

Review Article

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Standard Methods for Isolation, Identification, and Quantification of Microplastics in Cosmetic and Personal Care Products

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Abstract

The increasing awareness of microplastic contamination in personal care and cosmetic products has driven the need for standardized analytical methodologies to ensure reliable data generation and international comparability. Microplastics, intentionally added as exfoliating microbeads or unintentionally introduced through packaging degradation, represent a significant pathway of primary plastic pollution into aquatic environments. However, variations in sample preparation, extraction efficiency, and analytical identification have resulted in inconsistencies among reported data.

This paper provides a comprehensive review of existing standard methods and emerging practices for the isolation, identification, and quantification of microplastics in complex cosmetic matrices. Standardized approaches developed by ISO (e.g., ISO 24187:2023), ASTM (e.g., D8332–20, D8333–20), and national authorities such as Canada and the European Union are critically evaluated. Extraction techniques, including enzymatic and oxidative digestion, solvent dissolution, and density separation, are compared with respect to their selectivity, recovery efficiency, and polymer integrity. Analytical techniques such as μ -FTIR, Raman microspectroscopy, thermal analysis, and pyrolysis–GC/MS are discussed as tools for accurate polymer identification and quantification.

The review identifies key methodological gaps: absence of product-specific validation, limited interlaboratory reproducibility, and lack of harmonized reporting units. To address these, a multi-tiered standardization framework is proposed that integrates sample pretreatment, contamination control, and traceability requirements across laboratories. Such harmonization would enable consistent regulatory monitoring, support cosmetic industry compliance, and contribute to global efforts to mitigate microplastic emissions at the source.

Keywords Microplastics; Standard Methods; Personal Care and Cosmetic Products.

1. Introduction

From an analytical chemistry perspective, personal care and cosmetic products (PCCPs) represent one of the most challenging matrices for microplastic analysis, character-

ized by complex emulsions, high organic content, and a wide diversity of polymeric ingredients [1]. The proliferation of these synthetic polymers in PCCPs has raised substantial environmental and human health concerns,



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necessitating the development of rigorous, standardized analytical methods to detect, identify, and quantify them [2, 3].

1.1 Defining Microplastics in the Context of Personal Care Products

Although the term ‘microplastics’ generally refers to any plastic particle smaller than 5 mm [4], a more precise definition is required for analytical and regulatory purposes. The International Organization for Standardization (ISO) provides this, defining a microplastic as a “solid plastic particle insoluble in water with dimension between 1 μm and 1 000 μm ” [5].

It is crucial to distinguish between two main categories of microplastics (Fig. 1):

- **Primary Microplastics:** These are polymers that are intentionally manufactured at microscopic sizes for direct use in commercial applications, such as the microbeads in facial scrubs [5, 6]. Cosmetic products are a key source of primary microplastics entering the environment.

- **Secondary Microplastics:** These particles originate from the environmental degradation and fragmentation of larger plastic items, such as bottles, bags, or fishing nets, under the influence of UV radiation, wind, and microbial activity [5, 6].

1.2. Sources and Functions of Microplastics in Cosmetic Formulations

Microplastics are incorporated into PCCPs for a variety of functional reasons (Fig. 2). While exfoliating microbeads are the most well-known, these polymers serve numerous other roles within complex cosmetic matrices.

- **Exfoliating Agents:** The most recognized function is their use as “microbeads”, small, spherical scrubbing particles in products like facial and body scrubs [8]. These are typically 200–400 micrometers (μm) in diameter and are often made from polyethylene (PE) or polypropylene (PP) [9].

- **Functional Ingredients:** Beyond exfoliation, microplastics are used as binders, bulking agents, emulsifiers, film formers, opacifying agents, and viscosity regulators to achieve a desired product texture, stability, and appearance [8, 10]. The use of these polymers as emulsifiers and viscosity regulators creates a complex organic matrix that complicates subsequent extraction and digestion steps, necessitating robust sample preparation protocols as detailed in Section 3.2.

- **Packaging Degradation:** Secondary microplastics can also be introduced into the environment through the slow degradation and fragmentation of the plastic packaging used for cosmetic products [8].

