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Characterization of microplastics in skim-milk powders

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ABSTRACT

The diffusion of microplastics in the food supply chain is prompting public concern as their impact on human health is still largely unknown. The aim of this study was to qualitatively and quantitatively characterize microplastics in skim-milk powder samples ($n = 16$) from different European countries ($n = 8$) through Fourier-transform infrared micro-spectroscopy in attenuated total reflectance mode analysis. The present study highlights that the use of hot alkaline digestion has enabled the efficacious identification of microplastics in skim-milk powders used for cheese-making across European countries. The adopted protocol allowed detection of 29 different types of polymeric matrices for a total of 536 plastic particles. The most abundant microplastics were polypropylene, polyethylene, polystyrene, and polyethylene terephthalate. Microplastics were found in skim-milk powders in 3 different shapes (fiber, sphere, and irregular fragments) and 6 different colors (black, blue, brown, fuchsia, green, and gray). Results demonstrate the presence of microplastics in all skim-milk powder samples, suggesting a general contamination. Results of the present study will help to evaluate the impact of microplastics intake on human health.

Key words: microplastics, analysis, Fourier-transform infrared micro-spectroscopy, dairy

INTRODUCTION

Microplastics (MP) are defined as plastic fragments, fibers, and beads with a diameter smaller than 5 mm, deriving from the degradation of larger plastic debris (SAPEA, 2019). The presence of MP in the environ-

ment is an increasingly relevant topic. The fact that plastic particles are present in food products has led to their inevitable exposure to humans which has raised particular concern. In particular, MP have been observed in several food products, such as fish (Bessa et al., 2018; Peters et al., 2018), meat (Kedzierski et al., 2020), dairy products (Kutralam-Muniasamy et al., 2020; Da Costa Filho et al., 2021), water (Oßmann et al., 2018), energy drinks, and soft drinks (Shruti et al., 2020).

The effects of MP on human health depend on the way in which the plastic particles enter the body but also on the source of exposure. Scientific evidence has shown that humans can be exposed to MP through ingestion of contaminated food and water, inhalation, and direct dermal contact through personal care products, textiles or dust (Prata, 2018; Kutralam-Muniasamy et al., 2023). Still, the ingestion of contaminated food represents the primary route of entry for MP in the human intestine (Van Cauwenberghe and Janssen, 2014). Microplastics are also able to reach the respiratory tract through the ciliary movements of the mucosa after inhalation (Salim et al., 2013). The uptake of MP through inhalation represents a risk for human health due to inflammation, chemical toxicity, and infection of microorganisms introduced to the body via MP (Wright and Kelly, 2017). The accumulation of particles in the respiratory system can cause acute release of pro-inflammatory chemotactic factors that can induce chronic inflammation, known as dust overload (Prata, 2018). Other health risks may arise from the dermal contact of MP used in hand detergents, face washes, face masks, and toothpastes. In fact, the presence of MP in personal care products have been associated with skin damage due to local inflammation and cytotoxicity (Sharma and Chatterjee, 2017). However, due to the limited information on the potential uptake and effects of MP at human health level, tolerance limits are still not defined (Kirstein et al., 2021).

Considering the potential toxicity of MP and their absorption into human cells, it is important to investi-

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gate sources, quality, and quantity of plastics in food to protect the consumer (Kadac-Czapska et al., 2023). Despite an increasing public concern over the presence of MP in the food chain (SAPEA, 2019), consumer awareness is mainly focused on marine ecosystems and fewer studies have investigated food products other than fish or crustaceans (Bessa et al., 2018; Peters et al., 2018). This is partially due to the lack of standard methods for processing MP in food (e.g., dairy products, meat, honey, and soft drinks). In respect to sample preparation, different digestion solvents are used to remove food's organic and inorganic compounds to facilitate MP observation and identification, the most common being alkaline digestion with potassium hydroxide (KOH; Dehaut et al., 2016; Guo et al., 2022). As concerns the analytical phase, several identification methods exist, but which either lack adequate sensitivity for correct polymer identification or specificity to correctly discriminate organic matter from MP, thereby increasing the risk of false positives (Bai et al., 2022). These methods are microscopy-based techniques such as Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). Instead, spectroscopic techniques such as Fourier-transform infrared micro-spectroscopy (μ -FTIR) and Raman spectroscopy (RS) are alternative analytical methods that are sensitive and non-destructive, allowing for the identification and quantification of different polymer residues in food samples (Bai et al., 2022; Kadac-Czapska et al., 2023).

