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RESEARCH PAPER

Release of engineered nanomaterials from personal care products throughout their life cycle

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Abstract The impetus for this study was to provide release estimates that can serve to improve predictions of engineered nanomaterial (ENM) exposure for risk assessment. We determined the likely release of ENMs from personal care products (PCPs) through a consumer survey on use and disposal habits, and research on the types and quantities of ENMs in PCPs. Our estimates show that in the US zinc oxide (ZnO), with 1,800–2,100 mt yr^{-1}, and titanium dioxide (TiO_2), with 870–1,000 mt yr^{-1}, represent 94 % of ENMs released into the environment or landfills from the use of PCPs. Around 36–43 % of ENMs from PCPs were estimated to end up in landfills, 24–36 % released to soils, 0.7–0.8 % to air, and 28–32 % to water bodies. ENMs in sunscreen represent around 81–82 % of total release, from ZnO and TiO_2 as UV blockers, followed by facial moisturizer (7.5 %), foundation (5.7 %), and hair coloring products (3.1 %). Daily care products such as body wash, shampoo, and conditioner had by far the highest per capita and total use, but contributed little to the ENM release estimates as these products generally contain little or no ENMs. However, if ENMs are incorporated into these daily care products, this may substantially increase ENM release.

Keywords Sunscreen · Cosmetics · Zinc oxide · Titanium dioxide · Wastewater · Landfill

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Introduction

Personal care products (PCPs) currently represent one of the most significant applications of engineered nanomaterials (ENMs) in terms of human exposure and environmental implications (Keller et al. 2013; Keller and Lazareva 2014). In a 2011 database, 33.6 % of the 1,317 nanotechnology-based consumer products were in the cosmetic, personal care, or sunscreen categories (Rejeski 2011). The potential for exposure and the consequences to human health from ENMs in PCPs are not well quantified (Nazarenko et al. 2012). In addition to direct human exposure, the ENMs in these products can rapidly transport to the environment after single use and disposal (Gottschalk and Nowack 2011; Nowack and Bucheli 2007), where they may pose toxicity risks to organisms in water and soils (Gottschalk et al. 2009, 2013).

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The 2011 cosmetic and personal care industry worldwide market was estimated at $375 billion, and the market for cosmetic and PCPs containing nanomaterials was estimated at $17 billion (Future Markets 2012). The number of patents for nanobased cosmetics and PCPs increased fourfold from 2003 to 2010 (Future Markets 2012). In this study, PCPs encompass sunscreens, daily use products (toothpaste, shampoo, conditioner, body wash, facial moisturizer, face cream, body lotion, and serum), color cosmetics (foundation, concealer, blush, mascara, eye shadow, eyeliner, lip gloss, lipstick, lip liner, and nail polish), and hair coloring products. We used the common definition of an ENM as being an engineered material that has at least one dimension <100 nm.

Over 90 % of ENMs in PCPs are used in skin care and sunscreen for UV protection, "anti-aging" formulas, and as delivery vehicles for vitamins and other supplements (Future Markets 2012). An additional 3 % of ENMs are found in color cosmetics, while the rest are found in grooming, hair care, fragrances, and shower products. ENMs in these products enhance the stability of vitamins and penetration into the skin (e.g., in anti-aging creams), block UV light from reaching the skin (e.g., in SPF formulations), and make the product more esthetically appealing since they can be transparent (Mu and Sprando 2010). Common ENMs in cosmetics are TiO_2, ZnO, Ag, and SiO_2 (Bangale et al. 2012; Benn and Westerhoff 2008; Benn et al. 2010, 2011; Kaur and Agrawal 2007; McIntyre 2012; Yang et al. 2009).

