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Impacts of Temperature and Selected Chemical Digestion Methods on Microplastic Particles

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Abstract: Alkaline and wet peroxide oxidation chemical digestion techniques used to extract microplastics from organic matrices were assessed for recoveries and for impacts on ability to identify polymer types. Methods using wet peroxide oxidation generated enough heat to result in the complete loss of some types of microplastic particles, and boiling tests confirmed that temperatures >70 °C were responsible for the losses. Fourier transform infrared spectroscopy (FT-IR) confirmed minimal alteration of the recovered polymers by the applied methods. *Environ Toxicol Chem* 2018;37:91–98. © 2017 SETAC

Keywords: Microplastics; Aquatic toxicology; Contaminants; Personal care products; Marine plastics; Chemical digestion methods

INTRODUCTION

Microplastics, which are microscopic plastic particles less than 5 mm in size [1], comprise the majority of the plastic debris present in aquatic environments based on particle abundance [2], leading to concerns about exposure and effects in organisms. Microplastic contamination has been observed in marine and freshwater [3–5], sediments [6–8], fish [9–12], and filter-feeding invertebrates [13,14]. The methods used to isolate, identify, and quantify microplastics across these matrices vary across studies, and it is recognized that there is a need to standardize protocols for comparability and to ensure accuracy in assessments [15–17].

Early methods for separating and identifying microplastics from organic matter involved manual sorting and visual identification with or without microscopy [3,9,18,19]. Enhancements to aid separation included staining that colors organic matter while leaving plastics largely unaffected [20]; however, sorting is time consuming, and smaller particles can be missed or misidentified. Recently developed methods employ a variety of isolation (e.g., manual sorting, density separation, chemical digestion, and enzyme digestion) and identification techniques (e.g., visual identification, Fourier transform infrared spectroscopy [FT-IR], Raman spectroscopy, and scanning electron microscopy). The efficacy of several isolation techniques and their potential to impact microplastics have been considered and compared [21–30].

Density separation has been used to extract microplastics from sediments (beach sands and bottom sediments; [15,28,31,32]), and involves the flotation of microplastic particles and settling of denser materials (e.g., sand) in higher density solutions. Various solutions have been used that do not alter microplastic polymers, including hypersaline sodium chloride (NaCl; [15,31], sodium iodide [23], and sodium polytungstate [7,8]. However, some of these solutions (e.g., NaCl) are not effective for higher density polymers [21], and fouling by organic and inorganic materials can alter microplastic particle densities, requiring subsequent manual sorting [8]. Sufficient gradients may not be achievable to enable separations from biological tissues [12].

Chemical methods, which may involve wet peroxide oxidation or alkaline or acidic conditions, have been developed to break down natural organic matter and leave behind the more tolerant microplastic particles along with reduced amounts of nondigestible materials to increase processing efficiency and accuracy. The National Oceanic and Atmospheric Administration (Silver Spring, MD, USA; NOAA) Marine Debris Program has produced guidance for a wet peroxide oxidation method to extract microplastics from water and sediments [32] that uses 30% hydrogen peroxide (H_2O_2) and an iron (Fe(II)) catalyst, and the application of heat during digestion and drying of samples. Modified versions of this method have been used in a number of

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studies [5,33,34]. The wet peroxide oxidation method has been used in combination with alkaline (potassium hydroxide [KOH] 224 g/L) digestion for fish tissues (S. Mason, SUNY Fredonia, Fredonia, NY, USA, personal communication); the tissues were first heated in the KOH solution followed by the wet peroxide oxidation with 35% H_2O_2. Nuelle et al. [23] tested 30 and 35% H_2O_2 solutions for impacts on several microplastic polymers over a period of 7 d, demonstrating that there is potential for wet peroxide oxidation to impact polymers, with particles of polyethylene and polypropylene <1 mm in size losing 16 to 17% of their volume.

