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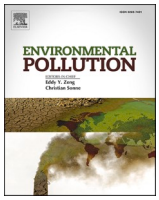

Source apportionment of microplastics in indoor dust: Two strategies based on shape and composition☆

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ABSTRACT

Microplastic (MP) pollution is widely distributed in the environment. However, methods for source apportionment of MPs are still lacking. In this study, the shape and size of 102,860 MPs in indoor dust from 39 cities of China were analyzed by laser direct infrared, and accordingly, a shape index (f_{shape}) based on MP's aspect ratio was developed to assess the contribution of textiles release. In addition, a composition index (f_{comp}) based on the ratio of the mass concentration of polyethylene terephthalate (PET) to polyamide (PA), which were detected by liquid chromatography–tandem mass spectrometry, was proposed. The contribution of textile source and non-textile source to the indoor MPs were also estimated based on f_{comp}. It is estimated by f_{shape} that 43% of MPs in indoor dust was released from textiles. Among the most abundant MPs in indoor dust, 98% of polyurethane, 94% of PA and 92% of PET come from the textile source, 76% of polypropylene and almost all of polyethylene come from the non-textile source. f_{comp} indicates that 83% of PET MPs comes from textile source, i.e., polyester. Considering the application proportion of PET in textile and non-textile industry, it is estimated that 59% of MPs in indoor dust comes from textile source, which is similar to the result obtained by f_{shape}.

1. Introduction

Microplastics (MPs) are plastic particles with sizes less than 5 mm and are recognized as an emerging contaminant widely distributed in the environment (Andrady, 2011; Sedlak, 2017). There are many emission sources of environmental MPs, such as plastic waste stacking and crushing (Zhang et al., 2021), sewage discharge (Gatidou et al., 2019), atmospheric settlement (Evangeliou et al., 2020), etc. However, the study of MP source apportionment is rarely (Grbic et al., 2020; Wang et al., 2021; Wang et al., 2019). Based on morphology and composition, Wang et al. proposed a classification system includes ten types of MPs to trace the sources of MPs in South China Sea (Wang et al., 2019). Nevertheless, this classification may not be specific to all detected MPs. Grbić et al. try to found some indicative MPs, such as nylon (polyamide, PA) fiber, to predict the emission source area of MPs found in the freshwater river (Grbic et al., 2020), however, this method fails to obtain the specific emission proportion among different sources.

Indoor dust is a crucial sink for MPs in living environments. Plastic products such as packing bags, synthetic textiles, and tableware are frequently used in households, which can lead to the release of MPs into indoor dust. Polyethylene terephthalate (PET) and PA have been reported in indoor dust with geomean concentrations of up to 23,000 and 179 mg/kg, respectively (Peng et al., 2020; Zhang et al., 2020). Other MPs, such as polypropylene (PP) and polyethylene (PE), are also frequently detected in indoor dust (Liu et al., 2019). Dust ingestion is an important way of human exposure to MPs (Yan et al., 2022). Because of MPs' toxic threat to environmental organisms and potential health associations with humans (von Moos et al., 2012; Yan et al., 2022; Yang et al., 2019), the occurrence level of MPs in the indoor dust deserved increased attention. Understanding the contributions of various emission sources is critical to appropriately manage the exposure risk of MPs in the indoor environment.

Different types of plastic products can lead to different forms of MP pollution. For example, fiber MPs will be released from textile after washing (Tian et al., 2021), while granular MPs were found in food containers after flushing (Du et al., 2020). Therefore, shape characteristics may help trace emission sources of MPs pollution. Moreover, component fingerprint is commonly used in tracing pollutants. For