Only a few studies have explicitly considered the contribution of PCPs to ENM release to the environment (Boxall et al. 2007; Gottschalk et al. 2010; Keller and Lazareva 2014; Keller et al. 2013). Boxall al. (2007) predicted concentrations of various ENMs from PCPs in soil, sludge, and water, while Keller et al. (2013) modeled total global ENM emissions from PCPs as well as emissions per ENM from PCPs. Gottschalk et al. (2010) modeled the fate of nanoTiO_2 and Ag emitted from cosmetics into wastewater treatment plants (WWTPs), waste incineration plants (WIPs), and water bodies. However, great uncertainty exists in estimates of ENM exposure from PCPs to specific environmental compartments (e.g., soil, water, air, landfill) because little information is available on the amount of ENMs in each product (Nazarenko et al. 2012) and ENM release during each life cycle stage—especially the use and disposal stages. Although several studies have surveyed how often consumers apply PCPs (Loretz et al. 2005, 2006, 2008; Hall et al. 2007, 2011; Manová et al. 2013; Biesterbos et al. 2013; Wu et al. 2010; McNamara et al. 2007; Pauwels et al. 2009), there is little or no information on how consumers remove or dispose of PCPs. Studies that model ENM exposure often use rough estimates for the disposal calculations that are not described in detail. For the amount of ENMs in each product, previous studies have used estimates of ENM concentration reported in a single personal care product formulation patent that likely does not represent the market as a whole (Boxall et al. 2007). The absence of reliable disposal and ENM concentration in personal care product data has been a hindrance to making more precise end-of-life estimates of ENM exposure to the environment and humans.

Our goal for this study was to refine the estimates of ENM release from PCPs to soils, water, air, and landfills in the USA We accomplished this by surveying consumers of PCPs about their purchasing, use, and disposal habits, and then combined survey results with research on container sizes of each PCP and amount of ENMs in each PCP. We then estimated the amount of ENMs emitted from PCPs per year into the soil, water, air, and landfills. The study also provided useful results on the flow of PCPs through various life cycle stages, which can be used to estimate releases of other PCP components into the environment.

Methods

Consumer survey

We employed a survey to better understand how consumers purchase, use, and dispose of PCPs. The survey was broken into four main sections: sunscreens, daily use products, cosmetics, and demographics. We asked questions regarding frequency of use (e.g., 5 days per week, once a month), and frequency of application for days where the PCP was applied. This approach filled in gaps from previous use studies that likely underestimated overall use by asking use in a single question that would typically be in a range of "several times per day, every day, 1 day per week," (Biesterbos et al. 2013; Manová et al. 2013). Frequency of product purchase was queried to have an additional method of determining use. To determine how consumers dispose of applied PCPs, they were given five choices: Explicit PCP removal by washing it off, removal using a disposable item (as is often done with facial cosmetics, in which case the ENMs go to landfill), did not remove the product (we assumed eventually washed off), used a non-disposable item (e.g., towel that would be washed), or "other" removal method.

To determine the amount of product left inside the container when discarded, we asked the respondents to estimate this on a scale of 0–100 %, at intervals of 10 %. This was an important question because a consumer will often not use the entire product before throwing away the container, due to having difficulty removing all the product, expiration date, or out of fashion. For sunscreens, we asked a specific question about how often the respondent went swimming in a water body, such as a river, lake, or ocean, after applying sunscreen. The demographics section asked for the respondent's gender, age, country, zip code, and approximate household income. The survey was conducted in the USA and in China. The full survey and details on its administration are provided in the Supporting Information (SI).

Estimating PCP use and disposal per capita

While the survey provided the cumulative distribution function (CDF) of per capita purchases, we determined it would be difficult for respondents to provide the typical size of each PCP container, or the amount of PCP used each time they applied it. Although market study information was considered, it is only available in financial terms (sales per product category), which cannot be easily translated to mass of PCP sold since different prices may be considered at the retail level for the same product in the same container size. To address this, we used two approaches. The first used peer-review data on amount applied for 8 types of PCPs (Loretz et al. 2005, 2006, 2008). To fill in the gap for the other products, we used data from two major US retailers to determine the most common container sizes (product content) for each PCP (additional details in SI). CDFs for product content were generated for each PCP type (Figures S5 and S6 in SI). CDFs from the survey data (purchase of a given PCP per year, amount left at disposal) and for container size were sampled via Monte Carlo simulation (10,000 runs) to obtain CDFs of per capita annual amount of PCP purchased, applied, and disposed. For the 8 PCPs with peer-reviewed studies, we compared estimates of PCP applied per capita from previous studies (Loretz et al. 2005, 2006; 2008) with those from the survey and market data.

