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Journal of Hazardous Materials 503 (2026) 141113

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

Hidden pollutants in food: Evidence of small microplastic particles (100–5 μm) in refined sugar from the Italian market

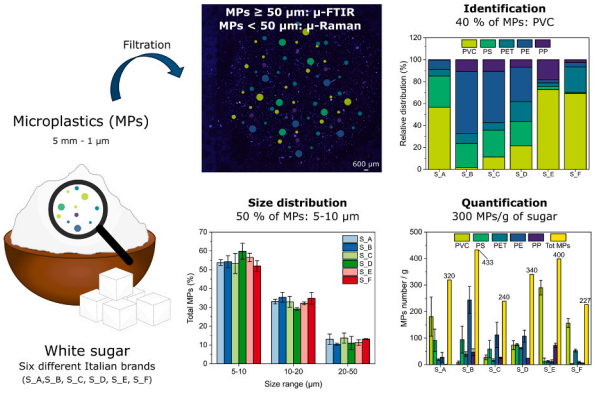
Marta Fadda , Alessio Sacco , Andrea Mario Rossi , Andrea Mario Giovannozzi *

Quantum Metrology and Nano Technologies Division, Istituto Nazionale di Ricerca Metrologica (INRiM), Torino 10135, Italy

HIGHLIGHTS

- Microplastics (MPs) were detected in six leading Italian brands of white sugar.
- Simple prep: sugar dissolved in hot water and filtered, no chemical digestion.
- MPs ≥ 50 μm identified by μ-FTIR; MPs < 50 μm down to 5 μm quantified by μ-Raman.
- μ-Raman revealed ~300 MPs/g sugar, mostly in the 5–10 μm size range.
- PVC was most abundant, raising food safety concerns and EU policy implications.

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:
Microplastics
Sugar
Vibrational spectroscopy
Fourier-transform infrared spectroscopy
Raman spectroscopy
Polyvinyl chloride

ABSTRACT

Microplastics (MPs) are emerging contaminants increasingly reported in food, including sugar. This study presents a novel analytical approach enabling the detection of MPs < 20 μm without chemical digestion, preserving particle integrity through simple dissolution in ultrapure water. White sugars from major Italian brands and distributors were analyzed using μ-FTIR for MPs ≥ 50 μm and μ-Raman for particles between 5 and 50 μm. The μ-Raman results are semi-quantitative and based on sub-sampling of 34 % of the filter area. While μ-FTIR detected relatively few MPs, μ-Raman revealed that the majority of particles fell below 20 μm, with a strong predominance in the 5–10 μm range. PVC emerged as the most abundant polymer, followed by PE, PS, PP and PET. The presence of MPs in a widely consumed product raises questions regarding food safety and human exposure. The predominance of MPs < 20 μm - a fraction not fully addressed by current EU regulatory frameworks - underscores the need for harmonized, standardized methodologies for monitoring MPs in food.

1. Introduction

Microplastics (MPs) - plastic particles with diameters between 5 mm and 1 μm, according to ISO/TR 21960:2020 [1] - have emerged as a growing global concern due to their adverse implications for both environmental and human health. While MPs were initially recognized

* Corresponding author.
E-mail address: a.giovannozzi@inrim.it (A.M. Giovannozzi).

https://doi.org/10.1016/j.jhazmat.2026.141113
Received 14 September 2025; Received in revised form 8 January 2026; Accepted 9 January 2026
Available online 10 January 2026

for polluting the oceans and harming marine ecosystems roughly a decade ago [2–4], they have more recently been detected throughout the global food chain [5–7]. Contamination has been reported in seafood [8,9], drinking water [10,11], table salt [12,13], and even fruits and vegetables [14,15], raising increasing concern about their potential impact on food safety and human exposure. Due to their small size, they can infiltrate food production systems through soil, water, and air, ultimately affecting both agricultural systems and natural ecosystems [5, 16–18]. Although the exact health risks associated with dietary exposure to MPs are still under investigation, recent studies suggest that these particles may induce inflammatory responses, disrupt endocrine functions, and act as vectors for harmful chemicals and additives [19]. Addressing the presence of MPs in food therefore requires coordinated improvements in waste management, industrial processes, agricultural practices, and consumer behavior.

Sucrose ($C_{12}H_{22}O_{11}$) is a naturally occurring disaccharide found in fruits, vegetables and nuts and is also produced on a large commercial scale [20]. Industrial sugar production mainly relies on sugar beet and sugar cane, processed through multi-step refining operations that transform raw plant material into crystalline sugar [21–25]. Across this chain - from harvesting to crystallization and final packaging - MPs may be introduced from processing equipment, filters, storage units and packaging materials, including composite paper–plastic laminates [21]. Consequently, refined sugar has emerged as a potential yet insufficiently studied source of dietary MP exposure.

Several studies have examined MPs in commercial sugars. Makhdoumi et al. reported 57.7 ± 20.6 particles/kg by optical microscopy and 226 ± 99.5 particles/kg after Nile red staining, predominantly PE and PP fragments between < 500 μm and > 5 mm [26]. Yurtsever et al. analyzed 19 store-bought beet sugars and found an average of 29,110 MPs/100 g, mainly fragments < 50 μm composed of PE, PP, PVC, PA, acrylic derivatives, additives and paints, using optical microscopy, Nile red staining, fluorescence microscopy, SEM and ATR-FTIR after peroxide treatment [21]. Patterson et al. identified 18–34 items/100 g in white sugar and 4–20 items/100 g in brown sugar (270 ± 81.8 and 103 ± 85 items/kg), mostly < 1 mm, dark, fibrous MPs made of PP, PET, cellulose propionate and poly(ether urethane), characterized by ATR-FTIR, SEM and EDAX [27]. Together, these studies indicate that sugars can be contaminated with MPs originating from processing, airborne deposition and packaging.

Despite these findings, current studies show two key limitations. Most analyses target MPs larger than 0.5 mm [26], or rely on optical and ATR-FTIR approaches that lack the sensitivity to detect MPs below 50 μm - particularly those < 10 μm, which pose the greatest concern due to their potential to cross biological membranes [27]. Additionally, many methods involve digestion steps such as peroxide oxidation, which may degrade or fragment MPs, leading to underestimation of smaller particles. These limitations underscore the need for analytical approaches capable of detecting MPs below 50 μm in sugar using high-resolution spectroscopy and minimal sample manipulation.

