DOI: http://doi.org/10.21698/simi.2019.ab26

DESORPTION OF PHTHALATES FROM MICROPLASTICS INTO SURFACE WATER. *IN VITRO* STUDY.

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Keywords: *desorption, microplastics, PET, phthalates, pollution*

Introduction

Microplastic pollution has been an environmental issue that extends and worsen with the development of the plastic industry which increased exponentially since the early 1950s. DEHP, one of the microplastic related additives, is included in the list of priority hazardous substances, being an endocrine disrupter, reproductive system, affecting development and mobility for both humans and aquatic animals. According to literature data, DEHP is the most desorbed compound on microplastics, especially from polyethylene terephthalate.

The aim of this study was to determine the level of phthalates (dimethyl phthalate - DMF, diethyl phthalate - DEF, dibutyl phthalate - DBF, di-2-ethylhexyl phthalate - DEHP, dioctyl phthalate - DOF) desorption from polyethylene terephthalate microplastics in surface water using *in vitro* laboratory tests. The samples were identified and quantified by gas chromatography coupled with mass spectrometry (GC-MS/MS).

Materials and methods

Polyethylene terephthalate (PET) from a commercial plastic bottle (1 L) was chosen as the plastic material because this is the most widely used and the most found microplastic in the environment. The phthalates present in the polyethylene terephthalate (PET) microplastics process were desorbed into the surface water and then identified and quantified by gas chromatography coupled with mass spectrometry (GC-MS/MS). PET microplastic samples used in the study were prepared in the laboratory using a cutter to obtain sizes between 0.5-5 mm. The experiment was performed using as surface water matrix (microplastics free) which was used throughout the *in vitro* study and supplemented with controlled amounts of microplastics earlier mentioned. This type of water has been chosen to reproduce desorption conditions of phthalates in microplastics in the aquatic systems where they are found. An amount of 10 g of PET microplastics were contacted with 1L of aqueous surface water for 5 days with periodic stirring, in two replicates, in normal conditions, avoiding of sun light. After 5 days microplastics were decanted from a water sample and a volume of 500 mL of water was extracted with liquid-liquid extraction by with a volume of 20 mL of hexane: dichloromethane (4:1 v/v); 100 µg/L PCB 209 was added as an intern standard.

The phases were shacked for 3-5 minutes and then were left to rest for 5 minutes to separate. The organic phase was passed over anhydrous sodium sulfate to retain traces of water, concentrated under a nitrogen stream to 1 mL volume and transferred quantitatively to a 2 mL GC vial. Determination of phthalates was performed with a Thermo GC-MS/MS TSQ 8000 EVO with a PTV injector, where 1 µL of sample was injected in splitless mode. The compounds were separated by a chromatographic column TraceGOLD TG-5SilMS with 5% diphenyl/95% dimethylpolysiloxane (60 m, 0.25 mm x 0.25 µm) from Thermo Scientific with 1 ml/min flow. The oven temperature program started from 50ºC (hold 2 min) and increased to 290ºC with 20ºC/min (hold 12 min). The MS/MS detector parameters were electronic impact ionization mode (EI, 70 eV), ionization temperature 250˚C, two ion transitions expressed as SRM (selected reaction monitoring).

Results and conclusions

Figure 1 shows the concentrations of phthalates [µg/L] desorbed from polyethylene terephthalate microplastics in surface water samples.

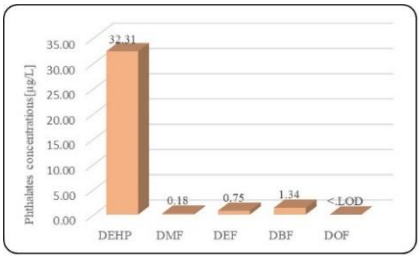

Figure 1. Phthalates desorbed from microplastics in surface water

After 5 days contact of surface water with microplastics it was observed the highest desorption of DEHP in approximately amount of 32 µg/L. The other compounds showed concentration values between 0.18-1.34 µg/L. Dioctyl phthalate was undetectable in the analyzed samples. According to Romanian law HG 570/2016, a program for the investigation of discharges, emissions and losses of priority hazardous substances, for di-2-ethylhexyl phthalate the maximum admissible concentration is 1.3 µg/L in surface waters.

The desorbed concentration of DEHP from *in vitro* microplastics is approximately 25 times higher than the maximum admissible concentration in surface water (HG 570/2016). Reported to the whole bottle weight (25 g) the DEHP concentration can be extrapolated to 80 µg/L. After 5 days exposure, 80 µg/L DEHP were desorbed from 1 L plastic bottle (25 g), while the total of other quantified phthalates (DMF, DEF, DBF, DOF) represents 93% less contamination than DEHP.

Acknowledgements

The authors acknowledge the financial support offered by The National Research Nucleu Program through contract 20N/2019, Project code PN 19 04 01 01.