A range of values for US consumption of each PCP was estimated using the CDF of per capita use, the US population by gender from US Census data, and the corresponding age group and fraction of each gender that uses the PCP (Figure S1 in Supporting Information, SI). To estimate the fraction of PCP disposed to WWTP or landfill, survey results (Table S1 for the USA and S2 for China) were multiplied by the PCP consumption data, accounting also for the amount of PCP left in the container when it is disposed to landfill (Table S3). Respondents also provided information on the frequency with which they swam in water bodies after application. We considered that 25 % of the amount applied was released to a water body based on an estimate of sunscreen lost when the body is exposed to water (Danovaro et al. 2008). The balance was considered removed via washing.

When extrapolating our survey results to the entire population, we assumed that age or geographic location differences were not significant to overall results since a number of previous studies on PCP use found no statistically significant differences between age groups and product use (Biesterbos et al. 2013; Loretz et al. 2006) and product use across geographic regions in the USA (Loretz et al. 2005, 2008). However, a couple of studies found statistically significant differences between age groups and hair conditioner use (Loretz et al. 2008) and geographic region and use of body wash and shampoo (Loretz et al. 2006).

ENM exposure estimates

To determine the concentration of ENMs in each PCP and the percentage of PCP on the market that contain ENMs, research of the scientific literature, market studies, and personal care product websites was conducted. Information in the scientific literature on PCPs that contain ENMs is scarce. However, on July 11, 2013, European Union (EU) cosmetics regulation 1223/2009 was implemented that requires a [nano] bracket in front of each ingredient on a product's labeling (http://www.nanotoes.eu/nanosafety-regulation/labeling-nano-products-mega-problem.html). Online stores in the EU began updating their product information indicating nanosized ingredients in the label; this greatly increased our knowledge of cosmetic products that contained nanomaterials. For example, silica dimethyl silylate (SDS) had not previously been identified in release or exposure studies as a nanosized ingredient in cosmetic products such as lip gloss and hair coloring products. We assumed that the content of ENMs in the same products sold in the USA would be equivalent to those in the EU. Table S7 presents the ENM content in the PCPs considered in this study, including type and concentration of ENMs as well as the reported fraction of the material that is nano versus bulk.

Per capita information, ingredient (nano and bulk) concentration, and percent of ingredient that is ENM (Table S2) were used to generate ENM exposure estimates for the US population for each type of ENM, based on:

$$\begin{aligned} &\text{U.S. ENM exposure per product} = \\ &\quad \text{Population Using PCP} * \text{Per Capita Use} \\ &\quad * \text{Fraction of product on the market that contains ENM} \\ &\quad * \text{Ingredient Concentration} \\ &\quad * \text{Fraction of ingredient that } \textit{is} \text{ 100 nm or less.} \end{aligned}$$

ENMs were routed to WWTP, landfill, or a waterbody based on the fate of the PCP, using the survey data. If the ENM passed through WWTP, its fate (fraction to effluent and biosolids, fate of biosolids) was determined based on previous studies (Keller and Lazareva 2014), with the transfer factors summarized in Table S8.

It is possible our methods underestimate the amount of products that contain ENMs, since only brands with stores in the EU with updated ingredient lists were considered. For certain PCPs, no data were available. In these instances, values similar to other products were used, or if it was difficult to ascertain the market share of a product, a value of 0.1 % was assumed. This value was used because the lowest brand market share in the market study used was 0.1 %.

Results

Survey results

The US survey had 491 respondents that provided useful data, 80 % female and 20 % male. 74 % of respondents were between the ages of 18–34, 16 % of respondents were 35–54, and 10 % of respondents were 55 and older. Respondents were overwhelmingly from the USA (94 %), and a majority from California (58 %). The survey conducted explicitly in China received 439 responses. Additional information on both surveys is presented in the SI.

