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Occurrence of azo-dyes, plasticizers, and PAH-bound microplastics: an emerging source and sink for hazardous compounds in indoor environments?

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Abstract
Indoor environments act as significant reservoirs for a wide range of potentially harmful substances, with microplastics (MPs) gaining increased attention in recent years. This study offers valuable insights into the role of MPs as both source and sink for hazardous contaminants within indoor environments. The analytical techniques employed encompass micro-Raman spectroscopy and high-resolution mass spectrometry. The results revealed a strong correlation between the levels of MPs with the size of the residential population. The dominant shape, colour and polymer type of MPs were fibre, white/transparent and polyamide, respectively. The risk of exposure to microplastics through ingestion was 22.5 times higher for infants compared to adults. In settlements where inhabitants utilized firewood as supplementary cooking fuel, an interesting observation was made: pyrene, which is a specific type of polycyclic aromatic hydrocarbon (PAH), was observed to be adsorbed by polystyrene (PS) MPs. This finding illustrates the capacity of MPs to serve as reservoirs for PAHs within indoor environments. Furthermore, the observation of PAH absorption onto MPs in households using firewood highlights a previously underexplored interaction between microplastics and pollutants in indoor settings. Organic micro-pollutants like di-ethyl hexyl phthalate (DEHP), monobutyl phthalate (MBP), 1,2-dihydro-2,2,4-trimethylquinoline and benzisothiazolone (BIT) were detected in both dust and MPs. It underscores the potential for organic micro-pollutants to move between settled dust and MPs within indoor settings, emphasizing the need for further research in this area.

Keywords Indoor dust · Residential settlements · Microplastics · PAHs · Plastic additives

Introduction

Over the past few decades, the use of plastic has increased exponentially. However, inadequate plastic waste management measures have caused their widespread contamination across various environmental matrices. Microplastics (MPs) are plastics having a size < 5 mm (Vethaak and Legler 2021). Based on the origin, MPs are classified into primary and secondary MPs. Primary MPs can be formed as a by-product of an industrial process or made on purpose to support the requirements of a product. Meanwhile, secondary MPs are formed by the breakdown of larger plastic items by the action of environmental factors (Lehtiniemi et al. 2018; Rillig 2012). Recently, another fraction of plastics known as nano plastics (NPs) has received much attention. There is no particular standardized cut-off size for NPs yet; however, based on some available literature, plastic particles can be considered as NPs if their particle size is < 1 µm or 0.001–0.1 µm (da Costa et al. 2016; Koelmans 2015; Rios Mendoza et al. 2018).

An array of auxiliary compounds is added throughout the manufacture of plastic products. These compounds help to improve their physical and chemical characteristics. As these compounds are not typically bound to polymers, they can migrate into the surrounding matrix (Kitahara and Nakata

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2020). Some of these auxiliary compounds are endocrine disruptors or carcinogens (Abdi et al. 2021; Hill et al. 2001; Kitahara and Nakata 2020; Warren et al. 1982). Most of the literature on microplastic (MP) pollution is focused on aquatic environments. However, the knowledge concerning their impact on indoor air quality is rather limited. Of late, there is an emerging interest in the investigation of MPs in indoor environments, as humans spend most of their lifetime in these spaces. The available literatures have showed that indoor settled dust may be a good sink for MPs (Dris et al. 2017; Liu et al. 2019a; Soltani et al. 2021; Zhang et al. 2020a). Settled dust is a reservoir for chemicals released by different sources in indoor spaces (Dalvand et al. 2023; Sreejith et al. 2021). It acts as a layer between the surface and air compartments (Shin et al. 2014). Thus, compounds present in these two (surface and air) compartments often migrate into the dust. High contaminant load can occur in settled dust due to lower degradation and ventilation rates indoors (Chao et al. 2016).