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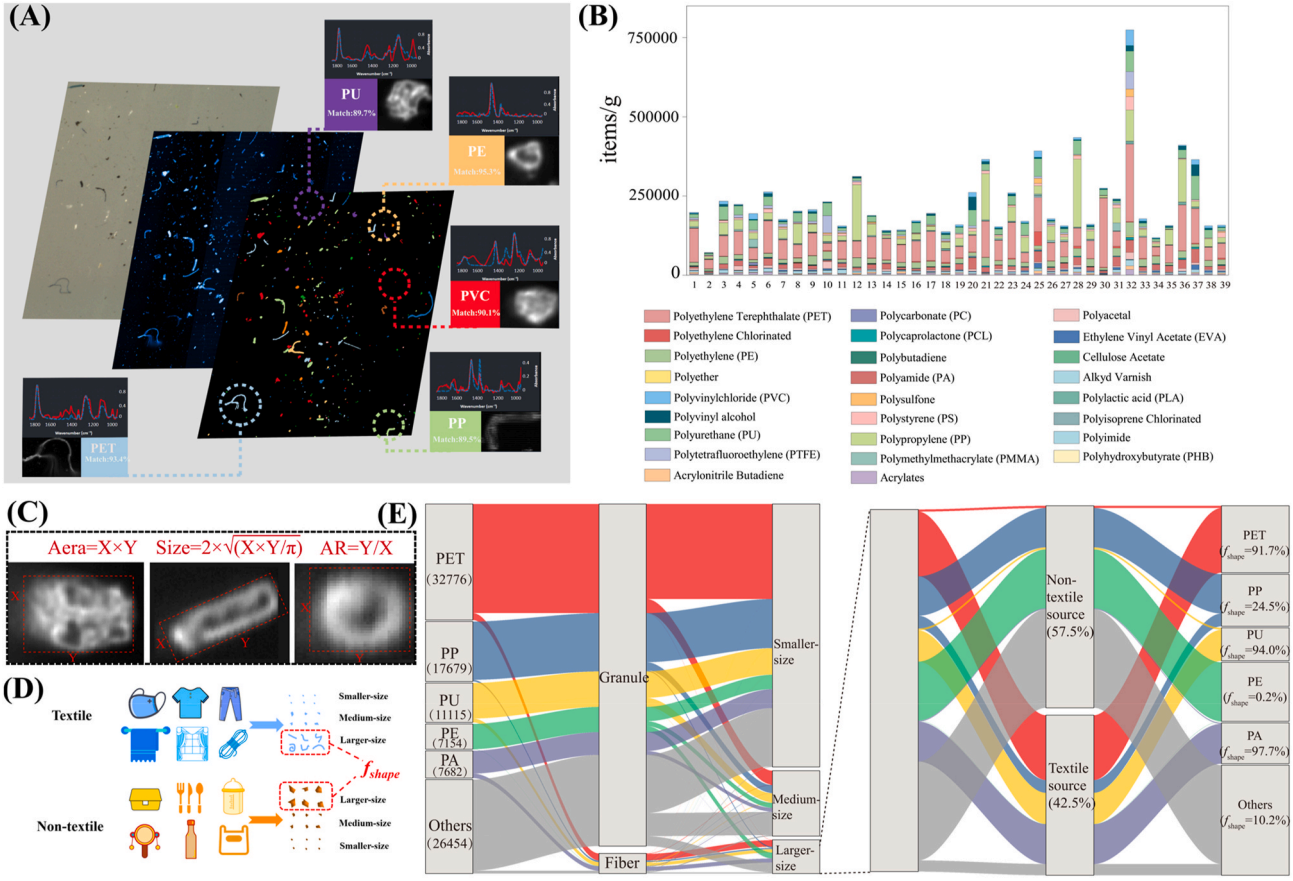

Fig. 1. LDIR scanning process (A) and detection result (B) of 39 indoor dust samples, and equations of area, size, and aspect ratio (AR) for each detected MPs (C). Results of size distribution and f_{shape} of MPs in 39 indoor dusts (D), and small MPs particles released from textiles may not be sufficiently long to be identified as fibers (E). f_{shape} was calculated by Eq. (2). Smaller-size: <50 μm, medium-size: 50–100 μm, larger-size: >100 μm.

instance, different sources would release polycyclic aromatic hydrocarbons (PAHs) with different rings, and the compositional characteristics of PAHs was used to trace their emission sources (Lammel et al., 2020). Plastic polymers also tend to have their own specific application. Polyvinyl chloride is primarily used as engineering plastics in indoor environments, while the main use of PA in indoor is textile (nylon) (Europe Plastics, 2019). Theoretically, component characteristics can be applied to source apportionment of MPs.

To establish a method for source apportionment of MPs in indoor dust, MPs in the collected dust samples were detected by laser direct infrared (LDIR) and liquid chromatography−tandem mass spectrometry (LC−MS/MS). Then, the shape index and composition index of MPs in indoor dust were described, respectively, and the emission sources of all detected MPs were analyzed. The contribution ratios of two indoor sources of MPs were quantified, and the results based on the two indices were compared and validated.

