

# Forensic determination of adhesive vinyl microplastics in urban soils

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## **Keywords**

soil contamination, microplastic hotspot, sticker, weathering material, density separation

## **Abstract**

Plastic production and consumption hubs are mainly concentrated in large urban centers, causing the soil in these places to become large sinks of fragments generated from their breakdown, known as microplastics (MPs). Adhesive vinyl polymers, widely used in various commercial sectors, as far as we know, have never been investigated scientifically from the perspective of their potential to form MPs. This proof-of-concept work started by studying the soil around a plaque made of adhesive vinyl that had been exposed to the weather for 8 years and was visibly degraded. We customized a selective methodology for separating specific MPs from the soil and obtained a range of  $2.6 \times 10^4$  fragments  $\text{kg}^{-1}$  of dry soil being produced from a maximum of 4.7

cm<sup>2</sup> of adhesive vinyl film. In addition: (1) it was observed that vertical and horizontal transport was not favored, causing the formation of a great potential for hotspots; (2) the area of the fragments did not influence vertical transport. Here, we presented a forensic assessment for the association between the presence of MPs and its source.

## **Introduction**

As more studies show the presence of MPs in places far from the hubs of production and consumption of plastics products, such as Antarctic<sup>1</sup>, deep-sea sediment<sup>2</sup>, in the cloud<sup>3</sup> and in caves<sup>4,5</sup>, more questions are raised regarding the impact of these emerging contaminants on ecosystems<sup>6</sup>. However, despite being found in remote regions, MPs are more abundant in more populated regions, which makes urban centers largely responsible for the production and fate of MPs in the environment<sup>7</sup>.

Thus, urban soils act as a pathway for MPs to flow into water bodies and groundwater, while also acting as sinks for MPs<sup>8,9</sup>. Therefore, studies involving the dispersion dynamics of plastic pollution in urban areas are essential for understanding the ways in which MPs are dispersed in different environmental compartments, supporting the development of mitigation strategies<sup>10</sup>.

Characteristics like shape, size, formulation, and density determine the potential of MPs to disperse. Additionally, the pathway through which they flow—such as air mass, groundwater movement, soil runoff, and water flow—also plays a crucial role in the dispersal of fragments<sup>11</sup>. Among these pathways, soil serves as one of the primary sinks for MPs<sup>11,12</sup>.

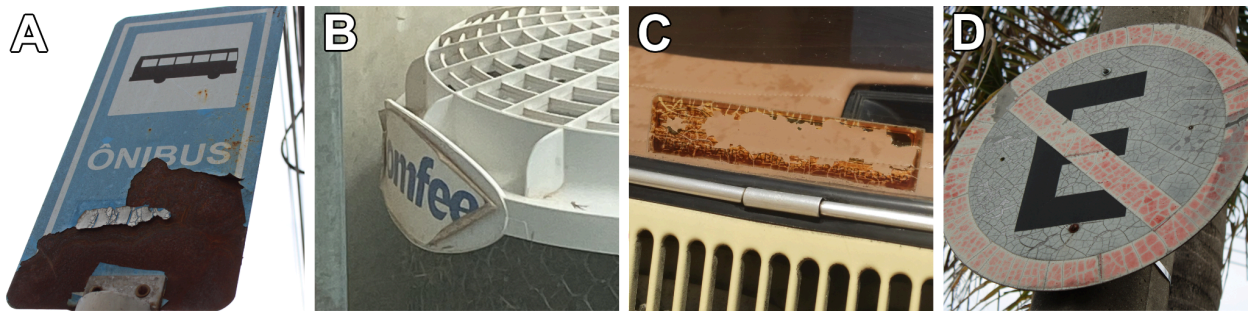
For these reasons, MPs with low transport capacity in soil are more likely to be neglected in monitoring studies and more prone to form hotspots, since they remain concentrated close to the source of dispersion. An example of this phenomenon was studied by Xu et al. (2022), who reported the highest concentration in the literature of MPs in soil nearest the Mauerpark graffiti wall in Berlin<sup>13</sup>. The graffiti paint particles studied have a high density compared to other types of MPs, which makes them difficult not only to disperse, but also to detect using conventional density separation protocols.

In 2022, PVC accounted for 12.7% of global plastic production, which reached 400.3 million tons, making it the third most produced type of plastic, only behind PP (18.9%) and PE (-LD and -LLD) (14.1%)<sup>14</sup>. Nevertheless, despite being among the most produced types of plastic, PVC was detected in 25% of types of soil and sediment analyzed in a review<sup>10</sup>, since its high density ( $\sim 1.5 \text{ g/cm}^3$ ) causes this material to deposit quickly into the sediment when it is released. This aspect raises the alert to the potential for hotspot formation by PVC MPs. Furthermore, it is worth noting that PVC is a type of plastic that carries a high load of additives in its composition, which makes PVC MPs a relevant vector of pollutants<sup>15</sup>.

One form in which PVC is incorporated is in adhesive vinyl. These adhesives can be used for visual communication, car adhesives, vinyl flooring and interior decoration (some examples are shown in Figure 1). Despite being commonplace in urban centers, there are no records in the literature evaluating the potential for MPs to form from these sources.