A substantial number of synthetic fibres in indoor environments pose the risk of inhalation or ingestion by occupants (Dris et al. 2017; Soltani et al. 2021). These fibres may host an assortment of hazardous contaminants, including dyes, flame retardants and pesticides. Nevertheless, information regarding the nexus between microplastics and their associated constituents in indoor environments under ambient conditions is notably sparse. Plastic additives, such as plasticizers, flame retardants, stabilizers, antioxidants, UV stabilizers, heat stabilizers, curing agents, blowing agents, biocides, colorants and organic and inorganic pigments, have the potential to diffuse within MPs and subsequently be released into the surrounding environment they come into contact with. This diffusion process is predominantly governed by Fick's law (Rahman et al. 2021) relying on the concentration gradient/flux between the polymer substrate and the surrounding medium. Moreover, the structural characteristics of additives, such as molecular mass, can significantly impact their diffusion rates within a polymer (Cheng et al. 2020). Factors like polymer thickness, crystallinity and surface structure also play pivotal roles in governing the migration rate of additives (Hahladakis et al. 2018). Within the dust, MPs may encounter various physical, chemical and biological stressors that could expedite the diffusion rate of additives into the dust matrix. Consequently, investigating indoor dust and MPs concurrently holds the potential to yield valuable insights into the intricate dynamics of plastic-associated compound transport between these two matrices.

Methodology

Study area and sample collection

The indoor dust accumulated on different indoor surfaces such as ceiling fans, window edges and floors was collected from 35 residential settlements (homes) in the Greater Cochin, India, during January 2021. The details regarding the location and number of occupants in the settlements are provided in supplementary material (Table. S1). Greater Cochin is an economic hotspot in the state of Kerala, hosting various commercial and industrial setups. It hosts a high population density of ~ 2531 people/km^2. The dust samples were gently swept onto a paper bag using a non-plastic (horsetail) brush. A representative dust sample was obtained from each settlement by combining the sub-samples collected from different locations, such as living room, bedrooms and kitchen. Following each sample collection, the brush was carefully washed with acetone to prevent any contamination and then dried for duration of 10 min. The dust collection bags were placed in tight zip lock covers after covering it with aluminium foil. In the laboratory, the samples were dried in an oven at a temperature not exceeding 30 °C for a period of 48 h (Rodrigues et al. 2019). The dried samples were sieved through a 700-µm stainless-steel sieve, and the samples were quickly subjected to further analysis.

Chemicals

Five reference polymers were used to determine the polymer type of MPs in the samples. Nylon 6,6 (PA) (CAS#:32,131–17-2), polyethylene (PE) (CAS#:9002–88-4), polypropylene (PP) (CAS#:9003–07-0), polystyrene (PS) (CAS#:9003–53-6) polyvinyl chloride (PVC) (CAS#:9002–86-2) (±)-metoprolol (+)-tartrate salt (CAS: 56,392–17-7), mono-butylphthalate (MBP) (CAS: 131–70-4), pyrene (CAS: 129–00-0), HPLC grade methanol (CAS: 67–56-1) and LCMS grade acetonitrile (CAS: 75 -05–08) were obtained from Sigma-Aldrich (USA). The ultra-pure water (18.2 MΩ.cm) used for the experiments was obtained from a Milli-Q ultra-pure system (ELGA LabWater, USA).

MP analysis

About 1 g of dried dust samples was weighed and transferred into a clean glass beaker. The organic matter in the samples was removed using wet peroxide oxidation (WPO) with 30% hydrogen peroxide solution. About 30 mL of NaCl solution ($d = 1.3$ g/mL) was added to the mixture for density separation, and the mixture was stirred vigorously with a glass rod for 5 min and was left undisturbed for 24 h. The resulting supernatant was filtered through a Whatman glass fibre filter paper (25 mm). Filter papers were dried at room temperature, and the shape and colour of MPs (100–700 µm) attached to it were monitored using a stereomicroscope (Nikon SMZ800N). MP suspects were transferred onto a glass slide with metal tweezers for Raman analysis. The analysis was carried out using a Micro-Raman spectrometer (WITec Alpha 300RA, Germany) equipped with a 532-nm

excitation laser, a grating of 600 groves/mm and 20 × objective. The instrument was calibrated using spectra of a silicon wafer (standard provided by WITec, Germany).

