PROJECT PERIODIC REPORT

Defining the baselines and standards for microplastics analyses in European waters

Project acronym: BASEMAN

Period covered: 01.01.2016 - 31.12.2016

Date of submission: 07.06.2017

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https://www.researchgate.net/project/BASEMAN-JPI-OCEANS-Defining-the-baselines-and-standards-for-microplastics-analyses-in-European-waters-Project-Coordinator-Dr-Gunnar-Gerdts-Alfred-Wegener-Institute-Helmholtz-Centre-for-Polar-and-Mar

Currently BASEMAN is implemented in the research network ResearchGate. A dedicated website is under construction and will be hosted by JPI-O (~July 2017)

PUBLISHABLE SUMMARY (MAX. 2 PAGES)

Aims of BASEMAN: Although microplastics (MP) are recognized as an emerging contaminant in the environment, currently neither sampling, extraction, purification nor identification approaches are standardized, making the increasing numbers of MP studies hardly -if at all- comparable. The overall goal of this interdisciplinary and international collaborative research project, is to overcome this problem through a profound and detailed comparison and evaluation of all approaches from sampling to identification of MP. Our collaborative research project combines experienced MP scientists (from different disciplines and countries) in a cutting edge project addressing the JPI Oceans (JPI-O) pilot call "Ecological aspects of MP in the marine environment". Our project tackles two major topics: 1) "The validation and harmonization of analytical methods" which is indispensable for 2), the "Identification and quantification of MP". The results of the project will equip EU authorities with tools and operational measures that may be applied to describe the abundance and distribution of MP in the environment. Such tools will permit JPI-O evaluation of member state compliance with existing and future monitoring requirements.

Current status: With respect to the first major topic -"validation and harmonization of analytical methods"- it was crucial to first gain an overview and discuss the analytical skills and capabilities of the BASEMAN project partners which were then individually consolidated in the respective labs. Since currently no standard operation procedures exist for purification, extraction and analyses of MP, it was expected, that the different procedures and approaches applied by the BASEMAN partners, directly reflect the diversity of approaches of the scientific community analyzing MP in the marine environment. As a consequence and since purification, extraction and analyses had to be seen *per se* as variable, for a validation or inter-comparison, samples used for validation, had to be standardized as much as possible. The prerequisites were I) standardized MP (specified polymers with specified sizes) and II) standardized matrices (from which polymers had to be extracted, purified and analyzed). Based on the discussions of the consortium during the BASEMAN kick off meeting, a final choice was made for the polymer-types to be implemented in the study: High Density Polyethylene (HDPE), Low Density Polyethylene (LDPE), Polypropylene (PP), Polystyrene (PS), Nylon6.6 (PA66), Poly(ethlyene Terephthalate) (PET), Poly(methyl methaacrylate) (PMMA), Polyvinyl chloride (PVC), and Polycarbonate (PC). These polymers represent I) a selection of the most common polymer types typically found as MP in the marine environment and II) a broad range of polymers with different densities facilitating a comparison of different extraction approaches (e.g. density separation). Concerning particle sizes, the consortium finally agreed on three categories -~1 mm, ~100 µm and ~20 µm- facilitating comparative MP analyses ranging from simple light microscopy and "expert knowledge based identification" (large particles) to "state of the art" analytical techniques like FTIR-and Raman-Microscopy or mass-spectrometry (PyGCMS) (small particles). In parallel to the definition of standardized MP, standardized matrices were defined by the consortium: I) three types of natural plankton, II) three types of sediment and III) three types of biota (e.g. fish- and mussel tissues). These matrices represent I) a selection of the most common matrices where MP have to be detected for instance in the framework of monitoring approaches and II) a variety of natural polymers (e.g. proteins, cellulose, chitin) and abiotic compounds facilitating a comparison of different extraction and purification approaches. All polymers were purchased as commercially available production pellets, characterized (by usage of TGA, DSC, FTIR, Raman, MVR and GPC), milled and sieved according to the specified size classes and finally provided for the consortium as single fractions (polymer, size class). For a validation and harmonization of analytical methods, an approach in analogy to ring trials was chosen. Hence the beforehand mentioned natural samples representing plankton, sediments and biota were spiked with a defined mixture of polymer particles and send to the participating labs. Analyses are ongoing and first results are to be expected in late autumn 2017.

A reliable identification of small MP particles (~< 500 µm) by simple light microscopy and "expert knowledge" is prone to misidentification and as a consequence data on numbers (and sizes) are not valid. Hence several "state of the art" analytical techniques like FTIR-and Raman-Microscopy or mass-spectrometry (PyGCMS) are applied in the framework of BASEMAN. Since identification of polymers by spectrometry (FTIR, Raman) is generally based on comparison with databases, "in house" databases for identification of polymers are generated. In contrast to commercially available databases, all data will be "free to use", be provided for the BASEMAN consortium and be available upon request for e.g. the scientific community or environmental agencies. The ATR-FTIR database (Bruker format and JCAMP-DX

format) currently comprises 312 spectra, the respective database in Perkin Elmer format comprises currently 100 spectra and the Raman database 209 spectra. Since environmental MP particles are exposed to several stressors (e.g. radiation, radicals, biological activity) the FTIR or Raman spectra of these particles display on the hand the general pattern of the respective polymer but on the other hand also “signals” deriving from an alteration of the polymer-backbone (e.g. carbonyl, hydroxyl introductions). Hence in 2016 a pilot-scale weathering system using UV/Vis metal halide lamps was installed for mimicking the environmental influence. Preliminary analyses on the simulated samples drawn from the weathering device have already carried out. Weathering influence will be examined by FTIR spectroscopy and data will be introduced in the databases (as weathered polymers). JPI-O BASEMAN and JPI-O WEATHERMIC will exchange experience and data here. The final aim will be to provide a comprehensive multi spectrometric database considering also hyperspectral and mass-spectrometric analytical approaches.

Furthermore an automated analysis pipeline for FTIR-Imaging data regarding identification, quantification and sizing of MP was developed and published. All scripts (Phyton, SimpleTK) are available as supplement of the publication and were provided for the consortium.

The evaluation of sampling methods for seawater, sediments & biota and standardization & intercalibration of sampling methods was partly performed on a research cruise in the Galway Bay in 2016. Another research cruise to the Bay of Biscay was conducted in April 2017.