Microplastic sample preparation methods for wastewater samples: Their applicability for sub-micro- & nano-plastic and the challenges ahead

Mohammed S. M Al-Azzawi\textsuperscript{1}, Oliver Knoop\textsuperscript{2}, and Jörg E. Drewes\textsuperscript{2}

\textsuperscript{1}Technical University of Munich – Arcisstrasse 21, D-80333 München, Germany
\textsuperscript{2}TUM Chair of Urban Water Systems Engineering – Technical University of Munich Am Coulombwall 3 85748 Garching, Germany

Abstract

Environmental samples are complex and a quantification of microplastics often cannot be performed unless the plastic particles are separated from the accompanying natural matrix. Unfortunately, sample preparation methods microplastic analysis are not standardized yet. Furthermore, the various sample preparation methods in literature often lack a comprehensive validation to ensure that the microplastics are not affected by the sample preparation methods. Additionally, nanoplastics, on account of their smaller size, are more difficult to quantify than microplastics. In a previous study, common sample preparation methods for microplastic analysis were validated, and two suitable methods were presented; Fenton reaction and hydrogen peroxide. The two methods were shown to be effective at removing organic matter from sludge matrices without affecting the microplastic characteristics. These methods were validated for microplastics with sizes 80-330 µm. However, there is a concern that smaller microplastics and especially nanoplastics, due to their larger surface area to volume ratios, are more vulnerable to sample preparation. Thus, their characteristics, especially size, could be adversely affected during sample preparation. Therefore, the current study was designed for smaller microplastics (< 10 µm) to investigate the effects of sample preparation methods on sub-micro and nano-plastics. The main concern is the change of size distribution of these particles, as they can be lost due to sample preparation. First experiments were performed on nano-PS spheres (107 nm, 78 nm) using Nanoparticle Tracking Analysis (NTA). The results show a decrease in the average size of particles of about 10-15 % after exposure to a treatment with hydrogen peroxide. Fenton analysis was not possible as the reaction generates nano-iron precipitates, which impeded the measurement of the size distribution. Thus, solutions are being investigated to deal with this problem and to increase the accuracy of the measurement to allow an accurate reporting on size changes for both treatments.

Keywords: Microplastics, sub, microplastics, nanoplastics, Fenton, Peroxide, Digestion methods