In this study, we addressed this gap by analyzing sugars from major Italian brands and distributors using a digestion-free protocol, based solely on dissolution of sugar in ultrapure water to preserve particle integrity. Samples were selected across different packaging formats, i.e. 1 kg paper bags, paper packets, and plastic packets, to capture a representative range of possible contamination sources. MPs measuring ≥ 50 μm were quantified and identified using μ-FTIR, whereas particles between 5–50 μm were analyzed using μ-Raman spectroscopy. Notably, the analysis revealed that 60 % of detected MPs fell within the smallest size class (5–10 μm). Only spectra with a Hit Quality Index (HQI) greater than 60 % were accepted, followed by manual expert verification to ensure reliability. Rigorous quality assurance and contamination control procedures were implemented following ISO 16094–2 [28], including ten procedural blanks analyzed on different days to assess potential contamination from laboratory environments and equipment. To date, no study has quantified MPs below 50 μm in refined sugar using a combined μ-FTIR and μ-Raman approach.

This work fills that gap by providing the first digestion-free, spectroscopically validated assessment of MPs down to 5 μm in refined sugar, with particular emphasis on the smallest size fractions of potential toxicological relevance.

2. Materials and methods

2.1. Particles and chemicals

Triton-X-100 was purchased by Sigma Aldrich. Ethanol and acetone were purchased by VWR Chemicals. Ultrapure water was obtained by a MilliQ QI-7000 purification system (Merck Millipore, Germany). Six of the most famous brands and distributors of white sugar in Italy were selected and purchased at local supermarkets.

2.2. Glassware and lab equipment cleaning

The glassware was rinsed with Triton X-100 (0.01 % w/v), making sure that all faces and edges were in contact with it. Then, glass containers and bottles were filled with Triton X-100 (0.01 % w/v) and sonicate in an ultrasound bath for 5 min at maximum power. Afterwards, they were rinsed with acetone, filled with ethanol (10 % v/v), sonicated in an ultrasound bath for 5 min at maximum power.

2.3. Sugar filtration

5 g of sugar were dissolved in 500 mL of water at 80 °C by stirring to make the process easier and the final sugar solution more homogeneous. Then, the solution was filtered through a silicon square filter with a square pore size of 5 μm (MakroPorP12M5–500; Smart Membranes), which was placed into a stainless-steel holder, and then sealed between two gaskets, by using a glass filtration system. The filtration system, consisting of a glass flask, a fritted glass, and glass funnel, was connected to a vacuum pump and placed under a laminar fume-hood to reduce environmental contaminations during the filtration procedure. The filtration system was conditioned three times with Triton X-100 (0.01 % w/v) and ethanol with the vacuum pump turned on. Then, the sugar solution was poured into the funnel, which was rinsed with Triton X-100 (0.01 % w/v) and ethanol to collect some sample residues in it. Finally, the vacuum pump was stopped, the filter was removed from the filtration system and dried in the oven for 30 min at 60 °C to make it ready for the following analysis. All the samples were labeled as reported in Table 1, with specific indication of the packaging material and form. The packaging material is expressed as paper-based, plastic-based or Paper–Polymer laminate referring to the prominent material which they are made of, since they are composites of more polymers and/or fillers made to improve the quality of the packaging itself. To exemplify, the samples are labeled as S_*, where S indicates sugar, and * the brand.

2.4. Quality assurance and control (QA/QC)

To reduce potential contamination, laboratory user equipment was cotton lab coats and gloves throughout all the experiment. All the

Table 1
Sample label with reference to the packaging material (paper-based, plastic-based and Paper–PE laminate) and packaging form (big bag or packets used in cafès).

Label of sample	Packaging material	Packaging form	Number of replicates
S_A	Paper-based	1 kg bag	4
S_B	Plastic / PE-based	0.5 kg bag	4
S_C	Paper–PE laminate	5 g packets	4
S_D	Paper–PE laminate	5 g packets	2
S_E	Paper–PE laminate	5 g packets	2
S_F	Paper–PE laminate	4 g packets	2

containers were made of glass or plastic-free and cleaned accurately before each experiment, as reported in 2.2. All the experiments were conducted under a laminar-flow fume hood to have the experiment environment controlled as much as possible. Each day, two sugar samples and one procedural blank were filtrated together to monitor the possible contamination due to the filtration procedure and environment, following the same procedure.

As recommended in ISO 16094–2:2025, Section 10.5, quantitative results were expressed relative to the Reporting Limit (RL) for each polymer determined. RL is defined as the minimum number of particles that can be confidently reported by the method, based on the distribution of MP identified in multiple procedural blanks. This approach is conceptually analogous to the classical limit of detection (LOD), as also discussed in Schymanski et al., 2021 (section III. Determination of the limit of detection) [29], that is derived from the mean blank value plus three times of its standard deviation. Both documents emphasize that blank subtraction should generally be avoided in MP analysis because it may produce negative artefacts and distort particle-size distributions, especially in the < 50 μm fraction. For these reasons, our study follows the methodological framework recommended by the ISO standard, explicitly applying an RL-based quality-control scheme. This approach is particularly important when analyzing complex matrices such as refined sugar, where the smallest MPs (<20 μm) represent the most abundant and analytically vulnerable size fraction. In line with best practices for spectroscopic MP quantification, the RL ensures transparent and reproducible reporting while preventing over-interpretation of values close to background contamination.

2.5. μ-FTIR characterization

A μ-FTIR Nicolet iN10 infrared microscope (Thermo Scientific™) equipped with a KBr/Ge beam splitter and a mercury cadmium telluride (MCT) detector. The whole filtration area was imaged with a 15 × objective (N.A. = 0.7, working distance = 16 mm), an acquisition time of 3 s × 16 in a spectral range of 650 – 4000 cm^{-1} (resolution of 8 cm^{-1}). The acquired FTIR spectra of each detected particle was classified by polymer type after their comparison with an internal library which collects all the main plastic polymers. At the end of this analysis, dimensional properties and type of polymer with corresponding percentage matching with internal library spectrum were considered. Spectra with a correlation coefficient greater than 80 % were automatically assigned to a polymer type based on the internal library, while those with HQI values between 60 % and 80 % were manually verified.

2.6. μ-Raman characterization

A confocal μ-Raman LabRAM Odyssey (HORIBA Scientific) equipped with an CCD detector, a 50 × long working distance objective (Olympus, NA = 0.5), dark field illumination system, and a 633 nm laser source (laser power at the sample 17 mW), an acquisition time of 3 s × 1 in a spectral range of 620 – 1760 cm^{-1}, and a 600 l/mm grating. In this approach, a sub-sampling strategy analyzing two regions covering 34 % of the filtration area was employed to speed up the process and allow for more detailed investigation of smaller particles down to 5 microns. The total number of particles was then estimated by normalizing with respect to the total filtration area. The acquired Raman spectra of each detected particle was classified by polymer type after their comparison with an internal library which collects all the main plastic polymers. For the classification, the spectral range 1100 – 1800 cm^{-1} was considered to exclude the peak associated to the second order of silicon, avoiding false positives. At the end of this analysis, dimensional properties and type of polymer with corresponding percentage matching with internal library spectrum, expressed as Hit Quality Index (HQI), were considered. Spectra with a HQI greater than 80 % were automatically assigned to a polymer type based on the internal library, while those with HQI values between 60 % and 80 % were manually verified.