Alkaline conditions have also been demonstrated to digest biogenic material [10,22,23,25,26,35]. Foekema et al. [10] used 10% KOH to digest fish digestive tracts over a 2- to 3-wk period, but did not evaluate treatment effects on plastic particles. Cole et al. [22] tested sodium hydroxide (NaOH) in comparison with enzyme-based and acidic conditions, finding that a 10 M NaOH solution, the most optimal alkaline conditions tested, impacted nylon, polyvinylchloride (PVC), and polyethylene particles. Catarino et al. [25] and Nuelle et al. [23] did not indicate that the alkaline conditions they tested impacted the tested microplastic particles, and Catarino et al. [25] were able to confirm identity of particles by FT-IR post treatment. Dehaut et al. [26] indicated minimal changes visually and by Raman spectroscopy to microplastics when using a 10% KOH, except for cellulose acetate.

Acidic conditions have also been applied to digest natural materials [14,21,22,23,25]. Claessens et al. [21] found that a strong, hot nitric acid (HNO_3) solution was effective, although polystyrene foam spheres melted somewhat, and nylon fibers were lost. Dehaut et al. [26] found that HNO_3 degraded polyamide (nylon). Catarino et al. [25] noted losses of nylon fibers and melding of polyethylene terephthalate and polyethylene particles. Enders et al. [27] evaluated a protocol that uses a 4:1 mixture of HNO_3 and perchloric acid ($HClO_4$) and found that several polymers degraded including polyamide, polyurethane, black tire rubber elastomer, and, to a lesser degree, acrylonitrile butadiene styrene, polymethyl methacrylate, and PVC. Collard et al. [36] used sodium hypochlorite (NaClO) and HNO_3 rinses and ultrasonication to digest fish stomach contents, finding it to be effective except for a 25% mass loss of PVC. Hydrochloric acid (HCl) has been evaluated in digestions, but was found to be the least effective method for reducing biogenic material [22,23].

There remains no clear consensus on the most effective chemical digestion method or standardized procedures. Alternatives to chemical digestion using enzymes have been evaluated for a limited subset of biological samples [22,25], and further development work is required. The studies summarized highlight the need to ensure that the chosen methods have minimal impact on the microplastic particles being isolated for identification and characterization.

The present study evaluates the impacts of alkaline and/or wet peroxide oxidation digestion conditions on several types of microplastic particles and microbeads isolated from consumer products. The recoveries of a known number of particles and impacts of treatments on ability to identify polymer type by FT-IR were evaluated. These tests were conducted to support selection of a method to be applied to digestive tracts from laboratory fish that were to be fed the tested microplastic particles and microbeads in a subsequent study. This systematic evaluation was undertaken after an initial wet peroxide oxidation test resulted in the loss of microbeads.

MATERIALS AND METHODS

Microplastic particles

A variety of microplastic particles were selected for testing for impacts of digestion conditions based on shape, but they also represented a variety of polymers, because they were to be part of a subsequent fish ingestion and retention study. Five types of microbeads were isolated from consumer face and body washes available in stores in Ontario, Canada by rinsing a volume of the products through a 125-μm metal sieve using deionized water (diH_2O). Three types of spherical microbeads (SB1, SB2, and SB3), irregular-shaped microbeads, and fragmented microbeads were collected. Based on product labels, SB1 was identified as cera microcrystallina, SB2, SB3, and irregular-shaped microbeads as polyethylene, and fragmented microbeads as oxidized polyethylene. Shavings were mechanically generated from a block of polyethylene using a pipe-threading tool in a drill press. Polystyrene foam spheres were taken from a sheet of expanded polystyrene foam insulation board. Synthetic fibers were cut from a nylon carpet sample. All microplastics included in treatments were within the 125-μm to 1-mm size range.

Digestion treatment conditions

Groups ($N_i = 20$ particles) of each type of microplastic were prepared for addition to each of 3 independent replicates/treatment (Figure 1). The treatments consisted of a control containing the microplastics in room temperature diH_2O and no chemicals added. The controls were also used to assess potential contamination through the isolation, handling, and counting stages. The remaining treatments consisted of boiling diH_2O, and 4 different chemical digestion methods. Based on literature findings, alkaline and wet peroxide oxidation-based methods were chosen. The alkaline methods used KOH at 2 concentrations for 14 d without additional heat and were adapted from Foekema et al. [10] and Rochman et al. [35]. The wet peroxide oxidation method was adapted from the NOAA protocols [32], and a digestion combining KOH (224 g/L) and 35% H_2O_2 used for digestion of Great Lakes fish tissues was included (S. Mason, SUNY Fredonia, Fredonia, NY, USA, personal communication).

