The influence of cosmetic microbeads on the sorptive behavior of cadmium and lead within intertidal sediments: A laboratory study

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HIGHLIGHTS

- Numbers of microplastics within Burrard Inlet, BC were 3120 and 5560/kg wet sediment.
- Seventy-five percent of recovered plastics were microbeads.
- Laboratory experiments where sediments were spiked with microbeads were applied.
- Less lead was recovered from pore water of treatment as compared to control microcosms.
- Sorption of metals such as lead to microplastics could introduce metals into food chains.

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ABSTRACT

Concentrations of microplastics within two geographically distinct urban locations within Burrard Inlet, British Columbia (BC), and the influence of facial scrub microbeads on lead and cadmium sorption within intertidal sediments were determined. Bulk intertidal sediment sampled from Cates Park (CP) located within the protected part of the inlet contained greater concentrations of microplastics (5560/kg wet sediment) as compared to Horseshoe Bay (HSB) (3120/kg wet sediment) located on the exposed open part of the inlet. Of the recovered microplastics ca. 75% were characterized as microbeads. Laboratory controlled microcosm experiments in which microbeads separated from a commercial facial scrub were added to bulk sediments collected from CP at ambient and 10-fold ambient (high) concentrations indicated that the microbeads acted as sorption sites. At ambient concentrations, less lead was recovered from pore water and surface water of treatment as compared to control microcosms. At high concentrations, the microbeads acted as a contaminant source to the microcosms, notably cadmium. Sorption of lead to microbeads has important implications for the potential role of microplastics, in this case microbeads acting as a yet quantified link in aquatic food webs.

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1. Introduction

Jambeck et al. (2015) recently quantified the yearly input of plastic waste from land to ocean, 4.8 million tons (Mt) and possibly as high as 12.7 Mt enters the ocean from land with the potential for cumulative impacts as high as 250 Mt by 2025. Once released into the environment, plastics continually breakdown through mechanical processes facilitated by UV radiation. In addition to the plastic particulates are microbeads which originate from a number of sources (e.g., cosmetic facial scrubs) and are released directly into the environment (Fendall and Sewell, 2009). Microplastics (<5 mm in diameter) are now ubiquitous within our aquatic environments and alarming numbers have been found within sediments of estuaries, freshwater and intertidal/coastal ecosystems (Eerkes-Medrano et al., 2015; Van Cauwenberghe et al., 2015; Jambeck et al., 2015; Cole et al., 2011).

Aquatic sediments have traditionally been characterized as being comprised of several different geochemical phases that can act as potential sinks for metals entering an aquatic environment. These phases include clay, silt, sand (i.e., grain size distribution), organic matter, oxides of iron and manganese, aluminum and silica, carbonates and sulfide complexes (Shea, 1988). Of these components, oxides of iron and manganese and organic matter have been considered the most important geochemical components controlling metal behavior including metal bioavailability to sediment ingesting organisms within the oxic portion of sediment (e.g., Thomas and Bendell-Young, 1998 and Bendell-Young et al., 1992). However, with the high concentrations of microplastics that now occur within coastal sediments (e.g., on heavily impacted...
beaches microplastics can make up 3.3% of the sediment by weight, Van Cauwenbergh et al., 2015, and the potential for these particulates to influence geochemical processes, sediments now must be viewed as being comprised not only of the classic geochemical parameters, but of microplastics as well.

Recent studies have identified the ability of microplastics to sorb trace metals from the aqueous environment (Holmes et al., 2012; Rochman et al., 2014). However, no studies as yet have attempted to determine their sorptive role within sediments under environmentally relevant conditions. Further, coastal sediments in which a high number of plastic particulates have been found are subject to tidal events and as a result, wide variations in temperature and moisture due to flood and ebb tide occur within these environments. Both these factors could result in different trace metal sorption characteristics as compared to studies which have isolated the microplastics from the sedimentary environment.

Hence, the objective of our study is through the application of microcosm studies where sediments are manipulated to simulate a tidal regime, to assess the role of microplastics within the sediments, in this case microbeads separated from a cosmetic product on the sorptive behavior of toxic trace metals within intertidal sediments.

2. Materials and methods

2.1. Sediment sampling

Bulk sediments were collected between April and July of 2015, at low tide, at two locations within Burrard Inlet, BC, Cates Park (CP) and Horseshoe Bay (HSB) (Fig. 1). CP is a residential park located east of a major shipyard/industrial region whereas HSB is a major ferry terminal. CP unlike HSB is subject to moderate tidal influences and is well protected within the inlet. HSB experiences full tidal regimes and is directly exposed to storm events. Approximately 20 kg of surficial (0–5 cm) bulk sediment was collected by shovel, placed into a bucket, transported to Simon Fraser University and stored in a cold room (4°C) and aerated until analysis.