We therefore carried out an analysis on the soil exposed to a specific source — which was a plaque coated with an adhesive vinyl that had been left outdoors for eight years — in order to

understand the fragmentation potential of this material. To track the MPs, we use the color and density as features to selectively separate them from the soil, adapting a protocol developed by Xu et al. (2022).



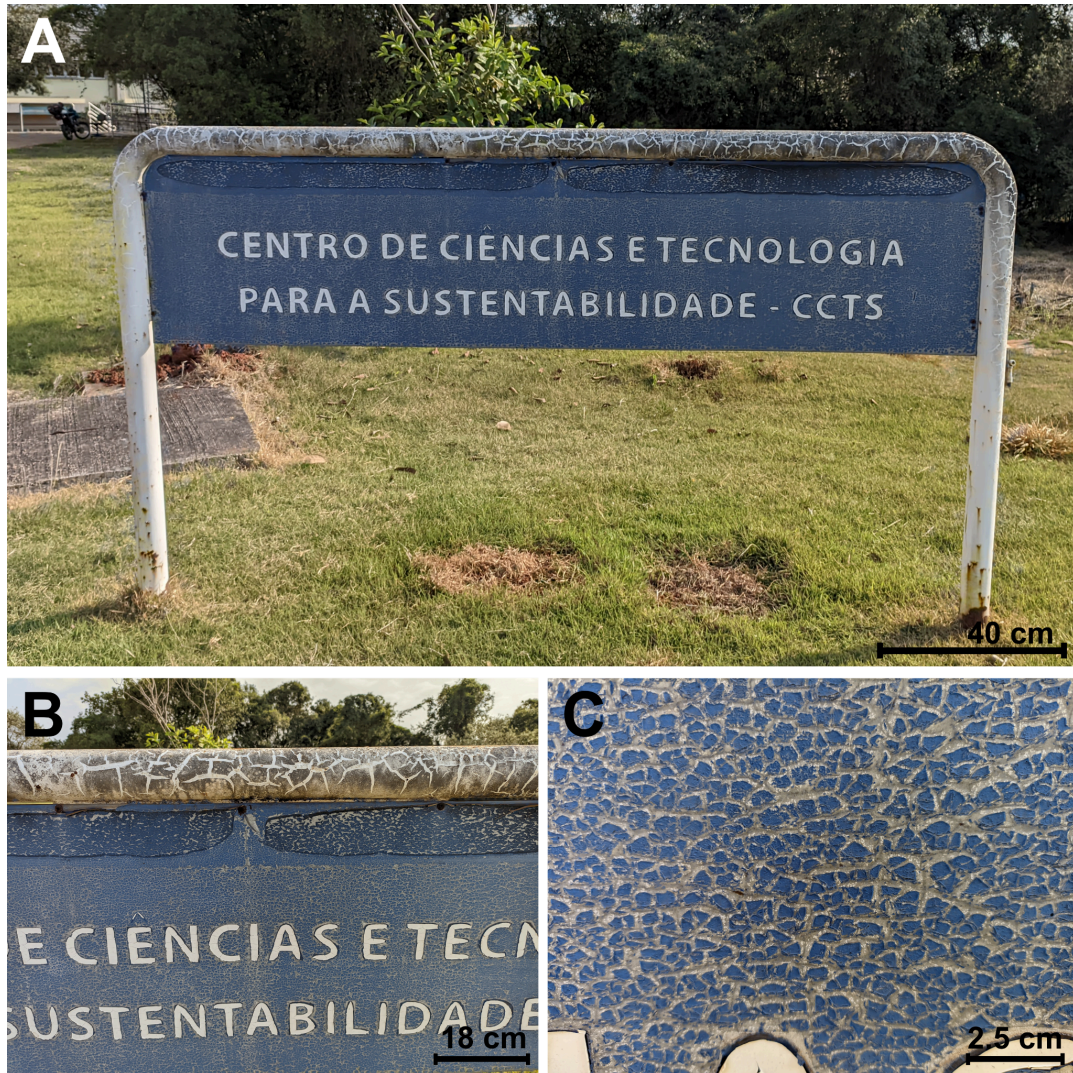
**Figure 1.** Examples of everyday objects with visually degraded parts made of adhesive vinyl films. (a) Bus stop sign; (b) Condenser coil brand sticker; (c) Sticker on a car window; (d) Stop sign. Photos authors: (a) and (d): Giovana Rocchi; (b) Walter Waldman; (c) Gabriel Prado.

## Materials and Methods

### Collection of soil samples

Soil samples were collected in Sorocaba, São Paulo, Brazil ( $23^{\circ}34'59.20''\text{S}$ ,  $47^{\circ}31'31.94''\text{W}$ ) around a plaque containing an adhesive vinyl film that had been exposed to weathering for eight years. We notice the whole area of the film was cracked, indicating that the polymer was highly degraded. In order to analyze horizontal transport, we collected samples from below the plaque (0 m), 1 m and 2 m distance towards the back of the image in Figure 2. To analyze the vertical transport, we collected samples up to 30 cm deep and divided them into layers of 10 cm. At each distance, the collection was carried out in triplicate with a soil sampler drill (Saci steel drill bit 1"

x 0-80cm). Soil samples were dried in an oven at 50°C for 48 hours and then sieved through a 2 mm sieve.