For each replicate and treatment, the groups of 20 particles for each microplastic type were transferred to plastic vials or glass beakers with diH_2O then treated as outlined in the following sections.

Control (room temperature diH_2O). Microplastic particles were transferred into polypropylene vials filled to a minimum of 15 mL with diH_2O, covered, and left standing in a fume hood at room temperature for 14 d.

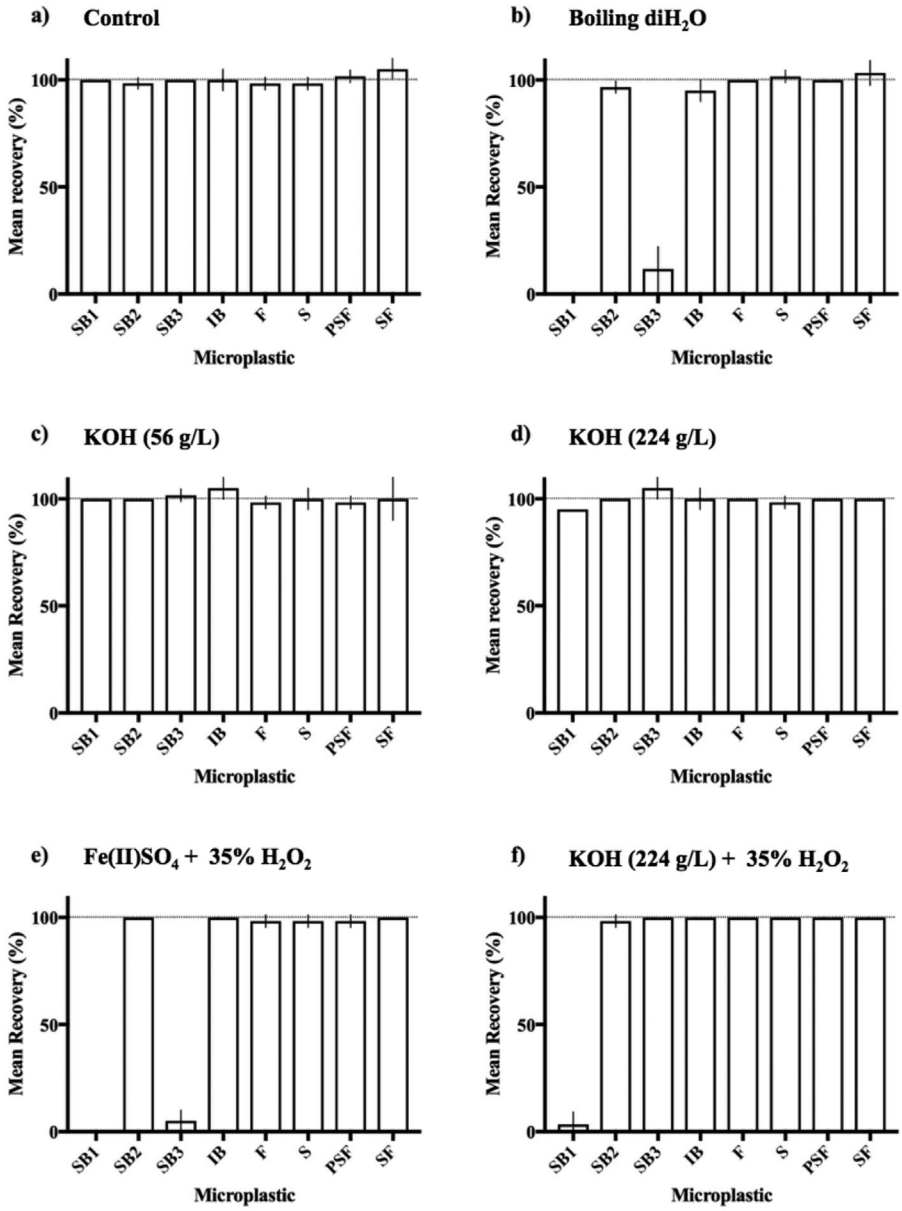

FIGURE 1: (a–f) Mean percentage of recovery of microplastic types ($n = 3$) from different consumer and industrial products across a range of chemical digestion methods, boiling water (~100 °C) and a room temperature diH_2O control. Microplastics tested include spherical microbeads (SB1, SB2, SB3), irregularly shaped microbeads (IB), fragmented microbeads (F), shavings (S), polystyrene foam (PSF), and synthetic fibers (SF).

Boiling diH$_2$O. Deionized H_2O (30 mL) was added to the microplastics in 250-mL glass beakers, which were heated on a hot plate to 100 °C and allowed to boil for 10 min. The contents were then sieved while hot.

KOH (56 and 224 g/L). A minimum of 15 mL of KOH at one of 2 concentrations (56 or 224 g/L), prepared from 85% (w/w) KOH pellets (Sigma-Aldrich), was added to the microplastics in polypropylene vials, covered, and left to stand for 14 d in a fume hood.

Fe(II)SO$_4$ + 35% H$_2$O$_2$. Under a fume hood, aliquots of 20 mL of a prepared solution of $Fe(II)SO_4 \cdot 7H_2O$ (15 g/L; Sigma-Aldrich) and 20 mL of 35% H_2O_2 (Sigma-Aldrich) were added to the microplastics in 250-mL beakers. Once the reaction settled (no boiling or bubbling) and the solution cooled somewhat, an additional 20 mL of 35% H_2O_2 was added, and this step was repeated until a total of 5 aliquots had been added. No additional heating was employed. Afterward, the contents of the beaker were rinsed into a 125-μm metal sieve to remove excess water, and then rinsed back in to the original beaker for a cleaning step. Microplastics were soaked in a 5:1 diH_2O:Contrad 70® liquid detergent (Fisher Scientific) solution for up to 24 h covered in a fume hood.

