirements for the competence and consistent operation of RM producers, covering the production of all RMs, including certified RMs, and ensuring that materials are produced under controlled conditions with traceable properties. According to ISO 33405:2024 [39], these materials must be homogeneous and stable in relation to one property of interest (e.g. mass or particle number) and sufficiently representative to be used in metrological studies, including the development of accurate analytical methods, calibration of measurement systems, and quality control testing.

This work presents an interlaboratory comparison (ILC) aimed at assessing the accuracy and comparability of μ -Raman measurements for the reliable identification and quantification of small microplastics (SMPs) in infant milk formula, using PET reference materials. The RM was produced in two batches with different loadings (high and low) within the framework of the European project 21GRD07 PlasticTrace [40], both tested for homogeneity and stability in terms of both mass fraction and particle numbers [38,39,41]. For the present work, the low load batch was primarily considered, as it better represents realistic food contamination scenarios. PET was selected as representative polymer due to its widespread use in food packaging [42,43] and to assess its limited stability under harsh oxidative conditions during sample preparation (e.g., temperatures above its glass transition and strong alkaline environments) [44,45] this was crucial to evaluate the impact of sample preparation on the recovery of smaller particle size fractions. The analytical approach combines an appropriate sample treatment based on enzymatic and chemical digestion, with particle characterization carried out by micro-Raman (μ -Raman) spectroscopy, chosen for its high spatial resolution and suitability for detecting SMPs. μ -Raman analysis was performed in an interlaboratory comparison involving two independent laboratories with different operators and instruments. Both laboratories followed the same sample preparation protocol to ensure reproducibility, comparability, and harmonization of the measurements.

2. Materials and methods

2.1. Materials

Ultrapure water was obtained by a Milli-Q® IQ 7000 purification system (Merck Millipore, Germany) equipped with a 220 nm polyethersulfone (PES) filter. This water was used for cleaning procedures and sample preparation. Infant powdered milk (Organic 1 First Infant Baby Milk Powder) was bought from a local supermarket and selected as the test matrix.

PET RM in the form of water-soluble tablets were produced and provided by Bundesanstalt für Materialforschung und-prüfung (BAM, Berlin, Germany).

In addition, the following reagents were used during sample preparation: a multi-enzymatic detergent (Prozyme Active Deconex, Borer Chemie AG, Switzerland); ethylenediaminetetraacetic acid sodium salt (EDTA-Na 0.5 M, pH 8, Invitrogen, ThermoFisher Scientific, USA); tetramethylammonium hydroxide (TMAH 25 % v/v Sigma-Aldrich, USA); sodium hydroxide (NaOH 2.5 M, PanReac AppliChem ITW Reagents, Spain); nitric acid (HNO_3 5 % v/v Carlo Erba Reagents GmbH, Germany).

Ethanol absolute anhydrous (99.9 %, Carlo Erba Reagents GmbH, Germany), Triton™ X-100 (0.1 % v/v in ultrapure water, Thermo Fisher Scientific, USA) and acetone (99.9 % v/v, Carlo Erba Reagents GmbH, Germany) were also used for the cleaning procedures.

2.2. PET RM tablets for method performance assessment

PET RM tablets containing PET particles in the size range of 5–100 μm , with sharp edges and irregular morphology, were provided by BAM in two different loadings: a high load batch (HLB) and a low load batch

(LLB). These different loadings were tested to evaluate the sensitivity and recovery of both environmental-like concentrations (HLB) and food-related concentrations (LLB) detectable by number-based spectroscopic methods. Particular attention was given to LLB, as its particle number is more representative of the levels potentially found in food matrices. Both batches were previously evaluated for homogeneity and stability for particle number according to ISO 17034:2016 [38] and ISO 33405:2024 [39], within the framework of the European project 21GRD07 PlasticTrace [40,41], and were found to fulfil the criteria of RMs. Detailed information on tablet dissolution, filtration, and characterization are described in a separate manuscript prepared within the 21GRD07 PlasticTrace project (under review) [46].

These PET RM tablets were used to establish reference particle numbers for assessing the analytical method performance in milk samples, and the values were expressed as absolute particle counts per tablet.