**Figure 2.** Visibly degraded plaque made of adhesive vinyl that generated the MPs in the soil targeted in this study. (a) plaque overview; (b) magnification of a; (c) magnification of b.

Separation protocol

In order to separate the blue MPs from soil, we adapted a customized separation protocol developed by Xu et al. (2022) which takes advantage of the high density of a plastic to separate it from the others. Firstly, we determined the density at which the microplastic would float by observing its buoyancy at various densities. Given this, the first separation was carried out with a saturated solution of NaCl ( $1.2 \text{ g/cm}^3$ ) to remove less dense MPs and organic matter. The subsequent separation was made using a saturated solution of NaBr ( $1.5 \text{ g/cm}^3$ ) to resuspend the PVC MPs in the dispersion. For this step, we weighed 2.5g of dry soil and added 7.5 mL of saturated solution of interest. Whenever we added a saturated solution to the falcon tube, we left them shaking gently on an orbital table for 15 minutes to break up soil aggregates that could be trapping MPs. The supernatant from the first separation (NaCl) was discarded while the supernatant from the second separation (NaBr) was vacuum filtered through a membrane filter of  $0.45 \mu\text{m}$  (GVS, Nylon disk 47 mm  $0,45 \mu\text{m}$ ). The separation using NaBr was performed three times to release as many MPs as possible from the soil. The membrane filters were observed in a Leica S8APO stereomicroscope for further analysis and measurements in ImageJ software.

#### Controls and precautions to avoid contamination

As long as MPs are everywhere, including inside laboratories<sup>16</sup>, we have adopted a few precautions. All reverse osmosis water used throughout the experiments was filtered three times with pores of  $5 \mu\text{m}$  using a vacuum filtration system, as well as saturated salt solutions. In addition, every step of processing the samples was conducted inside a laminar flow hood whenever the soil needed to be exposed to air, except for weighing for logistical reasons. This laminar flow hood was located in a room with a controlled flow of people where only cotton

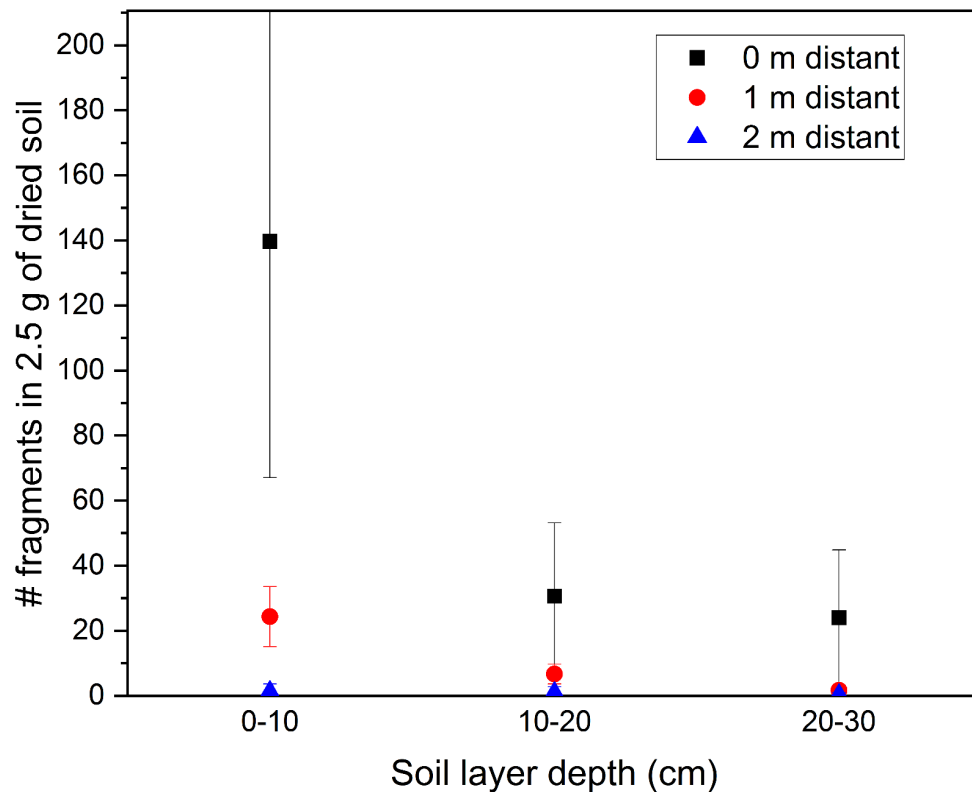
coats were allowed, and every time it needed to be used, a wet membrane was placed inside the flow workspace to monitor the MPs in the air as a procedural blank. Another precaution was to wash the falcon tubes, glassware and tools three times with filtered water.