2.7. Statistical data treatment

Pairwise comparisons of PE particle counts were performed using Welch's two-sample *t*-test, which does not assume equal variances between groups. To ensure statistical robustness, tests were restricted to samples with sufficient replication (S_A, S_B, S_C; n = 4 per group). Statistical significance was evaluated at $\alpha = 0.05$, and results are reported as p-values for the corresponding pairwise contrasts.

3. Results and discussion

Six types of sugar of different Italian brand and packaged in different materials (paper-based or plastic-based packaging) and forms (1 kg bag, 0.5 kg bag, 5 g packets, 4 g packets) were analyzed to improve the variability and significancy of our results. MPs in sugar samples were identified and characterized by vibrational micro-spectroscopy techniques. As previously reported by Ciornii et al., μ-FTIR is a reliable technique for the identification, characterization and quantification of MPs bigger than 50 μm, while for smaller particles results are not affordable, as it is diffraction limited technique [30]. For a more complete analysis of all the MPs smaller than 50 μm, μ-Raman was used, since from the state of the art it gives reliable results for particles bigger than 1 μm [31]. With μ-FTIR, the entire filter was analyzed, whereas with μ-Raman a sub-sampling approach was employed, as previously recommended by ISO 16094–2 [28], and Schymanski at al. [29]. This sub-sampling method is based on subdividing the filtration area into two regions covering 34 % of the filtration area itself, which were analyzed using a 50 × objective to investigate smaller particles in greater detail.

3.1. Procedural blanks analysis: μ-Raman and μ-FTIR

Ten procedural blanks were made in ten different days to monitor the plastic contamination due to the filtration procedure or to the environment. In this way, it was possible to understand if the MPs found in sugar samples derived from the sugar itself or from our procedure.

Regarding μ-FTIR analysis, as can be observed in Fig. 1A, the number of MPs bigger than 50 μm, was very low, with only 4 particles in day 7, and zero or one particles in the rest of the days. The relative distribution (Fig. 1B) of the identified polymer types in the blank samples highlights PET (33 %) as the most abundant, followed by PE and PP (22 %), and then by PVC and PS (11 %).

Regarding μ-Raman, as can be observed in Figure C, the number of MPs was variable from a minimum of 3 and a maximum of 80 on day 9 and day 6, respectively. This high variability is expressed by their average number (± standard deviation) as 29 ± 24. Furthermore, as can be noticed in Figure D, it was observed that PE is the most present polymer (66 %) followed by PP (20 %), whereas all the other polymers were in a little percentage, 3 % for PVC, 8 % for PS, and 2 % for PET.

As determined by ISO 16094–2 [28], the analysis of ten control blanks allows the determination of the reporting limit (RL) per each polymer type, as follows:

$$RL = Mean(X) + 3 * Standard\ Deviation(X)$$

where X is the array that contains the number of MPs for each specific polymer for each day. The need to conduct this analysis on a high number of samples and in different days derives from the randomness and variability of contamination. In addition, the evaluation of RL has the aim of avoiding the subtraction of the control blanks from the samples to have stronger quantitative results. The RL for each polymer type is reported in Table 2 for both the techniques. The RL will be used to evaluate if the contaminations in the sugar samples are representative of the samples themselves: for instance, if the amount of PVC MPs in sugar is higher than the RL of PVC, the number of PVC MPs is considered a quantitative and reliable result, therefore derived from the sugar sample itself.

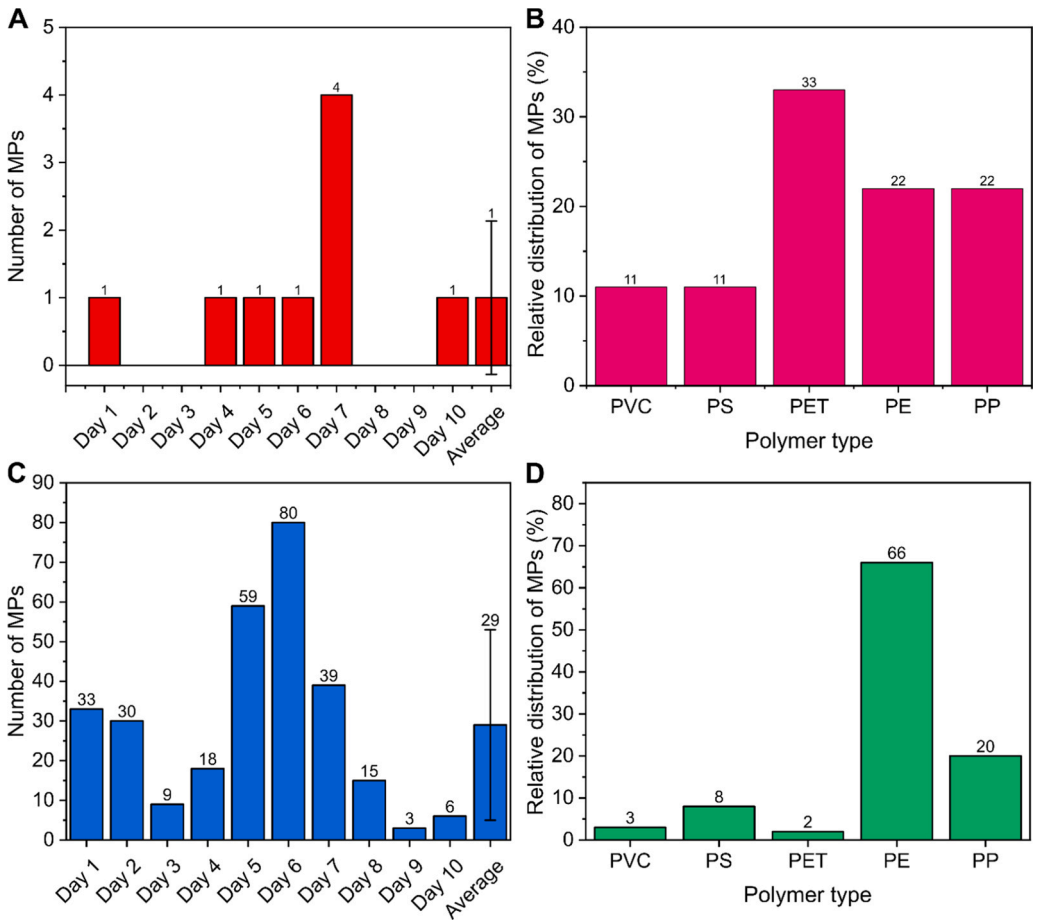


Fig. 1. Number of MPs for each procedural blank made in ten different days for (A) μ-FTIR and (C) μ-Raman. Relative distribution of the type of polymer in ten procedural blanks for (B) μ-FTIR and (D) μ-Raman.

Table 2
Reporting limit (RL) for each polymer type for μ-FTIR and μ-Raman expressed in MPs number.

