Development of methods to characterize & extract plastic microparticles from personal cleansing products

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INTRODUCTION

Plastics debris is reportedly the largest source of anthropogenic pollution found in the marine environment (Barnes et al. 2009). Plastic microparticles, hereafter referred to as microplastics, are defined as particles 5mm or less in size, and were first considered a minor pollution source derived from degraded plastic litter. However, manufactured microplastics are now found in many personal cleansing products (Fendall and Sewell 2009) and are the focus of this research. Given their prevalence and size, microplastics have become a ubiquitous contaminant, are biologically available to microorganisms and have shown to accumulate in the guts of various organisms (Eriksson and Burton 2003). Although long-term impacts of microplastics are currently unknown, ingestion of microplastics by organisms such as plankton, mussels, worms, fish and sea birds have been widely documented (Cole et al. 2013). Ingestion of plastics by smaller organisms can cause reduced food uptake, internal injury, and possibly death from intestinal blockages or starvation (Derraik 2002). Microplastics can also bind with toxic hydrophobic contaminants such as polychlorinated biphenyls (PCBs) at the water surface, and possibly serve as a vector for organic pollutants to enter aquatic systems (Fendall and Sewall 2009). Moreover, microplastics can externally bind to algae, and inhibit photosynthesis (Bhattacharya et al. 2010). Algae play a key role in aquatic food webs and therefore, high concentrations of microplastics could severely impair those ecosystems (Wright et al. 2013).

Plastics pollution is a well-documented topic in marine ecosystems, though there is a paucity of scientific literature on plastics pollution in freshwater ecosystems (Andrady 2011). The goal of this research was three fold: to measure, quantify, and harvest the microplastics from several brands of face wash and body wash. The data collected from these preliminary observations will form the basis for future research on microplastics pollution; specifically the mode of transfer from waste stream to aquatic ecosystems and their potential impact on the growth of freshwater algae.

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METHODS

Characterization of Microplastics

Microplastics were observed and characterized microscopically and by using an imaging particle analyzer. Before observing under the microscope, about 0.5g of sample was diluted with 25ml of distilled water and homogenized. A Zeiss ® Axioskop 40 research grade digital microscope station5 equipped with MediaCybernetics ® Image Pro Plus ® software version 5.1 was used to capture between one and ten photos of microplastics in each brand. The length of each particle was measured using the Image Pro Plus software. Magnification used ranged from 2.5x to 10x depending on particle size. Dial Powerscrub® microplastics also were photographed after the removal of soap residue.

Particle size distribution and shape characteristics for each product’s microparticles were determined using a FlowCAM Imaging Particle Analyzer6 (by Fluid Imaging Technologies). Prior to analysis, product samples were diluted in hot water, and then clarified of surfactants by alternating cycles of rinsing and harvesting of microplastics until the sample no longer produced suds.

A sample size of 1200 particles was used for all products. In some cases, more than one analysis run was required in order to achieve the desired sample size. A particle size filter was in place to exclude particles <20µm from the analysis in order to eliminate “false” particles associated with bubbles and non-plastics debris; based on the microscopic observations, no particles <30um were observed in any product. Particle size, as Estimated Spherical Diameter (ESD), was calculated for each particle by FlowCAM’s VisualSpreadsheet® Particle Analysis Software. Histograms illustrating particle size distribution for each product were created in VisualSpreadsheet® and data exported to Microsoft Excel.

Quantification and Harvesting

To determine the density of Dial Powerscrub®, the product was turned upside down, clamped, and allowed to drip into a pre-weighed beaker overnight to ensure as much product as possible was represented. In order to isolate microplastics from the soapy material of the product, various efforts were attempted. Universal to all attempts, between four and five grams of sample was diluted with 40ml of water.

The first method applied involved using vacuum filtration. Whatman™ GF/A Filter papers were dried in metal planchets in a 100ºC oven for twelve or more hours and pre-weighed. The diluted sample was poured slowly into the filtration unit and was allowed to filter, using several different vacuum pressures ranging from 5 to 14 psi. The filter paper containing plastics was dried at 100ºC for twelve or more hours and was weighed on a scale to the nearest microgram.