Milk and dairy products play a key role in human nutrition and development throughout life (Thorning et al., 2016). Such products are routinely tested for the presence of pathogens and other chemical substances that could harm human health. Nevertheless, the extent of MP contamination in milk and dairy products and their impact on human health remain largely unknown (Kutralam-Muniasamy et al., 2020; Rahman et al., 2021). Microplastics contamination may occur at different stages along the dairy supply chain, with milking procedures, technological treatments, and packaging representing the major points of contamination risk. This raises concern on the possible implications of MP on human health as ingested via milk and dairy products (Diaz-Basantes et al., 2020).

In respect to milk powders, only Da Costa Filho et al., 2021 and Zhang et al., 2023 determined MP in 2 cow milk powders and 13 infant milk powders, respectively. In this light, there is a lack of studies which attempted to characterize MP in skim-milk powder samples on a broader scale. Therefore, the aim of this study was to qualitatively and quantitatively characterize MP in skim-milk powder samples from different European countries.

MATERIALS AND METHODS

Sample Collection

Procedures adopted in this study are excluded from animal ethics evaluation, as they do not reach thresholds established in the 'Directive 2010/63/EU (Art. 1) of the European Parliament and of the council of 22 September 2010 on the protection of animals used for scientific purposes'.

Skim-milk powder samples ($n = 16$) were produced and collected from 8 different European countries, including 1 sample from Austria, 1 sample from Belgium, 1 sample from Germany, 9 samples from France, 1 sample from Ireland, 1 sample from Italy, 1 sample from Netherlands, and 1 sample from Poland. In particular, 9 samples from France were produced in 9 different French dairy plants. All samples (2 kg of net weight) were collected using a multilayer paper bag with a polyethylene inner bag.

Sample preparation and analyses of MP were carried out in the laboratory of European Center for the Sustainable Impact of Nanotechnology (ECSIN, Padova, Italy), as part of Mérieux NutriSciences Company (Chicago, USA).

Reagents

All the procedures involving reagents filtration, reagents handling, and glass washing were carried out in a dedicated clean room, according to the guidelines described in the ISO 14644-1:2015 class 7 (ISO, 2015). Ultrapure water (18.3 M Ω /cm resistivity at 25°C) was obtained through Zener Power III (Human Corporation, Garak-ro, Republic of Korea). All solutions used during the preparation phase were previously micro-filtered using a silver membrane filter (3.0 μ m pore-size, 25 mm diameter; Sterlitech Corporation, Auburn, United States) and kept in glass containers to avoid any contamination. Glassware was washed 5 times using liquid dishwashing, then rinsed 5 times with deionized water and finally rinsed 5 more times with ultrapure water.

Sample Quartering, Digestion and Microfiltration

All the procedures adopted for sample quartering, digestion and filtration were carried out in a dedicated clean room, according to the guidelines described in the ISO 14644-1:2015 class 7 (ISO, 2015). Before digestion and microfiltration steps, sample quartering was performed to obtain a representative aliquot. To remove organic compounds from skim-milk powder samples, a digestion protocol was set up (adapted from Dehaut et