2.2. Sediment parameters

Sediments (n = 6) were characterized for grain size and organic matter after methods of Bendell et al. (2014). In brief, 10 g of sediment was sieved through 4 sequential sieves to obtain grain separations of >1 mm; 1–0.5 mm; 0.5–0.25 mm; 0.25–0.063 µm. Retained sediment was dried at 40°C, weighed and percent grain size determined for each fraction. Organic matter was determined by loss on ignition (LOI), where 1 g of sediment was ignited at 550°C for 15 h and the difference pre and post burn representing % organic matter.

2.3. Microplastic recovery

Samples of sediment (n = 8), 50 g each of sediment were subjected to the “overflow/flotation” method (Claessens et al., 2013, Vianello et al., 2013, Nuelle et al., 2014) consisting of adding to the sediments a solution of sodium chloride (300 g/L) to separate low density particulates such as plastics from sediments. The solution was stirred at 5 and 30 min. This procedure was repeated 3 times. Recovered plastics were identified and counted using a Neubauer chamber at 40 fold magnification.

2.4. Separation of microplastics from cosmetic facial cleansers

Facial cleanser (Clearasil Ultra Rapid Action Scrub) was washed through a 0.063 µm sieve with warm tap water to separate the beads from the associated soap products and collected microbeads rinsed with distilled deionized water. Microbeads were comprised of polyethylene as indicated in the list of product ingredients provided on the product container. Active ingredients within the facial scrub also included salicylic acid (2%). Other ingredients as listed on the product container in order of importance are provided in the supplementary material.

2.5. Benchtop microcosm experiments

We applied methods of Bendell et al. (2014) to address the hypothesis that cosmetic microbeads would alter sediment sorptive characteristics. Sediments from CP were spiked with two concentrations of microbeads, ambient (6 microbeads/g sediment) and 10 fold ambient (64 microbeads/g wet sediment) where ambient is the approximate number of microplastics found in CP sediments. Six microcosms (12 cm length × 6 cm wide × 8 cm depth), three controls and three treatments were prepared by adding 775 g of sediment and 400 mL of seawater. Microcosms were established three days prior to any manipulation. All microcosms were aerated to ensure oxic conditions within surficial sediments. Simulated tidal conditions were applied after Bendell et al., 2014.
et al. (2014) (Fig. 2). Microcosms were flooded with seawater for 8 h. Surface and pore water was then sampled by recovering 10 mL of water with a syringe representing “ebb” tide (RI, RII, RIII). Exposed sediment within the microcosm was placed under a heat lamp to mimic warming conditions of the surface sediment at ebb tide. Maximum surface temperature over the 16 h exposure was 30 °C. After ebb tide, fresh seawater was added to mimic “flood” tide conditions (FI, FII, FIII) and surface water and pore water sampled as described above. Ebb and flood tides were sampled in triplicate. Samples were placed into 10 mL Falcon tubes and frozen until trace metal analysis.

### 2.6. Lead and cadmium analyses

Samples were analyzed by Agilent 7700 Series quadrupole ICP-MS at the Pacific Centre for Isotopic and Geochemical Research, University of British Columbia. Complete instrumental details can be found in Schude et al. (2015). All samples were diluted 20 fold in 15 mL Falcon tubes with distilled deionized water and 0.2 mL environmental grade nitric acid added to samples to obtain a 1% nitric acid solution prior to analysis. Ten μg L⁻¹ of ¹¹⁵In was added to all samples, blanks and standards for internal standardization and to compensate for instrumental drift (Schude et al., 2015). Each experimental run consisted of 10 samples followed by one blank and one standard.

### 2.7. Data analysis

SigmaPlot12 (Systat Software Inc.) was used for data analysis. Statistical significance was accepted at \( P < 0.05 \). All data were tested for normality (Shapiro–Wilk) and homogeneity of variances (Equal Variance Test). When assumptions of normality and equal variances were not met data were transformed as (\( \log (10) \) + 1). Simple student \( t \)-tests were applied to determine significant differences between HSB and CP for each sediment parameter (grain size, % organic matter), microplastic type and total number of microplastics recovered from the sediments. For each metal, two-way analysis of variance (ANOVA) were applied with Treatment (with microbeads, without microbeads) and Tide (Flood and Ebb) as the two factors. If significant differences (\( P < 0.05 \)) were detected Holm–Sidak post hoc multiple comparisons tests were applied to determine where differences occurred (SigmaPlot 12).