Polymer type	μ-FTIR - Reporting limit (MPs number)	μ-Raman - Reporting limit (MPs number)
PVC	1	5
PS	1	11
PET	3	4
PE	1	84
PP	1	30

3.2. MPs determination in sugar samples

3.2.1. MPs ≥ 50 μm: μ-FTIR

Microplastics larger than 50 μm were analysed using μ-FTIR, which is considered suitable for this size range [30], and allows scanning of the entire filtration area without loss of information. The analysis focused on PVC, PS, PET, PE, and PP, as these polymers are among the most frequently reported contaminants in food and water and are listed as priority materials in the Drinking Water Directive Delegated Act (EU) 2024/336 [32]. The total number of MPs detected in each sugar sample is shown in Fig. 2. As illustrated in Fig. 2F, the abundance of MPs ≥ 50 μm ranged from 10 to 35 particles per 5 g of sugar, depending on the sample.

Polymer-specific results are presented in Fig. 2A–E, together with their corresponding reporting limits (RLs, Table 2). For PET and PE (Fig. 2C and 2D), only a few particles were detected (typically fewer than five), making these results less reliable. For the remaining polymers, more robust observations could be made. A detailed comparison between particle counts and their polymer-specific RLs is provided in Table 3. To facilitate interpretation, values are classified as follows: i) Red, if the mean value is above the RL, indicating that MPs originate from the sugar sample; ii) Yellow, if the mean is near the RL and the standard deviation crosses the threshold, representing borderline cases; iii) Green, if the mean ± standard deviation remains entirely below the RL, suggesting that detected MPs do not derive from the sugar itself.

It is important to note that these observations are based on a relatively small number of detected particles and should therefore be interpreted cautiously from a statistical perspective. Nonetheless, the findings are analytically meaningful: the sugar samples contained comparatively few MPs larger than 50 μm, underscoring the need for more detailed investigations targeting smaller size fractions, which are considered more relevant for human exposure. While MPs in the 100 μm –1 mm range are generally ingested with limited systemic uptake, particles smaller than 100 μm, and particularly those approaching the micrometer scale, have a greater likelihood of translocating across biological barriers and reaching internal tissues.

3.2.2. MPs < 50 μm: μ-Raman

3.2.2.1. 2-regions model validation. To speed up the analysis and enable a more detailed investigation of smaller particles down to 5 μm, a subsampling strategy was employed by imaging two square regions (2 mm × 2 mm), covering a total of 34 % of the filtration area (see Figures S1–S6, Supporting Information). These regions were selected to minimize focus loss resulting from slight filter non-flatness and to reduce

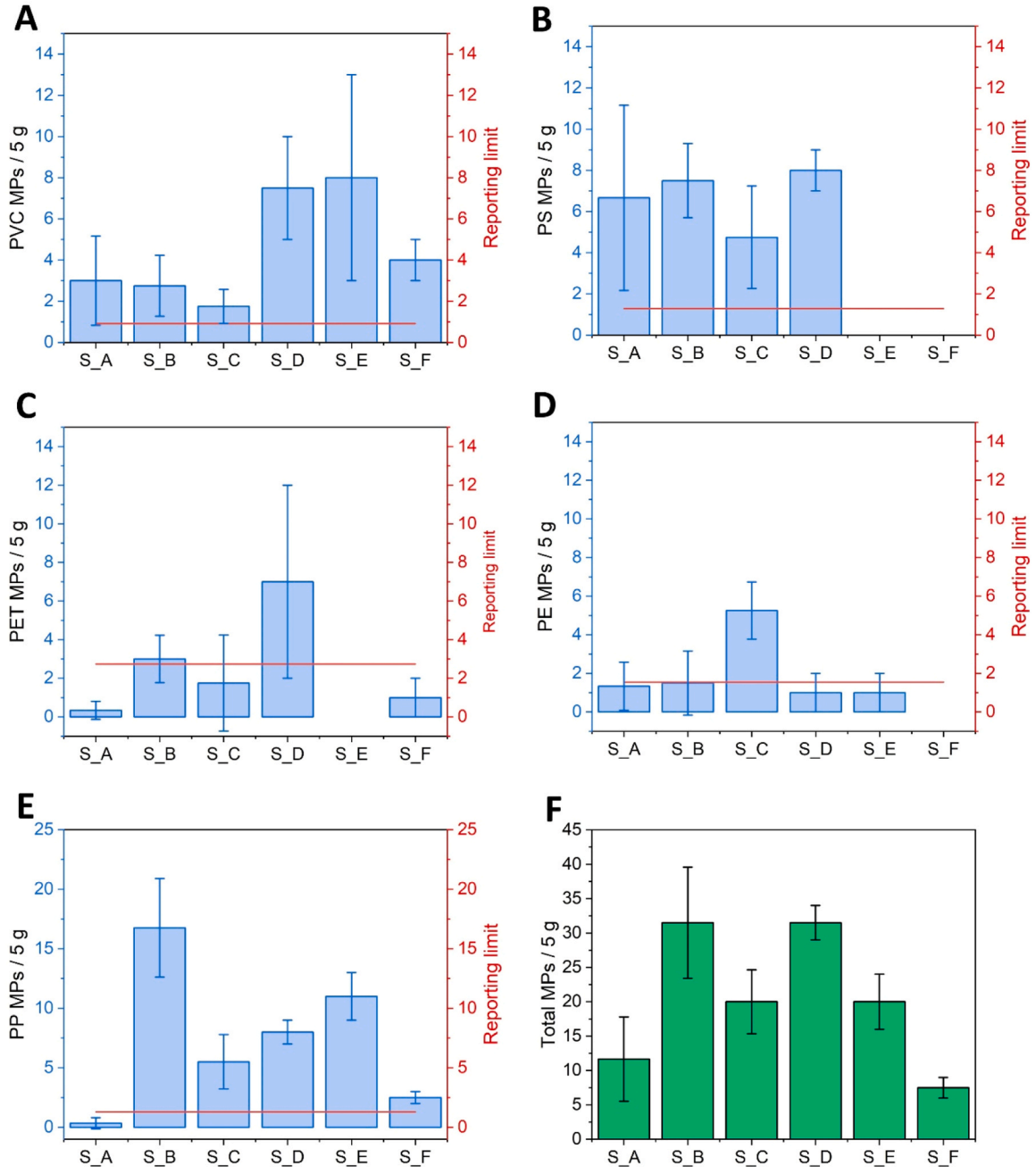


Fig. 2. μ-FTIR analysis: number of (A) PVC, (B) PS, (C) PET, (D) PE, (E) PP MPs with their respective reporting limit, and (F) total number of MPs detected in 5 g of each type of analyzed sugar.

overall analysis time. Particle counts obtained in these regions were then used to estimate the total number of particles on the filter. To verify whether analyzing 34 % of the filtration area provides a representative approximation of the total particle number, six random filters from different sugar samples were fully imaged at 20 × magnification. Both the entire circular filtration area (≈5.5 mm diameter) and the two selected 2 × 2 mm regions were scanned. Because particles smaller than 10 μm could not be reliably segmented at 20 × , a 10 μm cut-off was applied during image analysis to prevent error propagation. The same automated segmentation workflow (IsoData thresholding followed by morphological operations) was applied to all samples.

