PRODUCTION OF MICROPLASTIC REFERENCE MATERIALS

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Introduction: The validation of analytical methods for the identification and quantification of microplastics in environmental matrices is being hindered due to a lack of certified reference materials (CRM). These materials are required for the harmonisation of analytical methods and the generation of comparable and reliable data. As a result, errors in measurements within individual laboratories can occur due to contamination, over-estimation, and under-estimation of microplastics from environmental samples. This then has an evident impact in the ability to compare and interpret analytical data from different laboratories. Until now, the Norwegian Institute for Water Research (NIVA) have focused on making reference materials (RMs) in the microscale, i.e., from 50 µm and up to 1 mm of the following polymer types: polyvinyl chloride (PVC), polyethylene (PE), polyethylene terephthalate (PET), polystyrene (PS), and a mixture of polymers, in the form of soda tablets. These RMs have been used in a number of inter- laboratory comparison studies worldwide as well as in microplastic recovery tests in pyrolysis gas chromatography mass spectrometry (Pyr-GCMS). The CRMs have been analysed by different laboratories using different techniques, including light microscopy, micro-Fourier transform infrared spectroscopy (µFTIR), Raman and Pyr-GCMS.

Materials and Methods: Materials will be generated by cryo milling and size fractionation and will be characterised by several techniques. The materials will be made available through Chiron AS.

Results: Microplastic reference materials **(**CRMs) in the form of soda tablets were prepared in a clean room. Entrance to this room was restricted to avoid contamination. Prior to production, all surfaces and equipment were cleaned with a 70% ethanol solution. Contamination in the clean room was monitored by setting a wet filter on an open petri dish for 48 h. With this test it was concluded that contamination was insignificant.
The dissolvable soda tablets consist of a mixture of sodium hydrogen carbonate, citric acid and lactose. Then the different polymers, i.e., PET, PE, PVC and PS in specific size fractions between 50 and 300 µm were added, before being mixed in a shaker. The soda tablets were made manually in a tablet mold to avoid the use of lubricants in the machine as this would interfere with the analyses. The biggest size fractions between 425-1000 µm were added manually with a tweezer to the mold while making the tablets.

Discussion: Analytical quality control/quality assurance (QC/QA): The quality of the soda tablets was examined by checking 10% of the total tablet production in each batch. For this, the soda tablets were added to beakers containing 30 ml milliQ which were covered in aluminum foil and set into a shaker incubator at 40 °C and 100 rpm for approximately 30 minutes. Thereafter samples were filtered with a glass fiber filter with a pore size of 0.7 µm, before counting the particles under a microscope.

Conclusions: Different types of microplastic reference materials in the size fraction of 50 to 1000 µm were produced. Now and moving forwards we will focus on the size fraction below 50 µm.
These certified materials will ultimately be made available commercially by Chiron AS and via international research network.

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