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Ingestion of synthetic particles by fin whales feeding off western Iceland in summer

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HIGHLIGHTS

- Stomach content samples from 25 whales from western Iceland were analysed.
- The only ingested prey found in the samples was northern krill.
- The average concentration in krill was 0.057 synthetic particles (SP) per gram.
- The number of SP daily ingested by fin whales was estimated in the tens of thousands.
- SP presence in their diet might facilitate the exposure of whale populations to POPs.

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G R A P H I C A L A B S T R A C T



ABSTRACT

In this study we aim to assess the daily ingestion rates of synthetic particles by the fin whales (*Balaenoptera physalus*) that feed off the western coast of Iceland. To do so, we collected and analysed samples from the stomach content of 25 fin whales, consisting solely of northern krill (*Meganyctiphanes norvegica*). The particles found consisted of fibres and fragments, mainly blue, black and red, with an average size of 1.2 ± 1.3 mm. To confirm the synthetic nature of these particles, we used Micro-Fourier Transform Infrared Spectroscopy and comparison with a polymer library. The mean concentration of synthetic particles in the krill samples found in the stomachs of whales was 0.057 particles per gram, a value much lower than that previously reported for particle uptake by krill. From this concentration in krill, we estimated that the daily intake of synthetic particles for the North Atlantic fin whale would be ranging from 38,646 \pm 43,392 to 77,292 \pm 86,784 particles per day. Although at this level it is not possible to assess the impact of synthetic particles and their associated chemicals on the North Atlantic fin whale population, concentrations of these contaminants are likely to increase in the future, potentially causing adverse effects on whales and other marine mammals.

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1. Introduction

Marine litter, broadly classified in macro-, meso-, micro- and nano-litter, encompass a group of manufactured or processed

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fragments and fibres of different shapes, textures and colours. It is composed of over 80% of plastic items. Every year, between 4.8 and 12.7 million tons of plastic reach the oceans (Jambeck et al., 2015), currently making up the most widespread and chronic type of marine pollution (Alimba and Faggio, 2019).

Plastics can act as vector for the transport of chemical compounds, which can be either directly related to plastic manufacture. providing it with certain plasticizing properties (e.g., Phthalates such as mono and di-(2-ethylhexyl) phthalates (MEHP and DEHP, respectively) and Bisphenol A), or flame retardancy (e.g., Polybrominated diphenyl ethers (PBDEs) or Organophosphate esters (OPEs)), or are pollutants that adsorb on the plastic, such as heavy metals and hydrophobic organic pollutants (e.g., Polychlorinated biphenyls (PCBs), Chlorinated pesticides, Polycyclic aromatic hydrocarbons (PAHs)) (Avio et al., 2017). These pollutants, usually associated with plastic, may accumulate in the organisms and likely alter their biological processes. Among their multiple negative effects, they can act as endocrine disruptors, affecting organism reproduction and development (Mathieu-Denoncourt et al., 2015; Talsness et al., 2009), or depress the immune system, making it more vulnerable to viruses or other diseases (Aguilar and Borrell, 1994: Borrell et al., 1996).

Synthetic micro-litter is composed by microplastics, particles made of modified cellulose and cellulose combined with pigments (Lusher et al., 2020). Microplastics (*i.e.*, plastics items smaller than 5 mm) can be of primary origin (*i.e.*, beads, fibres or pellets) or of secondary origin (small plastic fragments derived from the breakdown of macroplastics) (Cole et al., 2013). Due to their small size, they are easily ingested by small aquatic organisms (*e.g.*, zooplankton) (Botterell et al., 2019; Khalid et al., 2020). This could represent a route to top predators through the food web (Nelms et al., 2018), although, to this date, studies certifying that this transfer involves biomagnification have not been produced (Provencher et al., 2019; Alava, 2020) and impacts from microplastics ingestion at high food web levels are not known (Reijnders et al., 2018).

Despite having been observed in laboratory within controlled feeding experiments (Cole et al., 2013), the ingestion of synthetic particles by euphausiids has not been fully confirmed in the field. Desforges et al. (2015) showed for the first time the ability of North Pacific krill (*Euphausia pacifica*) to ingest microplastics in the wild, suggesting that these animals may confuse microplastics with natural prey items when they are within the same size range.