We also adopted controls for saline solutions used in density separation. These consisted of adding the same amount of solution to a previously cleaned Falcon tube. After going through the same procedures as the other tubes (shaking and centrifuging), we filtered these solutions and analyzed them under a stereomicroscope. This control was carried out three times, each corresponding to the distance of the collections (0m, 1m and 2m). All these procedure blanks can help to avoid MPs, while air deposition blanks are important to track the quality of the working environment<sup>17</sup>.

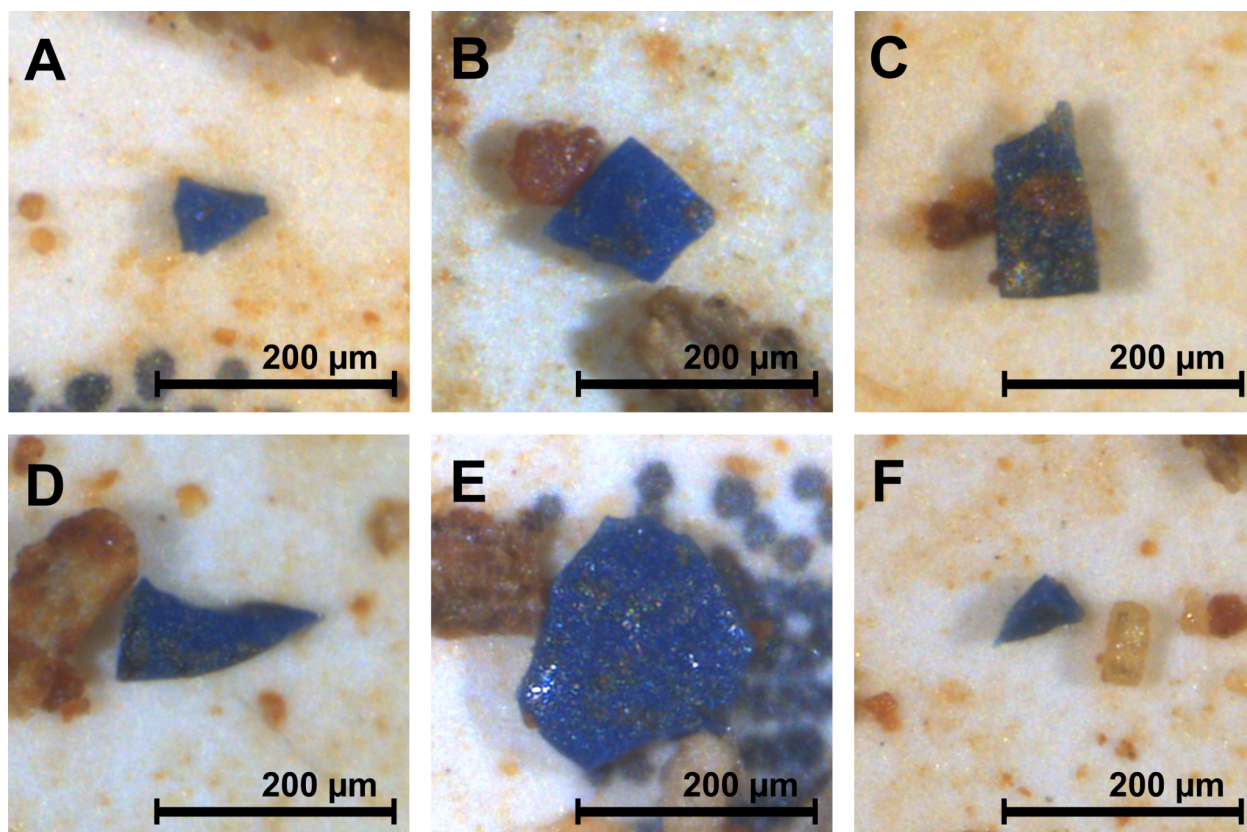
## **Results and Discussion**

Blue fragments number on the soil

At the distance of 0 m, we registered  $5.6 \times 10^4$  fragments  $\text{kg}^{-1}$  of dry soil on its topsoil layer (0-10 cm),  $1.2 \times 10^4$  fragments  $\text{kg}^{-1}$  of dry soil in the 10-20 cm layer and  $1 \times 10^4$  fragments  $\text{kg}^{-1}$  of dry soil in the 20-30 layer (as can be seen in Figure 3). The soil was located below the plaque and was the most contaminated by blue MPs (some of them can be observed on Figure 4).



**Figure 3.** Number of fragments in 2.5 g of dry soil (triplicates combined) at each depth of the soil layer. The topsoil layer was the most contaminated by blue MPs, with the soil below the plate (0m) showing the highest number of MPs, demonstrating the ability of adhesive vinyl films to form hotspots in proximity to the source. All data is available in the supplementary material (Spreadsheet S1).



**Figure 4.** Micrographs of blue fragments from the soil sampled below the adhesive vinyl film, at the 0-10 cm depth, which are also examples of MPs with sharp edges. All the micrographs taken for this study are available in the supplementary materials at “Micrographs” file.

At a distance of 1 m from the plate, the highest concentration of fragments was also in the topsoil:  $9.3 \times 10^3$  fragments  $\text{kg}^{-1}$ . A little further on, at 2 m from the plaque, we detected  $5.3 \times 10^2$  fragments  $\text{kg}^{-1}$  MPs in the topsoil layer. In every distance, the number of blue MPs was higher in the topsoil layer.