al., 2016 and EFSA, 2016). For all skim-milk powders, 15 g of sample were weighed in a 1000 mL glass flask. Then, 100 mL of ultrapure water and 10% KOH were added. To start the alkaline digestion process, samples were placed in Shake'n Bator (EuroClone, Milan, Italy) and heated up to 60°C overnight. Thereafter, 75 mL of 10% EDTA (**EDTA**) solution were added and samples were incubated in Shake'n Bator at 60°C for 2 h. Digested skim-milk powders were micro-filtered through a 3.0 µm pore-size silver membrane filter (Sterlitech Corporation, Auburn, United States) using a vacuum pump connected to a glass filter funnel. To enhance the separation of MP from organic compounds, a density separation step was applied. This action is aimed at re-suspending MP characterized by higher density. This step is performed by using an oversaturated solution of sodium chloride (NaCl) added to the same glass flask used for the filtration of the sample. After the density separation step of each sample, the solution containing MP was subjected to vacuum filtration. During filtration, the filtering funnel was covered with aluminum foil to minimize contamination. All filters were stored in previously decontaminated glass Petri dishes to prevent contamination. Finally, filters were left to dry at 70°C in an oven until analysis carried out in Fourier-transform infrared micro-spectroscopy in attenuated total reflectance mode analysis (**µ-FTIR-ATR**).

Detection and Identification of MP by µ-FTIR-ATR

Detection and identification of MP were carried out in a dedicated clean room, according to the guidelines described in the ISO 14644-1:2015 class 7 (ISO, 2015). The analyses to determine the polymeric matrix, the size (µm), the color and the concentration (MP/kg) of MP fragments were performed through FTIR Spectrometer Frontier coupled to a microscope Spotlight 400 (Perkin Elmer Italia Spa, Milan, Italy) in µ-FTIR-ATR analysis. The spectrum range was set between 4000 and 650 cm⁻¹ with 4 repeated scans for each measurement. The polymeric matrix of the detected particles was identified by comparing the collected µ-FTIR-ATR spectra with spectra reference libraries using Spectrum 10 software (Perkin Elmer Italia Spa, Milan, Italy). Each MP particle was considered correctly identified when the matching between MP spectra from skim-milk powders and MP spectra from reference libraries was >80%. In particular, identified MP residues included acrylonitrile butadiene styrene (**ABS**), ethylene-propylene diene monomer (**EPDM**), ethylene vinyl acetate (**EVA**), nylon, nylon: resin copolymer, polyacrylate, poly (chloro styrene), polyester, polyethylene (**PE**), polyethylene chlorinated (**CPE**), polyethylene-polyamide copolymer (**PE:PA**), polyethylene-polyethylene chlorinated copolymer (**PE:CPE**), polyethylene terephthalate (**PET**), polyisoprene, polyoxymethylene (**POM**), polypropylene (**PP**), polypropylene-polybutylene terephthalate copolymer (**PP:PBT**), polystyrene (**PS**), polystyrene-polyacrylate copolymer (**PS:Polyacrylate**), polystyrene-polyethylene chlorinated copolymer (**PS:CPE**), polystyrene-polyurethane copolymer (**PS:PU**), polytetrafluoroethylene (**PTFE**), polyurethane (**PU**), polyvinyl chloride (**PVC**), polyvinylidene fluoride, resin, silicone, styrene-butadiene-styrene (**SBS**), and Styrene-ethylene-butylene-styrene (**SEBS**).

High resolution images of MP fragments were electronically stored through Spectrum 10 software. The size of each MP was evaluated using ImageJ software. In particular, fibers were measured along their length whereas fragments characterized by irregular or spherical shapes were measured in their greater dimension. The identified MP exhibited a variety of colors such as black, blue, brown, fuchsia, green, and gray. No transparent MP particles were found in the skim-milk powder samples.

Blanks and Recovery

Blanks were prepared along with each skim-milk powder sample and according to the same protocol which included digestion, filtration, detection, and identification steps. Blanks were considered valid up to 5 MP fragment contaminants (in other terms, whenever 6 MP fragment contaminants were retrieved in a blank, the analysis of the corresponding sample was considered invalid and repeated again). In the analyzed blank filters, a maximum of 3 MP fragment contaminants was found. Polyethylene terephthalate was the most frequent polymer in the blank samples, representing 25% of total MP contaminants, followed by PP, PE, and polyisoprene (each representing 19% of total MP contaminants) and EVA, SBS, and polyvinyl alcohol (**PVA**) to an even lesser extent (each representing 6% of total MP contaminants). The number of MP contaminants detected in blanks were subtracted from the total MP fragments detected in the corresponding skim-milk powder sample.