Euphausiids make up the largest proportion of the diet of most baleen whales (Hewitt and Lipsky, 2018), which need to filter thousands of cubic meters of water every day to capture their food. During this activity, whales may ingest synthetic particles directly from the water (Fossi et al., 2012, 2014), or indirectly from their prey, if they are already contaminated with synthetic particles (Besseling et al., 2015; Germanov et al., 2018).

The fin whale (*Balaenoptera physalus*) is a cosmopolitan mysticete that carries out annual migrations from low-latitude breeding areas in winter to high-latitude feeding areas in summer (Aguilar and García-Vernet, 2018). The waters off western Iceland are a summer feeding ground for the North Atlantic fin whale population, which in this area feeds predominantly on the euphausiid *Meganyctiphanes norvegica* (Vikingsson, 1997). In this study, we investigated the presence of synthetic particles in the stomach of fin whales that feed off western Iceland. To do so, we analysed the krill obtained directly from the whales digestive tract, and basing on the results obtained, we assessed the magnitude of synthetic particle ingestion in this fin whale population.

2. Materials and methods

2.1. Sampling

Number, size, shape, type, and colour of synthetic particles were determined on different sets of samples of krill extracted from the forestomach (first compartment) of 25 fin whales and preserved frozen until analysis (Table 1). The whales sampled were caught during commercial whaling operations in the waters off western Iceland and flensed at the factory Hvalur H/F, located in Hvalfjörður, during summer 2018 (Fig. 1). Stainless steel material was used to cut through the stomach walls and manipulate the stomach contents, of which about 20 g per sampled whale were collected and placed in glass bottles. The krill extracted from the stomachs was carefully inspected in situ and no synthetic particles were observed in its jaw or exoskeleton. However, it was not rinsed with distilled water because we were interested in collecting all the synthetic particles from the samples. No field blanks were made, as weather and factory conditions did not facilitate this. The samples were stored at -20 °C, until their analysis in the laboratory.

2.2. Analysis of synthetic particles

Subsamples of krill of approximately 11 g (corresponding to ca 50 euphasiid individuals) were taken to guarantee a similar weight between samples and that sample was enough to perform other analyses (isotope and alkenone analysis). Samples were defrosted and placed into a glass beaker in $1:20 (w/v) H_2O_2 (15\% H_2O_2)$, Chem-Lab, Germany) and heated at 55-65 °C until H₂O₂ evaporation. Aliquots of 10 ml H₂O₂ were added gradually to the beakers until all the organic matter was digested (Tsangaris et al., 2020). Samples were then diluted with 50 ml Milli-Q and vacuum-filtered on fibreglass filters (pore size 1.2 µm, Whatman, GE Healthcare, UK), which were dried at room temperature for 24 h and subsequently stored in Petri dishes. For more details, consult Garcia-Garin et al. (2019) and Tsangaris et al. (2020). Tsangaris et al. (2021) performed a harmonization exercise on the two principal methods of microplastic extraction from biological samples (i.e., 15% H₂O₂ vs 10% KOH digestion), and microplastic recovery rates for the two methods were similar for each sample tested, with a mean recovery rate of 88.75% when using H₂O₂.

Filters were examined under a Nikon SMZ1000 stereomicroscope (10x to 40x) coupled with a DS-Fi2 camera. Synthetic particles found in the filters were photographed, counted, and classified by size (0.1–0.5, 0.5–1.0 and 1–5 mm), colour (blue, red, black and white) and shape (fragment, fibre and bead) (Lusher et al., 2020). Nineteen potential synthetic particles were analysed with a Thermo Scientific NicoletTM iNTM MX µFT-IR (Micro-Fourier Transform Infrared Spectroscopy) microscopy, and then compared against a polymer library to identify the type of polymer, at the *Centres Científics i Tecnològics* of the University of Barcelona (CCiT-UB).

