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Development and validation of an acid/alkaline digestion method for efficient microplastic extraction from wastewater treatment plant effluents: Sulfuric acid concentration and contact time do matter

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ABSTRACT

Accurate analysis of microplastic particles (MPs) in environmental samples requires removal of interferences during sample preparation. Wastewater samples are interference-rich and thus particularly challenging, with concentrated sulfuric acid currently deemed impractical as a reagent. Therefore, this study aimed to establish a straightforward, effective, and safe method employing concentrated sulfuric acid and potassium hydroxide to eliminate interferents from effluent samples obtained from wastewater treatment plants (WWTPs). We found that 80 % sulfuric acid at room temperature with a brief contact time of 5 min was viable through a qualitative spot test involving 37 plastics categorized into three types (I, II, and III) based on their polymer structure's oxygen position. A quantitative assessment revealed that treatments involving H_2SO_4 and KOH (20 %, 24 h, 48 °C), either separately or in combination, had no discernible physical impact on the overall plastics, except for a subtle one for Type III plastics (e.g., nylon and PMMA) known to be labile under harsh pH conditions. This acid/alkaline digestion (AAD) method, incorporating such conditions for H_2SO_4 and KOH treatments, yielded a high mass removal efficacy (97.8 ± 2.4 %, n = 13) for eliminating natural particle interferents for primary, secondary, and tertiary effluent samples. Furthermore, the AAD method allowed for the determination of MPs in effluents with high surrogate particle recoveries (e.g., 95.1 % for larger than 500 µm size fraction). This method is readily adaptable to create appropriate protocols for different types of environmental matrices.