In this study, we targeted five polymer types (polyamide (PA), polyethylene (PE), polypropylene (PP), polystyrene (PS) and polyvinyl chloride (PVC)) that find wide application in consumer products. The characteristic Raman signals used for the identification of MPs are ~ 2875, 2903 and 2928 cm^{-1} for PA; ~ 402, 2842, 2886 and 2961 cm^{-1} for PP; ~ 1059 cm^{-1} for PE; ~ 1000, 2855, 2907 and 3058 for PS and ~ 695 cm^{-1} for PVC (Kniggendorf et al. 2019; Sobhani et al. 2019). We further confirmed the polymer type of MPs by comparing the sample spectra with that of the reference polymers (polyamide (Nylon 6,6), polyethylene, polypropylene, polystyrene and polyvinyl chloride) (Figure S1).

For quality assurance and quality control, we followed the protocol as mentioned in an earlier report (Dehghani et al. 2017). The apparatus used for the MP extraction was acid washed and thoroughly cleaned with de-ionized water before and after each extraction. A glass petri-dish was kept open on top of the worktable within the laboratory to monitor the airborne MP contamination during the extraction procedure. The extraction solvents were used as blank samples and were subjected to the same extraction procedure used for the dust samples. Micro-Raman spectroscopic analysis of particles settled on petri-dish and glass fibre filter did not indicate any airborne/solvent MP contamination.

LC–Q–ToF analysis

About 0.1 g of dried dust sample was weighed and transferred into a glass centrifuge tube. Fifteen millilitres of HPLC grade methanol (extraction solvent) was added to the sample. Each dust–solvent mixture was sonicated for 30 min, followed by centrifugation at 4500 rpm for 5 min. The supernatant was concentrated by purging nitrogen gas, and the resulting solution was subjected to MS analysis. A similar protocol was used for the analysis of blank samples. Chromatographic separation was performed for the analysis using a reversed-phase BEH C18 column (50 mm × 2.1 mm × 1.7 µm). Flow rate was maintained at 0.3 mL min^{-1}. The mobile phase consisted of gradient elution of 0.1% formic acid in water (A) and ACN (B). The elution set at different time intervals is as follows, at 0 min, the mixture was 95% A and 5% B; subsequently, at 6 min, we shifted it to 5% A and 95% B, finally, at 9 min, we returned to 95% A and 5% B respectively. The injection volume was kept at 10 µL, and the desolvation gas flow and temperature were sustained at 9000 L/h and 350 °C, respectively. The m/z range and collision energy were set between 50 and 1000 Da and 5 and 30 eV, respectively. We used a similar protocol for the analysis of additives in MPs. The MPs were separated from dust samples using NaCl solution, but were not subjected to wet peroxide treatment to prevent the degradation of plastic additives (Fig. 1).

The flow chart (Fig. 1) presents an overview of the method followed for the experiments.

The identification of unknown compounds was conducted using accurate mass, elemental composition and MS/MS fragmentation data obtained through collision-induced dissociation with electrospray ionization in both positive (ESI +) and negative (ESI −) ionization modes. A full scan analysis of HPLC grade methanol, which served as the extraction solvent and field blank (paper bag, aluminium foil and zip-lock covers), was performed with a mass window ranging from 50 to 1000 Da in both ionization modes. The same protocol was followed for sample analysis. Similar peaks that appeared in the solvent, blank and sample were removed to isolate the peaks corresponding to unknown compounds. The charge state of the parent ion (whether single or multiple) was determined by examining the isotopic mass pattern. The tentative identification of compounds was based on the elemental composition predicted by Masslynx (V4.1), and the online database ChemSpider was utilized for further analysis.

Two analytical standards mono-butylphthalate (MBP) (CAS: 131–70-4) and (±)-metoprolol (+)-tartrate salt (CAS: 56392–17-7) were used to confirm the presence of identified compounds in MP and dust matrices. The linearity of the method was assessed by preparing standard solutions at five different concentrations (0.2–1 ppm). The coefficient of determinant (r^2) for the target analytes (MBP and metoprolol) is ≥ 0.99 as shown in figure S2 and S3. The method detection limit (MDL) and limit of quantification (LOQ) were calculated using Eqs. (1) and (2) respectively:

$$\mathrm{MDL(method\ detection\ limit)} = 3.3 \times (\mathrm{S.D.\ of\ the intercept/slope}) \quad (1)$$

$$\mathrm{LOQ(limit\ of\ quantification)} = 10 \times (\mathrm{S.D.\ of\ the intercept/slope}) \quad (2)$$

where,

S.D (standard deviation) = S.E (standard error) of intercept × $\sqrt{N}$.