2.3. Infant milk powder sample preparation

The milk digestion protocol for MPs extraction originally developed by Da Costa Filho et al., [17] was adapted with slight modifications to improve the removal of organic material while preserving the integrity of chemically sensitive polymers, such as PET, enabling the development of a new method for reliable MPs detection in complex matrices. The same protocol was applied in parallel by two independent laboratories, INRiM and University of Parma, to ensure comparable results.

Powder milk was reconstituted with ultrapure water (14 % w/v) and shaken at 40 °C for 15 min in a shaking water bath (Ultrasonic Cleaner DU-45, 180 W). Meanwhile, a PET RM tablet was dissolved in 20 mL of ultrapure water in a glass flask. After the complete dissolution of the tablet, 25 mL of reconstituted milk was added to the solution and heated to 40 °C while stirring to perform the digestion process. The milk sample was subjected to multi-enzymatic digestion by adding and stirring 2 mL of multi-enzymatic detergent (Prozyme) for 2 min, followed by the addition of 10 mL of calcium chelating agent sodium ethylene diamine tetra acetate (EDTA-Na 0.5 M, pH 8) for 3 min. Subsequently, 2 mL of alkaline solution tetramethyl ammonium hydroxide (TMAH 25 % v/v) were added under continuous stirring, and the temperature was raised to 80 °C to initiate hot alkaline hydrolysis.

Upon reaching 80 °C, the hot digested milk was immediately transferred to a filtration unit (100 mL funnel, glass holder with 13 mm fritted

support) and filtered under vacuum (N816 LABOPORT/100 W) using a circular silicon (Si) filter with a pore size of 5 µm (macroporous silicon membrane, 9 mm diameter, SmartMembrane) with an effective circular filtration area of approximately 20 mm² (~ 5 mm diameter).

Before filtration, the funnel was conditioned by rinsing with 15 mL of Triton™ X-100 (0.1 % v/v in ultrapure water) and 15 mL of ethanol (99.9 % v/v). The glass flask containing the digested milk was also rinsed with 15 mL of Triton™ X-100 (0.1 % v/v in ultrapure water) and 15 mL of ethanol (99.9 % v/v) to mobilize any MP PET particles adhering to its surfaces and collect them on the filter. The retained digested material on the Si filter was flushed sequentially with 5 mL of ultrapure water, 5 mL of nitric acid (5 % v/v in ultrapure water) and 10 mL of ultrapure water.

Finally, the funnel was rinsed again with 15 mL respectively of Triton™ X-100 (0.1 % v/v in ultrapure water) and 15 mL of ethanol (99.9 % v/v) to recover any particles potentially adhering to its surfaces. The filter was then stored in closed glass petri dishes until analysis. Following the same procedure, procedural blanks were performed using ultrapure water to evaluate any potential MP contamination introduced during the digestion procedure.

A comprehensive overview of the milk sample preparation is presented in Fig. 1.

As MPs are ubiquitous pollutants, preventing contamination during the analytical workflow is crucial for ensuring accurate results. To this end, all sample manipulations were performed under a laminar flow hood by qualified analysts adequately trained in sample handling and specific measurement techniques. Both laboratories followed the precautions recommended in [34], including washing hands, no make-up and wearing a cotton lab coat. All equipment (e.g. bottles, petri dishes, beaker, filters, tweezers, filtering units) was carefully cleaned before use and covered with aluminum foil. Reagents were also prepared under the same controlled conditions in clean beakers, using glass pipettes for transfers and aluminum foil protection before and after use to minimize potential contamination. Samples remained covered with aluminum foil during all different preparation steps, including filtration, except for brief exposure while gradually pouring.

The measurement results are expressed as the absolute number of PET particles detected by the analyzed sample, rather than being normalized to sample mass. This operational definition provides traceability through particle counting and maintains consistency for subsequent interlaboratory comparison.