Under these conditions, the total number of particles per filter ranged from 3050 to 6400, while the two 2 × 2 mm regions contained 980–2500 particles, corresponding to an average of (37 ± 5)% of the total count (Fig. 3). This matches closely the geometric area fraction (34 %), indicating that particle deposition on the filter was homogeneous at the relevant spatial scale. These findings support the use of the random sub-sampling model recommended in the European consensus paper on μ-spectroscopic microplastic analysis by Schymanski et al., 2021 [29].

Since the total particle number N was experimentally measured, the associated uncertainty of the sub-sampling strategy was quantified using the ISO 16094–2-aligned formulation of the random model:

$$e = \sigma\sqrt{P(1-P)\left(\frac{1}{n} - \frac{1}{N}\right)}$$

where

e is the absolute uncertainty on the microplastic fraction P

n is the number of particles analysed in the two regions (980–2500),

Table 3
MPs number (average ± s.d.) for each polymer type detected in sugar sample: they are highlighted in red (contaminants that derive exclusively from sugar) if the average (± s.d.) is higher than the RL, in green if the average (± s.d.) is lower than the RL, in yellow if the negative/positive s.d. is under/over the RL, while the average is higher/lower than the RL.

Sample	PVC	PS	PET	PE	PP
S_A	3 ± 2	7 ± 5	n.a.	1 ± 1	n.a.
S_B	3 ± 2	8 ± 2	3 ± 1	2 ± 2	16 ± 4
S_C	2 ± 1	5 ± 3	2 ± 3	5 ± 2	6 ± 2
S_D	8 ± 3	8 ± 1	7 ± 5	1 ± 1	8 ± 1
S_E	8 ± 5	n.a.	n.a.	1 ± 1	11 ± 2
S_F	4 ± 1	n.a.	Under	n.a.	3 ± 1

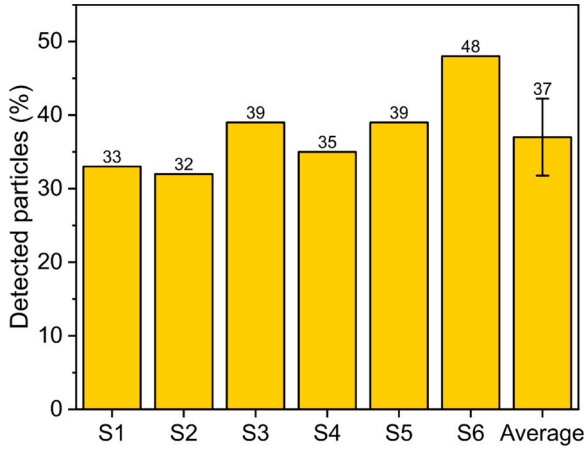


Fig. 3. Percentage of the particles detected in two areas (2 mm × 2 mm) respect to the number of the detected particles in the whole filtrating area for six sugar samples randomly chosen.

N is the measured full-filter particle count (3050–6400), and
$\sigma = 1.65$ corresponds to a 90 % confidence interval.

For the observed MP fractions (P = 0.07 - 0.28), the resulting absolute uncertainty is e = 0.004–0.022, corresponding to approximately 3 – 12 % relative uncertainty, with most samples falling around 5–8 %. It is important to note that this represents a conservative validation scenario, because the 20 × objective excludes the numerous particles in the 5–10 μm size range. In the actual μ-Raman workflow at 50 × , where particles down to 5 μm are detected, both the total number of particles *N* and the number of particles analyzed in the sub-sampled regions *n* increase substantially, and the microplastic fraction *P* may also increase. According to the random model, these increases would reduce the term $\left(\frac{1}{n}-\frac{1}{N}\right)$, leading to a lower sub-sampling uncertainty in the final 50 × measurements compared to the 20 × validation.

3.2.2.2. MPs detected in sugar samples. The following section reports the MPs identified by μ-Raman with a Hit Quality Index (HQI) > 60 %. After spectral comparison with the internal libraries, the most frequently detected polymers were PVC, PS, PET, PE and PP, whose abundances are shown in Fig. 4A–E. Table 4 reports the average (± standard deviation) number of MPs for each polymer and sample, together with the corresponding reporting limits (RL).

Across all samples, PVC consistently exceeds its RL and is one of the most abundant polymers, ranging from > 750 particles in S_A, S_E and S_F to between 250–500 in S_D, and < 250 in S_B and S_C. PS is present at intermediate levels (approximately 250–500 particles) in S_A–S_D, whereas for S_E and S_F the values fall below the RL, indicating that PS does not originate from these samples. PET is generally less abundant (typically <100 particles in S_A, S_C and S_E, and 100–300 in S_B, S_D and S_F), but for all samples its levels remain above the PET RL, confirming its presence in the sugars. PE represents the second most abundant polymer after PVC, with particularly high levels in S_B (~1250 particles), and substantial quantities in S_C and S_D (~500 particles). In S_A, PE counts are lower (~125 particles), while for S_E the results are inconclusive relative to the RL, and for S_F they fall below the RL. Finally, PP is detected in moderate numbers (100–350 particles) in S_B, S_C, S_D and S_E, while it remains below the RL in S_A and does not originate from the sample in S_F.

The total number of MPs quantified in the sugar samples is reported in Fig. 4F. All products contained more than 1000 MPs per 5 g of sugar, with concentrations ranging from approximately 1250 particles (S_C and S_F) to nearly 1500 (S_A), 1750 (S_D and S_E), and about 2000 particles in sample S_B. The relative uncertainties associated with the total MP counts were 10.5 % for S_A, 17.0 % for S_B, 24.7 % for S_C, 8.5 % for S_D, 7.6 % for S_E, and 8.1 % for S_F. Except for S_B and S_C, where the slightly higher variability reflects the intrinsic heterogeneity of their particulate composition, uncertainties remained within a narrow range of approximately 7–10 %. This behavior indicates that both the sampling and analytical workflow are reproducible, and that the particulate material is sufficiently homogeneous to support robust quantification. Regarding the sampling strategy, approximately 5 g of sugar were analyzed for each replicate, corresponding to the content of a standard single-serving sachet (≈1 teaspoon) and representing a realistic unit of daily consumer exposure. Although this quantity was not optimized through a dedicated mass-sampling variance study, the low inter-replicate variability demonstrates that it provides consistent, representative measurements for the purposes of this work.