### 3. Results

#### 3.1. Sediments

Sediments from CP tended to contain greater amounts of organic matter (Student’s \( t \)-test \( P = 0.1 \)) and with the exception of the largest grain size ( > 1 mm) were comprised of greater amounts of the three grain size fractions <0.5 mm as compared to HSB sediments (Student’s \( t \)-test \( P < 0.05 \)). HSB sediments contained significantly greater amounts of sediment greater than 1 mm diameter as compared to CP (\( P < 0.05 \), Table 1). CP sediments also contained significantly greater amounts of plastic chips, microbeads and total number microplastics as compared to HSB sediments (Student’s \( t \)-test, \( P < 0.05 \), Table 1). Numbers of fibers tended to be greater at CP as compared to HSB (Student’s \( t \)-test, \( P = 0.09 \)). At both locations, ca. 75% of recovered microplastics were identified as microbeads. Given their coastal locations (Fig. 1), these differences are not unexpected, as CP is located within a protected region of the inlet allowing for the deposition and accumulation of smaller sediment grain sizes, organic matter and microplastics as compared to the more exposed HSB.

### 3.2. Microcosm studies

#### 3.2.1. Lead and cadmium

Under ambient microbead additions, concentrations of lead were less in pore and surface water of treatments as compared to controls (Fig. 3(b) and (d) and Table 2). There was also a tidal affect with higher concentrations of lead recovered in flood as compared to ebb tides. No significant differences occurred for cadmium (Fig. 3(a) and (c) and Table 2). At microbead concentrations 10 fold those above ambient, cadmium was greater in pore water of treatments as compared to controls with ebb tide surface water concentrations being greater than flood tide concentrations. No significant differences (\( P > 0.05 \)) were observed for lead concentrations

### Table 1

Sediment characteristics of the two study location. HSB is Horseshoe Bay, CP is Cates Park. Values are averages with standard errors (S.E.). Percent grain size is in mm. OM is in percent. Microplastics are \# particles/50 g wet sediment.

<table>
<thead>
<tr>
<th></th>
<th>N</th>
<th>Grain Size</th>
<th>OM</th>
<th>Microplastics (# particles/50 g wet sediment)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>&gt;1</td>
<td>0.5–1</td>
<td>0.5–0.25</td>
</tr>
<tr>
<td>HSB</td>
<td>6</td>
<td>Average</td>
<td>64.8</td>
<td>13.07</td>
</tr>
<tr>
<td></td>
<td>S.E.</td>
<td></td>
<td>0.07</td>
<td>0.01</td>
</tr>
<tr>
<td>CP</td>
<td>6</td>
<td>Average</td>
<td>40.37</td>
<td>2.14</td>
</tr>
<tr>
<td></td>
<td>S.E.</td>
<td></td>
<td>0.2</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Fig. 2. Schematic of sampling protocol indicating “flood” and “ebb” tides (after Bendel et al., 2014).
Fig. 3. (a–d) Cadmium and lead (µg/L) in pore (a, b) and surface waters (c, d) of control versus treatment microcosms for “flood” (F) and “ebb” (R) tides. Values are averages of 9 (triplicate microcosms, triplicate tides) with S.E.’s. Significant differences are provided in Table 2. Ambient microbead concentrations (6 microbeads/g of wet sediment for ca. 5000 beads/microcosm). * indicates a significant difference.

Table 2
Results of the two-way analysis of variance (ANOVA) at ambient and high (10 fold ambient) microbead concentrations with Treatment and Tide as the two factors. NS is not significant, R is “ebb tide”, F is “flood tide”, T is treatment and C is control. Treat × Tide is the interaction term for Treatment × Tide. SW is surface water, PW is pore water.

