



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 microspectroscopy 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 cheesemaking 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 microspectroscopy, dairy

INTRODUCTION

Microplastics (MIPL) 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 MIPL in the environment 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, MIPL 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 MIPL 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 MIPL 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 MIPL 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., 2014). The uptake of MIPL through inhalation represents a risk for human health due to inflammation, chemical toxicity, and infection by microorganisms introduced to the body via MIPL (Wright and Kelly, 2017). The accumulation of particles in the respiratory system can cause acute release of proinflammatory chemotactic factors that can induce chronic inflammation, known as dust overload (Prata, 2018). Other health risks may arise from the dermal contact of MIPL used in hand detergents, face washes, face masks, and toothpastes. In fact, the presence of MIPL in personal care products has 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 MIPL at the human health level, tolerance limits are still not defined (Kirstein et al., 2021).

Considering the potential toxicity of MIPL and their absorption into human cells, it is important to investigate 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 MIPL in the food chain (SAPEA, 2019), consumer awareness is

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The list of standard abbreviations for JDS is available at adsa.org/jds-abbreviations-24. Nonstandard abbreviations are available in the Notes.

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 MIPL 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 MIPL 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 they either lack adequate sensitivity for correct polymer identification or specificity to correctly discriminate OM from MIPL, thereby increasing the risk of false positives (Bai et al., 2022). These methods are microscopy-based techniques such as scanning electron microscopy and transmission electron microscopy. Instead, spectroscopic techniques such as Fourier-transform infrared microspectroscopy (μ -FTIR) and Raman spectroscopy are alternative analytical methods that are sensitive and nondestructive, 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 MIPL contamination in milk and dairy products and their effect 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 about the possible implications of MIPL on human health as ingested via milk and dairy products (Diaz-Basantes et al., 2020).

With respect to milk powders, only Da Costa Filho et al. (2021) and Zhang et al. (2023) determined MIPL in 2 cow milk powders and 13 infant milk powders, respectively. There is a lack of studies that attempted to characterize MIPL in skim-milk powder samples on a broader scale. Therefore, the aim of this study was to qualitatively and quantitatively characterize MIPL 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 because 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 the Netherlands, and 1 sample from Poland. In particular, the 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 MIPL were carried out in the laboratory of the European Center for the Sustainable Impact of Nanotechnology (ECSIN, Padova, Italy), as part of Mérieux NutriSciences Company (Chicago, IL).

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 microfiltered using a silver membrane filter (3.0- μ m pore-size, 25-mm diameter; Sterlitech Corporation, Auburn, WA) and kept in glass containers to avoid any contamination. Glassware was washed 5 times using liquid dishwashing detergent, 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. In order 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 1,000-mL glass flask. Then, 100 mL of ultrapure water and 10% KOH were added. To start the alkaline digestion process, samples were placed in a Shake'n Bator (EuroClone, Milan, Italy) and heated to 60°C overnight. Thereafter, 75 mL of 10% EDTA solution were added and samples were

incubated in the Shake'n Bator at 60°C for 2 h. Digested skim-milk powders were microfiltered through a 3.0- μm pore-size silver membrane filter (Sterlitech Corporation, Auburn, WA) using a vacuum pump connected to a glass filter funnel. To enhance the separation of MIPL from organic compounds, a density separation step was applied. This action was aimed at resuspending MIPL characterized by higher density. This step was 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 MIPL 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 with μ -FTIR in attenuated total reflectance (ATR) mode.

Detection and Identification of MIPL by μ -FTIR-ATR

Detection and identification of MIPL 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 (MIPL/kg) of MIPL 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 4,000 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 MIPL particle was considered correctly identified when the match between MIPL spectra from skim-milk powders and MIPL spectra from reference libraries was >80%. In particular, identified MIPL residues included acrylonitrile butadiene styrene, ethylene-propylene diene monomer, ethylene vinyl acetate, nylon, nylon: resin copolymer, polyacrylate, poly(chlorostyrene), polyester, polyethylene (PE), polyethylene chlorinated (CPE), PE-polyamide (PA) copolymer, PE:CPE, polyethylene terephthalate (PET), polyisoprene, polyoxymethylene, polypropylene (PP), PP-polybutylene terephthalate copolymer, polystyrene (PS), PS-polyacrylate copolymer, PS:CPE copolymer, PS-polyurethane (PU) copolymer, polytetrafluoroethylene, PU, polyvinyl chloride, polyvinylidene fluoride, resin, silicone, styrene-butadiene-styrene, and styrene-ethylene-butylene-styrene. High-resolution images of MIPL fragments were electronically stored through Spectrum 10 software. The size of each MIPL was evalu-

