WCSR Advice 2025-20

SCIENTIFIC COMMITTEE REACH (WCSR)

SAMPLING AND ANALYSIS OF MICROPLASTIC GLITTERS AND BEADS IN PERSONAL CARE PRODUCTS

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1. Glitter

1.1. Summary

Microplastic glitters are an emerging environmental contaminant due to their widespread use in cosmetics, arts, textiles, and consumer products. Their unique physical and chemical properties pose challenges for effective sampling and analysis. This report provides guidance on standardized methodologies for sampling, treatment, and identification to support regulatory measures under the REACH framework. Key recommendations include the use of oxidation and density separation for sample preparation and the combination of spectroscopic and thermal analysis techniques for reliable identification.

1.2. Introduction

The global consumption of plastic has been rising steadily, with annual production expected to reach 500 million tons by 2025. As plastics enter the environment, they are exposed to sunlight, oxidation, and mechanical stress, breaking down into microplastics (MPs)—particles smaller than 5 mm that pose a growing environmental concern. MPs are categorized as primary (intentionally produced, such as microbeads in cosmetics) or secondary (formed by the degradation of larger plastics like synthetic fibers). These particles are widespread, found in air, water, and soil, even in remote locations like mountains and oceans. Among primary MPs, glitter is a distinct category, composed of layered plastic with thin metallic coatings, commonly used in cosmetics, clothing, and crafts. Due to its small size, low density, and water-insoluble properties, glitter easily disperses in the environment, persisting for years.

Despite growing awareness of MPs, glitter remains an overlooked pollutant, partly due to misconceptions about its composition. While its metallic sheen suggests mineral content, glitters are primarily plastic, making them a potential source of microplastic pollution. Studies have detected glitter particles in environmental samples, including dust, wastewater, and sediments, yet research on their contamination levels remains limited. A better understanding of glitter pollution requires standardized methods for sampling, preparation, and identification. This review focuses on current research in these areas to highlight knowledge gaps and provide recommendations for future studies.

1.3. Challenges in sampling and identification of glitters

1.3.1. Extraction and Sample Preparation:

Glitters exist in various environmental matrices, including water, sediments, air, and consumer products hindering the correct identification. Their small size and reflective properties complicate detection and isolation. The effective extraction techniques. is crucial, as some reagents can alter the color of glitter by dissolving metallic coatings, making visual identification difficult.

1.3.2. Physical Identification:

Glitter particles range from 50 µm to several mm, making it difficult to apply a single analytical technique. Determining the size, shape, and color of isolated glitters is typically done using stereomicroscopy which lacks standardized criteria for distinguishing glitters from other particles, and it struggles to detect nano-sized plastic particles. Metallic layers on glitter cause light reflection, making imaging and microscopic analysis difficult. Transparent and color-changing glitters complicate size and color classification. SEM provides detailed surface morphology and have a lower detection limit but requires extensive sample preparation and may introduce contamination.

1.3.3. Chemical Identification:

Confirming the polymeric composition of glitters is essential to differentiate them from other particulates. Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy are the most common methods. FTIR has a detection limit of about 10 µm, making it unsuitable for smaller glitters, while Raman spectroscopy can identify particles down to 1 µm but suffers from fluorescence interference due to additives and colorants. Some studies mitigate these issues by using multiple excitation lasers or combining Raman and FTIR analysis while there is still a lack of standardized protocols for spectral matching. Pyrolysis-GC/MS is highly effective for determining polymer composition and associated additives. However, selecting the pyrolysis temperature is challenging as it may cause loss of key polymer markers and generates interfering peaks making polymer identification difficult.

1.3.4. Filter Selection and Contamination Risks:

The choice of filtration material affects both extraction efficiency and compatibility with analytical techniques. Most studies use cellulose-based filters (e.g., cellulose nitrate or cotton), which have minimal fluorescence interference, making them suitable for Raman or FTIR analysis. However, differences in filter pore size (ranging from 0.2 µm to >1 µm) influence the ability to capture smaller MBs, potentially leading to underestimation. Moreover, contamination from airborne microplastics or from plastic-based laboratory materials (e.g., plastic pipettes) remains a concern, requiring rigorous blank controls.