2. Material and method

2.1. Samples and consumables

Thirty-nine indoor samples were collected in August 2020 from households in 39 cities in China (Table S1). The household selection criteria and detailed dust collection process were described in Text S1. The permission was received from each inhabitant, a questionnaire was administered to collect information on, for example, the time spent at home and the frequency of household cleaning. (Fig. S1). SCX and HLB solid phase extraction cartridges were purchased from ANPEL Inc. (Shanghai, China). HPLC-grade Ethyl acetate and ethanol were purchased from Sigma-Aldrich (Shanghai, China).

2.2. Microplastic detection by laser direct infrared imaging

A LDIR chemical imaging system (Agilent 8700, Agilent Technologies Inc., CA) equipped with a library of Microplastics Starter 1.0.1 were used to identify the MPs in samples. Pre-treatment of dust, namely digestion and flotation, was consistent with the previous literature (Zhang et al., 2021). In brief, 0.2 g dust was digested by H_2O_2, and MPs were separated from the dust by a $ZnCl_2$ solution. After pre-treatment, MPs were stored in 1.0 mL ethanol. Then the mixture was homogenized, and 0.1 mL of ethanol containing MPs was dripping on a high reflection window, and MPs were analyzed by LDIR after the ethanol has completely evaporated. The wavelength range of each spectrum was 975–1800 cm^{-1}, and the detection limit of the automatic workflow was 10–2000 μm. Results with a matching degree exceeding 65% were used for compositional identification.

2.3. Microplastics detection by LC−MS/MS

The mass concentrations of polyamide 6 (PA6), polyamide 66 (PA66), and PET polymers in the dust samples were detected by LC−MS/MS, and the detailed methods were described in our previous work (Peng et al., 2020; Wang et al., 2017). In summary, after 0.1 g dust containing PA and PET polymers was depolymerized, monomers of PA6, PA66, and PET, namely, 6-aminocaproic acid, adipic acid, and phthalic acid, were extracted using SCX or HLB solid phase extraction cartridges

or ethyl acetate, respectively, and after purification, these monomers were detected by LC−MS/MS. The masses of PA and PET were calculated using the concentration of monomers and equations. The total concentrations of PA6 and PA66 were recorded as the PA concentrations in the indoor dust.

2.4. Quality assurance and quality control

To avoid MP pollution, plastic containers were eliminated during the experiment, and the glass utensils were rinsed with acetone and methanol and muffled at 500 °C for 12 h. All procedures before detection by LDIR and LC-MS/MS were performed in a fume hood. Procedural blanks were analyzed with every batch of 15–20 samples for detection by both LDIR and LC-MS/MS, and the background was subtracted in the quantification of MPs.

2.5. Statistical analysis

Origin 2022 was used for the data statistics and graphical plotting. Linear regression and Pearson correlation analysis were performed by SPSS 19.0 Statistics to analyze the relationship between the abundance of MPs and living habits of inhabitants. The significance test was set to a p-value of <0.05.

3. Results and discussion

3.1. MPs in indoor dust and component distribution

Totally 254,682 particles (0.01–2 mm) in the 39 dust samples were identified by LDIR (approximately 6530 items per sample) (Fig. 1A&B), of which 102,860 were identified as MPs. There were 69,100–776,000 items/g of MPs, with a geomean (GM) of 207,000 items/g, in the indoor dust of this study, which is much higher than the distribution level of MPs in soils or sediments ($10-10^3$ items/g) (Corcoran et al., 2020; Mani et al., 2019; Zhang and Liu, 2018; Zhou et al., 2019). Twenty-six plastic polymers were identified (Fig. 1B), and PET, PP, polyurethane (PU), PE, and PA were the five most abundant polymers, with abundances of 22, 600–245,000, 5,590–215,000, 4,720–75,500, 5,640–42,700, and 3, 130–43,800 items/g, respectively. Through oral and nasal inhalation, high levels of MPs in indoor dust pose a high risk of human exposure. This can be explained by the frequent use of plastic products with these polymers in indoor environments, such as synthetic fiber fabrics (PET, PP, PU, PA), plastic containers (PET, PP, PE), and packaging materials (PP, PE) (Cao et al., 2014; Chen et al., 2022; Ge et al., 2015). The most abundant MP polymer compositions in dust have different applications. Therefore, it is necessary to quantify the contribution of different sources to MPs.