N = number of tests.

The MDL and LOQ for MBP were determined to be 0.005 ppm and 0.03 ppm respectively, while for metoprolol, they were 0.08 ppm and 0.254 ppm respectively. To monitor laboratory contamination, a procedural blank was analysed after each sample runs. The intra-day and inter-day relative standard deviation (RSD) values for MBP and metoprolol were both below 10%.

Statistical analysis

The calculations were performed using Microsoft Excel 2016 and IBM SPSS statistics 22 software. The

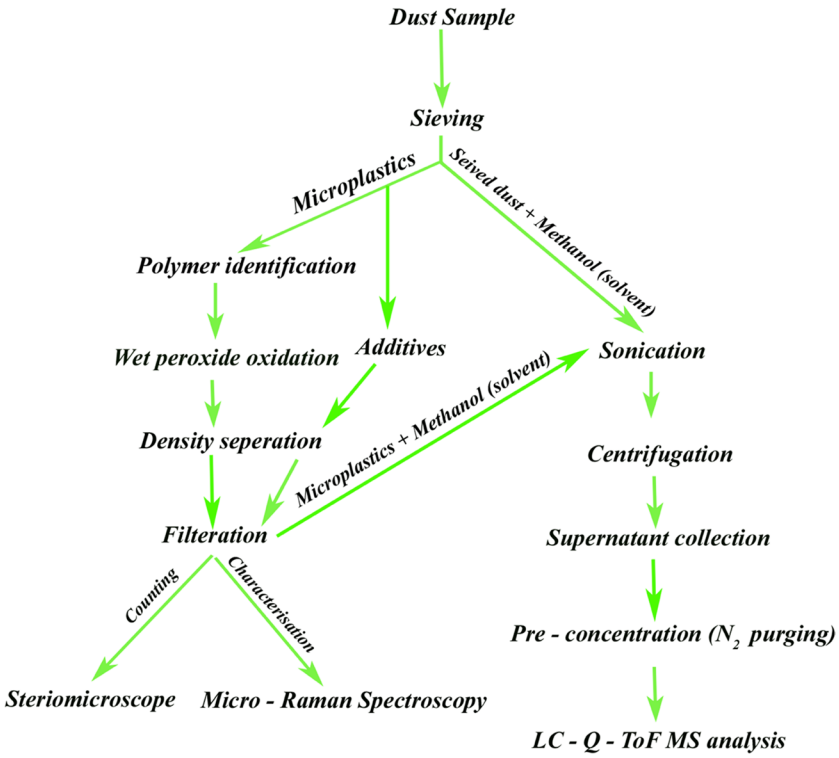


Fig. 1 Flow chart of the experimental protocol followed for the analysis

Shapiro–Wilk test was used to test the distribution of MPs across the sampling locations. Spearman rho correlation was applied to test the relationship between various groups. The tests were considered statistically significant when p values were less than 0.01.

Human exposure to MP via ingestion

The human exposure to MPs via ingestion was calculated for different age groups (infants (0.5–1 year), toddlers (2–3 years), children (6–12 years), teenagers (12–21 years), adults (≥ 21)) based on the Eq. (3) given below:

$$EDI_{ingestion} = \left(C * f * m_{di}\right)/BW \quad (3)$$

C = concentration of MPs in indoor dust (MPs kg^{-1} dust); f = indoor exposure fraction, m_{di} = indoor dust ingestion rate (kg day^{-1}) and BW = body weight (kg). The values used for calculating f and m_{di} for infants, toddlers, children, teenagers and adults were 0.88, 0.79, 0.79, 0.88 and 0.88 and 0.0004, 0.00004, 0.00003, 0.00002 and 0.00002 kg day^{-1}, respectively (Nematollahi et al. 2022). The average BW for infants, toddlers, children, teenagers and adults in Asian countries is estimated to be 5, 19, 29, 53 and 63 kg, respectively(Nematollahi et al. 2022).