The recovery of the method was performed by spiking ultrapure water with commercial standard of PS (Cospheric LLC, Santa Barbara, CA). Ten different spiking levels were tested ranging from 22 to 207 MP/Kg of water. Particle recovery ranged from 66 to 122%, with average recovery (standard deviation) of 84% (19.90 MP/Kg of water).

RESULTS AND DISCUSSION

Qualitative Features of Identified Microplastics

Results of the present study highlight a widespread presence of plastic particles in skim-milk powder samples from different European countries. Details about the identified MP, including dimensions, morphology, and colors are reported in Supplemental Table S1. Half of the identified MP were $\leq 60.50 \mu\text{m}$; nearly 30% of the identified MP were in the range of $60.50 - 99.00 \mu\text{m}$, whereas nearly 20% of them were $>99.00 \mu\text{m}$ with a maximum size of $1,444.00 \mu\text{m}$. Such dimensions are considerably smaller compared with those reported by Kutralam-Muniasamy et al., 2020, who detected the majority of MP in milk samples with a size $<500 \mu\text{m}$ (40%), followed by sizes comprised between $500 - 1,000 \mu\text{m}$ (28%) and between $1,000 - 2,000 \mu\text{m}$ (25%) using RS. In general, the size distribution of MP in food is affected by the different methods used for observing and measuring. Difficulties also arise when comparing results from various studies as bias may be introduced by different laboratory procedures and environments (Filella et al., 2015). In addition, it is possible to infer that physical processes adopted during skim-milk powder production may lead to the fragmentation and miniaturization of MP originally contained in the starting liquid milk. Although the majority of the particles identified in the present study consisted of irregular fragments (88%), spheres, and fibers were also observed (7% and 5%, respectively). This somewhat differs to the results reported by Kutralam-Muniasamy et al., 2020, who observed that fiber was the most represented shape (97.5%), while fragment accounted for only 2.5% in the 23 milk products analyzed. Figure 1 shows some $\mu\text{-FTIR-ATR}$ spectra and images of major species of MP found in skim-milk powder samples. All MP fragments characterized in the present study were pigmented, with brown, gray, black, and blue being the most common colors (about 51%, 40%, 6%, and 2.4% respectively; Supplemental Table S1). In particular, blue MP in food may be at least partly attributed to the work clothes and masks worn by personnel during food production processes (Bai et al., 2022).

Quantitative Extent of Microplastics Contamination

Table 1 reports count and descriptive statistics of microplastics in skim-milk powders. The most widespread MP characterized in the analyzed samples (Table 1) were PP (5,764.71 MP/kg), PS (1,081.00 MP/kg), and PE (466.00 MP/kg); their presence in skim-milk powder samples may be due to farm environment, milking procedures, milking apparatuses (Diaz-Basantes et al.,

2020), contamination from worker uniforms (generally made of PET) and hygiene caps and masks (commonly made of PP; Da Costa Filho et al., 2021). Other sources of MP contamination in milk can include pipes made of plastic materials commonly used for milk transport and storage at dairy industry level (Kutralam-Muniasamy et al., 2020).

Although MP were detected in all analyzed samples, MP types varied greatly among skim-milk powders, ranging from a minimum of 2 to a maximum of 13 MP species within sample (Figure 2). What is more, 29 different types of polymeric matrices were identified (Figure 2) for a total count of 536 particles (Supplemental Table S1) in the 16 analyzed skim-milk powders. Such results suggest a greater variability of polymeric matrices and a greater number of plastic particles compared with the results reported by Zhang et al., 2023. The same authors detected only PE, PP, PET, PA, and PVC in infant milk powder using $\mu\text{-FTIR}$. Furthermore, skim-milk powders analyzed in the present study originated from different countries, wherefore transport and the use of different packaging materials is a source of contamination to be taken into consideration. Results of the present study also highlighted that skim-milk powder samples from different countries had various types and levels of MP contamination (Figure 2). This could be due to different and specific conditions adopted for milk powders production, which include pre-treatment (skimming, homogenization, and pasteurization), concentration, and drying (including air filtering and energy supply; Moejes and Van Boxtel, 2017). In fact, the presence of a greater number of MP in powdered milk compared with liquid milk may be due to the release of plastic particles from polymeric membranes or filters with reduced performance used during the different phases of milk powder production (Kumar et al., 2013). A similar contamination is due to the degradation of PET milk bottles exposed to chemical or physical agents (particularly applied to reusable PET bottles), which indeed is presumed to lead to the release of plastic particles (Schymanski et al., 2018; Sobhani et al., 2020). Therefore, the exact knowledge of all the activities performed during milk powder production may help to shed light on the main sources of MP contamination in powdered milk samples.