<table>
<thead>
<tr>
<th></th>
<th>Cadmium</th>
<th></th>
<th>Lead</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SW</td>
<td>PW</td>
<td>SW</td>
<td>PW</td>
</tr>
<tr>
<td>Ambient</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Treatment</td>
<td>F 0.48</td>
<td>P 0.5</td>
<td>F 0.06</td>
<td>P 0.06</td>
</tr>
<tr>
<td>Tide</td>
<td>0.04</td>
<td>0.83</td>
<td>0.06</td>
<td>0.81</td>
</tr>
<tr>
<td>Treat × Tide</td>
<td>3.5</td>
<td>0.07</td>
<td>1.04</td>
<td>0.314</td>
</tr>
<tr>
<td></td>
<td>NS</td>
<td>NS</td>
<td>F &gt; R</td>
<td>T &lt; C</td>
</tr>
<tr>
<td>High</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Treatment</td>
<td>F 2.9</td>
<td>P 0.09</td>
<td>F 0.019</td>
<td>P 0.009</td>
</tr>
<tr>
<td>Tide</td>
<td>14.18</td>
<td>0.09</td>
<td>0.75</td>
<td>0.39</td>
</tr>
<tr>
<td>Treat × Tide</td>
<td>1.8</td>
<td>0.18</td>
<td>0.02</td>
<td>0.36</td>
</tr>
<tr>
<td></td>
<td>R &gt; F</td>
<td>T &gt; C</td>
<td>NS</td>
<td>NS</td>
</tr>
</tbody>
</table>
Fig. 4. (a–d) Cadmium and lead (µg/L) in pore (a, b) and surface waters (c, d) of control versus treatment microcosms for “flood” (F) and “ebb” (R) tides. Values are averages of 9 (triplicate microcosms, triplicate tides) with S.E.'s. Significant differences are provided in Table 2. High microbead concentrations (10 fold ambient, 64 microbeads/g of wet sediment for ca. 50,000 beads/microcosm). * indicates a significant difference.

in either surface or pore water although the trend was for greater amounts in treatments versus controls (Fig. 4(a)–(d) and Table 2).

4. Discussion

Microplastics are ubiquitous in our aquatic ecosystems and present real ecological threats to these environments (Eerkes-Medrano et al., 2015; Van Cauwenberghe et al., 2015; Jambeck et al., 2015; Cole et al., 2011). Among the many types of microplastics that are found within sediments, microbeads are one of most prevalent (e.g., Castaneda et al., 2014). Here, we assess their role on influencing the behavior of lead and cadmium within intertidal sediments. Unique to our study was attempting to elucidate the role of microbeads in the sorption of trace metals under tidal regimes, which occur within intertidal ecosystems. In our experimental system, flood tide (F) represents the addition of fresh seawater to the microcosm after an 8 h exposure period, and ebb tide (R) is the measurement of seawater and pore water after a 16 h inundation period.

4.1. Microplastics in sediments

Comparison of the total number of plastic particles found in sediments with other studies and as reviewed by Rocha-Santos and Duarte (2015) is hampered by different sampling methods and units used for expressing concentrations. As one point of reference however, total number of particles recovered in HSB and CP sediments (3120/kg and 5560/kg wet sediment respectively) were similar to those found Venice Lagoon, Italy (2175–6725 particles/kg Vianello et al., 2013). Of the particles recovered, 75% were identified as microbeads. Castaneda et al. (2014) also found high concentrations of microbeads in sediments from the St. Lawrence River at numbers that were comparable to the world’s most contaminated sites.

4.2. Differential behavior of cadmium and lead

At ambient microbead concentrations, less lead was recovered in surface water and pore water of treatments as compared
to controls. No differences were noted for cadmium. Lower concentrations suggest that lead is being removed from the aqueous phases, presumable onto the sorption sites afforded by the microbeads.

There are only a few studies that have addressed the ability of microplastics to sorb trace metals from aquatic environments. Ashton et al. (2010) determined the association of metals with plastic production pellets (PPP), sampled from four beaches in SW England and noted that pellets were enriched with cadmium and lead at two sites with PPP’s able to accumulate metals to concentrations which approached those of sediment and algal fragments. Holmes et al. (2012) assessed the interactions between trace metals and PPP’s, virgin and aged, under estuarine conditions and concluded that plastic pellets effectively sorb trace metals; short term which was attributed to adsorption of organic matter and long-term which incorporated the aging of the pellet. Rochman et al. (2014) compared the long-term sorption of metals among plastic types and found that in general all types of plastic tended to accumulate similar concentrations of metals and that over a 12 month study period concentrations of all metals increased over time and did not reach saturation. An important outcome of this latter finding is that plastic debris may accumulate greater concentrations of metals the longer it remains at sea.

Not known is the role of geochemistry in influencing the sorption of metals by microplastics within sedimentary environments such as intertidal systems. Partitioning of metals within sediments and the influence of this trace metal partitioning on metal bioavailability has traditionally been thought to be controlled primarily by the main sediment components of organic matter, oxides and iron and manganese (e.g., Thomas and Bendell-Young, 1998). More recent studies that have assessed the role of these sediment attributes on the sorption of trace metals have found that generally, lead sorbs more strongly than other metals such as cadmium (Diagboya et al., 2015; Xu et al., 2015).