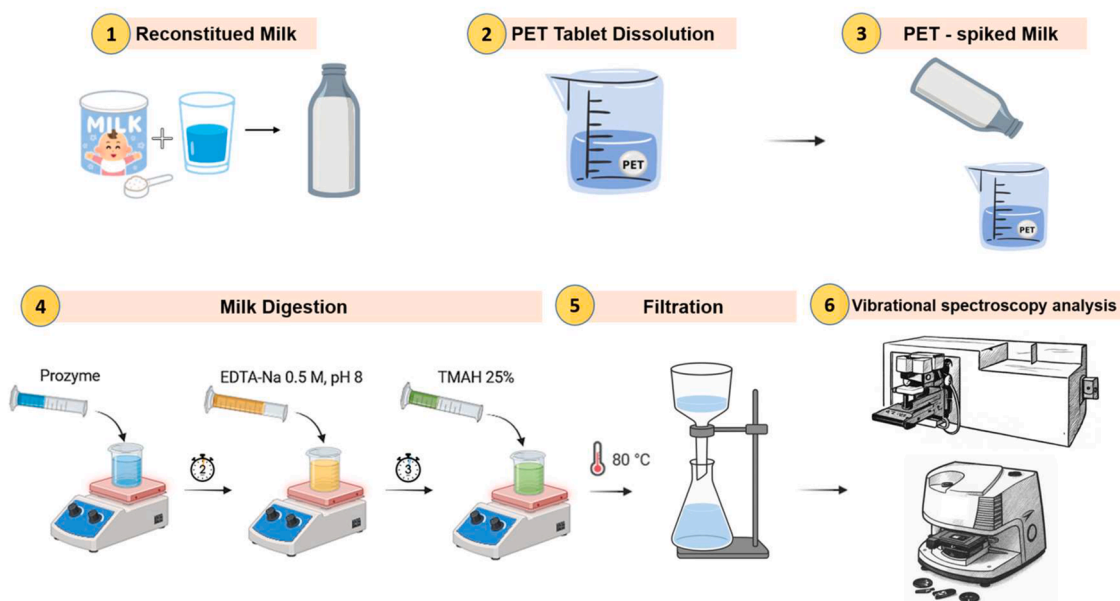


Fig. 1. Infant milk powder sample preparation, including PET RM tablet spiking, for identification and quantification of SMPs.

2.3.1. Interlaboratory comparison study of infant milk powder sample analysis

Infant milk samples were analyzed by Raman spectroscopy in two different laboratories, INRiM (Lab 1) and University of Parma (Lab 2), using their individual μ -Raman method but the same sample preparation protocol (Fig. 1).

Laboratory 1. Detection was performed on milk samples using a LabRAM Odyssey Raman spectrometer (HORIBA) equipped with a CCD detector. Initially the entire filter was imaged using a $10 \times$ objective (OLYMPUS, numerical aperture: 0.25) under darkfield illumination system to acquire a panoramic mosaic of the filter. All subsequent measurements were carried out with a $50 \times$ long working distance objective (OLYMPUS, numerical aperture = 0.5), also in darkfield mode in order to minimize the contribution of the filter pores and facilitate image analysis. Three regions were randomly selected across the effective filtration area (~ 5 mm diameter), each measuring $2.5 \text{ mm} \times 1.5 \text{ mm}$ (3.75 mm^2), collectively representing approximately 55 % of the filtering surface. The data obtained were scaled to estimate PET particle number across the full filtration area.

Spectra were acquired in the range of $620\text{--}1760 \text{ cm}^{-1}$ using a 633 nm, an excitation laser with a power of 15 mW, an exposure time of 1 s and one accumulation, and a 600 1/mm grating. Data acquisition was performed using LabSpec software (v. 6.8.1.9), while particle identification was carried out with IDFinder software (v. 4.2, HORIBA). Each detected particle was then classified as a specific polymer type by comparison with an internal spectral library containing reference spectra of the most common plastic polymers. Spectra with a Hit Quality Index (HQI) > 80 % were automatically assigned to PET while those with $60 \% < \text{HQI} < 80$ % were manually verified.

Laboratory 2. Detection was performed on milk samples using a LabRAM HR Evolution μ -Raman spectrometer (HORIBA) equipped with a liquid-nitrogen CCD detector. Daily calibration was performed using the characteristic silicon Raman band at 520.7 cm^{-1} .

An overview of the entire filter was obtained using a $10 \times$ objective (Olympus MPlan N $10 \times /0.25$) under brightfield mode. Based on this overview, two non-overlapping regions ($2.5 \text{ mm} \times 2 \text{ mm}$ each, 5 mm^2) were randomly selected across the effective filtering area (~ 5 mm diameter) and analyzed using a $50 \times$ objective (Olympus LMPlanFL N $50 \times /0.50$), covering approximately 50 % of the filtration surface.