ated using ImageJ software (ImageJ, 2022). In particular, fibers were measured along their length and fragments characterized by irregular or spherical shapes were measured in their greater dimension. The identified MIPL exhibited a variety of colors such as black, blue, brown, fuchsia, green, and gray. No transparent MIPL 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, including digestion, filtration, detection, and identification steps. Blanks were considered valid up to 10 MIPL fragment contaminants (that is, whenever 11 MIPL fragment contaminants were retrieved in a blank, the analysis of the corresponding sample was considered invalid and was repeated). In the analyzed blank filters, a maximum of 3 MIPL fragment contaminants was found. Polyethylene terephthalate was the most frequent polymer in the blank samples, representing 25% of total MIPL contaminants, followed by PP, PE, and polyisoprene (each representing 19% of total MIPL contaminants) and ethylene vinyl acetate, styrene-butadiene-styrene, and polyvinyl alcohol to an even lesser extent (each representing 6% of total MIPL contaminants). The number of MIPL contaminants detected in blanks was subtracted from the total MIPL 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 MIPL/kg of water. Particle recovery ranged from 66% to 122%, with average recovery (SD) of 84% (19.90 MIPL/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 MIPL, including dimensions, morphology, and colors are reported in Supplemental Table S1 (see Notes). Half of the identified MIPL were ≤ 60.50 μm ; approximately 30% of the identified MIPL were in the range of 60.50 to 99.00 μm , and approximately 20% of them were >99.00 μm with a maximum size of 1,444.00 μm . Such dimensions are considerably smaller compared to those reported by Kuttralam-Muniasamy et al. (2020), who detected the majority of MIPL in milk samples with a size <500 μm (40%), followed by sizes between 500 and 1,000 μm (28%) and between 1,000 and 2,000 μm

(25%) using Raman spectroscopy. In general, the size distribution of MIPL in food is affected by the different methods used for observing and measuring them. Difficulties also arise when comparing results from various studies because bias may be introduced by different laboratory procedures and environments (Filella, 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 MIPL 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 from the results reported by Kutralam-Muniasamy et al. (2020), who observed that fiber was the most represented shape (97.5%), and fragments accounted for only 2.5% in the 23 milk products analyzed. Figure 1 shows some μ -FTIR-ATR spectra and images of major species of MIPL found in skim-milk powder samples. All MIPL 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 MIPL in food may be at least partly attributed to the work clothes and masks worn by personnel during the food production processes (Bai et al., 2022).

Quantitative Extent of MIPL Contamination

Table 1 reports count and descriptive statistics of MIPL in skim-milk powders. The most widespread MIPL characterized in the analyzed samples (Table 1) were PP (5,764.71 MIPL/kg), PS (1,081.00 MIPL/kg), and PE (466.00 MIPL/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 MIPL contamination in milk can include pipes made of plastic materials commonly used for milk transport and storage at the dairy industry level (Kutralam-Muniasamy et al., 2020).

Although MIPL were detected in all analyzed samples, MIPL types varied greatly among skim-milk powders, ranging from a minimum of 2 to a maximum of 13 MIPL species within a sample (Figure 2). Additionally, 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 polyvinylchloride in

infant milk powder using μ -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 MIPL contamination (Figure 2). This could be due to different and specific conditions adopted for milk powders production, which include pretreatment (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 MIPL in powdered milk compared to 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 MIPL contamination in powdered milk samples.