1.4. Recommended technique

1.4.1. SAMPLING AND PREPARATION

- **Oxidation** treatment with H2O2 or Fenton reagent to remove organic materials
  *Preventing the acidic conditions during Fenton oxidation may degrade coatings on MPs, reducing visibility.
- **Density separation** (following the oxidation) by NaI solution
- **Vacuum filtration** for Isolating glitters on the surface of cellulose-based filters (non-plastic filters)

1.4.2. Physical Identification

- **Stereomicroscope** for morphology (shape, size, color); Common shapes are Hexagonal, rectangular, irregular polygonal, square, circular, and star-shaped particles.
  *requires standard criteria for MP identification based on shape, color, and size
- **Laser diffraction** for particle size analysis.

1.4.3. CHEMICAL IDENTIFICATION

- **Pyrolysis-GC/MS**: Highly effective for determining polymer composition and associated additives
  *Recommended temperature ~600°C to prevent analyte degradation.

2. Microbeads in personal care products

2.1. SUMMARY

Microplastic microbeads, typically defined as spherical particles under 5 mm in diameter, are commonly used in personal care products, household cleaners, and industrial applications. Due to their widespread use and environmental persistence, microbeads pose significant ecological and human health concerns. This report provides an overview of sampling techniques and analytical methods relevant to the detection and characterization of microbeads, based on a literature review, to support regulatory measures under the REACH framework..

2.2. Introduction

Improper disposal contributes to environmental contamination, as plastics persist in ecosystems for decades due to their resistant polymeric nature. Exposure to environmental factors such as sunlight, oxidation, and mechanical stress causes plastics to degrade into microplastics (MPs), which are defined as solid plastic particles or fibers smaller than 5mm. MPs are categorized into primary MPs, which are intentionally manufactured at microscopic sizes for specific applications like microbeads (MBs), and secondary MPs, which form through the breakdown of larger plastic waste.

MBs are commonly used in personal care products (PCPs) such as cosmetics, toothpaste, and skincare items as exfoliating agents, viscosity enhancers, and waterproofing additives. Despite first being identified as a pollutant in 1991, MBs have only recently gained scientific and regulatory attention. With an estimated 4,130 tonnes of MB-containing cosmetics consumed annually in the EU, Norway, and Switzerland, concerns are rising over their environmental impact. Many MBs are washed into wastewater treatment plants, where some are captured in sludge, while others escape into aquatic environments, posing risks to both ecosystems and human health. Growing awareness of these impacts has increased the demand for effective analytical methods to assess MB contamination, supporting the development of regulations to mitigate their effects. This short report highlights the challenges related to MBs detection and explores the currently available methods for sampling, preparation, and identification across various matrices to aid in REACH regulatory compliance.

2.3. CHALLENGES IN THE SAMPLING AND IDENTIFICATION OF MICROBEADS

2.3.1. EXTRACTION AND SAMPLE PREPARATION:

MBs, with a size range between 3µm and 5mm, are embedded in complex cosmetic matrices containing organic and inorganic compounds, requiring effective extraction methods. The choice of extraction method is crucial, as some solutions may alter or degrade certain polymers, leading to biased results. Variations in extraction protocols across studies also complicate the comparison of findings.

2.3.2. PHYSICAL IDENTIFICATION:

Determining the size, shape, and color of isolated MBs is typically done using stereomicroscopy, which lacks standardized criteria for distinguishing MBs from other particles and it struggles to detect nano-sized plastic particles. Scanning electron microscopy (SEM) provides detailed surface morphology but requires extensive sample preparation and may introduce contamination.