2.3. Quality assurance and quality control

To prevent contamination, researchers wore cotton lab coats and gloves. Air currents were reduced to a minimum throughout the analysis. The work was done in clean laboratory conditions. Glass or metal equipment was used instead of plastic (both in the field and in the laboratory). Glass beakers were rinsed with purified water and then dried before using. Krill samples were covered with aluminium foil during H₂O₂ digestion. A vertical laminar flow cabinet was used for sample filtration. Filters were protected with glass lids during stereoscope observation (Correia Prata et al., 2019). Procedural blanks, negative controls composed of 50 ml of 15%

Table 1
Biological parameters of the whales sampled, and synthetic particles (SP) found in their stomach content (i.e., krill)

Whale code	Catch day	Sex	Body length (m)	Weight of sample analized (g)	Number of SP per sample	Number of SP per kg of sample
F18004	jun-18	Female	16.2	12.2	1	82
F18008	jun-18	Female	18.3	10.9	1	92
F18009	jun-18	Male	18.6	11.9	0	0
F18012	jul-18	Male	18.9	12.9	1	78
F18016	jul-18	Female	19.8	11.3	0	0
F18017	jul-18	Female	18.9	11.4	0	0
F18019	jul-18	Female	18.6	11.4	1	88
F18020	jul-18	Male	19.8	11.9	1	84
F18030	jul-18	Female	19.2	10.3	2	194
F18036	jul-18	Male	18.3	11.7	0	0
F18047	jul-18	Female	18.0	11.3	0	0
F18048	jul-18	Female	18.9	11.4	0	0
F18052	jul-18	Male	16.8	11.2	1	89
F18060	jul-18	Male	18.3	11.0	0	0
F18071	ago-18	Female	19.5	12.3	0	0
F18073	ago-18	Female	19.2	11.2	2	179
F18075	ago-18	Male	17.7	11.5	0	0
F18083	ago-18	Male	18.9	12.9	1	78
F18086	ago-18	Male	18.3	11.0	2	182
F18092	ago-18	Male	18.3	10.5	1	95
F18098	ago-18	Male	18.3	12.9	0	0
F18099	ago-18	Female	20.4	11.3	1	88
F18110	ago-18	Female	18.3	13.3	0	0
F18111	ago-18	Male	14.3	10.6	1	94
F18114	ago-18	Female	19.5	12.6	0	0
mean				11.64	0.64	57
SD				0.81	0.70	64



Fig. 1. Locations of fin whales catches (red dots) and of the whaling factory where whales were flensed (black triangle).

 H_2O_2 (1 blank every 5 samples), or open petri dishes with a fibreglass filter (1 blank every 5 samples), were examined along with the samples (Correia Prata et al., 2019).

2.4. Quantification of synthetic particles ingested by fin whales

To quantify the synthetic particles ingested by fin whales, we based our calculations on the daily feeding rates estimated for the North Atlantic fin whale population by Víkingsson (1997), which ranged between 678 and 1,356 kg of krill, depending on the food transit time through the digestive system. We considered these values as the minimum and maximum amounts of krill ingested per day. To estimate the daily number of synthetic particles ingested by the whales, we multiplied these values for the number of synthetic particles detected in the krill samples (*i.e.*, 57 items/kg, Table 1).

3. Results

In total 19 particles were found in the 25 samples examined. One of them was excluded from the results as it was considered airborne contamination due to its similarity to one red fibre found in the blanks (Fig. S1). Out of the remaining 18 particles, one was a non-modified cellulose, one a silicate mineral, and the remaining 16 were considered synthetic particles (Fig. 2 and Fig. S2). Out of the 16 synthetic particles, five (37.5%) were identified as modified cellulose (*i.e.*, cellulose with pigments or rayon); three (18.8%) as polyethylene, three (18.8%) as polystyrene, three (18.8%) as poly-propylene and one (6.1%) as acrylonitrile (Fig. S2).

The number of synthetic particles ranged from 0 to 2 per sample (Table 1). The frequency of occurrence, calculated as the percentage of samples with synthetic particles from the total number of samples, was 52%. The average concentration of synthetic particles per sample, considering all samples, was 0.64 ± 0.70 (mean \pm SD), and that of synthetic particles per gram of krill was 0.057 ± 0.064 (Table 2).

The shape, colour and size of the synthetic particles extracted from the samples of stomach content are depicted in Fig. 3. The most frequent shape, colour and size of synthetic particles were fibres (69%), blue (62.5%) and the size smaller than 0.5 mm (44%), respectively.

The daily number of synthetic particles ingested by the whales was estimated to be between 38,646 and 77,292 (Table 2).