Spectral analysis was performed using the LabSpec software (v. 6.8.1.9). Particle counting, size measurement, morphological analysis, and spectral evaluation were carried out using the LabSpec Particle-Finder™ application. Compound identification was achieved by comparing the acquired spectra with both Horiba reference libraries and a custom-made spectral library.

User-defined morphological thresholds were applied to minimise noise and distinguish particles from the background using a real-time video image.

Spectra were collected in the $415\text{--}2060 \text{ cm}^{-1}$ range using a 532 nm excitation laser (25 mW power on the sample), with an acquisition time of 1 s and collecting two accumulations in order to remove spikes. A 600 lines/mm grating was used. The spectra were normalized and subjected to a polynomial baseline correction. Spectra with a Hit Quality Index (HQI) > 80 % were automatically assigned to PET. Spectra with HQI values between 60 % and 80 % were manually reviewed to confirm the identification.

2.4. Blank management and quality control

Despite strictly following the precautions recommended in [33], complete elimination of background contamination is not feasible. Therefore, it is essential to monitor any potential contamination introduced throughout the entire sample handling process [47]. Procedural blanks with ultrapure water were systematically performed in parallel

with the milk samples in both laboratories, in order to evaluate MP background contamination that could occur during enzymatic digestion, alkaline hydrolysis and filtration.

To ensure comparability and reliability of the results, each laboratory determined its own reporting limit (RL). The RL is defined as the minimum number of MPs that can be reliably quantified by the laboratory, depending on the polymer type, size class, and standard operating conditions [34]. The RL was established for each polymer based on the results of ten procedural blanks independently performed by each laboratory; however, in this study only the RL for PET was considered. It was determined taking into account the detection threshold imposed by the filter pore size, which in this case is $5 \mu\text{m}$, and calculated as follows:

$$RL [5 \mu\text{m}] = \text{mean} (10 \text{ blanks}) + 3 \times SD (10 \text{ blanks}) \quad (\text{Eq. 1})$$

All analyzed samples were compared against the corresponding RL determined for each laboratory. Values below the RL are considered not reliably quantifiable, while values above the RL can be reported.

3. Results and discussion

3.1. Reference values of PET reference materials

Reference PET particle numbers for HLB and LLB were previously determined by μ -Raman and μ -FTIR spectroscopy in a separate manuscript [46] prepared within the 21GRD07 PlasticTrace project [40]. Detailed instrumentation, acquisition parameters, and particle identification criteria are reported in Table S1, and PET particle numbers per tablet, categorized by size range ($5\text{--}10$, $10\text{--}20$, $20\text{--}50$, $50\text{--}100$, and $100\text{--}500 \mu\text{m}$) in accordance with ISO16094-2 [35,41], are summarized in Table 1. These PET RM values were used solely for spiking, method development, and accuracy evaluation through an interlaboratory comparison.

Given the inherent differences in spatial resolution between μ -Raman and μ -FTIR, and the additional challenges posed by the complex milk matrix, μ -Raman was selected as the primary analytical technique for the milk samples. For completeness, μ -FTIR measurements were also performed, supporting the observations previously observed. The corresponding μ -FTIR results are provided in the Supplementary Information (SI, Sect.1).

3.2. Raman spectroscopy

3.2.1. Evaluation of the sub-sampling strategy

To speed up the analysis and enable a more detailed investigation of smaller particles down to $5 \mu\text{m}$, a sub-sampling strategy was used that involved imaging three (Lab 1) or two (Lab 2) rectangular regions, covering a total of 55 % and 50 % of the filtration surface, respectively. These regions were selected to minimize focus loss caused by non-flatness of the filters and to reduce analysis time. The particle count obtained in the selected regions were averaged for each size range and then normalized to the entire filtration area in order to estimate the total number of particles. This method relies on the assumption that PET particles are homogeneously distributed across the filtration area to ensure accurate extrapolation. To verify whether imaging approximately 50 % of the filtration area provides a representative estimate of the total particle count, PET RM tablets from both HLB and LLB were dissolved in ultrapure water, filtered, and analyzed using the sub-sampling approach. The results were then compared to the known PET RM particle numbers for proper comparison. Fig. 2c and f show the PET RM tablets of HLB and LLB, respectively, particle size distributions extrapolated to 100 % of the filtration area, compared to the size distributions obtained from the analysis of the entire filters. No notable differences were observed between the two approaches in all size ranges, and the extrapolation accuracy was high for both batches: 87 ± 11 % and 97 ± 11 % for HLB, and 98 ± 21 % and 105 ± 32 % for LLB (Table 2), for Lab 1 and Lab 2, respectively. These results confirm that

Table 1Mean PET particle numbers per tablet (\pm SD, %RSD, $n = 10$) in HLB and LLB loading determined by μ -Raman and μ -FTIR.