The number of fragments in soil decreased proportionally to the increase of distance from the plaque, something that was observed as well in the work of Xu et al. on Mauerpark graffiti wall.

In both cases, the farther the source of MPs, the fewer the fragments. Distributions are shown in Figure 3.

Density is one of the fundamental properties to determine MPs transport<sup>10</sup>. We presume the high density of adhesive vinyl MPs did not foster horizontal transport, through making the fragments settle quickly. Meanwhile, fragments far from the plaque probably got there through the wind, the flow of water in the soil or even through the action of the biota. Earthworms, for example, are known to transport MPs through the soil<sup>18,19</sup>.

When comparing the highest concentration of fragments found in this study ( $5.6 \times 10^4 \text{ kg}^{-1}$  of dry soil) with the MPs concentrations in soil samples of urban soil and street dust analyzed by the review of Koutnik et al. (2021), we find out that the contamination caused by adhesive vinyl film is above the upper quartile of the gathered literature, showing the relevance of this so far neglected source of MPs.

Fragments area

#### Vertical transport

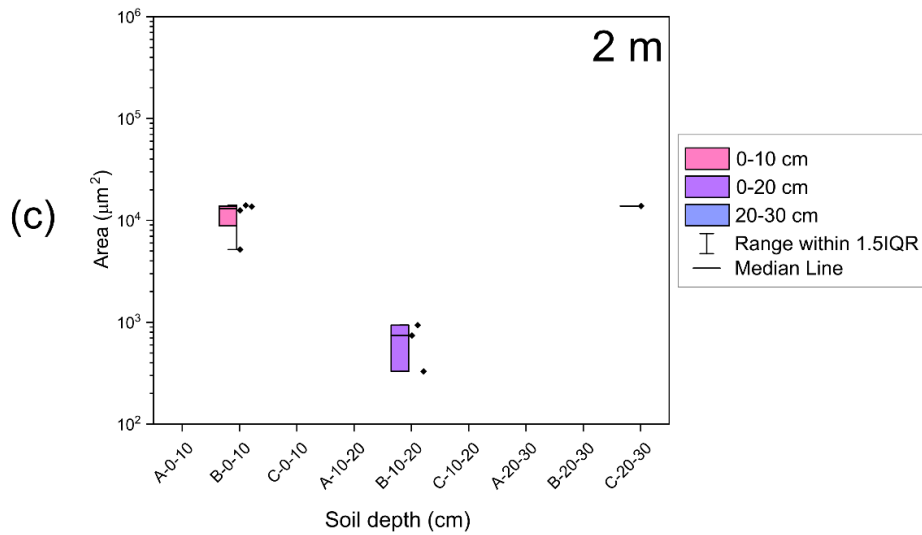
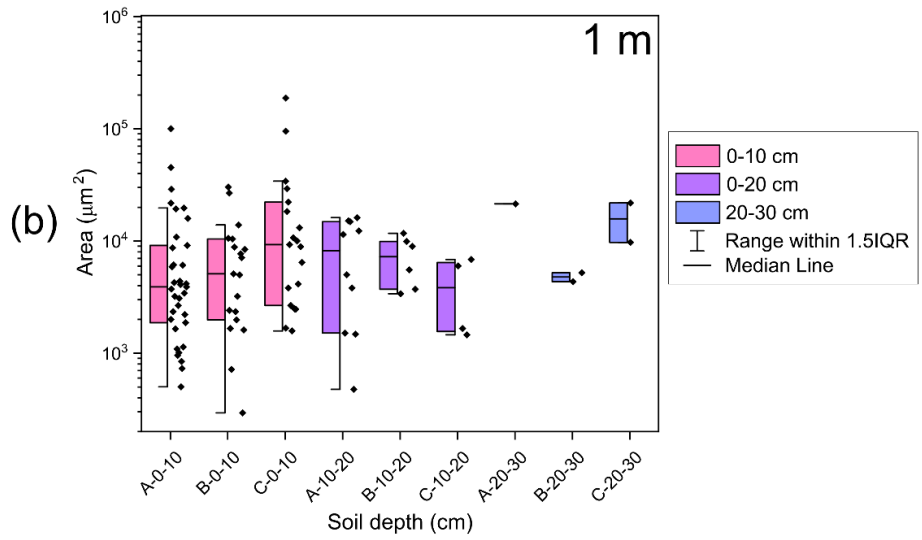
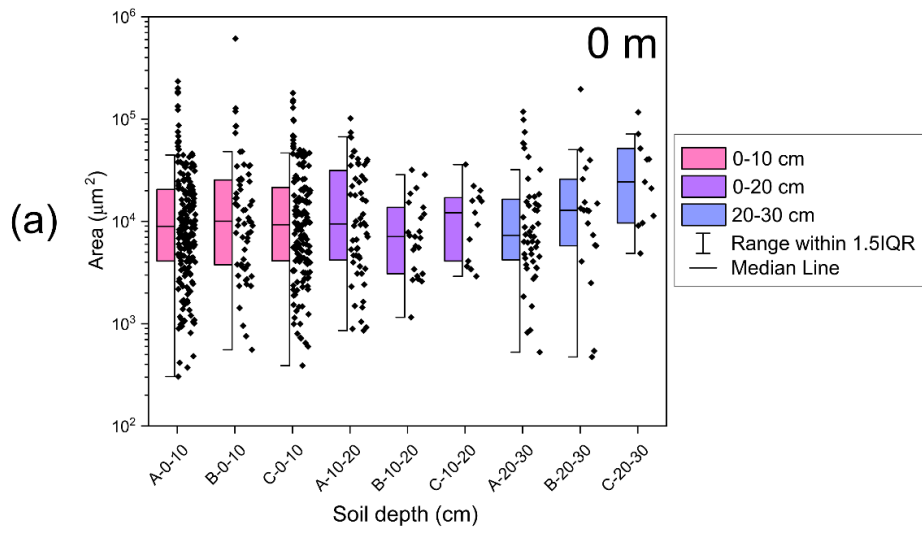
At each distance from the plaque, the fragments' area showed no difference depending on the depth (Figure 5). It seems that the soil structure was not capable of segregating different fragment sizes, at least for the size range studied. There is still the possibility that fragments outside our optical limit may have behaved differently, something that could be investigated in other studies with more sensitive equipment. This result, however, was different from the one found by Xu et al. (2022) with the spray paint particles. They noticed that the deeper the layer, the greater the