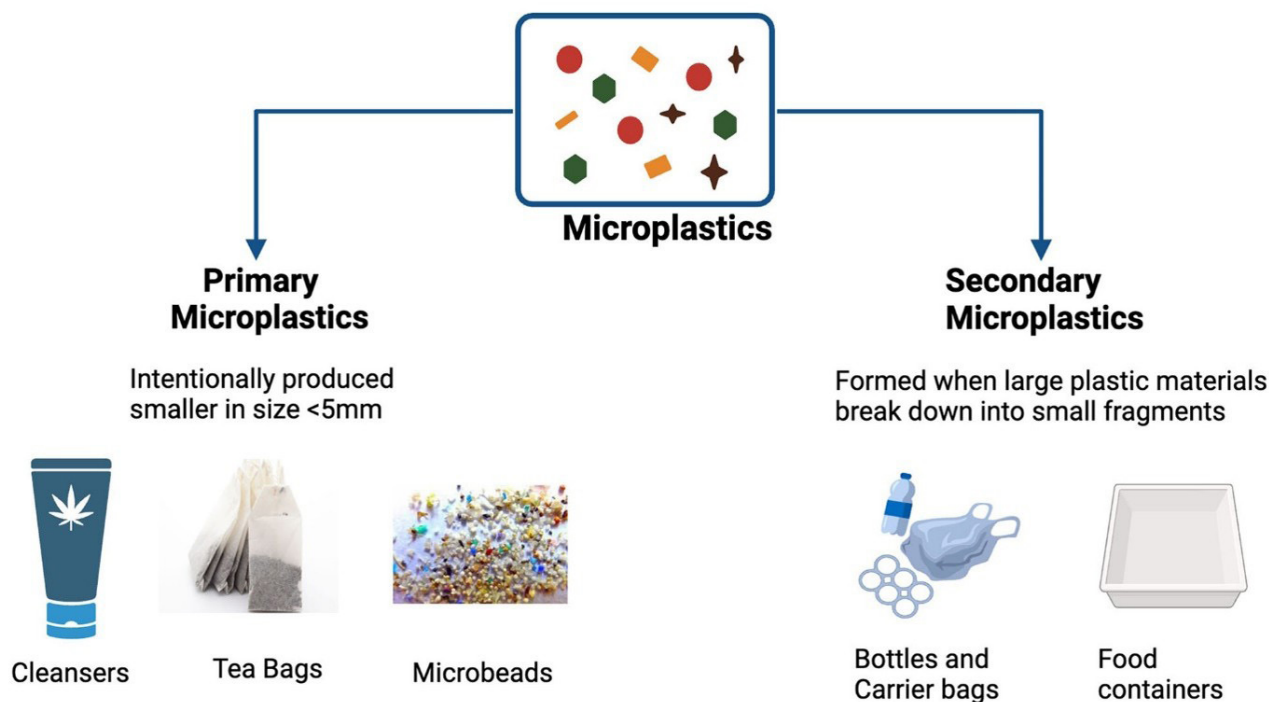


Fig 1. Two main categories of microplastics [7].