Spectroscopic method	Loading	5–10 μ m	10–20 μ m	20–50 μ m	50–100 μ m	100–500 μ m	Particle number per tablet
μ -Raman	HLB	111 \pm 49	330 \pm 62	889 \pm 58	361 \pm 29	68 \pm 14	1759 \pm 141 (8 % RSD)
μ -FTIR	HLB	0	0	393 \pm 152	280 \pm 56	78 \pm 11	751 \pm 194 (26 % RSD)
μ -Raman	LLB	26 \pm 10	38 \pm 5	69 \pm 9	26 \pm 9	2 \pm 1	160 \pm 22 (14 % RSD)
μ -FTIR	LLB	0	0	31 \pm 17	26 \pm 10	3 \pm 1	60 \pm 14 (24 % RSD)

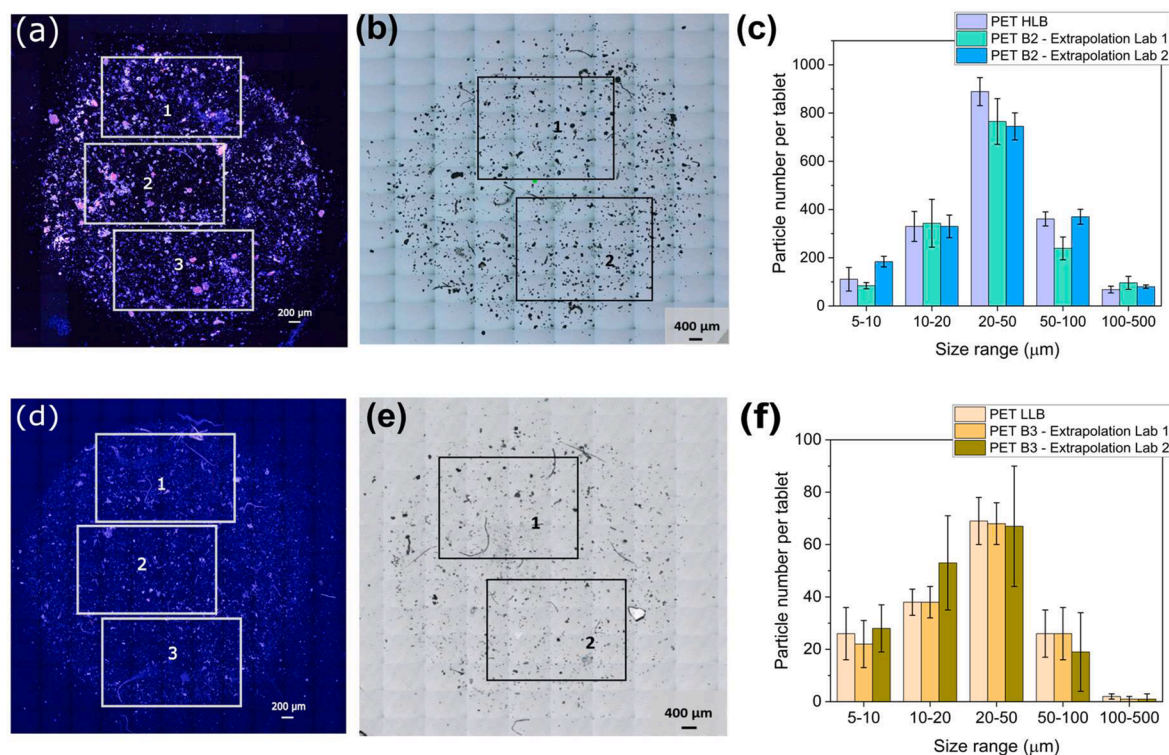


Fig. 2. Images of filters from high (a, b) and low (d, e) load batch showing the three (a, d) and two (b, c) selected regions located within circular filtration area (\sim 5 mm diameter) used for μ -Raman analysis. Bar graphs (c, f) report the PET particle size distributions obtained from the analysis of the entire filtration surface and from the extrapolation of the three selected regions to 100 % of filtration surface to the filtration area.