PCP per capita use and disposal

There are several orders of magnitude difference in the amount of PCP used in the USA for the 21 products considered (Fig. 1). Daily use products have the highest application amount per year per capita, followed by hair colorant, then moisturizing and sun

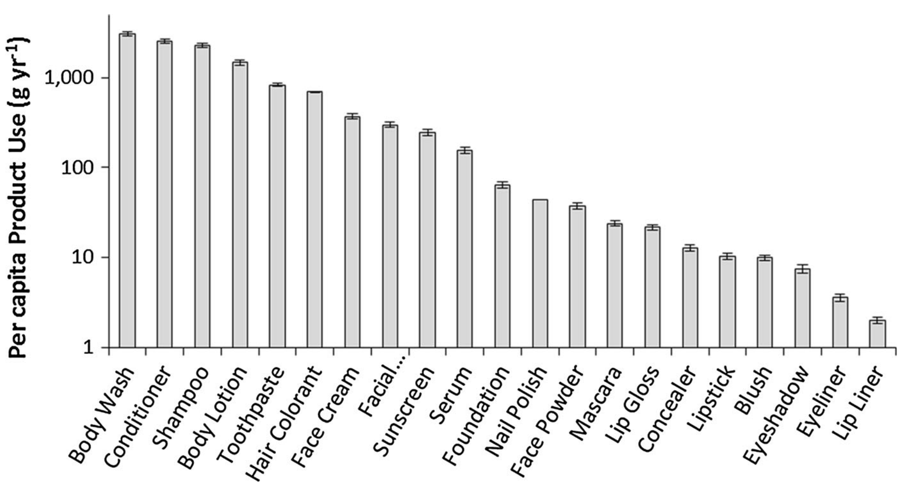


Fig. 1 Per capita use of 21 types of personal care products in the USA, based on 2013 survey data ($n = 495$). *Error bars* represent tenth and ninetieth percentile

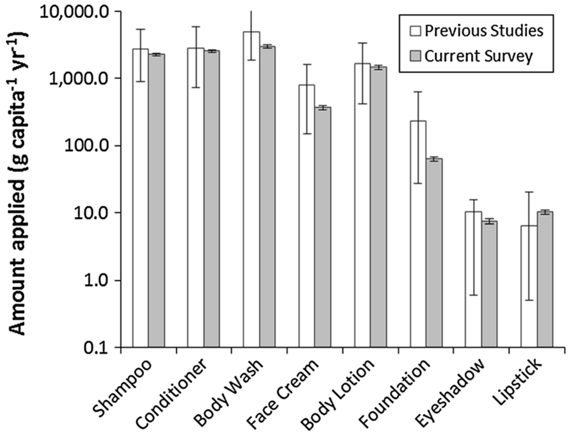


Fig. 2 Comparison of amount applied per capita (g yr^{-1}) between previous studies (Loretz et al. 2005, 2006, 2008) and the current US survey. *Error bars* represent tenth and ninetieth percentiles

protection creams, and at a much smaller scale cosmetics. A comparison of per capita annual application between survey results and previous studies on 8 PCPs (Fig. 2) indicated that the mean amount applied was quite similar for 7 of the 8 PCPs, except face cream. However, there was considerable more variance in previous studies. While the survey conducted in China provided data on frequency of PCP use (Fig. S2), per capita use in China could not be estimated due to incomplete information on the range of PCP container sizes sold in China.

In the USA nearly all sunscreen, daily use PCPs, and hair coloring products, are disposed to a WWTP after application (Fig. 3a), via direct wash or laundry wash of a towel used to remove the product. In contrast, in the USA, 25–41 % of color cosmetic products are disposed to landfills after application. A significant fraction of respondents indicated using disposable makeup removers for color cosmetics, while many respondents did not consciously wash off sunscreens and creams; we assumed these products are eventually washed off. When nail polish was not removed using a disposable item, it likely chipped off in the environment or in a household; its fate is undetermined. Swimming in a water body directly after applying sunscreen represents only a small fraction (5 %) of the release. In contrast to the USA, Chinese respondents indicated a much higher use of disposable wipes for their cosmetics, sunscreens, and skin care products (Fig. 3b). The most striking example is with sunscreens, where only 3 % of US respondents used a disposable wipe, while 34 % of Chinese respondents reported using disposable wipes to remove the product. Chinese tend to have a disposable wipe with them at all times and use it frequently throughout the day. Thus, it is important to consider cultural differences when making estimates of ENM release.

