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Editorial

Balancing New Approaches and Harmonized Techniques in Nano- and Microplastics Research

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Plastics, including microplastics (MPs) and nanoplastics (NPs), along with their associated chemicals, are being found in virtually every environmental compartment and location sampled to date, and their abundance is increasing annually.[1] In response, numerous jurisdictions and the global community are taking steps to stem ubiquitous plastic pollution.[2] These initiatives necessarily rely on sound data from which sources, temporal and geographic trends in abundance, and effects can be confidently discerned. However, plastics pose unique challenges relative to other forms of chemical pollution. Environmental plastics are comprised of a complex mixture of different polymers, sizes, shapes, surface functionalities, additive compositions, and degrees of weathering.[3] This complexity must be adequately captured to tackle issues ranging from source identification to understanding food web transfer and toxicity. However, this complexity defies reliance on existing methods of sampling and characterization that for other pollutants took decades to develop and validate. As such, there is a pressing need for well-validated methods that underpin building baseline environmental data and protocols for evaluating impacts. In response to this challenge, the complex and multifaceted topic of plastics pollution attracts scientists from diverse disciplines, ranging from analytical chemists to environmental scientists to human toxicologists and eco-toxicologists (to name a few).

This virtual issue draws together diverse studies unified by the goal of advancing harmonized methods for studying MPs and NPs. By harmonization, we refer to reducing variations between methods so that the results obtained can be quantitatively compared. In contrast, standardization refers to a set protocol or procedure and thus eliminating differences in practices. The latter is unlikely to be achieved in a timely fashion. However, the MPs and NPs community can strive toward using harmonized methods that allow for comparability. Along the road toward harmonization, studies are developing procedures for robust, quantitative, and nontargeted characterization and analysis of MPs in all environmental compartments as well as testing and probing impacts of MPs and NPs on biota. We anticipate that this virtual issue and other ensuing studies will lead to the availability and adoption of best practices and ultimately harmonization to enable us to confidently benchmark MPs and NPs pollution globally and to understand its environmental fate and impacts.

The papers presented here contribute to the development of harmonized test materials, analytical approaches, and experimental protocols for environmentally relevant complex mixtures of MPs and NPs. As with any new field of research, there is initially a diversity of approaches on how scientists define and tackle research questions and hypotheses and develop analytical, modeling, or experimental methods to understand environmental or health impacts of a pollutant. What is the purpose of this harmonization? Comparability of results is most often cited as the key reason for harmonization, and with good reason. Having readily available reference materials would allow for calibration of instrumentation and test methods. Consequently, harmonization of analytical equipment is beneficial to achieve a consensus of analytical detection limits and working parameters of different approaches. Harmonized best practices for sample collection, handling, and data processing ensure that results reported are unbiased and avoid analytical artifacts. In addition, harmonized methods can streamline the entire pipeline of characterization and quantification MPs (and eventually NPs) while having the added benefit of bringing new practitioners in the field up to speed faster (i.e., no one needs to reinvent the wheel). In summary, harmonization can help to understand and contextualize results across the field and would decrease uncertainty.

While it is important to strive for harmonized methods, it is also important to recognize that methods used must be tailored to the research question being asked. In essence, exploration at this stage is welcome and needed in terms of analytics, experimental design, modeling approaches, and exposure studies to address unique and targeted research questions. In addition, efforts should continue to improve methods by, for example, characterizing associated uncertainties and potential biases. As the field matures, so too do expectations for the use and clear explanation of quality assurance/quality control steps followed such as reporting limits of detection[4] and using environmentally relevant MPs for toxicity testing. Full descriptions and evidence of evaluation of sampling, sample preparation, characterization of MPs and NPs, and testing should be a routine part of all studies.[5] Ultimately, there is a balance between ever-evolving techniques and harmonization, where on one hand reaching for new techniques and exploration allows for new developments where harmonization allows for comparisons. Collectively, those working in plastics

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pollution research strive to achieve best practices in MPs and NPs research, which balances new approaches and harmonized techniques across the field.

In this virtual issue, we highlight interesting developments considering the dichotomy of research featuring new insights and fit-for-purpose techniques with harmonized approaches leading toward best practices, including publications on (1) analytical method development, (2) creating harmonized test materials, (3) experimental design, sampling, and data processing, and (4) how MPs impact technical and environmental systems.

Critical reviews of existing analytical methods and recommendations on paths forward are an easy entry pathway for those new to the field to appreciate the current state, challenges, and research gaps in the field of analysis of MPs and NPs, including focusing on matrix specific considerations of MPs in water, as summarized by Elkhaltib et al.[6] and Delgado-Gallardo et al.,[7] and soils, as assessed by Moeller et al.[8] Assessing ISO technical specifications for pyrolysis GC-MS for quantification of tire road wear particles, Rauert et al. found high variability in synthetic rubber and additive compositions across manufacturers.[9] Consequently, the authors found that using ISO methods had the potential to underreport environmental concentrations, and the authors suggested that further refinement of harmonization of analysis is needed.

New methods for the identification of MPs are continuously being suggested, as exemplified by Primke et al., who explored the rapid and reliable method for determination of the size, shape, and number of MPs by quantum cascade laser-based hyperspectral infrared chemical imaging.[10] Comparing the performance of this technique to that of the existing state-of-the-art Fourier transform infrared microscopy analysis in a number of different environmental samples, the authors suggest this approach as a viable path forward as a rapid MPs monitoring approach with a highly detailed data set. To reduce time demands on the imaging and characterization of MPs, Hufnagl et al. developed automating data analysis following spectroscopic measurements with machine learning tools.[11] In some laboratory-based studies, the challenges of analysis of NPs and MPs were circumvented by using a conservative tracer embedded in the polymer, such as a trace metal, to more easily quantify and understand the fate and transport by using the metal as a proxy for plastics. For example, Keller et al. assessed the transport of particles of NPs and fibers of MPs through unsaturated porous media[12] and Heinze et al. examined the transport of NPs in soil via bioturbation by *Lumbricicus terrestis*[13] using this approach, which allowed the authors to track smaller particles with greater speed and accuracy.