Table 2PET measurement accuracy using sub-sampling approach for area-based quantification for high ($n = 3$) and low ($n = 3$) load batch.

	PET particles per tablet	PET particles per tablet – Extrapolation (Lab 1)	PET particles per tablet – Extrapolation (Lab 2)	Extrapolation accuracy % (Lab 1)	Extrapolation accuracy % (Lab 2)
HLB	1759 \pm 141 (8 %)	1527 \pm 150 (10 %)	1709 \pm 142 (8 %)	87 \pm 11	97 \pm 11
LLB	160 \pm 22 (14 %)	156 \pm 25 (16 %)	168 \pm 45 (27 %)	98 \pm 21	105 \pm 32

the data obtained from the two or three selected regions (50–55 % of the filtration surface), when extrapolated to the entire filtration area, reliably represent the actual PET particles distribution, supporting the assumption of a homogeneous particle distribution across the filtration area. Based on this assessment, the sub-sampling method was systematically applied by both laboratories to all Raman measurements, including procedural blanks.

3.2.2. Evaluation of PET background

The evaluation of the PET RL and the analysis of the unfortified milk matrix were conducted by μ -Raman to assess potential background contamination and verify the suitability of the milk matrix for the analytical assessment using the PET RM.

In both laboratories, the RL for PET was determined via μ -Raman analysis of the procedural blanks from each laboratory, corresponding to

a total of 25 particles for Lab 1 and 21 particles for Lab 2. The unfortified milk matrix showed a PET number of 18 ± 5 as calculated in Lab 1 and 2 ± 2 in Lab 2. The slight differences observed in the values are most plausibly attributed to varying levels of background contamination resulting from the different operating environments of the two laboratories. The number of PET particles detected in the unfortified milk matrix in Lab 1 and Lab 2 was found to be below the RL calculated by each laboratory (Fig. 3). In addition, values of PET particle count measured by both laboratories are very low and negligible compared to the PET particle numbers per tablet for both batches (HLB: 1759 ± 141 (8 % RSD), LLB: 160 ± 22 (14 % RSD)). This confirmed the absence of significant background interference for the suitability of the analytical method for PET detection in infant milk powder, which will be confidently attributed to the tablet itself.

RL values for the most common polymer types were calculated using

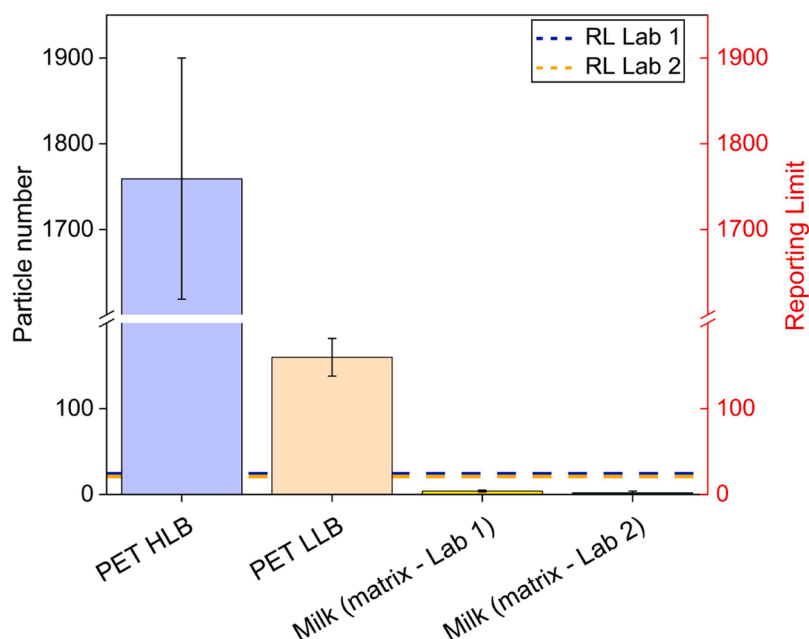


Fig. 3. Evaluation of PET background levels in unfortified milk compared to PET RM tablets in ultrapure water (HLB and LLB) by μ -Raman.