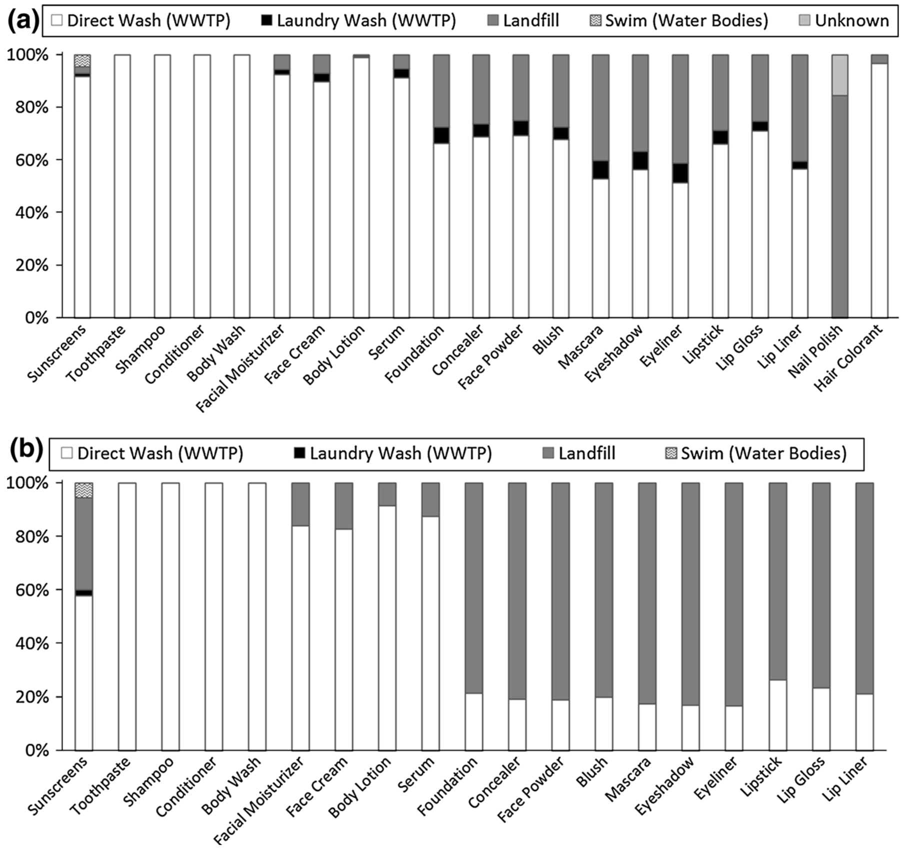
PCP life cycle material flows through the US economy

Total PCP material flows for the USA were estimated from the per capita information, considering the US population and differences in use based on gender. Table S9 presents the range of production estimates for all 21 PCPs, in metric tons per year (mt yr^{-1}). Figure 4 provides a graphical representation of the material flows for these PCPs from production and use, through intermediate processing (WWTP and WIP), to landfill or environmental releases, considering emissions from WIP to air and WWTP to water bodies (via effluent) or soils (via biosolids applied to land). Daily use products represent the majority of the material flows, followed by sunscreen, hair colorant, and other color cosmetics, which combined are <5 % of the material flow. More than 96–98 % of the PCP material flows pass through the WWTPs, while only ~2–4 % of the PCPs go directly to landfill after use, mostly from skin care and color cosmetics. Flows to landfill also include product left in the container that is disposed of by the consumer (Table S3). Figure 4 presents the high effluent emission estimates from a WWTP, i.e., when a higher fraction of the PCPs are disposed to the sewer system.