Understanding how macroplastics degrade into MPs and NPs can help in the generation of test materials that can be used across multiple studies, as reviewed by Bacha et al.[14] In this context, Pfohl et al. adapted a NanoRelease protocol using industry-relevant polymers to develop a parametrized mechanistic fragmentation model using varying ultraviolet doses and cryo-milling to measure the rates of fragmentation.[15] A number of studies addressed confounding factors for experimental design and artifacts when conducting ecotoxicity tests. For example, Hermsen et al. critically reviewed the quality criteria for MPs in biological samples, where the authors noted the variability and inability to directly compare results between studies may be due to the lack of harmonized methods.[16] Subsequently, the authors suggest 10 criteria to benchmark future studies in an effort to harmonize best practices in the future. Confounding factors in toxicity tests were assessed by Petersen et al., especially in relation to artifacts of dosing of NPs and MPs and the lack of control experiments.[17] Issues related to preservatives in commercial formulations were also highlighted by Pikuda et al., who specifically noted the acute toxicity of nondialyzed MPs to organisms, and consequently, toxicity was mainly associated with sodium azide, a preservative in the stock suspension of nanoplastics, and not the particles themselves.[18] However, in some cases, chemicals in MPs can play a role in toxicity, as noted by Kim et al.[19] Here, the authors found that adverse effects were mainly attributable to the extractable additives, and when these additives were extracted before organism exposure, the acute toxicity of test materials significantly decreased.

Other papers highlighted in this virtual special issue assess experimental design to facilitate comparison of results across different studies. For example, Kooi and Koelmans note that many studies struggle with the diversity of MPs in environmental samples and current classification approaches do not capture the essentially continuous and diverse nature of environmental MPs.[20] Consequently, the authors created a three-dimensional probability distribution, and although this is a simplified concept, it could be especially helpful in probabilistic risk modeling. To better align methods used in MPs research for previously incomparable data sets with respect to MPs exposure assessments, Koelmans et al. suggest an approach to correct for differences in particle size, number, volume, and mass in different exposure tests to account for different types of MPs used to create a species sensitivity distribution.[21] Likewise, sample heterogeneity of MPs in environmental samples can also impact sampling and analysis efforts. Bai et al. critically analyzed sampling methods with the goal of developing harmonized methods with a specific focus on capturing the variable spatiotemporal riverine fluxes of MPs,[22] and Kittner et al. conducted a comprehensive screening of MPs in the Danube River basin with a harmonized analytical approach.[23] Morgado et al. assessed and modeled the random and systematic effects affecting the quantification of MPs using Poisson-log-normal distributions and the uncertainty from particle counting and provided a user-friendly spreadsheet to facilitate harmonization between research groups.[24]

Finally, it is evident that the diversity of MPs, NPs, and plastics-added chemicals combined with the complexity of environmental test systems has resulted in conclusions that differ from study to study. Above, we have outlined several attempts at harmonization in the field, but one which is still large lacking is test materials that can be compared among laboratory groups. Currently, most laboratories are producing their own MPs and NPs in house, but harmonization would also benefit from a larger repository of well-characterized standards for analytical calibration and environmental fate and biological uptake comparisons. Another issue that requires further attention is the impact of various processes on the physiochemical surface characteristics of MPs and NPs, which can impact both their environmental transport and interactions with biological organisms. Plastics can also have indirect impacts on technical and environmental systems,[25−27] which is another area that should be explored in more depth in the future, as well. It is worth noting that analytical and experimental harmonization for other particulate pollutants, such as environmental health and safety assessments of engineered nanomaterials, took many years to develop and

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in some regards is still ongoing. Nevertheless, there are many lessons learned that could be incorporated into MPs and NPs research, including, for example, designing environmentally relevant test materials and exposure systems.[28] In the coming years, we look forward to seeing how harmonization progresses in this field of research to enable best practices that support both academic research and eventually more widespread monitoring and testing of MPs and NPs environmental contamination in the future.

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Notes

Views expressed in this editorial are those of the authors and not necessarily the views of the ACS.

Biography

Denise M. Mitrano is an Assistant Professor at ETH Zurich, Switzerland, in the Environmental Systems Science Department. As an environmental analytical chemist, she focuses on the distribution and impacts of anthropogenic materials in technical and environmental systems. She is particularly interested in developing analytical tools to systematically understand the mechanisms and processes driving the fate, transport, and biological interactions of particles, such as engineered nanomaterials and nano- and microplastics. In this context, her research group uses these results to assess risks of anthropogenic materials across various ecosystems and scales. An interest in a safer by design approach for both nanomaterials and plastics is exemplified by working on the boundaries of environmental science, materials science, and policy to promote sustainability and environmental health and safety of new materials. Her work has recently been recognized by the Swiss National Science Foundation Marie Heim Vögtlin Prize for Outstanding Young Woman Researcher of the Year (2022), the 2022 James J. Morgan Early Career Award for Outstanding Contributions to Environmental Science from *ES&T* and the ACS Division of Environmental Chemistry, and the 2022 Emerging Investigator in Atomic Spectroscopy Award. She is currently serving as a Topic Editor with *ES&T Letters*.

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