The size distribution of MPs detected in the sugar samples is shown in Fig. 5 for each polymer type. Considering the total MP counts (Fig. 5F), approximately 60 % of all particles fall within the 5–10 μm range, about 30 % between 10–20 μm, and the remaining ~10 % between 20–50 μm. This distribution is consistent across polymers and samples.

PVC shows the strongest contribution, especially in S_A, S_E, and S_F, where its abundance clearly exceeds that of the other polymers. In contrast, S_B is characterized by a marked predominance of PE, whereas S_C and S_D display a more balanced distribution across polymer types. In addition to identifying polymers in the particles isolated from sugar, the packaging materials of all samples were characterized using μ-FTIR and μ-Raman spectroscopy, focusing specifically on the internal layers that are in direct contact with the product. These spectra, included in the Supplementary Information (Fig. S7-S8), show that the big-bag packaging used for sample S_A is predominantly cellulose-based, whereas sample S_B consists of polyethylene (PE). For samples S_C–S_F, which are commercially described as paper sachets, the analyses consistently revealed the presence of an inner PE layer, clearly detectable in ATR-FTIR and Raman spectra despite the increased fluorescence associated

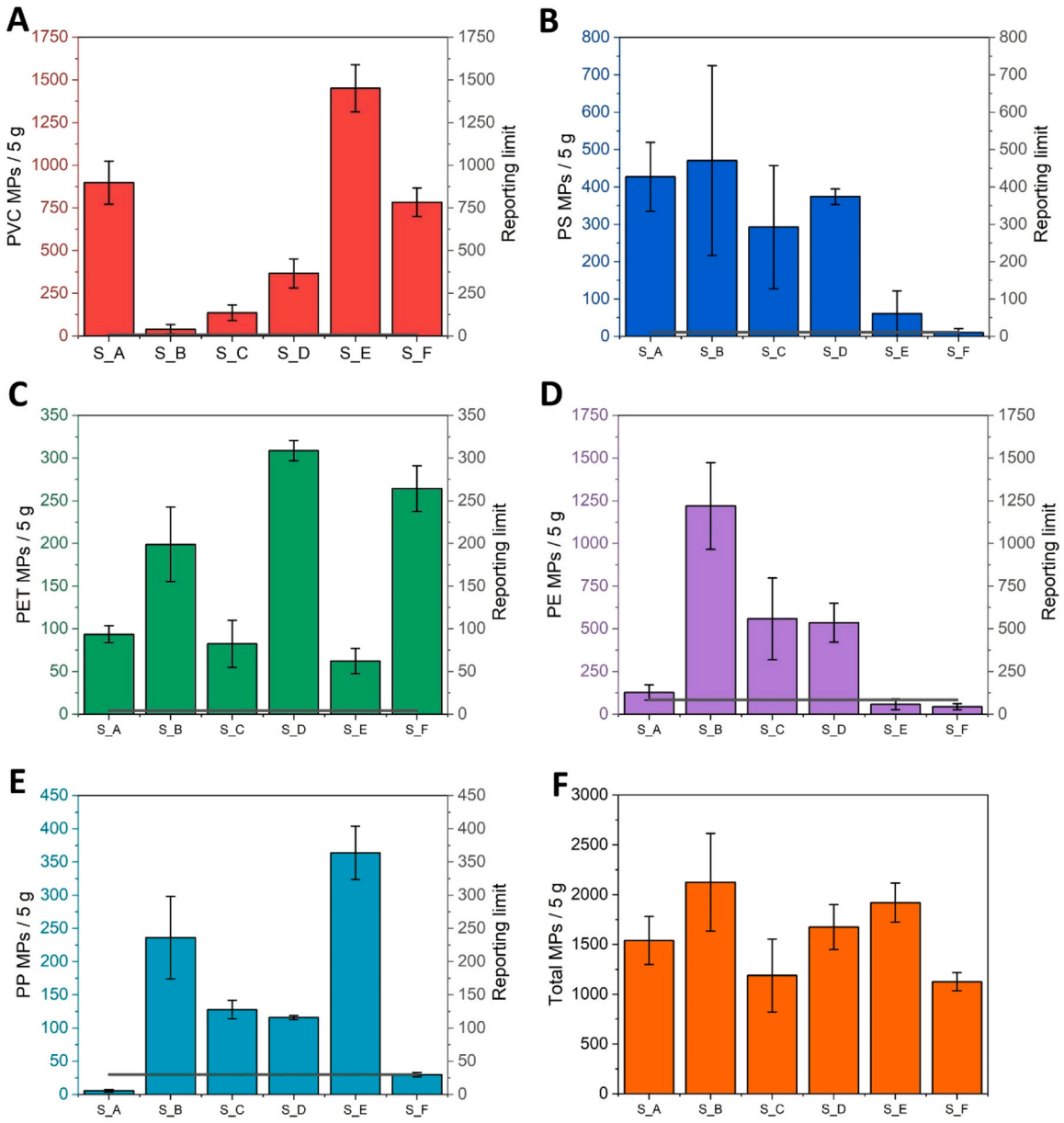


Fig. 4. μ-Raman analysis: number of (A) PVC, (B) PS, (C) PET, (D) PE, (E) PP MPs with their respective reporting limit, and (F) total number of MPs detected in 5 g of each type of analyzed sugar.

Table 4
MPs number (average ± s.d.) for each polymer type detected in sugar sample: they are highlighted in red (contaminants that derive exclusively from sugar) if the average (± s.d.) is higher than the RL, in green if the average (± s.d.) is lower than the RL, in yellow if the negative/positive s.d. is under/over the RL, while the average is higher/lower than the RL.

Sample	PVC	PS	PET	PE	PP
S_A	898 ± 126	427 ± 92	94 ± 10	127 ± 44	Under
S_B	39 ± 28	471 ± 254	199 ± 44	1219 ± 254	236 ± 62
S_C	136 ± 46	292 ± 165	82 ± 28	559 ± 239	128 ± 14
S_D	367 ± 85	374 ± 21	309 ± 12	536 ± 114	116 ± 3
S_E	1450 ± 138	Under	62 ± 15	58 ± 31	364 ± 40
S_F	784 ± 83	Under	264 ± 27	Under	30 ± 3

with cellulose-rich materials. The combined packaging analysis therefore supports a plausible correspondence between the PE identified