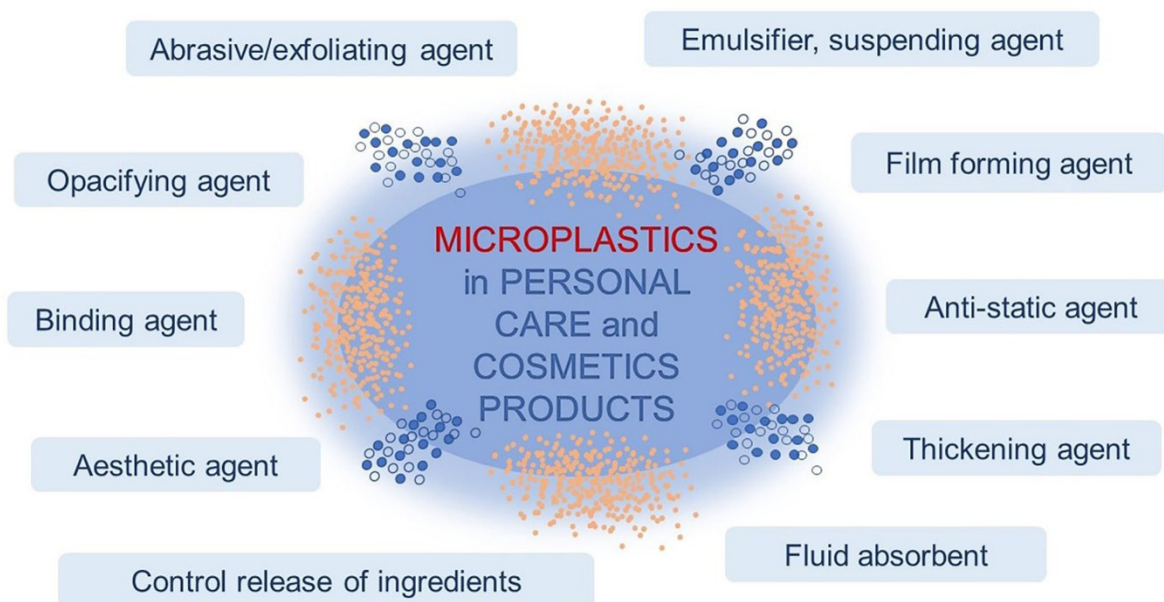


Fig 2. Variety of functional reasons that microplastics are incorporated into PCCPs [11].

1.3 Environmental Pathways and Health Concerns

The primary environmental pathway for microplastics from rinse-off PCCPs, such as facial scrubs, is through domestic drains. These particles enter the wastewater stream and are transported to wastewater treatment plants (WWTPs), where removal is often incomplete [12], allowing a significant quantity of microplastics to be discharged directly into aquatic ecosystems [13].

The environmental and health implications of this release are a subject of growing concern. Once in the environment, these persistent particles can be ingested by a wide range of organisms. The concern extends to human health, as recent studies have provided alarming evidence of microplastics being found not only in human blood [14] and placenta [15] but also in semen, testis, sputum, kidneys, and urine [8].

This evidence underscores the critical need for standardized analytical methods to accurately assess the scale of contamination and inform effective regulatory and mitigation strategies.

This article reviews the current international standardization efforts, details established laboratory procedures for sample preparation and analysis, evaluates the primary analytical techniques, and provides recommendations for future harmonization, based on a critical review of international standards and peer-reviewed literature from the past decade. These international standards form the necessary framework for generating comparable and reliable data on a global scale, a prerequisite for effective risk assessment and regulation.

2. A Review of International Standardization Efforts

In response to the global scale of microplastic pollution, a concerted effort has emerged among international and national standardization bodies to develop methods that ensure data quality, consistency, and comparability. The harmonization of analytical protocols is essential for creating a robust evidence base to support both scientific research and regulatory action. This section critically reviews the key initiatives from leading organizations, such as ISO and ASTM International, highlighting their distinct contributions to the development of a standardized framework for microplastic analysis.

2.1 The International Organization for Standardization (ISO)

ISO has been central to creating a harmonized framework for microplastic analysis, publishing a series of documents that provide principles and specific guidance for the entire analytical workflow.

• **ISO/TR 21960:2020:** This technical report serves as a foundational document, summarizing the state of knowledge on macro- and microplastics in the environment. It provides a comprehensive overview of the available methods for sampling, sample preparation (including separation and enrichment), and analysis, covering spectroscopic, thermo-analytic, and chemical extraction techniques [16].

• **ISO 24187:2023:** This international standard is a critical document that establishes general principles for the

entire analytical process. Its key recommendations provide a baseline for quality and consistency, including [5]:

- The mandatory use of plastics-free or low-plastics working conditions to prevent sample contamination.
- The need to clearly define analytical objectives, such as determining mass content versus particle number, which dictates the choice of subsequent methods.
- Guidance on sample preparation, including density separation and methods for removing organic matter from complex matrices.
- A framework for analytical quality assurance and control (QA/QC), emphasizing the use of reference materials and the performance of interlaboratory comparison tests to validate methods and ensure data reliability.

