Optimisation of cryomilling parameters to produce microplastic particles

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Abstract

Microplastics (MPs, polymer particles < 5 mm) received high attention in the last years, but to this day there is no standardized method to prepare MP particles, which often hampers the comparability between different studies. Either for method evaluation, environmental analysis or ecotoxicological studies, a comparable preparation of MP particles, which are well-characterized and representative for the particles in the environment, is necessary. MPs comprise particles of different plastic types in various shape and size, while fragments usually dominate in the environment. Cryogenic milling is a method able to create MPs in fragment shape of virtually all plastic types present. This technique is frequently used in the MP community, however, the parameters used for milling differ among studies, resulting in different particle size distributions and particle characteristics. Here, we present a ready-to-use protocol, which can be used for standard in-house preparation of MPs, aiming to gain more comparability in MP research. The protocol contains the cryomilling parameters for 11 different polymer types. The cryomilling parameters are optimized for three different (small < 60 μm, medium < 120 μm, large > 200 μm) size classes of each polymer type, so the protocol is applicable for multiple purposes. Pristine plastic beads were milled, and the size distribution was determined by dynamic light scattering (DLS). The particles were characterized before and after the milling by Fourier transform infrared spectroscopy (FTIR), Raman-spectroscopy and the morphology was analyzed by scanning electron microscopy (SEM) measurements. Spectra gained from FTIR and Raman did not change due to the milling, regardless of the milling time, indicating that the chemical composition of the polymers did not change during the procedure. The final protocol was tested on five different plastic packaging of different polymers used in food industry, to compare the effect of the initial shape of the material added to the milling jar.

Keywords: Cryogenic milling, Microplastics (MPs), Size distribution, DLS, FTIR, Raman, SEM, reference particles

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