amount of large MPs found, suggesting that the smallest particles were kept in the topsoil layers as they probably interact more with the soil structure<sup>13</sup>.

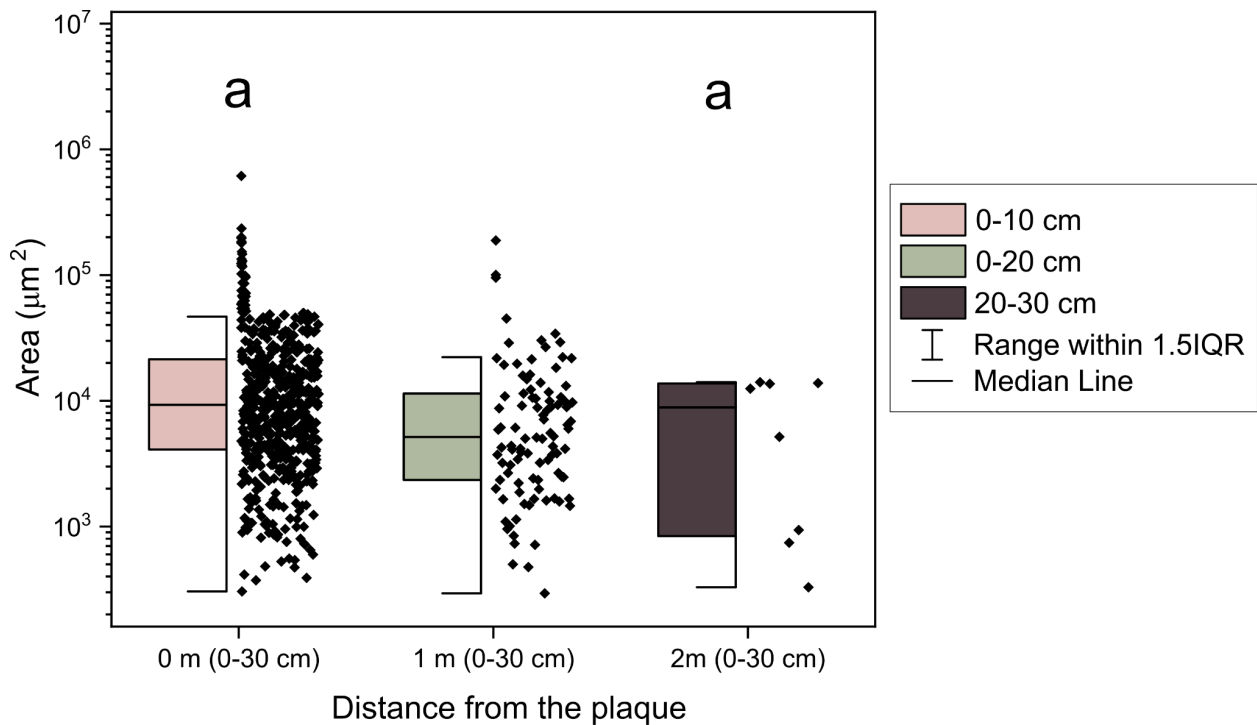
### Horizontal transport

Since no difference was detected between the area of the fragments as a function of the depth, we combined all the triplicates from each distance to analyze horizontal transport (Figure 5). Fragments at 0 m showed 20,405  $\mu\text{m}^2$  of average, at 1 m 11,913  $\mu\text{m}^2$  and at 2 m 7,651  $\mu\text{m}^2$ . This result shows a trend for the area to decrease as a function of the distance from the source of contamination, which may indicate that smaller particles have a greater dispersion capacity than the larger ones.