Method Feasibility and Advantages

The digestion protocol used in this study, after sample solubilization through water addition, consisted of a hot alkaline digestion, which allowed removal of organic and inorganic residues (i.e., proteins, lipids, minerals, and cellulose), thus facilitating the identification and the quantification of MIPL in skim-milk powder samples. Overall, KOH, which is alkaline in nature, is the most common digestion solution used in food matrices (Guo et al., 2022), with less common acid digestion based on either hydrogen peroxide (H_2O_2) or nitric acid (HNO_3). These strong acids can destroy or damage polymers with a low pH tolerance, such as PS and PA (Cole et al., 2014). Their application in the analysis of MIPL is thus limited (Cole et al., 2014). In addition, in a study aiming at the characterization of MIPL in seafood, Dehaut et al. (2016) confirmed that KOH-based extraction is an effective and practical method to separate plastic particles from OM.

To date, the μ -FTIR technique is the most frequently used approach in MIPL identification and quantification (Chen et al., 2020; Bai et al., 2022) and represents a promising tool for automated MIPL 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 analysis time required for

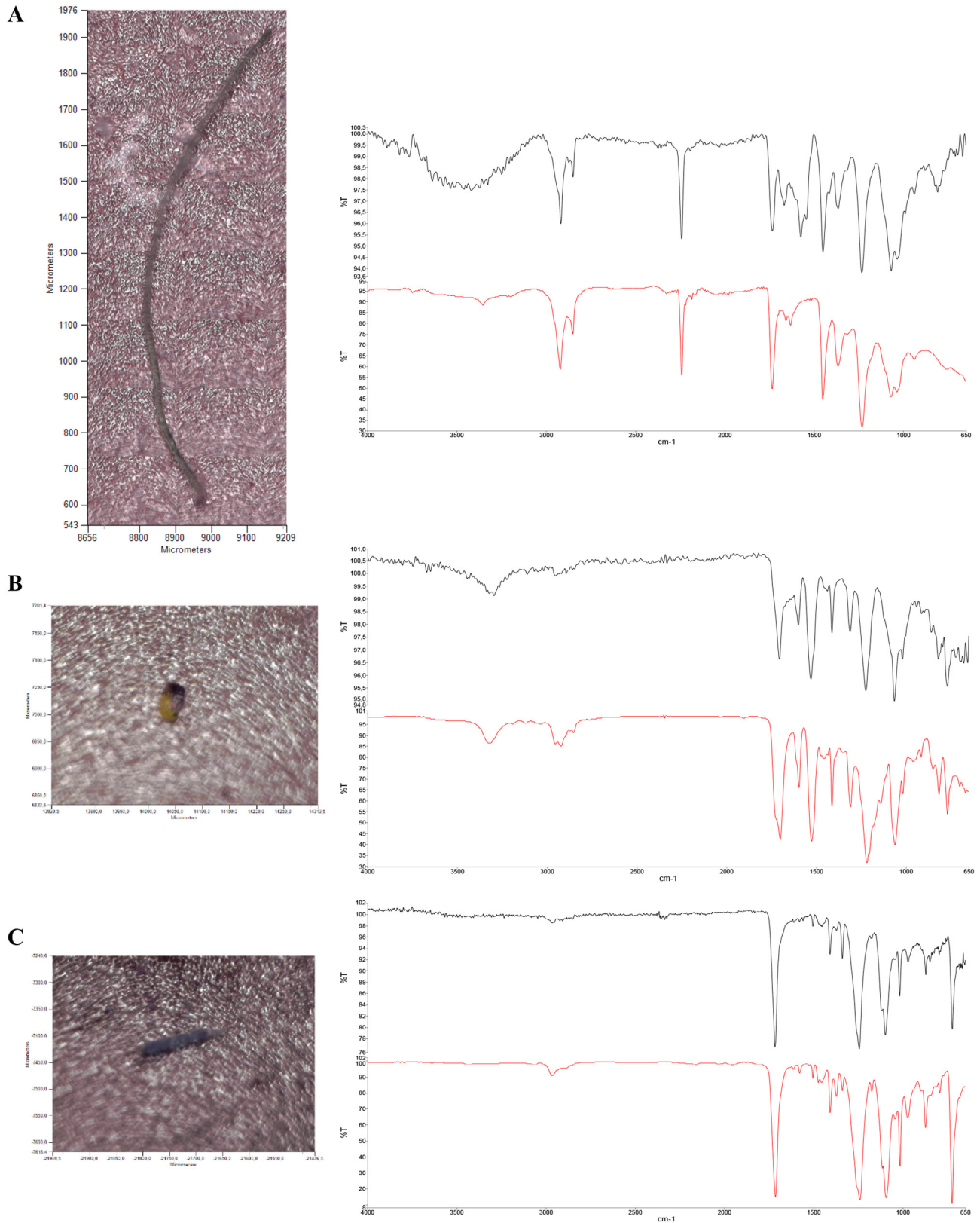


Figure 1. Images and μ -FTIR-ATR spectra (black line = microplastic spectra from skim-milk powders; red line = microplastic spectra from reference libraries) of the major species of microplastics in skim-milk powders: (A) polyacrylate, (B) PU, (C) PET, (D) polyisoprene, (E) PP, (F) PS, and (G) styrene-ethylene-butylene-styrene.