4. Discussion

This study investigates the ingestion rate of synthetic particles through the stomach contents of fin whales that feed in the waters off western Iceland during summer. The prey found in the stomach content of the sampled whales consisted exclusively of krill, consistently with a previous study on whales captured from the same population between 1967 and 1989. Of the 1,609 stomachs analysed in that study, 96% contained only euphausiids, 99% of which belonged to the species *M. norvegica* (Sigurjónsson and Víkingsson, 1997).

4.1. Synthetic particle abundance in krill

M. norvegica feeds by filtering from the water dense patches of prey organisms, especially copepods, while moving through the water column (Mauchline, 1967; McClatchie, 1985). This type of feeding could facilitate the ingestion of synthetic particles similar in size to their prey (Cole et al., 2013). The ingestion of synthetic particles by marine zooplankton is well documented through laboratory experiments (Cole et al., 2013), and it has been suggested to

be a significant pathway of plastics into marine food webs (Seträ'l'ä'et al., 2014). However, the levels of synthetic particles in zooplankton within natural marine environments remain largely unknown (Botterell et al., 2019).

The current study reports for the first time the presence of synthetic particles in *M. norvegica* in field samples. To our knowledge, only two studies assessed microplastic ingestion in other euphausiids species under natural conditions (Desforges et al., 2015; Sun et al., 2018). Desforges et al. (2015) analysed the ingestion of microplastics in North Pacific krill (E. pacifica) from the northeast Pacific Ocean, finding 0.058 microplastics/individual, and 0.83 microplastics/g. The large difference between this concentration and that of 0.057 synthetic particles/g we found in M. norvegica may be due to several factors. On the one hand, since the concentration of microplastics in the zooplankton correlates with that of seawater (Desforges et al., 2015), it could reflect different levels of synthetic particles and/or microplastic concentration in seawater, that is, higher concentrations in the Pacific than in the Atlantic Ocean (Van Sebille et al., 2015). On the other hand, it could reflect a greater capability of E. pacifica, for the acquisition and accumulation of synthetic particles, similarly to the capacity that this euphausiid has with respect to copepods (Desforges et al., 2015). More likely, it could be a combination of both factors.

Shape, colour and size of synthetic particles ingested by organisms should also vary, reflecting those of the particles in seawater. However, the types of particles found in the North Pacific krill (68% fibres, 32% fragments) was very similar to that of the synthetic particles found in the North Atlantic krill (69% fibres, 31% fragments), as well as their colour, mainly blue, black, and red (Desforges et al., 2015). On the other hand, the microplastics found in *E. pacifica* were considerably smaller (816 ± 108 µm) than those found in *M. norvegica* (1,148 ± 1,334 µm). This could derive from differences in the size of the litter in the respective marine waters (Desforges et al., 2015), as well as from morphological characteristics of the two species, such as the length of the feeding appendix and the size of the mouth. Both sizes are larger in *M. norvegica* than in *E. pacifica* (Hewitt and Lipsky, 2018), which would allow the former to ingest relatively larger particles (Frost et al., 1983).

The other study reporting microplastic ingestion in krill under natural conditions analysed 10 zooplankton groups from the China Sea, including Euphausiidae spp. (Sun et al., 2018). The concentration of microplastics found in the krill was 0.2 items/krill (53% fibres), a much higher figure than the 0.058 reported by Desforges et al. (2015) and the 0.013 we found in the present study, probably reflecting the high level of contamination by plastics in the China Sea, up to 19.7 ± 22.4 microplastics/m³ (Sun et al., 2018). Sun et al. (2018) did not specify differences between krill species, making any comparison between species unfeasible.

4.2. Number of synthetic particles ingested daily by whales

Fin whales are characterized by their extreme lunge-feeding behaviour that involves the engulfment of a large volume of prey-laden water (Goldbogen et al., 2007). To feed, the whale opens its mouth widely and collects dense shoals of prey (such as krill), along with large volumes of water. Then, it partially closes its mouth and presses its tongue against the upper jaw, forcing the water to pass sideways through the baleen, sieving out the prey, which are then swallowed. Since from the krill analyses we cannot distinguish the particles ingested by the krill from those attached to it from the surrounding water, the current estimate might include also part of plastics filtered directly from the water and retained with the prey.