Results and discussion

MP levels in indoor dust

MPs were detected in all the indoor dust samples ($n = 35$). An overview of shape, colour and polymer type of MPs in dust samples is presented in Table 1 and Fig. 2. The minimum and maximum concentrations of MPs in the dust samples ranged from 9 ± 3.2 (H1 and H6) to 23 ± 3.2 (H25) MPs g^{-1} dust. Interestingly, a significant positive association ($\rho = 0.691$, $p < 0.01$) was obtained between the number of occupants in each settlement with total MP concentration. Thus, the resident population number was observed to contribute to MP abundance in the study area.

The shape of microplastic particles (MPs) can offer valuable insights into their origins. For instance, fibrous particles are often associated with synthetic fabrics (Klein et al. 2015). Fibres were the prevailing form of microplastics (MPs) observed in the samples, as indicated in Table 1. In the majority of the sampling sites, fibres accounted for over 50% of the composition, as illustrated in Fig. 2a. It is noteworthy that the distribution of fibres in the samples followed a normal distribution ($p > 0.01$), indicating a consistent prevalence of fibres in the composition across the various settlements. These findings emphasize the possible role of fabrics as a significant source of

Table 1 Outline of characteristics (shape, colour and polymer type) of MPs in dust samples (H1–H35)

Characteristics	Per cent	Total number of MPs monitored in dust (H1-H35) samples (*N*)	Min (*N*)	Max (*N*)	Mean (*N*)	Median (*N*)	S.D
ΣMPs	100	476	9	23	13.6	14	3.2
Shape							
Fibre	66.2	315	5	15	9	9	2.4
Fragment	33.8	161	2	9	4.6	5	1.7
Polymer							
Polyamide	36.1	172	2	9	4.9	5	1.7
Polyethylene	20.8	99	1	5	2.8	3	1.2
Polypropylene	25	119	0	8	3.4	4	1.6
Polystyrene	7.1	34	0	4	1	1	1
Polyvinyl chloride	10.9	52	0	5	1.5	1	1.2
Colour							
White/transparent	44.5	212	2	14	6.1	5	2.6
Red/pink	14.9	71	0	7	2	2	1.8
Blue	13	62	0	5	1.8	2	1.5
Green	15.3	73	0	6	2.1	2	1.7
Orange/yellow	5.3	25	0	5	0.7	0	1.1
Black/grey	6.9	33	0	5	0.9	0	1.4

N number of MPs from 100 to 700 µm

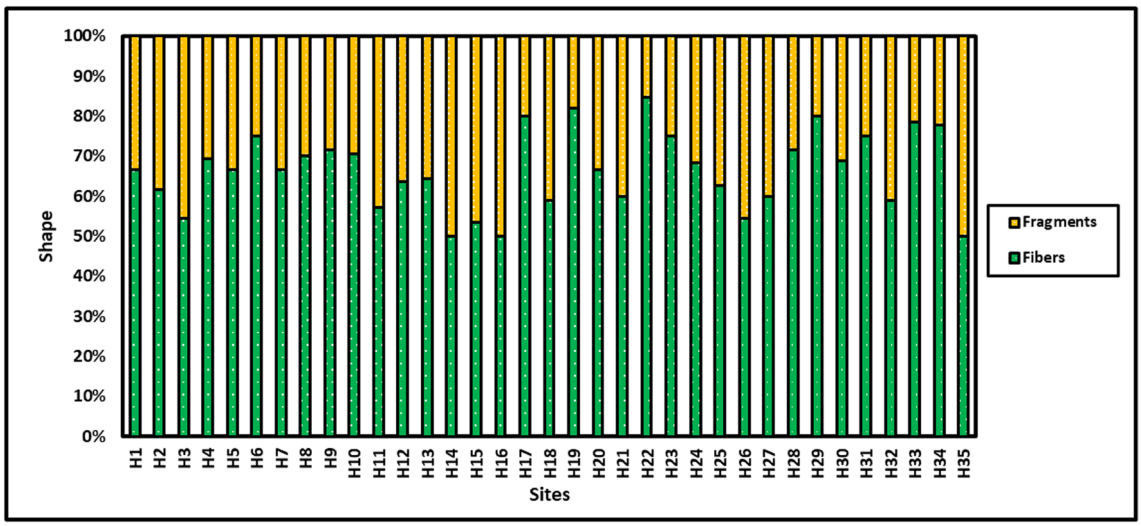
fibrous microplastics within indoor environments. (Dris et al. 2017).