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The second method applied involved using centrifugation. Each brand was placed into the Thermo Scientific™ Sorvall™ Legend™ X1 centrifuge to determine which separated best, Dial Powerscrub® was chosen for the harvesting portion of this experiment. The diluted samples were placed in 50ml centrifuge tubes and centrifuged for 15 minutes at 9500 x g after several trials using less gravity and less time which were less successful. As much supernatant that could be removed without disturbing the microplastics was decanted precisely using a 10ml pipet; any remaining material from the sample was added to the centrifuge tubes. This process was repeated several times to guarantee the removal of soapy materials from the microplastics. All water decanted in this method was later analyzed for lost plastic particles which, if any, were recovered and accounted for. When all soapy materials were no longer detectable by shaking with a Thermolyne Scientific™ MaxiMix II, the microplastics were decanted into pre-weighed planchets containing aluminum foil using a 10ml pipet. Aluminum foil was used because it helped reduce hydrostatic interactions that made the microplastics affix to edges of materials. The planchets were dried in an oven; any remaining material in the centrifuge tubes was added to the dry planchets. The planchets were then dried in an 100°C oven for twelve or more hours and weighed to the nearest microgram the next day.

RESULTS AND DISCUSSION

Characterization

Use of the centrifuge to extract microplastics for imaging analysis was attempted but ultimately was deemed unsuccessful; microplastics were distorted and compacted, thus measured characteristics would not be representative of the product’s contents. The results presented here were acquired using the diluted samples viewed microscopically (for particle characterization) and those processed with the Flowcam® particle analyzer (for size distribution).

Figures 1 and 2 include examples of microplastics viewed microscopically. Data of the distribution and identity of particles in each product sampled is shown in Table 1.

Figure 1. Two microplastic particles from Dial Powerscrub© viewed at 5x magnification.
Table 1. Minimum and maximum particle sizes and materials detected

<table>
<thead>
<tr>
<th>Product</th>
<th>Minimum size detected (µm)</th>
<th>Maximum size detected (µm)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>St. Ives ®</td>
<td>~60</td>
<td>~988</td>
<td>Walnut shell powder</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Corn kernel meal</td>
</tr>
<tr>
<td>Softsoap ®</td>
<td>~20</td>
<td>~981</td>
<td>Acrylates</td>
</tr>
<tr>
<td>Olay ®</td>
<td>~20</td>
<td>~1003</td>
<td>Acrylates</td>
</tr>
<tr>
<td>Neutrogena ®</td>
<td>~20</td>
<td>~995</td>
<td>Acrylates</td>
</tr>
<tr>
<td>Dial Powerscrub ®</td>
<td>~60</td>
<td>~1010</td>
<td>Acrylates</td>
</tr>
<tr>
<td>Clean and Clear®</td>
<td>~60</td>
<td>~948</td>
<td>Acrylates</td>
</tr>
</tbody>
</table>

Every product had variability in the size of its particles, with a minimum range of almost 900 microns (µm). The largest particles were found in Dial© Powerscrub and the smallest were found among Softsoap®, Olay® and Neutrogena® Deep Clean™. Each product used a form of acrylates as their abrasives except St. Ives®, which used natural alternatives such as walnut shell powder and apricot extracts. Figure 3 indicates the particle size distribution within random samples of each product.
Common to each product except St. Ives®, the most frequent particle sizes ranged from 20-150 microns. The frequency of particles of larger sizes began to taper off after about 100 microns. The Flowcam reported that particles less than 30 microns in length were present. However, no particles smaller than 30 microns in length were observed microscopically. This apparently is an artifact of the Flowcam system. Overall, St. Ives contained the largest average particles, though these particles are non-plastic, natural particles.

**Harvesting and Quantification**

The centrifugation method was superior to the filtration method, which sometimes led to large loss of plastic by the particles lodging in filter paper pores. A total of 3.2483 grams of microplastics were harvested from about 450ml of Dial Powerscrub®. Figure 4 displays the frequency of weight of microplastics in 4-5g samples of Dial Powerscrub®.
The mean concentration of plastics per sample was $8.12 \pm 1.21 \text{mg microplastic/g}$ of Dial Powerscrub®. The average shower uses about 70 gallons of water (WSSC 2014); if the average person uses about 4.7g of Dial Powerscrub about 0.116mg of microplastic is used per gallon of water. If a person uses the product every day for a year about 2.96g of microplastic is used. If person in the United States used the product daily for a year over 945 metric tons of microplastic would be used. This does not account for more than one product being used per shower, or people who do not use products containing microplastics, but illustrates the potential for large quantities of plastic debris to enter the waste stream. They must be removed or dealt with in the environment.

**CONCLUSION**

It is worth noting that wastewater from two wastewater treatment plant was examined for microplastics. While none were found in the effluent examined further study on this subject is needed. More work that can be conducted includes experimentation with methods to degrade microplastics, such as heat and UV radiation in hopes to suggest microplastic eradication treatments at wastewater treatment facilities.

**REFERENCES**


Barnes, D.K.A., Galgani, F., Thompson, R.C., and Barlaz, M. 2009. Accumulation and


