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Methodology for identification of plastic fragments (PET) in aqueous media through computerized microtomography

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1. Introduction

PET material is widely used in various sectors of the economy, such as the food, automobile, textile, chemical, electronics and civil construction, among others [1]. The importance of PET in the packaging industry is highlighted, especially in the bottle format, as it presents advantageous physicochemical properties, low cost, good resistance and lightweight (easy to handle). The properties of resistance to degradation and durability of plastics make their complete degradation by nature very difficult, making this material just smaller and smaller, generating the so-called microplastics (MPs) [2].

According to Montagner in 2018 [3], microplastics are everywhere, often invisibly, having shapes of fragments, spheres, small pieces of films or plastic fibers, with a length or diameter smaller than 5 millimeters, and may also be micrometric and nanometric. These substances can be classified into two categories, the primary (released into the environment as small particles), and the secondary (resulting from the degradation of larger objects). They can be detected in breathing air, in land or water environments (fresh or salt water), in tap or bottle water, in sea salt, in fish and seafood eaten by man, in honey, in beer, and, consequently, in the feces of human beings all over the planet. The presence of microplastics in the oceans has only been known since the 70s, with the seas being the repository for a good portion of the microplastic produced on land, when they receive water from rivers, streams and sewage.

Turra, in 2014 [4], highlights that the challenges of research with microplastics are related to the diversity of types, fonts, shapes and sizes of plastics. The researcher also reports that, in addition to microscopic fragments, there are those whose dimension is in the nanometer scale (less than 1 thousandth of a millimeter), capable, in theory, of entering the bloodstream and reaching organs such as the liver, kidneys and brain. However, he claims that, so far, neither the technology to monitor these particles nor knowledge about their effects on ecosystems and biodiversity is available. In addition to physical effects, microparticles ingested or inhaled by humans and animals can be vectors of microorganisms and contaminants, such as persistent organic pollutants (POPs), synthetic compounds resistant to degradation in the environment. There are two types of substances associated with the particles: those inserted in the plastic itself to obtain special properties, such as phthalates and bisphenol A, which are capable of modifying hormonal functioning; and substances adsorbed by microplastics, such as heavy metals and POPs. Phthalates (plasticizers) have the characteristic of making PVC flexible, and bisphenol A is the raw material for polycarbonates, being used in the manufacture of long-life products, such as electronics and construction material (CAMPOS, 2019 [5]).

According to Montagner, in 2018 [3], POPs are abundant in the environment and can accumulate in organisms. These contaminants are released into the environment by pharmaceuticals, pesticides, hormones, personal care products and illegal drugs, and can present concentrations in rivers at the same level as untreated sewage, according to a survey carried out by the author.

As stated by MacLeod, in 2021 [6], potential impacts from poorly reversible plastic pollution include a lot of possible negative outcomes in the areas where they should be found, as: changes to carbon and nutrient cycles; habitat changes within soils, sediments, and aquatic ecosystems; co-occurring biological impacts on endangered or keystone species; ecotoxicity; and related societal impacts.

As claimed by Brazilian Association of the PET Industry, ABIPET, in 2015 [1], although plastic has so many uses, its unbridled and careless disposal is causing severe consequences for the environment. The problems involved in the improper disposal of this material in nature (on beaches, rivers, lakes, lakes and oceans), and all the risks of microplastics both for the ecosystem and for the living beings. It is extremely important to know a methodology that makes it possible to quantify this material when present in aqueous media, so that it is possible to account for the level of contamination provided by them in natural environments such as lakes, lakes, rivers, seas, beaches and riverbeds. According to Saitoh, in 2021 [7], different analytical approaches have been performed for the separation, characterization, and identification of microplastics, in order to verify environmental pollution caused by MPs.

Computerized microtomography (microCT) is a non-destructive method, which allows to know the internal structures of an object with relative reliability. The X-ray microtomography technique applied to PET material aims to quantify this material when present in aqueous media, so that it is possible to account for the level of contamination provided by this polymer in natural environments such as lakes, rivers and oceans.

Thus, the aim of this study is to present a methodology developed in the laboratory to identify and quantify plastic fragments (PET) present in an aqueous media. For this, Phantoms were developed with plastic fragments up to 2 mm in diameter, which were later analyzed using the computerized microtomography technique.

2. Methodology

Three different samples were analyzed using the microCT technique, all with plastic fragments up to 2.0 mm in diameter. One of these samples was made with fragments of higher density, the second with those of lower density, and the third had all mixed densities.

Methodology in order to quantify the PET material present in aqueous media:

1st part) PET material crushed into fragments of different sizes and densities was subdivided by the size of the grain diameter. The separation of the grains was made in the sieves according to the opening of the meshes contained in a vibrating base brand GRANUTEST, acting for a period of 15 minutes. The sample of interest for this study was the one contained in the largest sieve, which contained particles up to 2.0 mm in diameter, called, from now on in this study, 2.0 mm PET.

2nd part) It proceeded with the preparation of three samples with 2.0 mm PET in an eppendorf. For the first sample, 1.03g of this substance was used, the volume of which was determined by the variation of this quantity in distilled water, in comparison with what it had before adding the PET. Then, the sample was frozen. To make the second and third samples, the 2.0 mm PET was immersed in distilled water. Then, the two substances were mixed, the PETs of higher and lower densities were separated (those that floated from those that sank), the PET was removed from the water and allowed to dry naturally. Subsequently, the procedure performed in the first sample was repeated, but with 0.84g of PET 2.0 mm of lower density and 1.51g of PET 2.0 mm of higher density, respectively, for the second and third specimens .