• **ISO 5667-27:2025:** This international standard provides specific guidance on sampling from water. It distinguishes between grab sampling, where a discrete volume of water is collected, and volume-reduced sampling methods like cascade filtration or net sampling, which concentrate particles from a much larger volume of water at the point of collection [17].

• **ISO 16094 Series (Drafts):** This series of draft standards is being developed to provide detailed protocols for specific analytical techniques. Part 2 focuses on identification using vibrational spectroscopy (FTIR and Raman) [18], while Part 3 addresses thermo-analytical methods for quantifying polymer mass content in waters with low suspended solids [19].

2.2 Other Standardization Bodies and National Methods

Alongside ISO, other organizations, such as ASTM International, have contributed to and are actively developing standardized methods for microplastic analysis. At the national level, some countries have established their own protocols; for example, Canada's Method 623.1 provides a specific procedure for this purpose [20]. Furthermore, organizations like the Organisation for Economic

Co-operation and Development (OECD) have developed well-established methods for assessing the biodegradability of materials (e.g., OECD 301 F), which are highly relevant for evaluating the environmental persistence of both conventional and alternative polymers used in PCCPs. While these individual efforts signify important progress, the overall landscape remains fragmented. This reinforces the need for greater international alignment to ensure that data generated in different regions and by different laboratories can be reliably compared. These overarching standards and methods directly inform the specific procedures used in the laboratory for the analysis of PCCPs.

3. Standardized Laboratory Procedures for Microplastic Analysis in PCCPs

A successful analysis hinges on a meticulous, multi-step laboratory process designed to isolate microplastics from cosmetic products while preventing contamination [21].

3.1. Sample Collection and Contamination Control

In the field of microplastics analysis, contamination is not merely a risk but a certainty that must be rigorously controlled. The ubiquity of airborne synthetic fibers and particles means that a result is only as reliable as the contamination mitigation procedures employed. As mandated by ISO 24187:2023, preventing contamination is the most critical first step. Essential precautions include [5]:

- **Avoidance of Plastic Lab Equipment:** Use glass, metal, or ceramic beakers, funnels, and tools whenever possible.
- **Non-Synthetic Protective Equipment:** Wear personal protective equipment made of natural materials, such as cotton lab coats, to avoid shedding synthetic fibers.
- **Strict Handling Protocols:** All glassware should be rinsed three times with particle-free water before use, and samples must be kept covered at all times to prevent contamination from airborne dust and fibers.

3.2. Sample Preparation: Extraction and Matrix Digestion

The first analytical step, which depends on the product's

Table 1. Methods for sample preparation

Method	Protocol and Source
Alkaline Digestion	A 10 wt% potassium hydroxide (KOH) solution at 55 °C with stirring for 5 days. This method was found to be highly effective (>95% efficiency) for cream-type cosmetics and did not cause chemical or physical damage to PE microspheres [23].
Oxidative Digestion	Use of Fenton reagent ($\text{FeSO}_4 + \text{H}_2\text{O}_2$) to break down organic-rich components in the matrix [22].
Solvent Extraction	Use of organic solvents such as ethyl acetate and chloroform to separate parabens and other soluble organic components from the solid particles [22].

formulation, is to separate the solid microplastic particles from its complex organic and aqueous matrix. For products with a simple matrix, such as some exfoliating scrubs, microplastics can be extracted by dissolving a known weight of the product (e.g., 5–10 grams) in warm water (45–50 °C) and subsequent filtration [22], whereas for more complex formulations like creams, lotions, and foundations, the organic matrix must be chemically removed (digested) to isolate the plastic particles. Several methods exist (Table 1), each with specific applications and efficiencies.

The conflicting reports on the efficacy of different digestion methods highlight the matrix-dependent nature of sample preparation; a method effective for one type of environmental sample, such as acidic digestion, may be entirely unsuitable for the organic-rich matrices of PC-CPs [23], underscoring the need for method validation specific to this product category.

3.3. Isolation: Density Separation and Filtration

After the initial extraction or digestion, further steps are needed to isolate the microplastics.

1. Density Separation: To remove dense inorganic materials (e.g., minerals, silica) from the sample, a technique called density separation is used. The sample is suspended in a saturated metal salt solution. Because polymers have a relatively low density (0.9–1.6 g/ml) compared to most minerals (>2 g/ml), the plastic particles will float while the heavier inorganic materials sink, allowing for their separation [16].