2.3.3. CHEMICAL IDENTIFICATION:

Confirming the polymeric composition of MBs is essential for differentiating them from other particulates. Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy are the most common methods, but each has limitations. FTIR has a detection limit of about 10 µm, making it unsuitable for smaller MBs, while Raman spectroscopy can identify particles down to 1 µm but suffers from fluorescence interference due to additives and colorants. Some studies mitigate these issues by using multiple excitation lasers or combining Raman and FTIR analysis. However, there is still a lack of standardized protocols for spectral matching, as some studies relying on commercial libraries, while others use literature-based spectra.

2.3.4. FILTER SELECTION AND CONTAMINATION RISKS:

The choice of filtration material affects both extraction efficiency and compatibility with analytical techniques. Most studies use cellulose-based filters, such as cellulose nitrate or cotton, which have minimal fluorescence interference, making them suitable for Raman or FTIR analysis. However, differences in filter pore size (ranging from 0.2 µm to >1 µm) influence the ability to capture smaller MBs, potentially leading to underestimation. Moreover, contamination from airborne microplastics or from plastic-based laboratory materials (e.g., plastic pipettes) remains a concern, requiring rigorous blank controls and, where possible, the use of a clean room to minimize external contamination.

2.4. Recommended techniques

2.4.1. Sampling and preparation

- **Dissolution in Warm/Boiling Water:** The most common method for extracting microbeads from cosmetic products.
- **Vacuum Filtration:** Utilizes cellulose or nitrocellulose filter papers to collect microbeads.
- **Oxidation Treatment (Optional):**
    - **Fenton Reagent ($FeSO_4 + H_2O_2$)**: Highly effective in breaking down organic matter.

2.4.2. Physical Identification

- **Stereomicroscope (40-100× Magnification):** Most commonly used for assessing size, shape, and color.
- **Scanning Electron Microscopy (SEM):** For high-resolution morphology and surface roughness analysis.
- **Laser Diffraction (LDF):** For determining particle size distribution, including the nanometer size range.

2.4.3. Chemical Identification

- **Pyrolysis-GC/MS (Not used in reviewed studies but recommended):** Highly effective for determining polymer composition and associated additives.
- **Fourier Transform Infrared Spectroscopy (FTIR):**
    - **ATR-FTIR (Attenuated Total Reflectance-FTIR):** Used for thick samples.

    - **µ-FTIR (Micro-FTIR):** Enables simultaneous physical and chemical analysis at a microscopic scale.
    - Particularly sensitive to functional groups, providing insights into molecular composition.
- **µ-Raman Spectroscopy:**
    - **Excitation Lasers (455-785 nm):** Multiple lasers recommended to minimize fluorescence interference.
    - Enables simultaneous physical and chemical analysis at a microscopic scale.
    - Provides information on molecular structure and bonding, often complementing FTIR data.

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CONFLICT OF INTEREST

No member has declared any conflict of interest.

RAPPORTEUR(S)

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ADOPTION OF THE ADVICE

The Scientific Committee REACH advice was adopted by consensus on 28/05/2025.

LEGAL FRAMEWORK OF THE ADVICE

Cooperation agreement of 17 October 2011 between the Federal State, the Flemish Region, the Walloon Region and the Brussels Capital Region concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH)

Ministerial decree of 8 July 2014 appointing the members of the Scientific Committee REACH established under Article 3, § 3 of the Cooperation Agreement of 17 October 2011 between the Federal State, the Flemish Region, the Walloon Region and the Brussels Capital Region concerning the Registration, Evaluation, Authorisation and restriction of Chemicals (REACH)

Ministerial decree of 2 June 2016 on dismissal and appointment of members of the Scientific Committee REACH

Ministerial decree of 5 October 2016 on appointment of members of the Scientific Committee REACH

Ministerial decree of 13 June 2016 on appointment of members of the Scientific Committee REACH

Ministerial decree of 10 April 2019 on appointment of members of the Scientific Committee REACH

Ministerial decree of 28 July 2022 on appointment of members of the Scientific Committee REACH

Ministerial decree of 8 February 2024 on appointment of members of the Scientific Committee REACH

Ministerial decree of 8 October 2024 on appointment of members of the Scientific Committee REACH

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