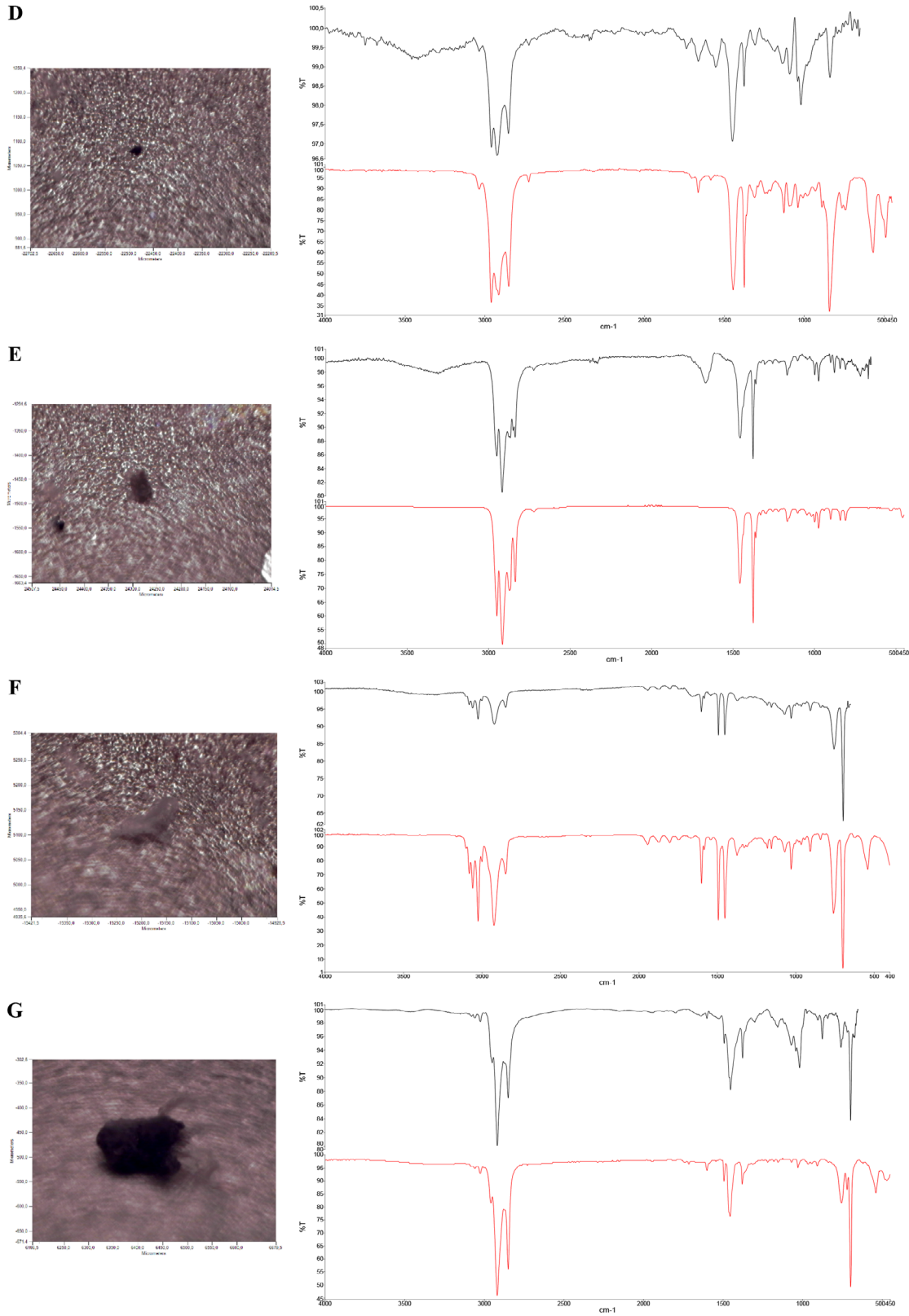


Figure 1 (Continued). Images and μ -FTIR-ATR spectra (black line = microplastic spectra from skim-milk powders; red line = microplastic spectra from reference libraries) of the major species of microplastics in skim-milk powders: (A) polyacrylate, (B) PU, (C) PET, (D) polyisoprene, (E) PP, (F) PS, and (G) styrene-ethylene-butylene-styrene.

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

MIPL	MIPL count	Mean	SD	Minimum	Maximum
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

¹The MIPL count was calculated as the sum of MIPL retrieved in all analyzed filters. The mean, SD, minimum, and maximum values are expressed as number of MIPL per kilogram of sample.