KOH (224 g/L) + 35% H$_2$O$_2$. Under a fume hood, a 30-mL aliquot of KOH (224 g/L) solution was added to the microplastics in 600-mL beakers, covered, and magnetically stirred on a hot

plate at 60 °C for 1 h. After cooling, a 5-mL aliquot of 35% H_2O_2 was added to each beaker, and the solutions were stirred for 15 min and then allowed to stand covered for 2 h.

Post treatments, all samples were transferred to a 125-μm metal sieve, rinsed thoroughly with diH_2O, then transferred to aluminum dishes, and dried in an oven at 60 °C until dry. During the course of the tests, aluminum trays were replaced with 10-μm polycarbonate filters (Fisher Scientific) to increase efficiency by reducing dry time. The remaining microplastic particles were counted under a dissecting microscope (×10–80 magnification; Leica S8 APO Stereozoom; Leica Microsystems Canada).

FT-IR analysis

To indicate whether treatments impacted the polymer materials, control and selected microplastic particles ($n = 3$) of each type subjected to each treatment were analyzed by a Bruker Vertex-70 FT-IR spectrometer (Bruker Optics) using the Platinum attenuated total reflectance (ATR) accessory equipped with a diamond crystal plate and operated in ATR mode. Recorded spectra were compared with spectral library databases (S.T. Japan, Hummels) and the top identified material listed based on hit quality. Spectra from control and treated particles were also compared via correlation analysis provided within the OPUS Spectroscopy Software (Ver 7.2; Bruker Optik).

Data analysis

Percentage of recovery was determined from the number of microplastics remaining in each sample (N_f) following exposure to each method using the following equation

$$\%\,\text{Recovery} = \left(\frac{N_f}{N_i}\right) \times 100\%$$

Three independent replicates were used in each test. The mean percentage of recovery and standard deviation were calculated for triplicates from each treatment.

Kruskal–Wallis rank sum tests, nonparametric tests similar to a one-way analyses of variance, were performed in R to determine whether there were significant differences ($\alpha = 0.05$) among microplastic types for each treatment.