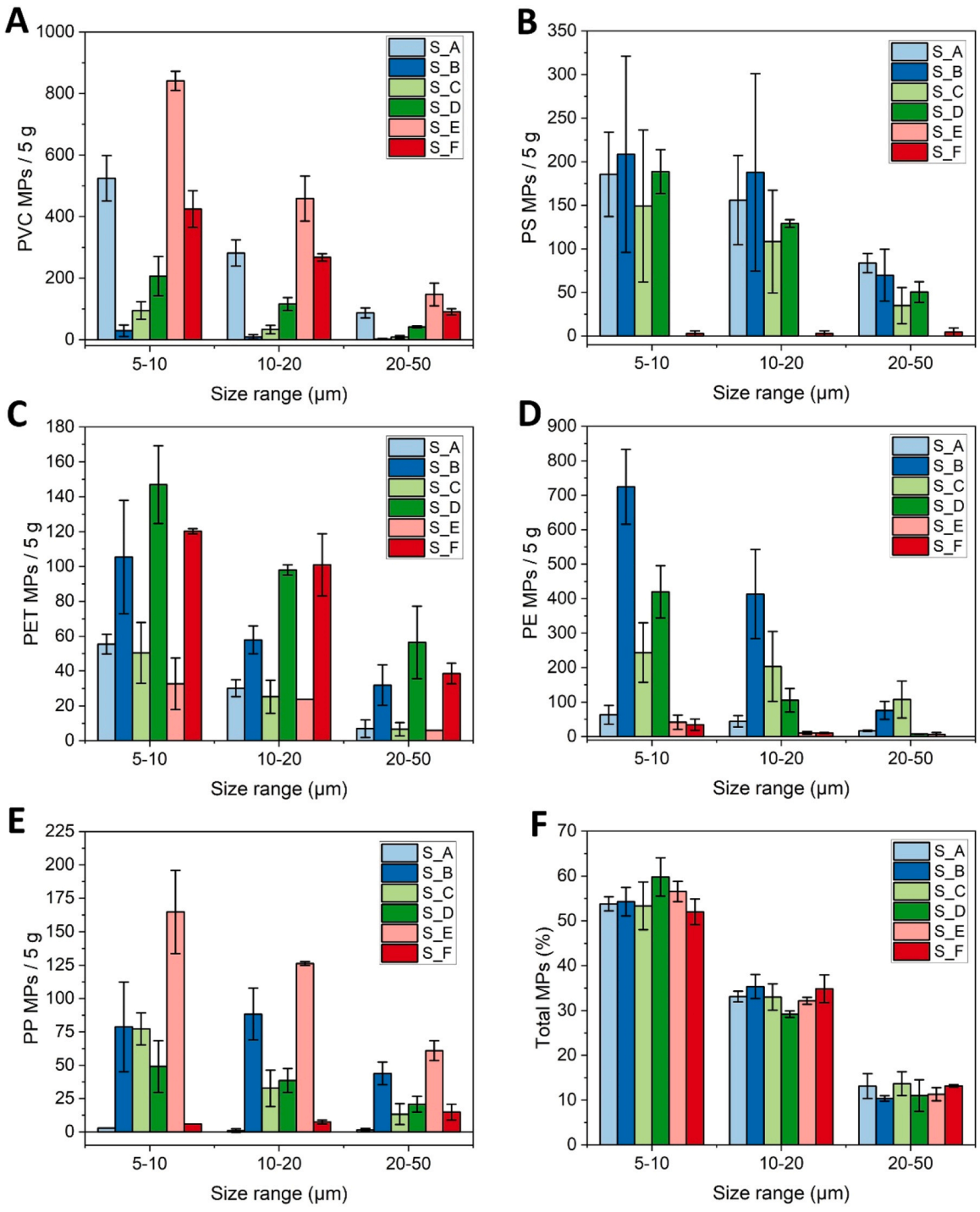


Fig. 5. µ-Raman analysis: size distribution of (A) PVC, (B) PS, (C) PET, (D) PE, and (E) PP MPs in 5 g of each type of analyzed sugar. (F) Relative distribution (%) of the total MPs for each size range.

within the sugar and the PE present in the packaging structures. To investigate whether PE abundance differed significantly among products, we restricted the statistical analysis to the three samples (S_A, S_B, and S_C) for which an adequate number of replicates ($n = 4$) was available, allowing a more reliable comparison. These samples also represent the three main packaging categories encountered in this study, i.e. paper-only (S_A), fully plastic/PE-based (S_B), and paper–PE laminate (S_C), and therefore provide a meaningful basis for assessing the potential contribution of packaging type to PE contamination. The results showed that sample S_B contained significantly higher PE particle counts than both S_A and S_C ($p < 0.01$). In addition, S_C exhibited significantly higher PE levels than S_A ($p < 0.05$). These findings indicate a clear difference in PE abundance among the three packaging categories. These results support the hypothesis that packaging configuration influences PE contamination, particularly when sugar is stored in direct contact with polyethylene layers, as in the fully plastic packaging of S_B. However, even if this conclusion can be drawn with higher confidence for S_B, the present dataset does not allow us to determine whether the PE detected in the paper–PE laminated product (S_C) originates from the inner polyethylene coating or from earlier stages of sugar processing. Dedicated packaging-transfer and migration experiments will therefore be required to differentiate packaging-derived

contributions from those introduced during production.

To provide a comparable quantitative measure, MP abundances were normalized to one gram of sugar (Fig. 6).

When expressed in MPs/g, our results reveal substantially higher contamination levels than previous reports, largely driven by the inclusion of particles < 50 μm, which earlier studies did not quantify. For example, Makhdoumi et al. reported ~0.058 MPs/g, focusing mainly on particles > 500 μm (fibers and fragments of PE, PP, PS, and PA). In contrast, our study detected roughly 300 MPs/g, dominated by PVC in the 5–10 μm class, representing an increase of approximately five orders of magnitude. Similarly, Yurtsever et al. measured concentrations around 0.1–1 MPs/g for particles mainly > 50 μm. Our results exceed these values by 2–3 orders of magnitude owing to the much larger number of small particles detected. Patterson et al. reported < 0.1 MPs/g, largely composed of fragments > 50 μm (PE, PP, PET). Using complementary μ-FTIR and μ-Raman, we found only 10–35 particles ≥ 50 μm per sample but more than 1000 particles < 50 μm, with PVC as the dominant polymer, an increase of 1–2 orders of magnitude relative to their reported levels. Altogether, these quantitative contrasts show that including the smaller size fractions and applying high-resolution spectroscopic techniques dramatically changes both the magnitude and the polymeric profile of MP contamination in sugar, underscoring the need for analytical approaches capable of detecting particles below 50 μm.