Method Feasibility and Advantages

The digestion protocol used in this study, after sample solubilization through water addition, consisted in a hot alkaline digestion, which allowed to remove organic and inorganic residues (i.e., proteins, lipids, minerals, and cellulose), thus facilitating the identification and the quantification of MP in skim-milk powder samples.

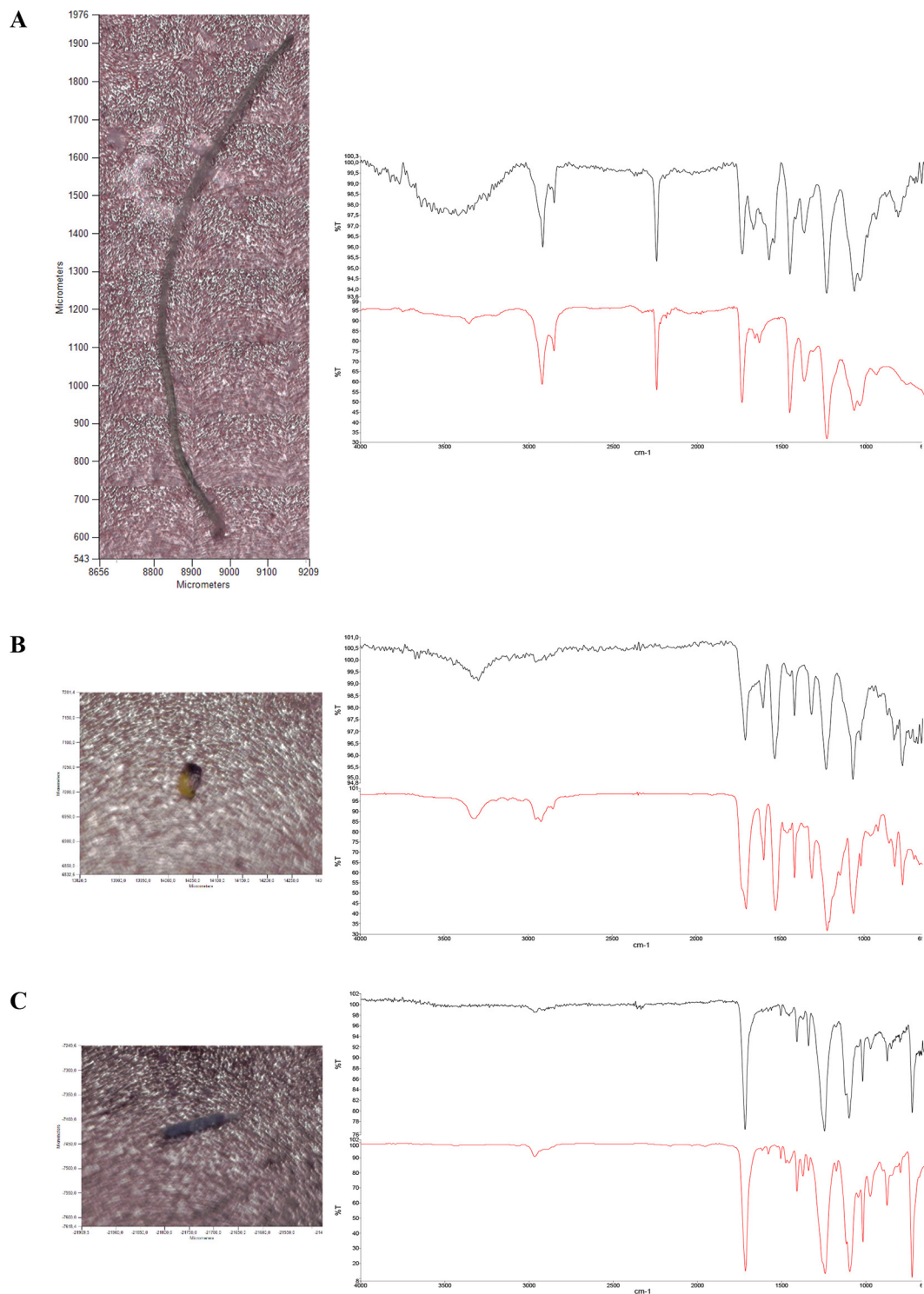


Figure 1. Images and μ -FTIR-ATR spectra (black line for microplastic spectra from skim-milk powders, red line for microplastic spectra from reference libraries) of the major species of microplastics in skim-milk powders: (A) polyacrylate, (B) polyurethane, (C) polyethylene terephthalate, (D) polyisoprene, (E) polypropylene, (F) polystyrene, and (G) styrene-ethylene-butylene-styrene.