The area of MPs from 0 m distance and 2 m distance was the only one that did not differ, while we detected a difference between the particle size between 0 m and 1 m and between 1 m and 2 m. Since the number of fragments at 2 m was very small ( $n=8$ ), the statistics cannot reflect this comparison properly.



**Figure 5.** Dispersion of areas as a function of depth for all triplicates at a distance of 0m, 1 m and 2 m from the plaque. No significant differences were found between the triplicates of each distance by applying Kruskal-Wallis test.



**Figure 6.** Area of blue fragments at each distance from the plaque. The farther the source, the smallest the fragments. Same letter indicates no significant difference, while different letters indicate difference from the others.

Association between the sizes and the area of the source

Having a forensic approach, this study has a very unique aspect: as we know the source of the MPs we are working on, it is possible to associate the area that has been lost by the plaque with the number of fragments generated per kg of dry soil. At the distance with the highest

concentration of MPs (0m), an area of 4.7 cm<sup>2</sup> from the plaque has generated 78.534 fragments/kg of dry soil at 0m (0-30 cm depth). However, as the MPs from samples located further away from the plate presented fragments with smaller areas, it is possible to establish that, for these regions, the areas that originated, respectively, 13065 (1 m distant) and 1066 fragments kg<sup>-1</sup> of dry soil (2 m distant) were smaller than 1 cm<sup>2</sup> – 0.52 cm<sup>2</sup> and 0.032 cm<sup>2</sup>, respectively. It indicates that adhesive vinyl films have a great potential for fragmentation and release of MPs to the environment, something that hasn't been studied yet.

### Brittleness

The MPs here investigated had apparently already lost much of their malleability and were more brittle. One piece of evidence is that when handling the removal of any contamination on a fragment, this caused the fragment to break. Another evidence that indicates this characteristic is the presence of many edgy fragments, typical of fragile breaking, as shown in Figure 4.

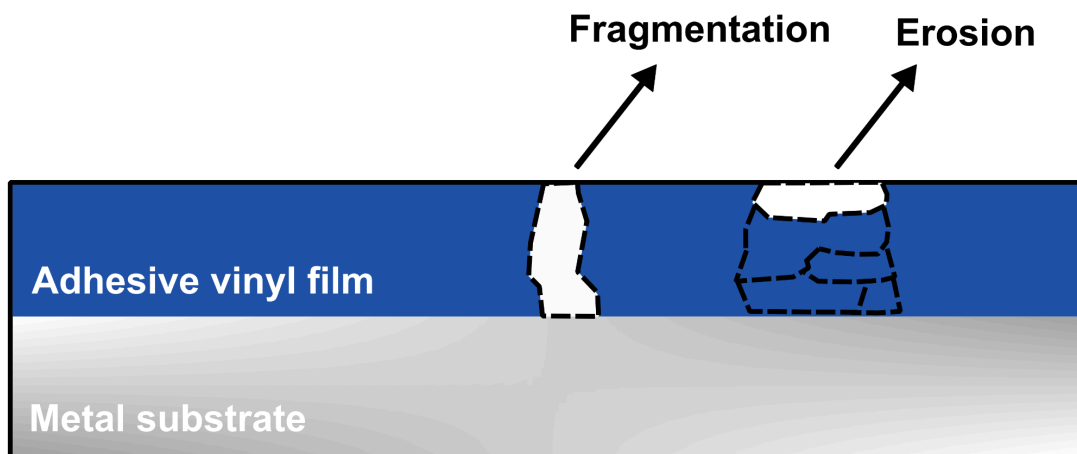
### Thickness

It was possible to measure the thickness of 11 fragments that were facing upwards in the filter. All photos and measures are available in the supplementary material as Figure S1 and in the Spreadsheet S1). The average thickness of the fragments was 30±7 µm, with a maximum of 40 µm, a minimum of 20 µm.

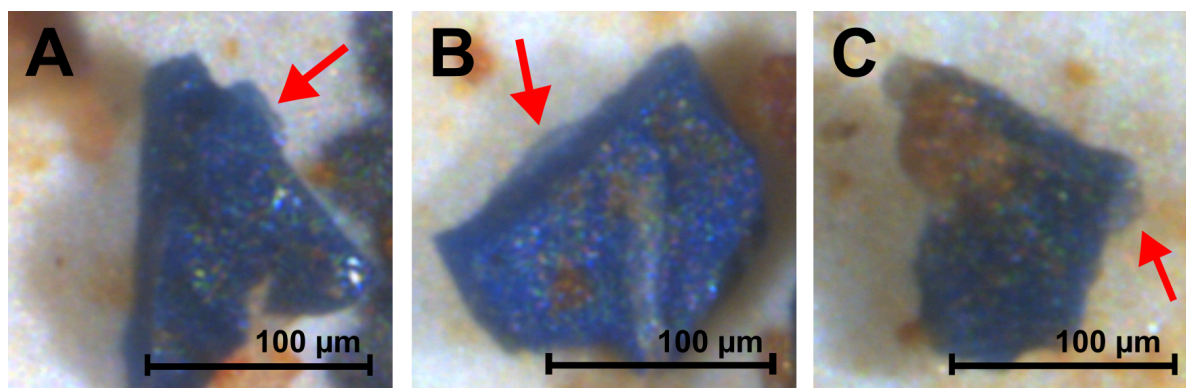
Sometimes different thicknesses could be seen on the same fragment (Figure S1). This could indicate that in addition to the fragmentation, erosion could be also taking place on the formation of MPs, i.e., several MPs may be forming in the same area, as represented in Figure 7.