RESULTS

Recoveries from treatment methods

Average recoveries for triplicate treatments of the various types of microplastics are presented in Figure 1 and the Supplemental Data, Table S1. Recoveries among microplastic types were significantly different, as confirmed by the Kruskal–Wallis rank sum tests ($\chi^2 = 27.883$, 7 *df*, $\alpha = 0.05$, $p < 0.001$) for the wet peroxide oxidation test, KOH (224 g/L) followed by wet peroxide oxidation, and for the boiling tests. Differences were driven by SB1 and SB3. Complete or nearly complete recoveries (95–100%) of particles were observed across all shapes and material types for the control (room temperature diH_2O) and both KOH (56 and 224 g/L) treatments. Recoveries that differed slightly from 100%, particularly those in the control group, represent the method error associated with handling, transfers, sieving, and counting. Some error may also be partly attributed to observed breakage of some particles during processing, with the result that a single particle can be counted as more than one particle. Losses may also have occurred if broken pieces were small enough to pass through the sieve.

Control. For almost all microplastic types, 100% recovery was observed (Figure 1A). Differences in recoveries among microplastic types were not significant ($\chi^2 = 27.883$, 7 *df*, $\alpha = 0.05$, $p > 0.001$).

Boiling diH_2O. Differences in recoveries among microplastic types were significant ($\chi^2 = 27.883$, 7 *df*, $\alpha = 0.05$, $p < 0.001$). During heating, SB1 began to melt at approximately 60 °C and no beads remained after boiling (Figure 1B). The SB2 particles melted and adhered to the stir bar. The temperature of the metal in the stir bar may have been hotter than the surrounding water, causing SB2 to melt. The test was repeated without the stir bar and no melting or adhering was observed (Figure 1B). Particles of SB3 were completely lost in one replicate, with only a few recovered in the others (~12%; Figure 1B), but a small white, waxy mass was observed in the beaker in each replicate. For all other microplastic types, nearly 100% recovery was observed (Figure 1B).

KOH (56 and 224 g/L). Differences in recoveries among microplastic types were not significant ($\chi^2 = 27.883$, 7 *df*, $\alpha = 0.05$, $p > 0.001$) for both concentrations of KOH solutions (56 and 224 g/L; Figure 1C and D). Complete or nearly complete recovery (95–105%) was observed for all microplastic types. Minor discoloration of some microplastics, particularly SB1 and SB2, was noted under the microscope following the more concentrated KOH (224 g/L) treatments.

Fe(II)SO_4 + 35% H_2O_2. Differences in recoveries among microplastic types were significant ($\chi^2 = 27.883$, 7 *df*, $\alpha = 0.05$, $p < 0.001$). Mean recoveries of 0 and 5% were observed for SB1 and SB3, respectively (Figure 1E). For all other types, complete or nearly complete recovery was observed (Figure 1E). After the first aliquot of H_2O_2 was added, the highest recorded temperatures ranged from 72 to 89 °C, and typically increased for subsequent aliquots. The peak temperature was recorded at 93 °C. The low recoveries of SB1 and SB3 were consistent with the boiling treatment results.

KOH (224 g/L) + 35% H_2O_2. Differences in recoveries among microplastic types were significant ($\chi^2 = 27.883$, 7 *df*, $\alpha = 0.05$, $p < 0.001$). The mean recovery of SB1 was 3% (Figure 1F). Melting began at the 63 to 68 °C temperature range and particles agglomerated into an indistinguishable mass as melting progressed. For all other microplastic types, complete or nearly complete recovery was observed (Figure 1F).