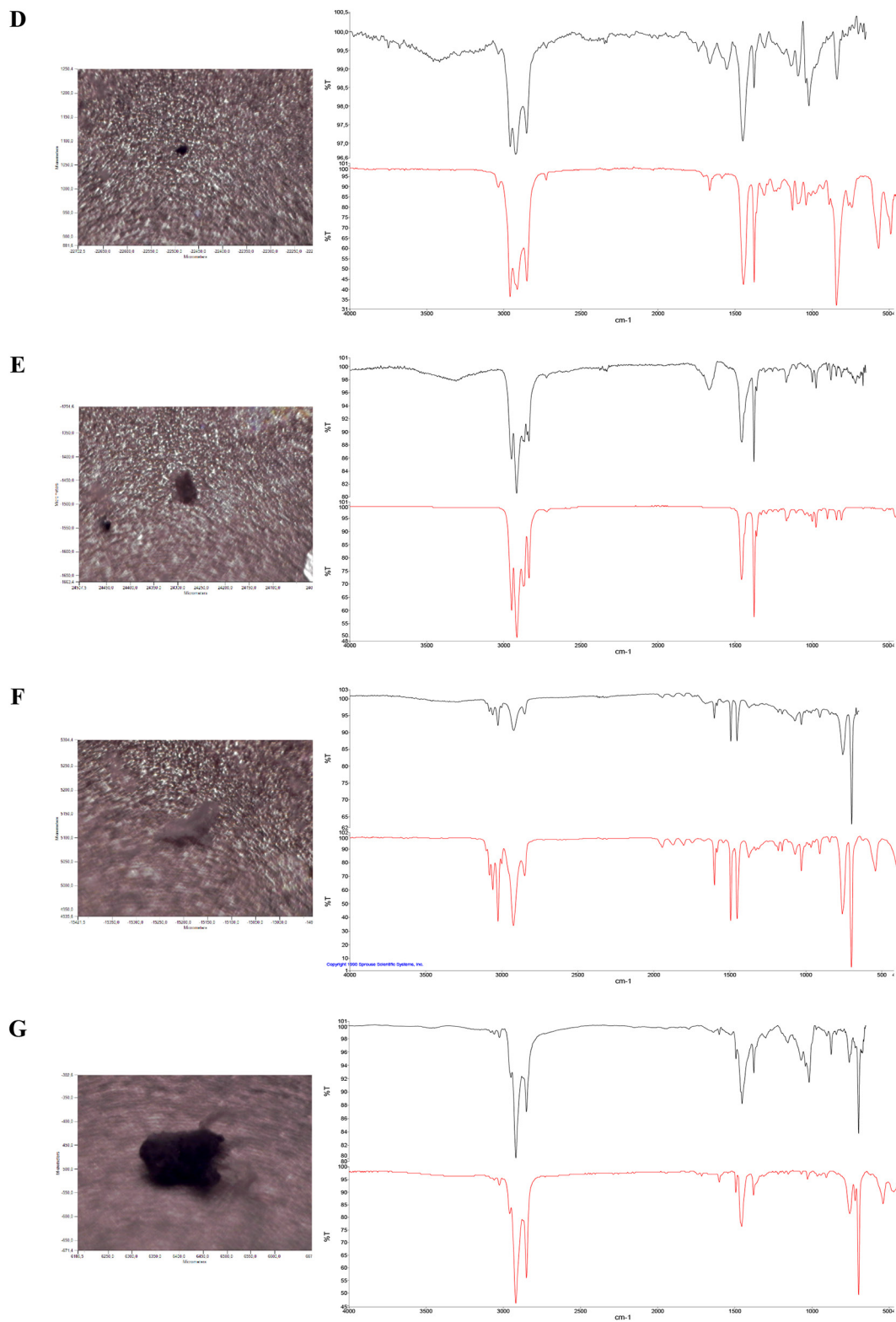


Figure 1 (Continued). Images and μ -FTIR-ATR spectra (black line for microplastic spectra from skim-milk powders, red line for microplastic spectra from reference libraries) of the major species of microplastics in skim-milk powders: (A) polyacrylate, (B) polyurethane, (C) polyethylene terephthalate, (D) polyisoprene, (E) polypropylene, (F) polystyrene, and (G) styrene-ethylene-butylene-styrene.

Table 1. Count and descriptive statistics of microplastics in skim-milk powders (at least detected in 2 samples)

Microplastics	Count of microplastics ¹	Mean ²	Standard deviation ²	Minimum ²	Maximum ²
Ethylene vinyl acetate	7	219.83	198.84	67.00	562.00
Nylon	11	276.00	149.10	133.00	562.00
Polyacrylate	2	119.50	19.09	106.00	133.00
Polyester	8	370.50	341.80	106.00	843.00
Polyethylene	33	466.00	454.39	133.00	1,785.00
Polyethylene chlorinated	2	231.50	177.48	106.00	357.00
Polyethylene terephthalate	23	368.40	229.19	133.00	714.00
Polyisoprene	13	239.33	194.08	40.00	581.00
Polypropylene	334	5,764.71	12,837.49	67.00	47,765.00
Polystyrene	43	1,081.00	2,052.00	133.00	7,071.00
Polyurethane	9	227.43	216.62	133.00	714.00
Polyvinyl chloride	4	240.75	94.90	133.00	357.00
Polyvinylidene fluoride	9	578.50	237.52	357.00	843.00
Resin	6	441.50	252.38	133.00	714.00
Silicone	5	172.40	103.85	106.00	357.00
Styrene-butadiene-styrene	11	343.25	356.19	106.00	867.00