ENMs in PCPs life cycle material flows through the US economy

While a number of PCPs contain ENMs, the most ENM intensive application is sunscreen, with 81–82 % of the total ENM mass flow (Fig. 5). Table S7 presents a compilation of ENM use in the various PCPs, including ENM concentration. ZnO and TiO_2 are the most commonly used ENMs in PCPs, found in sunscreens, facial moisturizer, and foundation. Together these two ENMs represent ~94 % of ENM use in PCPs. SDS is used in several PCPs, most notably in hair coloring products, which rank fourth in ENM use in PCPs at

Fig. 3 PCP disposal methods in a the USA; and b China. Note that nail polish and hair colorant were not included in the survey in China



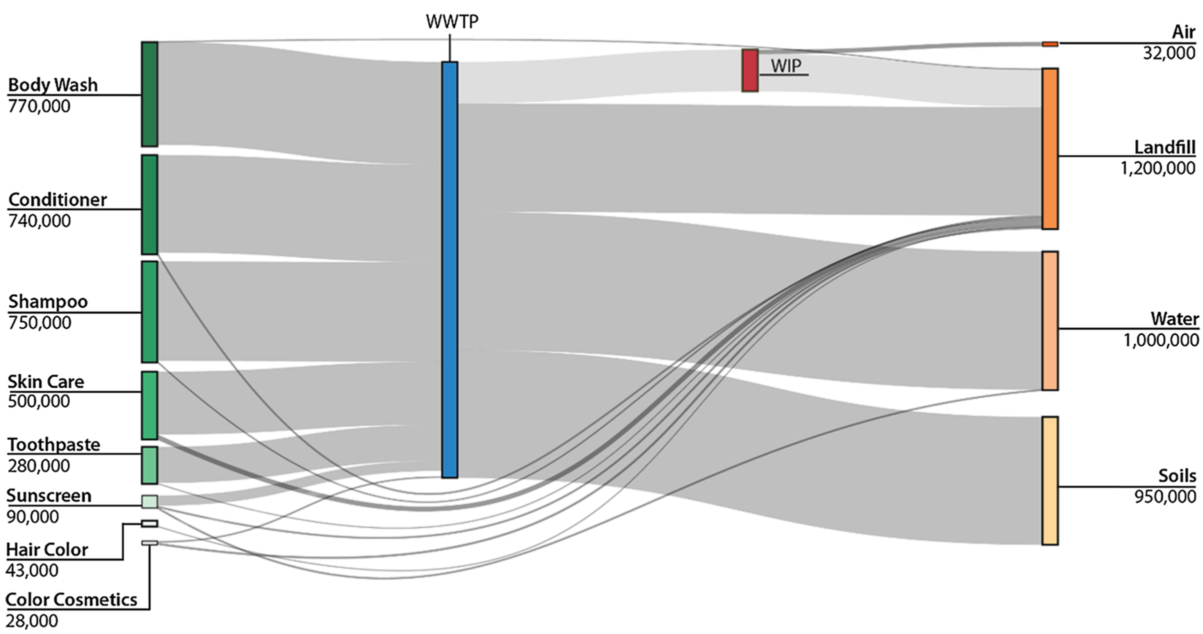


Fig. 4 US material flows of 21 PCPs in 2013 (mt yr^{-1}), considering use estimates derived from the survey and high WWTP effluent emission estimates