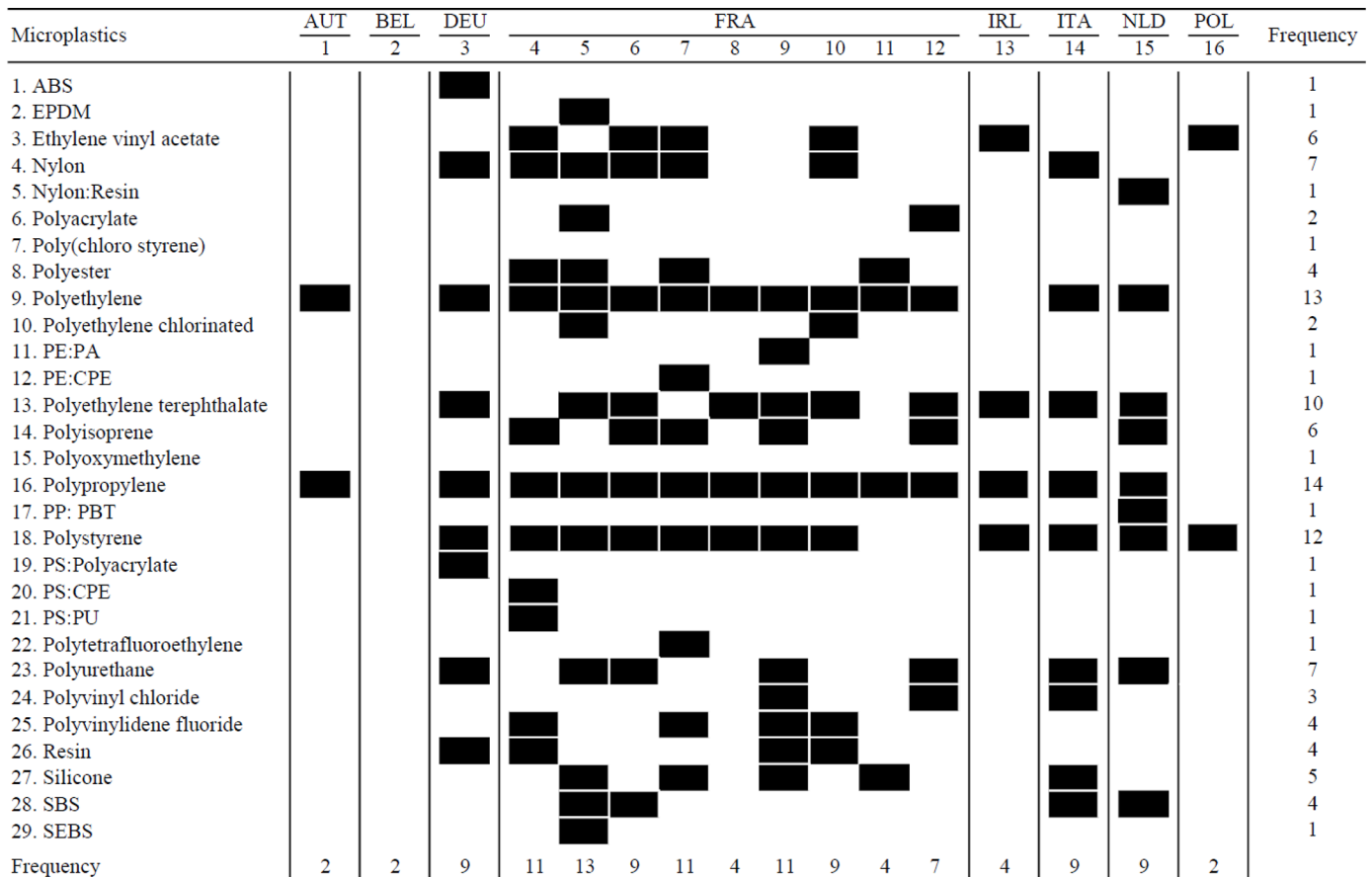


Figure 2. Microplastics characterized in 16 skim-milk powder samples from 8 different European countries. The “Frequency” column on the right refers to each type of microplastic across samples; frequency numbers listed across the bottom refer to numbers of microplastics particles within each sample. The number listed under each country is the sample number. Microplastics: 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. Countries: AUT = Austria; BEL = Belgium; DEU = Germany; FRA = France; IRL = Ireland; ITA = Italy; NLD = the Netherlands; POL = Poland.

each filter obtained at the end of sample preparation was long (about 2 working days per filter), it is undisputed that this technique allows more accurate and efficacious detection of microsized 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 because the MIPL may otherwise be covered or trapped by sample fibers, leading to an underestimation of their quantification.

Further research should be devoted to improving automated analysis methodology and to reduce the identification time. In addition, it is imperative to standardize appropriate methods of identification and characterization of MIPL 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 our knowledge, this is the first contribution addressing a qualitative and quantitative characterization of MIPL in skim-milk powder samples from different European countries. Skim-milk powder samples used for cheesemaking in different European countries were