2. Filtration: The final isolation step involves passing the processed sample through a filter to capture the solid particles. This is typically done using vacuum filtration with specific filter types, such as Whatman filter paper (grade 42 or N°4) [22, 24]. For samples with high particle loads, cascade filtration, using a series of filters with progressively smaller pore sizes, may be necessary to prevent clogging [17].

3.4. Analytical Identification and Quantification

Once isolated on a filter, the particles must be identified (what polymer are they?) and quantified (how many are there, or what is their total mass?). A combination of spectroscopic and thermo-analytical techniques is typically employed.

3.4.1. Spectroscopic Methods

These non-destructive methods identify polymers based on their interaction with light. It should be noted that the stated size limits are instrument-dependent and represent the practical thresholds for routine analysis, below which signal-to-noise ratios and diffraction effects become pro-

hibitive, especially for FTIR.

• **Fourier Transform Infrared (FTIR) Spectroscopy:**

FTIR spectroscopy identifies a polymer by matching the vibrational frequencies of its constituent chemical bonds—which produce a characteristic absorption spectrum in the mid-infrared range—to entries in a validated spectral library [22, 9]. The lower particle size limit for conventional FTIR microscopy is typically 15–20 µm [18]. Modern FTIR imaging systems can automatically scan an entire filter, identifying and measuring thousands of particles in a single run.

• **Raman Spectroscopy:** Raman is a complementary vibrational spectroscopy technique that works by analyzing inelastically scattered light. It is particularly advantageous for identifying very small particles, with a detection limit down to 1 µm [18].

3.4.2. Thermo-analytical Methods

These destructive methods provide mass-based quantification of polymers.

• **Pyrolysis-Gas Chromatography/Mass Spectrometry (Py-GC/MS):** In this technique, the sample (e.g., the entire filter) is heated to very high temperatures in the absence of oxygen (pyrolysis). The pyrolysis thermally fragments the polymer into a reproducible set of smaller molecules (pyrolysate) whose pattern is unique to the original polymer, thus serving as a chemical fingerprint for identification and quantification [16]. A key advantage is its ability to analyze an entire sample at once, though it requires a minimum polymer mass of approximately 0.1–2 µg [19].

3.4.3. Staining and Microscopy

• **Nile Red Staining:** For particles that are too small or numerous for individual spectroscopic analysis, fluorescence staining can be a powerful tool. Nile Red is a dye that selectively binds to plastic surfaces, causing them to fluoresce under specific wavelengths of light. This allows for the rapid identification and counting of small microplastics (<106 µm) using a fluorescence microscope [23].

3.4.4. Data Processing and Validation

Identification relies on comparing sample spectra to reference libraries. The quality of this comparison is often expressed as a Hit Quality Index (HQI), which indicates the confidence of the match [9, 18]. For imaging techniques that generate enormous datasets, automated algorithms and machine learning are used for processing. The performance of these algorithms must be validated, for example, by using a confusion matrix to assess the rate of correct and incorrect classifications [5]. The selection of an appropriate analytical technique is ultimately dictated by the specific research question, as each

Table 2. Summary of existing international standards and technical guidelines relevant to microplastic analysis