3.2. Shape index of MPs in the indoor dust and the source analysis

LDIR can provide composition information and capture images of each detected particle, and shape indexes of different MPs were attempted to be described based on the composition and appearance information. According to the appearance of detected MPs (n = 102,860) in the indoor dust, MPs can be divided into two categories, i.e. synthetic fibers and non-fiber granules, and they are assumed to come from textile and non-textile sources, respectively. MP's shape can be characterized by its aspect ratio (AR), which can be calculated automatically by LDIR, according to Equation (1).

$$AR = Y/X \quad (1)$$

where X and Y are the width and length of detected particles, respectively (Fig. 1C). Particles with an AR greater than 3 were considered as fibers, and those with an AR lower than 3 were considered as non-fibers (Cole, 2016). Notably, some fibers may bend on glass slides during detection by LDIR, causing these fibers' AR to be lower than 3. In the subsequent correction, the Y and X of these MPs were manually measured through their scanned images, and the ARs were recalculated. A shape index, f_{shape}, was proposed as Eq. (2), which describes the proportion of the fibers' number to the total number of MPs. Assuming that all the fibers were contributed by the textile source and all the non-fibers came from the non-textile source, f_{shape} can be used to identify the contribution of textile and non-textile to indoor MPs.

$$f_{shape} = \text{Number of fibers} / \text{Total number of MPs} \times 100\% \quad (2)$$

Of the 102,860 MPs with shape information, only 4.5% were directly identified as fibers (AR>3), indicating a contribution of 4.5% of textile to indoor MPs. This proportion is obviously lower than the expected contribution of synthetic fibers to the indoor MPs. For example, the daily use of PA was mainly textiles (Ge et al., 2015). However, 83.5% of the detected PA was non-fibers (Fig. 1D). This abnormal result is due to that a great part of small MPs particles released from textiles may not be sufficiently long to be identified as fibers, as illustrated in Fig. 1E. Smaller-sized MPs (<50 μm) accounted for 72.4% of all MPs, and the non-fibers with AR<3 were account for 99.1% of these small MPs (Fig. 1D). This may cause the under estimation of f_{shape}, which cannot properly represent the contribution of textiles. The small non-fibers MPs can come from the breakage of fibers, and can no longer maintain the shape of the fibers. In comparison to the small MPs, larger MPs tend to come directly from the shedding of plastic and can better reflect their original source of plastic products. In the larger-sized PA MPs of >100 μm, 97.7% of the PA particles of this study were fibers (Fig. 1D). This is in line with the expected use of PA polymers in indoor environments, i.e. 75.3% of PA is used to produce nylon fiber (Statista, 2022a; Statista, 2021), and the predominant PA usage indoor is textile. The abundance of larger-sized PA MPs was significantly positively correlated with that of the smaller-sized PAs (Table S2, $p = 0.001$), suggesting that these smaller sized granular PAs have the same source as larger-size PA, that is, textiles.

Therefore, only f_{shape} of the larger-sized MPs (>100 μm) was considered in assessing the contribution of textile release to the indoor MPs, which indicates that 42.5% of the indoor MPs comes from textiles (Fig. 1D). Accordingly, among the most abundant polymers, most of the larger-size PET (f_{shape} of 91.7%), PU (94.0%), PA (97.7%) were fibers (Fig. 1D), demonstrating that these three MP polymers, were mainly from textiles. The dominant usage of PET, PA, and PU are to produce synthetic fibers of polyester, nylon, and spandex. Larger-sized PP MPs consisted of fibers (24.5%) and non-fibers (75.5%), suggesting a mix source of PP MPs in indoor dust. Larger-sized PE MPs were also observed, of which 99.8% was non-fiber ($f_{shape} = 0.02\%$, Fig. 1D). PP and PE are the world's highest annual output of plastics (approximately 50 Mt) (Europe Plastics, 2019), whose main application are in packaging materials and containers. A small part of PP is used to produce synthetic fiber (e.g., 3.0 Mt in 2021) (Statista, 2023a), which can explain the textile source of PP MPs.