As one gram of sugar corresponds to half a teaspoon, we could infer that approximately 300 MPs could be ingested with this small quantity, regardless of the sugar type. Based on intake data reported by the World Health Organization (WHO, 2022), free sugar consumption is ~45–60 g/day for adults and ~45–75 g/day for children [33,34]. Using mean values of 50 g/day and 60 g/day, the estimated MP intake is therefore ~15 × 10^3 MPs/day for adults and ~18 × 10^3 MPs/day for children. These exposure levels are non-negligible and indicate a potential for human ingestion that warrants targeted toxicological evaluation. In terms of polymer distribution, PVC accounts for approximately 40 % of the MPs detected in the sugar samples. Within the European Union, PVC used in food contact materials is regulated under Commission Regulation (EU) No. 10/2011, which sets strict limits on vinyl chloride monomer migration, not allowing detectable levels above 0.01 mg/kg in food. Vinyl chloride monomer is a confirmed human carcinogen and exhibits documented toxicity to both humans and the environment [35–38]. The recurrent detection of PVC particles smaller than 50 μm across samples raises justified concerns regarding potential sources and pathways of contamination. Possible origins include abrasion or wear of processing equipment, such as PVC-based pipes, ducts, and filtration units commonly employed in sugar refining and drying lines, which have been reported to release micro-scale PVC fragments during operation [39]. Airborne deposition represents an additional route, as industrial dust and MP-rich particulate matter have been shown to settle on open product streams or processing surfaces in food manufacturing facilities [40,41]. These mechanisms are consistent with peer-reviewed studies documenting MP contamination in commercial sugars and processed foods, which similarly identify PVC among the dominant polymer types and confirm that such particles can occur even under controlled, plastic-free laboratory conditions [42].

Finally, although our analytical results are methodologically robust and consistently show measurable contamination across multiple independent sugar samples, the current number of analyzed products remains limited, and the dataset cannot yet support conclusions about the ubiquity of microplastic contamination across all sugar types or markets. Additional constraints include the semi-quantitative nature of the μ-Raman data (i.e. sub-sampling strategy), which, despite providing reliable MPs quantification, introduces uncertainty in absolute particle counts, as well as the inability of the present workflow to detect particles smaller than 5 μm, a size range that may substantially contribute to total particle abundance and potential human exposure. Taken together, these limitations highlight the need for broader, large-scale monitoring efforts to determine the generality of these observations. Only once a more comprehensive exposure baseline is established can dedicated toxicological studies be meaningfully designed.

4. Conclusions

This study provides the first quantitative assessment of microplastics down to 5 μm in commercial table sugar using a combined μ-FTIR and μ-Raman workflow supported by polymer-specific reporting limits. Procedural blanks allowed us to distinguish true sample-derived MPs from analytical background, ensuring robust attribution of polymer types. While μ-FTIR detected only limited numbers of particles ≥ 50 μm, often below their respective reporting limits, μ-Raman revealed a substantially higher abundance of smaller particles, with more than 1000 MPs per 5 g sugar sample and a size distribution dominated by the 5–10 μm fraction. PVC emerged as the most abundant polymer across all products, followed by PE, PS, PP, and PET.

Beyond reporting contamination levels, the study demonstrates the analytical importance of resolving MPs in the < 20 μm size range, which accounted for ~90 % of all detected particles. These findings underscore the need for improved standardization of analytical workflows and for expanding current regulatory monitoring frameworks, such as Commission Delegated Decision (EU) 2024/1441 on drinking water, which currently addresses MPs only down to 20 μm, to reliably capture this

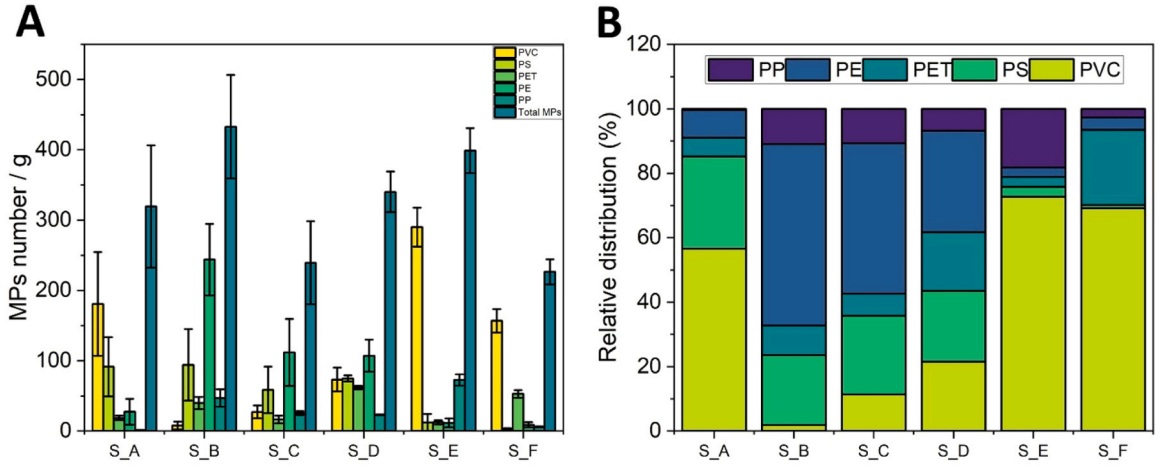


Fig. 6. μ-Raman analysis: (A) Number of MPs in one gram of each analyzed sugar, divided for polymer type and the corresponding total amount. (B) Relative distribution (%) of each polymer type for each analyzed sugar.

smaller and more numerous fraction in food matrices.

Overall, our work highlights both the novelty and necessity of high-resolution spectroscopic approaches for understanding microplastic contamination in refined sugar and provides a methodological foundation for future exposure assessments and regulatory development.

Environmental implication

This study demonstrates that refined sugar, a widely consumed food product, contains significant amounts of microplastics, with PVC emerging as the dominant polymer. The detection of high abundances of particles < 20 μm highlights the potential for human ingestion and raises concerns about cumulative environmental exposure through the food chain. These findings underline the urgency of harmonised analytical methods and stricter monitoring of microplastics in food. They also call for policy actions aligned with the EU Zero Pollution and Farm to Fork strategies to mitigate plastic contamination at its environmental sources.

CRediT authorship contribution statement

Andrea Mario Giovannozzi: Writing – review & editing, Supervision, Resources, Project administration, Methodology, Conceptualization. **Alessio Sacco:** Writing – review & editing, Validation. **Marta Fadda:** Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Andrea Mario Rossi:** Writing – review & editing, Supervision, Funding acquisition.

Funding

The PlasticTrace project has received funding from the European Partnership on Metrology (grant no. 21GRD07 PlasticTrace, Funder ID: 10.13039/100019599), co-financed by the European Union's Horizon Europe Research and Innovation Programme and the Participating States. METROFOOD-IT project has received funding from the European Union-NextGenerationEU, PNRR-Mission 4 "Education and Research" Component 2: from research to business, Investment 3.1: Fund for the realisation of an integrated system of research and innovation infrastructures-IR0000033 (D.M. Prot. n.120 del 21/06/2022).

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Andrea M. Giovannozzi reports financial support was provided by National Institute of Metrological Research. Andrea M Giovannozzi reports a relationship with National Institute of Metrological Research that includes: employment. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors thank Matteo Berruto (INRIM) for the support on the sample preparation of the analyzed samples, and Alina Maltseva (Horiba France) for the support on sugar sample preparation methodology and spectral library database selection.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jhazmat.2026.141113.

Data availability

The datasets generated and/or analysed during the current study are available in the Zenodo repository under the DOI: https://doi.org/10.5281/zenodo.18231214.

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