Organization	Reference	Year	technical committee	Topic	Ref.
ISO	TS 21396	2017	ISO/TC 45	TRWP: mass concentration	25
ISO	TR 21960	2020	ISO/TC 61/SC 14	MPs and macroplastics: definitions, occurrence in the environment and biota	16
ISO	4484-1	2023	ISO/TC 38	MPs from textiles: washing test	26
ISO	4484-2	2023	ISO/TC 38	MPs from textiles: quantitative and qualitative analysis	27
ISO	4484-3	2023	ISO/TC 38	MPs from textiles: domestic washing	28
ISO	24187	2023	ISO/TC 61/SC 14	MPs in the environment: minimum requirements until specific standards are available	5
ISO	6775	2024	ISO/TC 61/SC 5	Plastics: identification	29
ISO	22638	2024	ISO/TC 45	TRWP: generation and collection in a road simulator	30
ISO	5667-27	2025	ISO/TC 147/SC 64	MPs in water: sampling	17
ISO	16094-2	DIS	ISO/TC 147/SC 2	MPs in water: microspectroscopic analysis	18
ISO	16094-3	DIS	ISO/TC 147/SC 2	MPs in water: thermoanalytical analysis	19
ASTM	D8332	2020	D19.06	MPs in water: sampling	31
ASTM	D8333	2020	D19.06	MPs in water: sample preparation	32
ASTM	D8489	2023(e1)	D19.06	MPs in water: particle/fiber size, distribution, shape, and concentration	33
ASTM	D8402	2023	D19.06	MPs in water: reference samples	34
ASTM	D8401	2024	D19.06	MPs in water: mass concentration	35
ASTM	87463	WK	D19.06	MPs in water: IR spectroscopy	36

method offers a unique balance of qualitative and quantitative information.

Table 2. Summarized existing ISO and ASTM standards and technical guidelines relevant to microplastic analysis.

4. Comparison and Evaluation of Standardized Methods

A critical evaluation of the primary analytical methods is necessary to understand their suitability for different research questions. The choice of method involves trade-offs between the type of information obtained, sample throughput, and potential interferences. In Table 3, spectroscopic and thermal methods used for the analysis of microplastics are compared.

This comparison highlights the complementary nature of these techniques and underscores the need for a harmo-

nized, multi-method approach to fully characterize microplastic contamination on a global scale.

5. Conclusions and Recommendations

The analysis of microplastics in cosmetic products is supported by a foundational framework of international standards, principally ISO 24187:2023. However, significant variations in specific laboratory procedures for sample preparation and analysis persist across different studies. To advance the field and generate globally comparable data, the following recommendations, grounded in the principles of analytical quality assurance outlined in the source documents, should be prioritized.

1. Develop Certified Reference Materials (CRMs): The creation and use of CRMs and matrix reference ma-

terials (RMs) are crucial for method validation and ensuring analytical accuracy. As specified in ISO 24187:2023 (Clause 15.1), these materials are essential for laboratories to verify method performance. However, developing such materials is uniquely challenging due to the vast diversity of polymer types, shapes, sizes, additive compositions, and weathering states of microplastics found in real-world samples.

2. Promote Interlaboratory Comparison: Laboratories must be strongly encouraged to participate in interlaboratory comparison tests, also known as proficiency testing. This process, governed by standards like ISO/IEC 17043 and recommended in ISO 24187:2023 (Clause 15.2), is the most effective way to assess and improve data comparability between different analytical facilities.

3. Harmonize Definitions and Scope: There is an urgent need to harmonize the definition of microplastics in regulatory frameworks to ensure consistency. This should include broadening the scope to address materials like synthetic waxes, which can be analytically challenging to distinguish from conventional plastics and may pose similar environmental risks [9].

4. Standardize Data Processing and Reporting: The algorithms and software used to process analytical outputs (e.g., spectra from FTIR, chromatograms from GC/MS) require standardization and validation. As suggested in ISO 24187:2023 (Clause 14), developing standardized data processing workflows, including those based on machine learning, will ensure that raw data is interpreted consistently, leading to more robust and comparable final results.

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Consent for publication

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Competing interests

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 33. ASTM D8489:2023e1 Standard Test Method for Determination of Microplastics Particle and Fiber Size, Distribution, Shape, and Concentration in Waters with High to Low Suspended Solids Using a Dynamic Image Particle Size and Shape Analyzer.
 34. ASTM D8402:2023, Standard Practice for Development of Microplastic Reference Samples for Calibration and Proficiency Evaluation in All Types of Water Matrices with High to Low Levels of Suspended Solids.
 35. ASTM D8401:2024, Standard Test Method for Identification of Polymer Type and Quantity of Microplastic Particles and Fibers in Waters with High to Low Suspended Solids Using Pyrolysis-Gas Chromatography/Mass Spectrometry.
 36. ASTM WK87463, New Test Method for Spectroscopic Identification and Quantification of Microplastic Particles in Water Using Infrared (IR) Spectroscopy.

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