The composition of MPs based on polymer type followed the order: PA (36.1%) > PP (25%) > PE (20.8%) > PVC (10.9%) > PS (7.1%) (Table 1). Representative Raman spectra of different MPs identified from the samples are provided in the supplementary material (Figure S1). The most abundant polymer types at the sampling sites were PA and PP MPs, as illustrated in Fig. 2b. The distribution of both PA and PP MPs followed a normal distribution ($p > 0.01$) pattern across the sampling sites. Residents in the study area commonly practice outdoor laundry drying during the day by suspending garments on nylon ropes. It is essential to acknowledge that prolonged exposure to UV radiation from sunlight can lead to the degradation and weakening of both fabric materials and nylon ropes (Nematollahi et al. 2022). Consequently, when the clothes are dried in this manner, they have the potential to release a significant number of microplastic (MP) fibres. Meanwhile at night, indoor drying methods are employed, such as hanging fabrics on ropes or spreading them on the floor, often with the aid of ceiling fans to expedite the drying process. These activities create conditions conducive to the shedding and dispersion of microplastics from the fabrics into the surrounding environment. Additionally, the COVID-19 pandemic has led to widespread use and, in some cases, reuse of polypropylene-based protective masks, including surgical and N95 masks. As these masks age, they can become a source of fine polypropylene (PP) fibres.

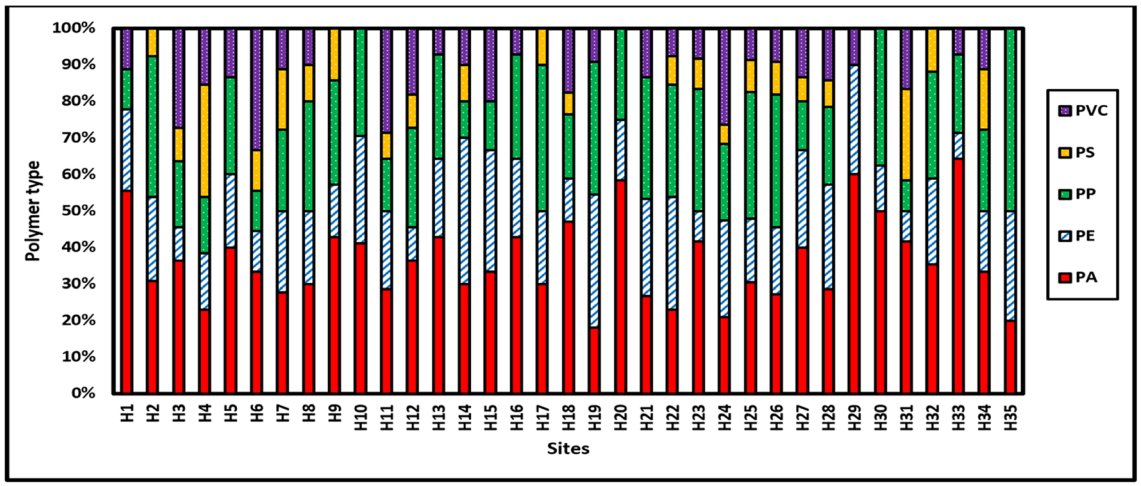
Interestingly, the study results unveiled a significant positive correlation between the number of PA fibres and overall fibre numbers ($\rho = 0.529$, $p < 0.01$), as well as between the number of PP fibres and fibre numbers ($\rho = 0.462$, $p < 0.01$). Furthermore, there was a notable positive association between the total microplastics concentration and fibre numbers ($\rho = 0.827$, $p < 0.01$), as well as between fibre numbers and the number of occupants ($\rho = 0.546$, $p < 0.01$).

The positive correlation observed between polypropylene (PP) and polyamide (PA) fibres with fibrous-shaped particles can be attributed to their extensive use in the manufacturing of synthetic textiles. These findings strongly suggest that synthetic fabrics, particularly those used in clothing, represent the primary source contributing to the presence of fibrous microplastics within these settlements.

