A methodological protocol to extract microplastics from river sediments (bed, bank and floodplain)

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Abstract

Identifying and quantifying the entry of microplastics into the environment is fundamental to develop effective mitigation strategies. So far, microplastic research has mainly focused on marine environments and large rivers. However, it is the small-scale rivers systems, often adjacent to agricultural areas and waste water treatment plants, which have been suggested to play an important role in the entry of microplastics to aquatic habitats. Therefore, the aim of our ongoing research is to map the Hessian river Lahn, exemplary as small-scale river system, in high resolution to assess microplastic pollution and to identify sources. As river sediments differ from marine sediments, a different methodological protocol is required. Here, we present a methodological protocol to purify samples from the river bed, bank and flood plain and show first results of microplastic pollution. These results indicate that small-scale rivers act as (possibly temporary) sink for terrestrial microplastics.

Keywords: microplastics, methodological protocol, river sediments, small-scale spatial distribution

1. Introduction

Microplastic pollution has been reported in ecosystems worldwide and impacts on wildlife and, potentially, human beings [1]. In order to manage the environmental damage on local and global scales, it is necessary to quantify entry pathways and the spatial distribution of microplastics. To this end, different types of ecosystems, terrestrial and aquatic, marine and freshwater, have to be monitored.

While research has focused on marine habitats [2] and large streams so far, the role of small-scale confluences is yet to be investigated. At the same time, the microplastic research community faces a twofold challenge: Reliable and precise analysis techniques have to be developed, and they shall be standardized to allow for inter-study comparisons. For these reasons, the aim of our research in Marburg is to develop simple and efficient methods to quantify microplastics in sediments and to understand entry and spatial distribution of microplastics in river systems. This work provides preliminary data of a case study on microplastic pollution on a small spatial scale of the Hessian river Lahn, a tributary of river Rhine.

2. Methods

Sediment samples were obtained near the district Niederweimar along a transect covering river bed, bank and floodplain. The transect was applied to three different river bank types: undercut slope, slip-off slope and a straight river bank.

Sediments were removed via density separation with the so-called MicroPlastic Sediment Separator MPSS (Hydro-Bios Apparatebau GmbH; after [3]) and sodium chloride ($\rho \approx 1.19$ g ml^{-1}) as separation liquid. The motor of the MPSS was switched off after one hour of stirring and – in contrast to beach or sea floor sediments – larger pieces of mostly vegetational matter were removed from the lower standpipe. The total separation process lasted 12–15 hours. Afterwards, samples were transferred to a cascade of stainless steel sieves (diameter: 20 mm; Retsch GmbH) with the following mesh sizes: 2.5 mm, 0.5 mm and 0.315 mm. The two large size fractions were dried (max. 70 °C) and transferred to a glass jar for visual inspection. The smallest size fraction was vacuum-filtered onto a quartz filter and stained with the fluorescent dye Nile red [4] (concentration: 20 µg/ml; solvents: acetone and ethanol, ratio 50:50). Since visual detection of the middle size classes turned out to be very time-consuming, these were later subjected to fluorescent labeling with Nile red, too.

The two large size classes were scanned for potential microplastics with a magnification of 2,5x to 30x (hand magnifier; stereo microscope, Wild Heerbrugg AG). The stained size fraction was excited energetically with a blue LED (450 nm) followed by an excitation filter (FWHM: 430–470 nm; Thorlabs GmbH). Resulting fluorescence emission was guided through a long pass filter (532 nm; Thorlabs GmbH) to block the excitation light. Those particles which emit fluorescence can not only be microplastics, but also biological matter. Suspicious particles from all three size classes were subjected to an ATR-FTIR spectroscopic measurement (Tensor 37; Bruker Optics) to validate their synthetic origin and to identify the polymer type. Data were analysed with Microsoft Office Excel.

All laboratory glassware encountering the samples was cleaned with Milli-Q (Merck KGaA). The separation liquid was cleaned prior to encountering the environmental sample by means of a density separation

in the MPSS (lasting five hours). Laboratory coats made of c **A** ere worn all the time. Three blank runs were performed to estimate process contamination. In total, one PP particle was found showing a very low contamination level.