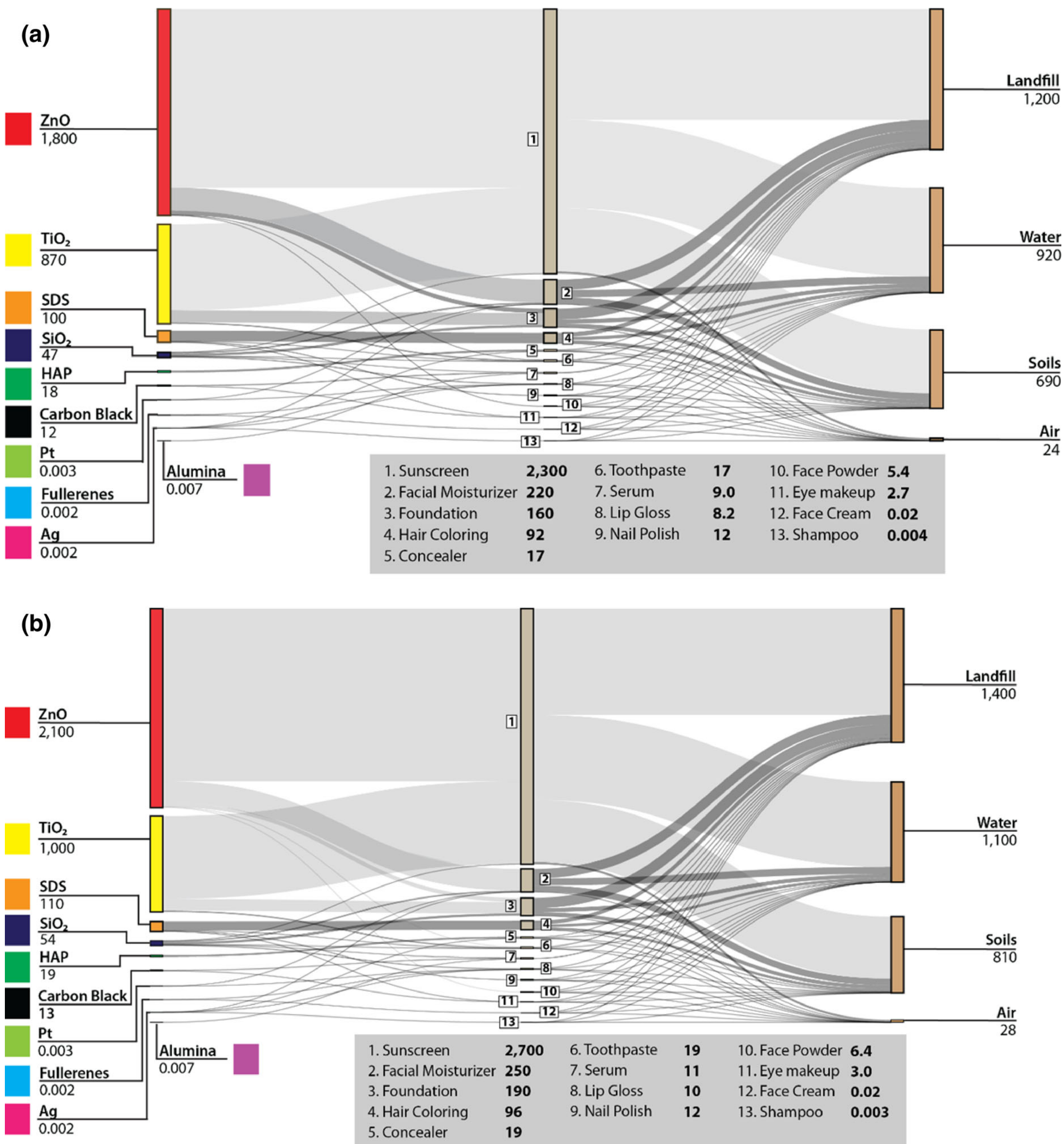


Fig. 5 US material flow of ENMs used in PCPs in 2013 (mt yr^{-1}): **a** low estimates; and **b** high estimates, considering use estimates derived from the survey and high WWTP effluent emission estimates. *SDS* silica dimethyl silylate and *HAP* hydroxyapatite

3 %. Silica (2 %) is used in several PCPs, while nanoscale hydroxyapatite (HAP) is mostly used in toothpaste (Tschoppe et al. 2011; Poggio et al. 2013). Carbon black (0.4 % of ENM mass flows in PCPs) is used mostly in nail polish and eye makeup. Other ENMs (e.g., platinum, fullerenes, silver, and alumina) are used in very minor amounts in PCPs. In terms of end-of-life fate, around 36–43 % of these ENMs will end up in landfills, either from direct disposal or from sludges collected from WWTP that are landfilled. Around 920–1,100 mt yr^{-1} of ENMs (28–32 % of total ENM mass flows) are likely to be discharged into

water bodies, with the vast majority passing through WWTPs. Given the high solubility of ZnO (Fairbairn et al. 2011; Li et al. 2011a; Bian et al. 2011; Meulenkamp 1998) and the residence time (hours) in WWTP processes (Ma et al. 2014; Musee et al. 2014), one would expect most of the Zn to be either as Zn^{2+}, or transformed to ZnS (Lowry et al. 2012) and zinc phosphate compounds (Li et al. 2011b; Lv et al. 2012). TiO_2 ENMs are expected to be agglomerated by the time they are emitted from the WWTP due to the very high ionic strength of this medium (Brar et al. 2010; Westerhoff et al. 2011; Kiser et al. 2009; Limbach et al. 2008). Air emissions are estimated at 0.7–0.8 % and reflect emissions from the production phase of the ENMs and WIP. The low effluent emission scenario (Fig. 5a) considers more ENMs in sludge, which leads to more ENMs released to soils and air, while the high effluent emission scenario (Fig. 5b) has less ENMs in sludge and thus more ENMs being released into the water bodies via treated WWTP effluent.