Diagboya et al. (2015) assessed the effects of time, soil organic matter and iron oxides on the relative retention and redistribution of lead, cadmium and copper in soils. They found that lead was preferentially sorbed over cadmium (and copper) with this preference decreasing with time. Of further note, organic matter was found to determine immediate sorption, whereas oxides of iron determined the long term redistribution of metals in soils. Xu et al. (2015) compared the sorption behaviors of cadmium and lead from water onto oxides of manganese and iron and noted that lead sorption was ca. two and four fold greater versus cadmium onto oxides of manganese and iron respectively. Results of our study suggest that microbeads are effectively a fourth sediment component and as suggested by Ashton et al. (2010) can compete for sorption sites, in this case for lead. The differential sorption characteristics of lead versus cadmium observed by Diagboya et al. (2015) and Xu et al. (2015) could explain why cadmium was not different in surface or pore waters between treatment and control microcosms.

In contrast to ambient concentrations of microbeads, for the 10 fold ambient concentrations, there was either no difference between treatment versus control (for lead) or concentrations were greater in treatment as compared to control microcosms (cadmium in pore water). Cadmium was also greater in surface water collected at ebb versus flood tide. One explanation is that at higher microbead concentrations, rinsing the facial scrub with warm water was not sufficient to remove all the various additives which makeup the cosmetic product. Further, higher concentrations of cadmium in treatments versus control suggest that the facial scrub may have actually been a source of cadmium to the microcosms. Higher concentrations of cadmium in ebb tides supports this as the 16 h the sediments are inundated would allow for the movement of cadmium from the spiked sediments to the surface water. As noted in the product information (listed in supplementary material), cosmetic facial scrubs contain greater than 20 components including propylene glycol. Johnson (2001) in a final report on the safety assessment of propylene glycol derivatives noted that impurities that were detected during the manufacture of the polymers included iron, cobalt, nickel, cadmium and arsenic at a maximum of 10 mg/L combined. Other constituents within cosmetic microbeads that could affect the sorption characteristics of lead and cadmium within sediments include EDTA (Ethylene diaminetetraacetic acid) which is known to strongly complex metals, notably lead (Chrstny et al., 2008).

5. Conclusions

Through application of controlled microcosm studies we found that microbeads separated from a cosmetic cleanser provided sorption sites for lead within intertidal sediments. Seltenrich (2015) recently reviewed the literature on Marine Plastic Pollution and Seafood Safety. It was noted that several studies have demonstrated plastics’ ability to sorb persistent, bioaccumulative, and toxic substances present in all water bodies and that the plastics as well as the chemicals and metals that are sorbed can travel into the bodies of marine organisms. Our findings suggest that within sedimentary environments, the toxic metal lead can sorb to microbeads and if the microbeads are ingested could provide an additional point of entry of this contaminant into the food chain. An important caveat and as noted by Katsnelson (2015), to date, almost all studies which have attempted to elucidate the impact of microplastics in aquatic environments have been lab based and it is as yet known if results can be extrapolated to real environmental conditions. It is unknown whether microplastics are indeed as suggested by Seltenrich (2015) a new vector in the transfer of toxic materials through the food chain or if amounts are insignificant compared to food and water. Clearly, much research is required to shed some light on the role of microplastics in aquatic environments and what threats are actually posed by these now recognized components of intertidal sediments.

Acknowledgment

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Appendix. Supplementary material

Other ingredients contained in the facial cosmetic scrub as listed on the product container in order of importance include; PPG 15 Stearyl Ether, Glycerin, Mica, Stearyl Alcohol, Cetyl Stearate, Cetearyl Alcohol, Distearlyldimonium Chloride, Sodium Lauryl Sulfate, Alcohol, Steareth 21, Sodium Chloride, Behenyl Alcohol, Synthetic Wax, Steareth 2, Xanthan Gum, Fragrance, Dimethyl Palmamidopropylamine, Hydrolyzed Milk Protein, Ferric Ammonium Ferrocyanide (CI 77510), Propylene Glycol, Magnesium Nitrate, Disodium EDTA, Isopropyl Alcohol, BHT, Diphopropylene Glycol, Phenoxyethanol, Sodium Benzoate, Methylchloroisothiazoline, Magnesium Chloride, Methylisothiazolinone, Ethylparaben, Propylparaben, Blue 1 Lake (CI 42090), and Sodium Sulfate.

References