3. Results

This section outlines three key findings of our study. Firstly, microplastics were found at all riverine structures sampled: in the river bed, the bank and the floodplain. Secondly, concentrations at the *straight* river section varied only between 2 and 6 particles per dry weight, while concentrations of the *slip-off* **B** were higher at the *bank* reaching up to 38 particles per kg of dry weight (

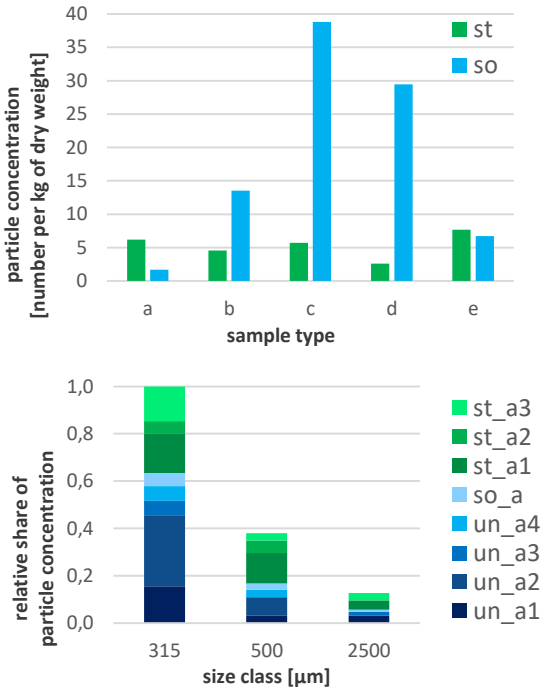

Figure *1*A).

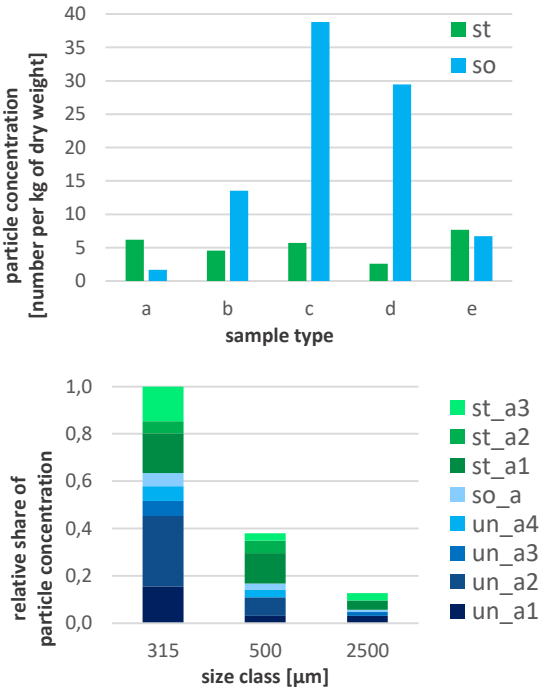

Figure 1. ***Sample names:*** *a: river sediments (note that the number of samples per river bank type differs due to limited accessibility to river bed sediments), b–d: bottom, middle and upper section of the river bank, e: floodplain. The abbreviations st, so and un refer to the river bank type (**st**raight river bank, **s**lip-**o**ff slope and **un**dercut slope).* ***(A)*** *Absolute number of microplastic particles (per kg of dry weight) of all three size classes found at the slip-off slope (blue) compared to the straight river section (green)* ***(B)*** *Relative share of microplastic particles (per kg of dry weight) found per size class (> 315 µm, > 500 µm and > 2500 µm), normalized to the total particle concentration of the smallest size class.*

Thirdly, next to spatial distribution, information on size distribution was obtained. A comparison of the different size classes of particles found in the river *bed* reveals an increasing number of particles with decreasing particle size (**Σφάλμα! Το αρχείο προέλευσης της αναφοράς δεν βρέθηκε.**1B). Furthermore, almost all river bed samples contained microplastics of the smallest and the medium size class, albeit with varying share, whereas only about the half of all river bed samples contained plastics of the large size class.

4. Discussion and Conclusions

First data on microplastic pollution in the Hessian river Lahn are shown here. The larger abundance of microplastics in the river bank at the slip-off slope compared to the straight river section is plausible and can be explained with a lower flow velocity. An increase in particle number with decreasing particle size, as observed here, was also reported in other studies [e. g. 4]. However, this trend might not continue in the smaller size classes because the flow velocity of a river prohibits sedimentation of smaller particles. Generally, the absolute concentrations must be interpreted with caution. Firstly, this sample set served to implement a methodology in our laboratorium and, as pointed out

above, was subjected to some minor changes within sample processing. Secondly, the small number of samples was not suited for a statistical analysis. Nevertheless, the method of this work, consisting of density separation via MPSS, sieving, detection via Nile Red and identification via ATR-FTIR spectroscopy, seems to be suitable to analyze microplastics in river sediments.

As a conclusion, different morphological structures (as river bed or bank) of a rather small-scale river in a rural area can be polluted and act as a sink for microplastics. In order to reliably quantify entry pathways and sinks a) the analytical method used here requires further improvement and b) much more samples have to be investigated to perform statistical analyses and confirm observations.

5. References

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