Discussion and conclusions

We estimated the release of ENMs from PCPs throughout their life cycle, considering sunscreens, daily use products, cosmetics, and hair coloring products. The results provide a detailed understanding of the use and disposal habits of consumers who use these products, and the fate of ENMs in those products. ENMs in PCPs have a different fate than ENMs in other consumer products. A previous study found that 63–91 % of the ten most significant types of ENMs in over a dozen ENM applications were discarded in landfills (Keller et al. 2013). Here, we estimated that in the USA, only 36–43 % of ENMs from PCPs end up in landfills, with the remainder passing through WWTPs and eventually to the air, water, or soils. This difference is explained by the higher disposal of PCPs in the sink or shower, with 96–98 % of the PCP material flows in the USA passing through WWTPs. Our results also provide data on the amount of PCPs used per year per person, which are useful for future exposure studies.

While it is quite certain that most ENMs from PCPs will be transported to WWTPs, their fate in these facilities is not well understood. While a number of studies have begun elucidating the fate of ENMs in WWTPs, there is data for only a few ENMs (Kiser et al. 2009; Westerhoff et al. 2011; Jarvie et al. 2009; Shafer et al. 1998; Kaegi et al. 2011; Brar et al. 2010; Ma et al. 2014; Musee et al. 2014). In addition, coatings and other ENM characteristics may be quite different in various PCPs, which results in a high uncertainty in the estimates of ENM removal to biosolids or transfer to treated effluent water.

Although previous studies (Keller and Lazareva 2014; Piccinno et al. 2012; Tovar-Sánchez et al. 2013) considered TiO_2 to be the most commonly used ENM from PCPs, this study showed ZnO may in fact be more commonly found in these products. Our evaluation of over 50 sunscreen formulations sold in the US containing ENMs indicated ZnO was more commonly used and at higher concentrations than TiO_2. Some studies indicate ZnO is a better UV blocker because it is more transparent and covers a broader UVA spectrum than TiO_2 (Morabito et al. 2011). However, others question its effectiveness as a photoblocker (Lewicka et al. 2013), and there are also some health concerns (Osmond-McLeod et al. 2013). Nevertheless, there has been a steady and significant increase in the use of ZnO in sunscreens since its approval in 1997 (Wang et al. 2013).

While the current use of ENMs in daily care products (shampoo, conditioner, body wash, and body lotion) is very low, the PCP material flows indicate that these are very significant overall. If these products are modified to contain photoblockers (e.g., TiO_2 and ZnO), antimicrobials (e.g., Ag), or hair regrowth enhancers (fullerenes) (Boxall et al. 2007; Future Markets 2012), there could be significant increase in the release of ENMs to the environment.

A possible caveat to our estimates of sunscreen product used per person and ENM exposure to the environment and landfills is that the majority of our respondents were from California. With warm and sunny weather year round, it is likely that respondents use more sunscreen than other areas of the country with colder and stormier winters. One study based in California found no significant difference between use of sunscreen in the summer and winter (Wu et al. 2010), but that assumption cannot be extrapolated to the rest of the USA

The lack of information on the amount and type of ENMs in PCPs presents limitations to the accuracy of our study. Requiring manufactures to report the type and amount of ENMs in their products would allow for more accurate estimates of exposure of PCPs to humans and the environment throughout their life cycle. The EU cosmetic regulation requiring [nano] to be listed next to ENM ingredients significantly improved our estimates; similar regulations around the world would be extremely valuable. Further development of technology and best practices in measuring ENMs in the environment will help validate release estimates.

Supporting information available

Detailed explanation of data used to estimate PCP and ENM estimated releases, including 9 tables and 6 figures.

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