Furthermore, some fragments had lighter colored regions on the edge (Figure 8), indicating that an adjacent part of the fragment had broken off and left a piece there. These lighter-colored regions could be an indication of smaller thickness and therefore less absorption of the visible light, according to the Beer-Lambert law.

With erosion, the area (4.7 cm<sup>2</sup>) that generated the 78534 fragments/kg of dry soil may be an overestimation, since in that calculation we considered that a certain area of film from which the fragment would have detached would have come off the metal entirely, as named in Figure 7 as “fragmentation”. One major point to note is that although we don't know how much this erosion interferes with the process of MP formation, the scenario is still concerning, since a small area generates a large number of MPs.



**Figure 7.** Scheme representing the supposed dynamics of fragmentation and erosion for the formation of MPs from adhesive vinyl films. Here, we consider fragmentation as the complete breaking of the fragment from the surface to the substrate and erosion as the partial breaking of the fragment's extension. The white areas represent parts of the film that have detached.



**Figure 8.** Blue fragments with lighter colored regions at the edge (indicated by red arrows), pointing out possible erosion through an adjacent part of the fragment that broke off.

#### Method perspective

The shape of polymers is often affected by the reagents used in the process of digesting organic matter. Acidic reagents such as HCl, HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub> and basic reagents like NaOH and KOH can be associated with an increase or decrease in polymer mass and structural changes<sup>21,22</sup>. Because of this, we decided to conduct the first separation by density in order to remove excess organic matter from the sample, most of which is composed of plant remains. We believe this method causes milder changes to the MPs. Additionally, as our protocol is based on the characteristics of the MP source to trace the fragments, we have no problem removing less dense MPs at this stage, which would be a problem for other types of environmental studies.

Nevertheless, we have to consider that the shaking and centrifuging stages are probably those involving the greatest mechanical stimuli between MPs in the sample and soil particles, a phenomenon that could be investigated further in future studies.

Another shortcoming of the technique is related to the optical limit we have for counting and analyzing the particles in the stereomicroscope. Due to this, the number of MPs may be underestimated.

## Controls

All controls adopted throughout the procedural stages and atmospheric deposition monitoring stage in the laminar flow hood registered absence of contamination by blue MPs. We believe that all the steps and precautions taken were important for this result, especially the stage of cleaning the reagents and solvent. When manufactured, these products lack MPs quality control and are packaged in plastic, which further increases the risk of contamination<sup>20</sup>.

In addition to the procedural controls mentioned previously, we replaced the original blue-capped conical tubes, employed in the pilot tests for density separation, with orange-capped tubes. This way, even if the tubes generated some kind of contamination during handling—something we have experienced— we avoid counting this MP as a false positive. Having a specific MP that we can trace, ignoring any other type of contamination that may appear, is one of the main advantages of this method, and it is also what characterizes this procedure as a forensic approach, once we established a relation between the source of contamination and its presence on the environment.

## Final considerations

More research is needed to assess the potential impact of these MPs on biota and the environment. We encourage the establishment of standards that set a maximum limit for the

permanence of this material in the environment, by establishing a time limit for replacement with a new adhesive, as well as the development of new materials that generate minimal contamination by MPs in the environment.

## **Conclusion**

In this work, the potential formation of MPs from a plaque containing adhesive vinyl was identified using a new forensic soil analysis protocol. It was observed that this material generates a significant load of MPs in the environment, which can contaminate the soil to the order of  $5.6 \times 10^4$  fragments  $\text{kg}^{-1}$  of dry soil in the topsoil layer. Furthermore, it was possible to establish a relationship between the area lost from the plaque and the number of MPs generated by analyzing the area of the fragments, where an area of  $4.7 \text{ cm}^2$  generated  $2.6 \times 10^4$  MPs  $\text{kg}^{-1}$  of dry soil, which demonstrates the great potential for the formation of hotspots from vinyl adhesives.

## **Acknowledgment**

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## **Abbreviation**

MP, microplastic.

## Supplementary material

All micrographs of microplastics, Figure S1 for thickness and spreadsheet S1 are available on

[https://drive.google.com/drive/folders/10yBJFHyPf33MeaaOqWs8eogVoXTcCH\\_x?usp=sharing](https://drive.google.com/drive/folders/10yBJFHyPf33MeaaOqWs8eogVoXTcCH_x?usp=sharing)

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### **Synopsis**

This study investigates the potential for forming microplastics from adhesive vinyl films, revealing a production of up to  $5.2 \times 10^{-4}$  microplastics  $\text{kg}^{-1}$  of dry soil from  $1 \text{ cm}^2$  of adhesive vinyl exposed for 8 years, which demonstrates its potential for the formation of microplastics.