A fin whale stomach can contain up to 600 Kg of krill (Vírkingsson, 1997), making the analysis of synthetic particles



Fig. 2. Particles found in the 25 fin whales' stomachs. µFT-IR analysis revealed that the particles were composed of modified cellulose (H, L, M, N, O, P), polyethylene (A, Q, R), polystyrene (B, E, F), polypropylene (C, I, J), acrylonitrile (G), silicate mineral (D) and non-modified cellulose (K) (Fig. S2).

Table 2

Frequency of occurrence, characteristics and abundance of synthetic particles (SP) in the krill samples of stomach contents extracted from 25 fin whales from SW Iceland and estimation of total number of SP ingested daily by fin whales.

Parameter	Value					
Number of samples containing SP	13					
SP frequency of occurrence (%)	52					
SP number	16					
SP dimension length range (mm)	0.1-4.9					
SP mean length (mm) (±SD)	1.2 ± 1.3					
SP abundance in krill (mean \pm SD):						
Number of SP per sample in all samples examined	0.64 ± 0.70					
Number of SP per gram in all samples examined	0.057 ± 0.064					
Number of SP per individual of krill (50 individuals/sample)	~0.0128					
Calculation of synthetic particles ingested:						
Minimum-maximum kgs of krill ingested daily (minmax.)	678-1,356					
Number of SP per kg of krill (mean \pm SD)	57 ± 64					
Number of SP ingested daily (min. \pm SD)	$\textbf{38,646} \pm \textbf{43,392}$					
Number of SP ingested daily (max. \pm SD)	$\textbf{77,292} \pm \textbf{86,784}$					



Fig. 3. Shape, size and colour of the synthetic particles detected in all the krill samples examined.

contained in the whole stomach content of 25 whales impossible to perform. For this reason, 11-g aliquots of the stomach content of each whale, equivalent to approximately 50 krill individuals, were analysed, and results were extrapolated to the total amount of krill ingested per day. Such small samples of krill per whale resulted in only 16 synthetic particles, which reduces the strength of the results obtained. Although the calculations may be poorly adjusted due to the factors discussed above, the extrapolation from the number of synthetic particles detected in the krill samples to the potential particles ingested daily by the whales results in an amount of several tens of thousands of particles per day.

Few studies have approached synthetic particle ingestion by whales, due to the difficulties involved. Fossi et al. (2014) calculated the potential amount of microplastics ingested by Mediterranean fin whales from the concentration of microplastics in the water where they were feeding, obtaining an average of 3,653 microplastics/day. However, they did not assess the microplastics ingested by the krill that the whales feed on, which probably produced a strong bias in their calculation.

Similarly to our approach, Desforges et al. (2015) calculated the ingestion of microplastics by humpback whales (*Megaptera novaeangliae*) off the coast of British Columbia, basing their estimations on the potentially ingested krill (*E. pacifica*). Since the krill was collected from seawater and not from the whales' stomach, their approach was made indirectly. Furthermore, the authors did not consider that these whales are generalist predators and that

they likely exploit fish species in addition to zooplankton (Witteveen et al., 2011). The authors estimated a much higher intake of microplastics (above 300,000 items/day) than that of fin whales, despite the daily intake of krill by humpback whales is lower, probably because north pacific krill contained a larger amount of plastics than northern krill, as already discussed above.

On the other hand, Besseling et al. (2015) analysed the stomach content of a stranded humpback whale in the Netherlands. They found a total of 16 microplastics in samples from a gastrointestinal tract that represented only 5–10% of the intestine total length, which lead them to estimate a total of 160 microplastics in the whole intestine. This low number could be partly related to the fact that, since the whale spent four days agonizing stranded on a sandbank without ingesting anything, few remains of fish remained in its digestive tract. Moreover, the authors did not consider synthetic fibers in their analysis, which can also be a cause of the low estimate of microplastics in the gastrointestinal tract of the stranded whale.

Finally, Burkhardt-Holm and N'Guyen (2019) evaluated the possible uptake of microplastics by the common minke whale (*Balaenoptera acutorostrata*) and the sei whale (*Balaenoptera borealis*) based on the load of microplastics of their prey, but they did not quantify the number of microplastics ingested.