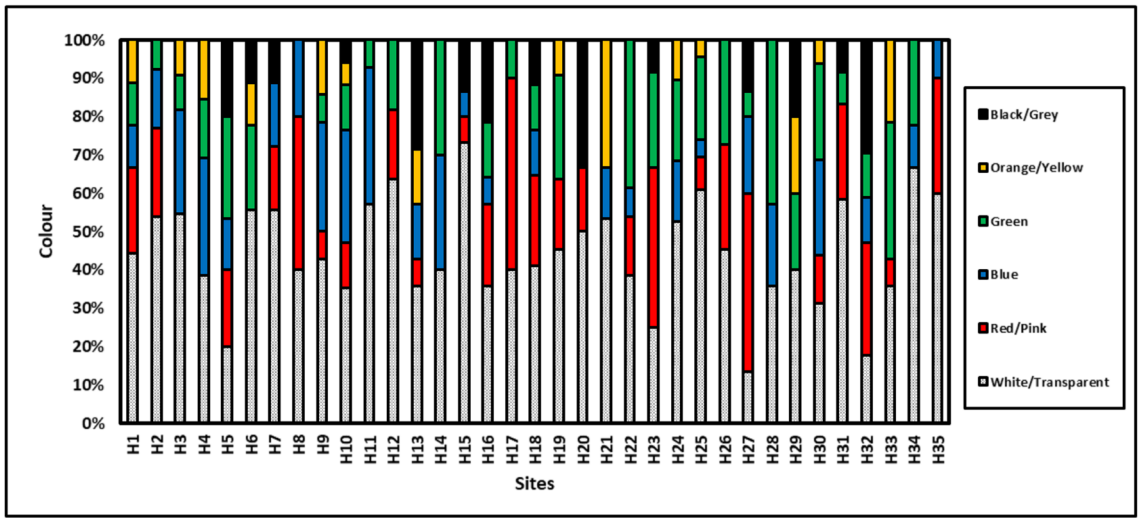
Fragments displayed a significant positive association ($\rho = 0.478$, $p < 0.01$) with PE. PE polymers are widely utilized in the production of single-use plastic materials such as carry bags, food packaging films and sweet and snack wrappers. These materials are extensively consumed within the study area. Disposable plastics are susceptible to wear and tear from both human activities and environmental factors in indoor spaces, which can lead to rapid disintegration into smaller fragments (Nematollahi et al. 2022). The presence of PS MPs in the settlements can be attributed to various sources, including eating utensils, foamed cups and plates. On the other hand, PVC MPs found in the dust may result from the disintegration of pipes, toys, cable insulation and walls and floor coverings.



(a)



(b)



(c)

Fig. 2 Percentage composition of MPs sorted based on **a** shape, **b** polymer type and **c** colour type within indoor dust in residential settlements (H1-H35)

The distribution of PE, PS and PVC MPs across the sampling sites followed a non-normal ($p < 0.01$) pattern. These results indicate that the distribution of these polymer types did not follow a consistent pattern across the settlements and is, instead, site-specific. This suggests that local factors (ventilation, airflow, resuspension of microplastics and cleaning habits) and sources (single use plastics, curtains, sofas, chairs and flooring) likely influence the presence and distribution of these specific types of microplastics in the indoor environment of each settlement (Jessieleena et al. 2023).

The colour of MPs has the potential to function as a meaningful marker for determining the age of plastics and influencing their degradation process and the formation of microplastics through interactive processes (Zhao et al. 2022). The dominance of white/transparent MPs in most of the sites is clearly highlighted in Table 1 and Fig. 2c. The total composition of MPs sorted by colour type followed the order: white/transparent (44.5%) > green (15.3%) > red/pink (14.9%) > blue (13%) > black/grey (6.9%) > orange/yellow (5.3%). All the coloured MPs had a non-normal distribution ($p < 0.01$). It is proposed that the colour of MPs in indoor/outdoor spaces may not be static and can change in response to prolonged photo bleaching. Thus, colour-based identification of the origin of MPs might be not accurate (Nematollahi et al. 2022).