μ -Raman (Table S2) and used as benchmarks to assess possible background contamination from the unfortified milk matrix. PP, PS and PE were detected, but the particle count for these polymers in the unfortified milk matrix were all lower than their respective RLs, confirming the absence of MPs contamination in milk powder (Table S3).

3.2.3. Identification and quantification of PET SMPs in infant milk powder by μ -Raman

The reconstituted milk samples fortified with PET RM from both batches and subjected to digestion treatment (Fig. 1) were analyzed by Lab 1 and Lab 2 using μ -Raman to assess PET particle number and size distribution after the sample treatment protocol. Measurements were performed using the validated sub-sampling method, in which two or three representative regions of the filtration area were analyzed and the results extrapolated to estimate the total particle count over the entire filtration area. Results are reported as absolute particle counts per analyzed sample, following the operational definition in the Materials

and Methods (Sect. 2.3). Both laboratories yielded comparable results, with an average number for HLB of 1448 ± 253 (17 %) in Lab 1 ($n = 3$) and 1501 ± 203 (13 %) ($n = 3$) in Lab 2, and for LLB of 132 ± 17 (13 %) in Lab 1 ($n = 6$) and 140 ± 26 (19 %) in Lab 2 ($n = 6$) (Fig. 4). Both laboratories applying the same digestion treatment and using different laser sources and sub-sampling areas provided values that align well with the reference particle numbers obtained from PET RM tablets dissolved in ultrapure water (HLB: 1759 ± 141 (8 % RSD), LLB: 160 ± 22 (14 % RSD)). For both batches, no notable differences were observed across all size ranges between the two laboratories or when compared to the RM in ultrapure water. However, in the case of LLB, a more detailed comparison between laboratories, reveals some variability in the smaller size ranges (5–50 μm), as indicated by differences in their standard deviation values. The lower number of PET particles in this batch may increase the relative uncertainty within each size class. When particle numbers within a given size class are low, even a variation of one or two particles across replicates can substantially affect the standard

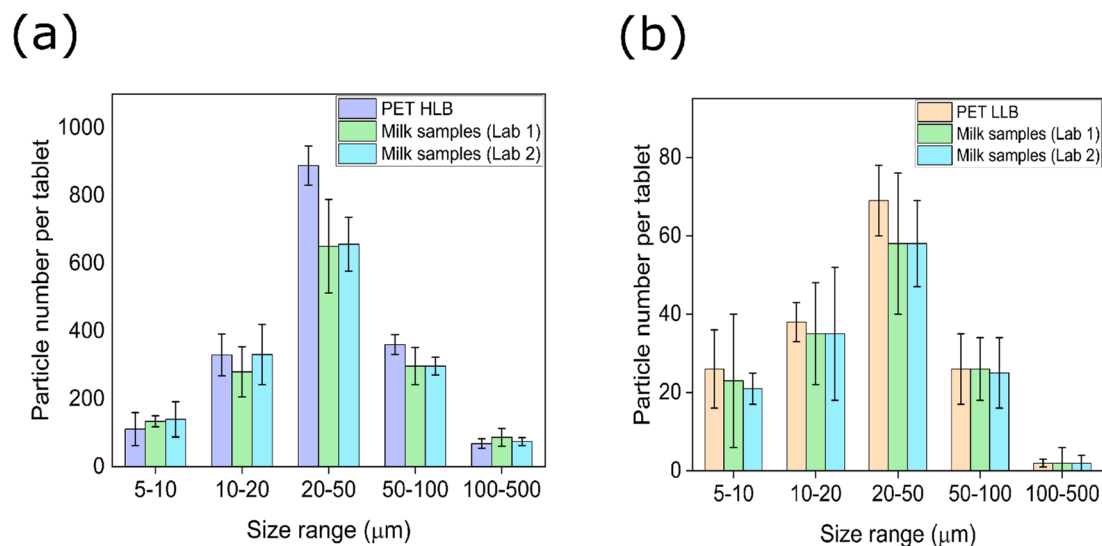


Fig. 4. . PET size distribution in treated infant milk after digestion procedure for high (a) ($n = 3$) and low (b) ($n = 6$) load batch by μ -Raman as measured by Lab 1 and Lab 2.