FT-IR evaluation

Selected microplastic particles remaining after exposure to treatment conditions were subjected to FT-IR analysis to confirm that conditions minimally impacted the polymers and the materials could still be identified. The results of the spectral correlation analyses between post-treatment particles and controls are listed in Table 1. Example spectra of each particle type by treatment are provided in Figure 2 for fragment microbeads and in the Supplemental Data, Figures S1 to S7, for the other particle types. Listings of assigned particle identities and hit qualities are in the Supplemental Data, Table S2. Post-treatment spectra exhibited few differences from control spectra, with percentage similarities >95% across all treatments for particles SB1 and SB2, the remaining melted waxy material for SB3, irregular-shaped microbeads, fragmented microbeads, and shavings (Table 1), and as illustrated in Figure 2 for fragmented microbeads. There may be some indication of polymer modification of fragmented microbeads, with small bands appearing at approximately 1450 and approximately 1550 cm^{-1} for the 2 KOH treatments (Figure 2). Similarities were lower (71–82%) for polystyrene foam subjected to the more concentrated alkaline treatment and those using wet peroxide oxidation. The spectra for polystyrene foam also indicated that some minor polymer modification may be occurring. The polystyrene foam spectra contained a broad band from 3400 to 3500 cm^{-1} in treatments with wet peroxide oxidation and KOH (224 g/L), which may be indicative of hydrogen bonding after hydrolysis during digestion, but could also indicate the presence of some water remaining encapsulated in the foam given that this feature was also present in the control sample. The wet peroxide oxidation and KOH (224 g/L) treated polystyrene foam also had bands appearing at approximately 1450 and approximately 1550 cm^{-1}, and the 2 treatments with KOH (224 g/L) had small bands appearing at approximately 1000 cm^{-1}. The nylon fibers (synthetic fibers) had slightly lower percentages of similarities (88–97%; Table 1) and hit qualities (Supplemental Data, Table S2), but this may have been influenced more by lower responses on FT-IR because of the narrow cross sections of the fibers (Supplemental Data, Figure S7). Assigned identities from the library searches varied within particle types but were generally consistent across treatments, indicating that particle identification was essentially unaffected by the treatments (Supplemental Data, Table S2).

DISCUSSION

The choice of chemical digestion method used to remove natural organic matter and isolate microplastic particles, and the conditions under which the method is applied, can significantly impact the recovery of some types of microplastic particles. In the present study, heating conditions were found to impact 2 of 5 types of microbeads from personal care products tested. The beads SB1 and SB3 melted in treatments where solution temperatures from application of heat or heat generated from the exothermic oxidation reactions increased above 60 °C. Others have noted losses or changes in the microplastics particles when heat has been applied. In addition to complete loss of nylon microplastic particles, Claessens et al. [21] noted melting and clumping of polystyrene spheres when using a strong HNO_3 solution at 100 °C, and Catarino et al. [25] observed fusing of some polyethylene terephthalate and high-density polyethylene particles under similar conditions. Avio et al. [12] found that only approximately 4% of polyethylene and polystyrene was recovered from fish tissues digested in HNO_3 at 100 °C. The losses and changes in physical particle character were attributed to the aggressive nature of nitric acid [12] and the combined effects of temperature and reaction with the strong acid [25]. In this case, the losses of the microbeads were solely attributed to the heat applied or generated during the digestions, based on obtaining similar results with boiling water only. The wet peroxide oxidation and alkaline conditions used in the present study did not impact recovery of polystyrene foam or nylon particles, nor did temperatures up to 100 °C.

The alkaline and wet peroxide oxidation digestion conditions in the present study did not impact the ability to correctly identify the polymeric character of the particles remaining after treatment based on FT-IR spectral correlations between treated particles and controls (Table 1) and library assignments (Supplemental Data, Table S2). Dehaut et al. [26] and Catarino et al. [25] similarly did not observe impacts on particles by Raman spectroscopy and FT-IR respectively, from alkaline digestions that affected identification. However, the FT-IR spectra did indicate some modifications to polystyrene foam that reduced spectral similarities to controls.