The shape index fluctuates wildly among samples (13.4%–74.1%, Fig. S2). There was no obvious relationship between the source of MPs in households and the geographical location of the city in which the households were located. Based on the results of the householder questionnaire (Fig. S3), f_{shape} was positively correlated with the time spent at home and the preference for using synthetic textiles ($p = 0.025$ and 0.12, Table S3) and was negatively correlated with the time windows opening and the preference for single-use plastics ($p = -0.023$ and -0.17, Table S3). Long time spent in indoors will increase the probability of fiber shedding, while single-use plastics tend to release non-fiber MPs. This also shows that f_{shape} is reasonable as an index of source apportionment.

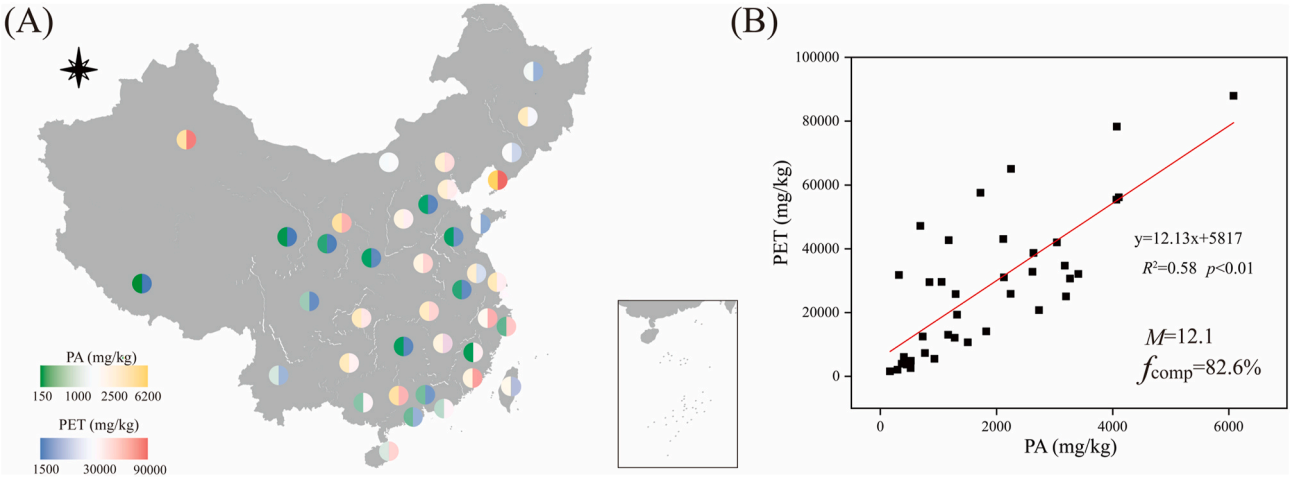

Fig. 2. Mass concentration (A) and component index (B) of PET and PA in the indoor dust. *M* was obtained based on Eq. (4) and linear fitting of concentration ratios of PET to PA in 39 samples, and f_{comp} was obtained by *M* and Eq. (5).

3.3. Component index of the PET and PA and the source analysis

The index of component distribution is commonly used for the source apportionment of some contaminants in different environments. For instance, fluoranthene (Fla), pyrene (Pyr), indenol[1,2,3-c, d]pyrene (IcdP), and benzo[g, h, i]perylene (BghiP) were used as indicative components of PAHs, and Fla/(Fla + Pyr) ratio below 0.4 and IcdP/(IcdP + BghiP) ratio below 0.2 represent a petroleum source of PAHs (Qiao et al., 2021). In this study, a component index of polymers is tried to be established to analyze the sources of indoor MPs.

PA and PET are two of the most abundant MP components in the indoor dust (Fig. 1D). The annual production of raw PA in 2021 was 7.80 Mt worldwide (Statista, 2022a), of which 5.87 Mt were used to produce nylon fiber (Statista, 2021). Only a small part of PA was used to produce engineering plastics (Europe Plastics, 2019), which is seldomly used indoor. Therefore, PA MPs in the indoor dust is thought to mainly originate from textile products. Differently, a great amount of PET polymer is used in polyester fiber production (global annual production of 60.5 Mt) (Statista, 2022b), while its usage in the packaging manufacturing, such as plastic bags and films, is also high (35.3 Mt) (Statista, 2023b). These packaging materials are frequently used in the indoor environment, and are potential sources of MPs (Geyer et al., 2017).