¹Calculated as the sum of microplastics retrieved in all analyzed filters.

²Expressed as number of microplastics per kg of sample.

Microplastics	AUT	BEL	DEU	FRA									IRL	ITA	NLD	POL
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1. ABS			■													
2. EPDM					■											
3. Ethylene vinyl acetate				■	■	■	■			■			■			■
4. Nylon			■	■	■	■	■			■				■		
5. Nylon:Resin															■	
6. Polyacrylate					■							■				
7. Poly(chloro styrene)																
8. Polyester				■	■		■				■					
9. Polyethylene	■		■	■	■	■	■	■	■	■	■	■		■	■	
10. Polyethylene chlorinated					■					■						
11. PE:PA									■							
12. PE:CPE							■									
13. Polyethylene terephthalate			■		■	■		■	■	■		■	■	■	■	
14. Polyisoprene				■		■	■		■		■		■	■	■	
15. Polyoxymethylene																
16. Polypropylene	■		■	■	■	■	■	■	■	■	■	■	■	■	■	■
17. PP: PBT															■	
18. Polystyrene			■	■	■	■	■	■	■	■			■	■	■	■
19. PS:Polyacrylate			■													
20. PS:CPE				■												
21. PS:PU				■												
22. Polytetrafluoroethylene							■									
23. Polyurethane			■		■	■			■	■		■		■	■	
24. Polyvinyl chloride				■					■	■		■		■		
25. Polyvinylidene fluoride			■	■			■		■	■						
26. Resin			■	■					■	■						
27. Silicone					■		■		■	■		■		■		
28. SBS					■	■								■	■	
29. SEBS					■											
Frequency	2	2	9	11	13	9	11	4	11	9	4	7	4	9	9	2