Losses or melding of some types of microplastic particles because of digestion and heating conditions may lead to reports of microplastic concentrations/abundances that are biased low,

TABLE 1: Percentage of similarity (mean ± standard deviation) of Fourier transform infrared spectroscopy spectra for each microplastic type across 4 different chemical digestion methods and boiling compared with the control treatment ($n = 3$)

	Boiling diH_2O	KOH (56 g/L)	KOH (224 g/L)	$Fe(II)SO_4 + 35\%\ H_2O_2$	KOH (224 g/L) + 35% H_2O_2
SB1	N/A	95.2 ± 0.1	99.8 ± 0.0	N/A	N/A
SB2	100.0 ± 0.0	99.7 ± 0.2	99.9 ± 0.1	99.9 ± 0.0	99.9 ± 0.1
SB3	N/A	99.0 ± 0.3	99.8 ± 0.1	N/A	97.8 ± 0.8
IB	99.8 ± 0.3	99.4 ± 0.1	96.2 ± 0.5	99.6 ± 0.4	99.5 ± 0.0
F	99.9 ± 0.0	99.2 ± 0.5	98.9 ± 0.9	99.6 ± 0.2	99.9 ± 0.0
S	98.5 ± 1.9	99.1 ± 0.7	98.2 ± 0.9	96.9 ± 2.5	99.3 ± 0.3
PSF	95.5 ± 0.9	94.1 ± 2.6	71.3 ± 9.0	81.7 ± 8.3	77.5 ± 8.4
SF	94.1 ± 0.1	87.6 ± 13.6	96.8 ± 0.9	91.3 ± 1.2	88.0 ± 2.5

SB1, SB2, SB3 = spherical microbeads; IB = irregularly shaped microbeads; F = fragmented microbeads; S = shavings; PSF = polystyrene foam; SF = synthetic fibers; N/A = not available.

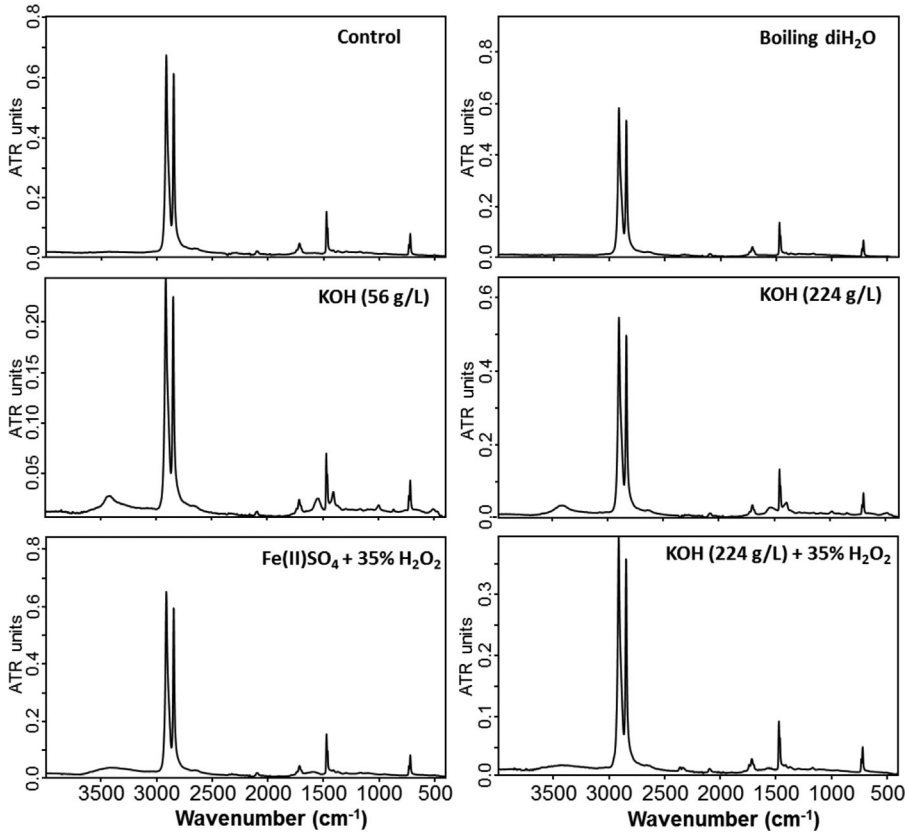

FIGURE 2: Fourier transform infrared spectroscopy–attenuated total reflectance spectra of individual microbead fragments from a body wash product subjected to the treatment processes. ATR = attenuated total reflectance.

as suggested by Catarino et al. [25], and errors in microplastic proportions by categories, which may represent specific source types. Based on our results, the microbead source category may be under-represented in studies that use or generate heat above 60 °C. In addition to the 2 microbeads melting of the 5 that were tested across treatments, 6 of 14 off-the-shelf microbead-containing personal care products had microbeads that melted at temperatures ranging from 70 to 98 °C when subjected to boiling tests, suggesting that up to 40% of the different types of microbeads used in products could be lost under elevated temperatures.