3rd part) Each sample was placed inside an expanded polystyrene (EPS) box, in order to avoid rapid thawing of the frozen mixture, when performing microCT in the GE-Phoenix Vtomex equipment with the

parameters Voltage = 70 kV, current =250 µA, Power=17.5 W, Timing= 333 ms, number of frames=5. After this procedure, the images were reconstructed using Phoenix Data X2 Reconstruction software.

4th part) Then, in order to analyze the volume of PET that can be detected with microCT, the CTAn software was used. Thus, a comparison was made between the volume actually placed in each of the samples and the amount detected by the process used in this study. Making it possible to verify the reliability of the method used, and also to verify whether there is an increase in accuracy when using samples made only with PETs of similar densities in each eppendorf.

3. Results and Discussion

For the three samples, the same plug-ins were used in the CTAn software, which are tools that have the function of treating the image, in order to obtain more accurate results by removing the excess of existing noise. The plug-ins are described below, according to Manual for Bruker-microCT CT-Analyser v. 1.13 [8] in the order in which they were performed:

Thresholding- this plugin is used to segment the foreground from background to binary images.

Despeckle- In the despeckle plugin it is possible to select a range of object sizes – both white and black voxel objects / pores. You can either delete objects / pores within a specified range, or delete all objects / pores outside the range. In this study, both white and black despeckle were used:

- Remove white despeckle- removes objects that are white on the basis of their size
- Remove black despeckle- removes objects that are black on the basis of their size

Morphological Operations- involve adding or removing pixels/voxels to or from the surface of all selected binarised objects (called erosion and dilation respectively). In this study, the dilation was used.

In sample 1, which contained different densities of 2.0 mm PET, it was necessary to perform the thresholding in two parts, as the less dense PET was at the top of the sample and the denser one at the bottom. Adding the volume of each part, a total volume of 590 mm^3 was obtained in this sample. The volume initially placed was 800 mm^3. In sample 1, it was possible to identify around 74% of the 2.0 mm PET actually inserted in the eppendorf.

For sample 2, containing PET 2.0 mm of lower density, only one Thresholding was necessary and the result obtained was 99 mm^3. The initial volume inserted into the sample was 750 mm3. In sample 2, it was possible to identify only 13% of the 2.0 mm PET actually inserted in the beaker. And in sample 3, with PET 2 mm of higher density, only one Thresholding was performed, and the volume found was 1060 mm3. The actual sample volume was 1250 mm^3. In sample 3, it was possible to identify around 85% of the 2.0 mm PET actually inserted in the eppendorf.

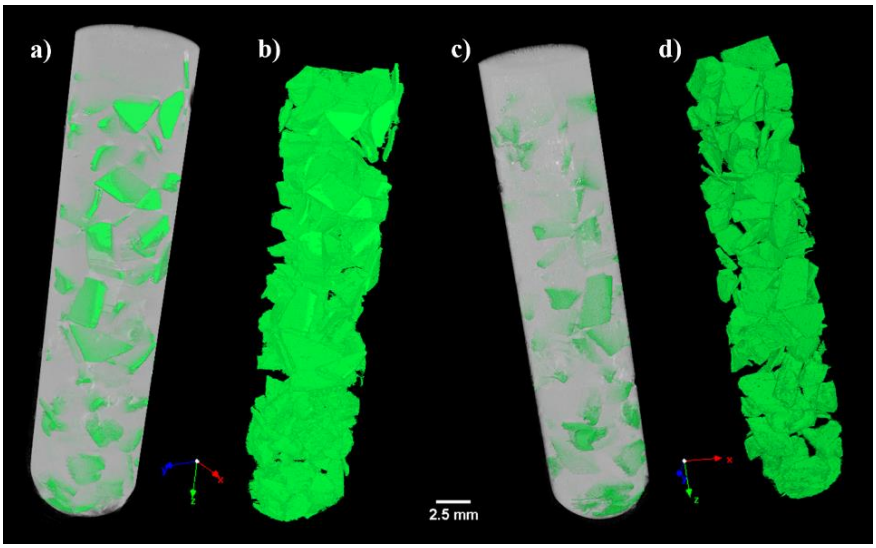

Figure 1: items a) and c) are 3D model of 2mm PET higher density; items b) and d) represent the plastic fragments highlighted.

4. Conclusions

The methodology developed in the laboratory had the aim to identify and quantify plastic fragments (PET) present in an aqueous media.

With this methodology, it was possible to identify the following percentage of PET actually inserted in each eppendorf: around 74% of the 2.0 mm PET of different densities; 13% of the lower density 2.0 mm PET and 85% of the higher density 2.0 mm PET.

The high resolution computed microtomography results provided a three-dimensional view of the plastic fragments (PET). However, the use of the same density fragments in the samples can improve the results obtained to quantify plastic fragments present in an aqueous media. Still, it was shown that samples with lower densities could give us much less satisfactory results as the substance has a lower contrast to ice in microCT due to similar density of them both.

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