Figure 2. Microplastics characterized in 16 skim-milk powder samples from 8 different European countries. Frequency on the right column is referred to each type of microplastics across samples; frequency on the bottom line is referred to microplastics particles within each sample. Abbreviations for microplastics are: ABS: Acrylonitrile butadiene styrene; EPDM: Ethylene-propylene diene monomer; PE:PA: Polyethylene-polyamide copolymer; PE:CPE: Polyethylene-polyethylene chlorinated copolymer; PP:PBT: Polypropylene-polybutylene terephthalate copolymer; PS:Polyacrylate: Polystyrene-polyacrylate copolymer; PS:CPE: Polystyrene-polyethylene chlorinated copolymer; PS:PU: Polystyrene-polyurethane copolymer; SBS: Styrene-butadiene-styrene; SEBS: Styrene-ethylene-butylene-styrene. Abbreviations for countries are AUT: Austria; BEL: Belgium; DEU: Germany; FRA: France; IRL: Ireland; ITA: Italy; NLD: Netherlands; POL: Poland.

Overall, KOH, which is alkaline in nature, is the most commonly digestion solution used in food matrices (Guo et al., 2022), with less common acid digestion basing on either hydrogen peroxide (H₂O₂) or nitric acid (HNO₃). These strong acids can destroy or damage polymers with a low pH tolerance, such as PS and polyamide (PA; Cole et al., 2014). Their application in the analysis of MP is thus limited (Cole et al., 2014). In addition, in a study aiming at the characterization of MP in seafood, Dehaut et al., 2016 confirmed that KOH-based extraction is an effective and practical method to separate plastic particles from organic matter.

To date, μ -FTIR technique is the most frequently used approach in MP identification and quantification (Chen et al., 2020; Bai et al., 2022) and represents a promising tool for automated MP analysis. It allows concurrent identification and quantification of polymer types (Sridhar et al., 2022), while also inferring information on their chemical characteristics and structure (Bai et al., 2022). Although the long analysis time required for each filter obtained at the end of sample preparation (about 2 working days per filter), it is undisputed that this technique allows more accurate and efficacious detection of micro-sized particles compared with other microscope-based techniques (Song et al., 2015; Lee and Chae, 2021). The μ -FTIR technique requires good sample filter surface conditions in terms of wrinkles, folds, and organic compounds, as the MP may otherwise be covered or trapped by sample fibers, leading to an underestimation of their quantification.

Further research should be devoted to improve automated analysis methodology and to reduce the identification time. In addition, it is imperative to standardize appropriate methods of identification and characterization of MP in foods using reliable protocols with strict quality assurance and blank control. This would provide repeatable, sensitive, and accurate results to guarantee a relationship of trust between the food industry and consumers.

CONCLUSIONS

To authors' knowledge, this is the first contribution addressing a qualitative and quantitative characterization of MP in skim-milk powder samples from different European countries. Skim-milk powder samples used for cheese-making in different European countries were analyzed to detect and identify MP through μ -FTIR-ATR. Results demonstrated a wide diffusion of MP in the analyzed samples, which may be transferred to cheese products and, ultimately, ingested by consumers. A great variability of polymeric particles exists, both in terms of quality and quantity. Based on the presence of these MP across country, the most frequent are PP

(n = 14), PE (n = 13), PS (n = 12), and PET (n = 10). In conclusion, due to the uncertainty surrounding the effect of MP on human health, it is of most importance to identify the main MP contamination in skim-milk powder, so a selective reduction in the use of plastic along the supply chain can be made. This will ultimately prevent, or at least minimize the amount of MP ingested by consumers.

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