analyzed to detect and identify MIPL through μ -FTIR-ATR. Results demonstrated a wide diffusion of MIPL 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 MIPL across countries, 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 MIPL on human health, it is of most importance to identify the main MIPL 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 MIPL ingested by consumers.

NOTES

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ber 2010 on the protection of animals used for scientific purposes. The authors have not stated any conflicts of interest.


Nonstandard abbreviations used: ABS = acrylonitrile butadiene styrene; ATR = attenuated total reflectance; AUT = Austria; BEL = Belgium; CPE = polyethylene chlorinated; DEU = Germany; EPDM = ethylene-propylene diene monomer; FRA = France; IRL = Ireland; ITA = Italy; MIPL = microplastics; μ -FTIR = Fourier-transform infrared microspectroscopy; NLD = the Netherlands; PA = polyamide; PE = polyethylene; PE:PA = polyethylene-polyamide copolymer; PE:CPE = polyethylene-polyethylene chlorinated copolymer; PET = polyethylene terephthalate; POL = Poland; PP = polypropylene; PP:PBT = polypropylene-polybutylene terephthalate copolymer; PS = polystyrene; PS:CPE = polystyrene-polyethylene chlorinated copolymer; PS:Polyacrylate = polystyrene-polyacrylate copolymer; PS:PU = polystyrene-polyurethane copolymer; PU = polyurethane; SBS = styrene-butadiene-styrene; SEBS = styrene-ethylene-butylene-styrene.

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