deviation. Moreover, the 532 nm laser used by Lab 2 offers higher spatial resolution than the 633 nm laser used by Lab 1, potentially allowing for more accurate detection and separation of small or overlapping particles, particularly in the 5–10 μm , and especially in the presence of residual organic matter from the matrix that interferes with particle recognition. Nevertheless, these variations remain within an acceptable range and do not affect the overall comparability of results between the two laboratories. One-way ANOVA confirmed no significant differences ($p > 0.05$) across the three size ranges (5–10, 10–20 and 20–50 μm) (Fig. S3) between the two laboratories. The recovery rate was $82 \pm 16\%$ and $85 \pm 14\%$ for HLB in Lab 1 and in Lab 2, respectively, and $82 \pm 15\%$ and $88 \pm 19\%$ for LLB in Lab 1 and Lab 2 (Table 3).

4. Conclusions

The lack of certified RMs remains a critical challenge for ensuring comparability of analytical methods for quantification of MPs in complex matrices. This study demonstrated the reliability and comparability of μ -Raman measurements for SMPs $> 5 \mu\text{m}$ in infant milk powder using well-characterized PET RM tablets, produced and characterized in accordance with ISO 17034 and ISO 33405 principles. The approach supports harmonized MP measurements across laboratories without the immediate need for a standardized method, provided that reference materials are available to underpin traceability.

The method was successfully applied across two laboratories, yielding comparable results in terms of particle number and size range distribution. Its successful application to PET, a polymer known for its sensitivity to digestion conditions involving high temperatures and the use of chemical reagents, demonstrates the robustness of the approach and suggests its potential applicability for quantifying other types of polymers with different sensitivities. Strict quality assurance and quality control measures, including procedural blanks and the calculation of reporting limit, were implemented to reliably distinguish PET RM MPs from potential background contamination.

Overall, this study not only demonstrates the feasibility of applying an analytical method for small PET MPs in a complex matrix but also highlights the importance of interlaboratory comparability and provides a practical foundation for harmonization of MPs quantification methods in complex matrices. With appropriate sample preparation and reporting limit, this methodological approach could represent a reliable analytical tool which can support routine monitoring of MP contamination across the dairy supply chain, aiding efforts to reduce both the number and types of MPs in consumer products.

CRedit authorship contribution statement

Mara Putzu: Conceptualization, Formal analysis, Investigation, Methodology, Validation, Visualization, Writing – original draft. **Marta Barbaresi:** Formal analysis, Investigation, Methodology, Writing – review & editing. **Marta Fadda:** Methodology, Writing – review & editing. **Alessio Sacco:** Writing – review & editing. **Maurizio Piergiovanni:** Investigation, Writing – review & editing. **Matteo Masino:** Methodology, Writing – review & editing. **Federica Bianchi:** Formal analysis, Writing – review & editing. **Korinna Altmann:** Writing – review & editing. **Nizar Benismail:** Formal analysis, Writing – review & editing. **Laureen Coïc:** Formal analysis, Writing – review & editing. **Ivana Fenoglio:** Supervision, Writing – review & editing. **Monica Mattarozzi:** Investigation, Methodology, Validation, Writing – review & editing. **Andrea Mario Rossi:** Supervision, Writing – review & editing. **Maria Careri:** Conceptualization, Funding acquisition, Methodology, Resources, Supervision, Writing – review & editing. **Andrea Mario Giovannozzi:** Conceptualization, Funding acquisition, Investigation, Project administration, Supervision, Writing – review & editing.

Table 3

PET recovery rate calculated by Lab 1 and Lab 2 in treated infant milk for high ($n = 3$) and low ($n = 6$) load batch by μ -Raman.

	PET particles per tablet	PET particles per tablet in Milk – Lab 1	PET Particles per tablet in Milk – Lab 2	Recovery % - Lab 1	Recovery % - Lab 2
HLB	1759 \pm 141 (8 %)	1448 \pm 253 (17 %)	1500 \pm 204 (14 %)	82 \pm 16	85 \pm 14
LLB	160 \pm 22 (14 %)	131 \pm 16 (13 %)	140 \pm 24 (17 %)	82 \pm 15	88 \pm 19

Declaration of competing interest

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

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.talo.2025.100586](https://doi.org/10.1016/j.talo.2025.100586).

Data availability

The data that support the findings of this study are openly available in Zenodo at <https://doi.org/10.5281/zenodo.17483289>

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