There is some ambiguity both in the labeling of products that contain microbeads and in the definition of microbeads in relation to microplastics. Of the 5 microbeads tested, one (SB1) was listed in the ingredient list as "cera microcrystallina," whereas the others were listed as polyethylene (SB2, SB3, irregular-shaped microbeads) or oxidized polyethylene (fragmented microbeads). The library database search assignments for each of the 5 microbeads were similar in nature, with each having either polyethylene wax or paraffin wax among the listed assignments, along with some version of polyethylene or polyethylene copolymer (Supplemental Data, Table S2). Microbeads listed in products as polyethylene are considered as microplastics, whereas those listed as cera microcrystalline (also called microcrystalline wax and/or synthetic wax) may not be considered microplastics. Waxes are more likely to melt at lower temperatures than most plastics. For example, microcrystalline waxes have a melting point range of 62 to 102 °C [37]. Given the ambiguity in product labeling, and the possibility that many microbeads thought to be plastic may be waxes (at least as assigned by FT-IR), there is merit in ensuring that microbeads are broadly captured within methods to monitor and assess microplastics, especially given recent voluntary and regulatory efforts to remove plastic microbeads from products. A more in-depth analysis may be merited to better characterize the materials used as microbeads and potential replacements in products for their stability in the environment and to determine whether there is the potential for ingestion and concerns similar to those for polyethylene microbeads.

Alkaline and wet peroxide oxidation-based digestions have both been shown to be effective in the removal of biological tissues to isolate microplastic particles [10,26,27,35] (S. Mason, SUNY-Fredonia, Fredonia, NY, USA, personal communication). The wet peroxide oxidation methods developed by NOAA [32] are also very effective at removing a variety of plant-based and other organic matter in water, wastewater, and sediment samples [5,23,33,38]. Methods that recommend heating to temperatures >60 °C or have the potential to reach such temperatures during digestion reactions should be revised to maintain lower temperatures, to ensure that all materials are retained that may be considered microplastics, especially microbeads. For example, we have found that using an ice bath to cool digestion beakers when temperatures begin rising above 50 °C has prevented high temperature spikes in samples subjected to wet peroxide oxidation protocols. This does require constant monitoring by those carrying out the

digestions, but has allowed the wet peroxide oxidation process to effectively remove algae and other plant material while preventing stronger exothermic reactions with abundant organic matter from wastewater and surface water samples. Inclusion of an ice bath and lower temperatures within wet peroxide oxidation protocols could enable these methods to be applied to biological tissues, ensuring that particles such as microbeads from personal care products are not lost during processing steps.

CONCLUSIONS

It is essential to test chemical digestion protocols for recovery of microplastics before proceeding with sample processing and analysis. Given the results of our study, we caution against any chemical digestion methods that apply heat or generate temperatures greater than 60 °C in either the digestion or drying stages. Based on recoveries observed for the different microplastic types and methods tested, alkaline digestions at room temperature, or incubated at temperatures less than 60 °C, may be best suited for tissue digestions. Wet peroxide oxidation remains an effective method for digesting samples with plant matter in marine and freshwater samples (water, sediment), and may be effective for tissues as well, but temperatures must be controlled (eliminating spikes in temperature during reactions) at or below 60 °C to minimize the loss of any constituent microplastics, particularly microbeads from personal care products. Assessments of the occurrence, types, sources, and impacts of microplastics may be incomplete if method-processing conditions selectively remove some types of materials.

Supplemental Data—The Supplemental Data are available on the Wiley Online Library at DOI: 10.1002/etc.3935.

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Data availability—Data files can be accessed by request via email to the corresponding author (keenan.munno@mail.utoronto.ca).

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