Apart from the above cited, we are not aware of any other research estimating the amount of synthetic particles ingested by mysticetes. While the effects of macro-litter ingestion in cetaceans are well known (e.g. Baulch and Perry, 2014), micro-litter ingestion in these species, especially in mysticetes, remains poorly studied (due to difficulty in sampling and analysing and lack of standardization, among others) (Zantis et al., 2021). Although most of the ingested particles are excreted in the faeces, their rates of ingestion and excretion are unknown. The likely disintegration of these particles, and the release and subsequent absorption of lipophilic contaminants through the gastrointestinal wall of the animal almost certainly depends on the transit time in the digestive system. Advancing these types of studies, and harmonizing the quantification systems to allow more accurate intra- and interspecific comparisons among baleen whales, should be a priority for a future in which the quantity of synthetic particles will exponentially increase in the marine environment.

4.3. Review of the chemical compounds found in Icelandic fin whales related to synthetic particles

Plastics can pollute the environment or the organisms that ingest them by releasing several additives and chemical compounds that are attached to them. Contaminants associated with marine litter include chemical additives, such as plasticizers, antioxidants, flame-retardants and UV-stabilizers, and chemicals that accumulate from the surrounding ocean waters (Avio et al., 2017; Rochman, 2015). Most of these compounds are highly recalcitrant, such as the so called 'persistent organic pollutants' (POP), meaning that their chronic acquisition produces an accumulation over time along the trophic webs and ends up depressing the immune system and acting as endocrine disruptor in terminal predators. Ingestion of these compounds usually occurs through food, but since they are bound to plastics, a high exposure to microplastics can lead to an increase in the body loads of these pollutants (Hermabessiere et al., 2017).

Many of these pollutants have previously been detected in the tissues of the Icelandic fin whale population (Borrell, 1993; Garcia-Garin et al., 2020; Rotander et al., 2012). Thus, Garcia-Garin et al. (2020) recently found organophosphate esters in samples of muscle of fin whales and of krill from Icelandic waters at concentrations of 1,060 (SD = 2,564) and 949 (SD = 1,090) ng/g lw, respectively.

Furthermore, Borrell (1993) had previously found organochlorine compounds (PCBs and DDTs) in concentrations of few $\mu g/g$ lw, in the blubber of individuals caught in 1986 from the same population. Finally, Rotander et al. (2012) found organobrominated compounds (PBDEs) in the blubber of individuals sampled during the 1980s and 2006–2009 in Iceland. The highest levels of these compounds were found in the most recent samples (8.4, 1980s vs 22 ng/g lw, 2006–9), which possibly reflected the increase of the global production of technical PBDE mixtures during those years. Given that in 2009 the Parties of the Stockholm Convention for POPs included the commercial PBDEs in the list of prohibited substances, it would be interesting to investigate the current progression of these pollutants in the Icelandic fin whale population.

Some of the aforementioned pollutants found in the whales' tissues could derive from the ingestion of plastic particles throughout the life of the animals. No direct effects of these toxic compounds have been described in this species yet, but other marine mammals feeding on higher trophic level prey (*e.g.*, dolphins and seals) tend to accumulate higher amounts of POPs and have shown reproductive and immunosuppressive effects (*e.g.*, Reijnders, 1986; Aguilar and Borrell, 1994; Jepson et al., 2016). However, the effects of synthetic particles in the natural environment and implications for the food web remain poorly understood (Hermsen et al., 2018). Further studies are needed to evaluate the possible toxic effects caused by the ingestion of synthetic particles and their adhered pollutants by mysticete whales.

5. Conclusions

A total of 57 synthetic particles per kg krill were found in the stomach content samples of fin whales from the waters off western lceland, which, according to our estimations, would imply that an individual in this population could ingest between 38,646 and 77,292 synthetic particles per day. Despite no toxic effects have been reported for these organisms as caused by the ingestion of micro particles, this amount of litter seems high enough to fear that pollutants associated with synthetic marine litter could be transferred to fin whale tissues, potentially causing adverse effects, in an uncertain future.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2021.130564.

Credit author statement

Odei Garcia-Garin: Conceptualization, Formal analysis, Methodology, Investigation, Writing - review & editing. **Alex Aguilar:** Funding acquisition, Writing - review & editing. **Morgana Vighi:** Writing - review & editing. **Gísli A. Víkingsson:** Providing samples, Writing - review & editing. **Valerie Chosson:** Providing samples, Writing - review & editing. **Asunción Borrell:** Conceptualization, Funding acquisition, Writing–Original Draft, Writing review & editing.

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