Assuming the usage frequency of polyester and nylon fibers in the indoor environment can be expressed by their production, and these two fibers release MPs in the same way (as there's no available information of the releasing process), an ideal mass ratio of polyester to nylon (M_{ideal}) can be calculated according to Eq. (3).

$$M_{ideal} = P_{polyester}/P_{nylon} = 10 \quad (3)$$

As the global annual production of polyester ($P_{polyester}$) and nylon (P_{nylon}) were reported as 60.5 and 5.87 Mt (Statista, 2022a; Statista, 2022b), M_{ideal} of 10 can be estimated.

$$M = C_{PET}/C_{PA} \quad (4)$$

where C_{PET} and C_{PA} are the mass concentrations of PET and PA in dust, respectively. Therefore, a component index of PET (f_{comp}), describing the contribution of PET fibers (polyester) to PET MPs of indoor dust, can be calculated according to Equation (5).

$$f_{PET} = M_{ideal}/M \times 100\% \quad (5)$$

If the f_{PET} is close to 1, that is *M* is close to 10, indicating that PET MPs in dust are mainly from textile source. On the contrary, the farther *M* deviates from 10, the greater the contribution of non-textile sources to PET in indoor dust.

In this study, mass concentrations in the samples were 2,080–78,300 mg/kg (PET) and 166–6,080 mg/kg (PA) (Fig. 2A), respectively, and the mass ratio of PET to PA (*M*) obtained by the linear fitting of the 39 indoor dust samples was 12.1 (Fig. 2B). Accordingly, f_{comp} of PET can be calculated as 82.6% (Eq. (5)), indicating that PET MPs in indoor environments were mainly derived from textiles. This is consistent with the estimated result of the shape index of PET MPs, which suggests 91.7% of PET MPs in indoor dust of this study were textile-source (Fig. 1D).

Based on the occupancy of PET polymers in textile and non-textile industries, the results of PET source apportionment in indoor dust can be extrapolated to the total plastic polymers. It is assumed that 83% of indoor PET MPs come from textiles and 17% from non-textiles (mainly packaging materials). The occupancy of PET fiber in chemical fiber market is 82% (60.5 Mt in 73.4 Mt, 2018) (Statista, 2022b), while PET packaging materials in plastic packaging industry share of 25% (35.3 Mt in 144 Mt, 2018) (Europe Plastics, 2019; Statista, 2023b). Assuming that other packaging plastics and synthetic textile fibers have the same ability to produce MPs as PET, then the source of synthetic textile of MPs in indoor dust (f_{comp}) can be calculated as 58.5% by Eq. (6), which is comparable to the result of the f_{shape} that 42.5% of the indoor MPs comes from textiles.

$$f_{comp} = (P_{tex}/P_{tex-PET} \times f_{PET}) \Big/ \left[(P_{tex}/P_{tex-PET} \times f_{PET}) + P_{pac}/P_{pac-PET} \times (1 - f_{PET})\right] \times 100\% \quad (6)$$

Meanwhile, the mass concentrations of PET and PA can be accurately detected by LC-MS/MS according to our published methods (Peng et al., 2020; Wang et al., 2017). The detected mass ratio of the two polymers (*M*) in dust can be calculated by Eq. (4),

Where P_{tex}, $P_{tex\text{-}PET}$, P_{pac}, and $P_{pac\text{-}PET}$ are the global plastic production of synthetic textile fibers, PET fibers, packaging plastics, and PET packaging plastics, respectively.

The f_{comp} doesn't require the size information of MPs, and the LC-MS detection of indicative MPs (PET and PA) is more efficient than LDIR. Notably, because the choice of synthetic textile by consumers is either random or has a clear preference, it is not appropriate to compare the M value of a single household indoor dust with the M_{ideal}, which was derived from the global production of synthetic fiber. Therefore, f_{comp} is significant only for the analysis of a large sample size in a large-scale study, whereas the study of a small sample size in a specific environment lacks applicability.

Author contributions statement

Chu Peng: Conceptualization, Methodology, Data collection and analysis, Writing–Original Draft Preparation, Visualization; **Xinyi Zhang**: Data collection and analysis; **Mengxi Li**: Data Collection; **Yuan Lu**: Data Collection; **Chunguang Liu**: Writing-Review & Editing; **Lei Wang**: Writing-Review & Editing, Project administration, Supervision, Funding Acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

I have shared the link to my data.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2023.122178.

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