The occurrence of MPs in indoor environments have been identified in several countries such as Paris, Australia, Colombia, South Korea, Japan, China, Vietnam, India, Pakistan, Kuwait, Saudi Arabia, Romania, Greece and the USA (Dris et al. 2017; Soltani et al. 2021; Zhang et al. 2020a). However, only limited information is available concerning the distribution of MPs in indoor dust in Indian conditions. A recent study highlighted the presence of polyethylene terephthalate (PET) (mean concentration: 2000 µg/g) and polycarbonate (PC)-based MPs (mean concentration: 20 µg/g) in indoor dust collected from settlements in Patna (Zhang et al. 2020a). In the outdoor environment, MPs were detected in airborne particulates over the eastern Indian Ocean (mean concentration: 0.4 ± 0.6 items/100 m^3) (Wang et al. 2020) and street dust (mean concentration: 227. 94 ± 91.37/100 g) (Patchaiyappan et al. 2021). In our study, we got a mean concentration of 13.6 ± 3.2 MPs g^{-1} of dust.

Exposure to MPs through dust ingestion

Table 2 presents the EDIs of fibres, fragments and total MP concentration (ΣMPs) for infants, toddlers, children, teenagers and adults. Mean EDI (MP kg^{-1} day^{-1}) for ΣMPs followed the order: infants (0.09) > toddlers (0.023) > children (0.011) > teenagers (0.005) > adults (0.004). The risk of microplastic (MP) exposure through ingestion was found to be 22.5 times higher for infants compared to adults. This increased exposure in younger age groups, such as infants and toddlers, can be attributed to their lower body weight and higher rate of ingestion (Nematollahi et al. 2022). Additionally, when considering both fibres and fragments, we observed a consistent pattern where the highest exposure risk was for infants, followed by toddlers, children, teenagers and adults. In the comparison of EDIs of MPs between fibres and fragments across these age groups, fibres posed a greater risk. This elevated risk associated with fibres can be linked to their prevalence in the dust samples analysed in the study. These findings underscore the importance of understanding MP

Table 2 The EDIs of fibres, fragments and ΣMPs for different population (infants, toddlers, children, teenagers and adults)

Age group	MP type	Min	Max	Mean	Percentile		
					5th	50th	95th
Infant	Fibre	0.035	0.106	0.063	0.040	0.063	0.094
Toddler	Fibre	0.008	0.025	0.015	0.009	0.015	0.022
Children	Fibre	0.004	0.012	0.007	0.005	0.007	0.011
Teenager	Fibre	0.002	0.005	0.003	0.002	0.003	0.004
Adult	Fibre	0.001	0.004	0.003	0.002	0.003	0.004
Infant	Fragment	0.014	0.063	0.032	0.014	0.035	0.049
Toddler	Fragment	0.003	0.015	0.008	0.003	0.008	0.012
Children	Fragment	0.002	0.007	0.004	0.002	0.004	0.006
Teenager	Fragment	0.001	0.003	0.002	0.001	0.002	0.002
Adult	Fragment	0.001	0.003	0.001	0.001	0.001	0.002
Infant	ΣMPs	0.063	0.162	0.096	0.068	0.099	0.129
Toddler	ΣMPs	0.015	0.038	0.023	0.016	0.023	0.030
Children	ΣMPs	0.007	0.019	0.011	0.008	0.011	0.015
Teenager	ΣMPs	0.003	0.008	0.005	0.003	0.005	0.006
Adult	ΣMPs	0.003	0.006	0.004	0.003	0.004	0.005

exposure risks in different age groups and the potential health implications, particularly among the most vulnerable, such as infants and young children.

Recent literature has drawn attention to the substantial deposition rates of airborne MPs in various indoor environments, such as office (1.8×10^3 MPs/m^2/day), dormitory (9.9×10^3 MPs/m^2/day), corridor (1.5×10^3 MPs/m^2/day) and homes (22–6169 fibres/m^2/day) (Soltani et al. 2021; Zhang et al. 2020b). The contamination of food resulting from the deposition of airborne microplastics (MPs) is a significant concern. Ingested microplastics can lead to severe health issues, including damage to the intestinal barrier function, reduced secretion of intestinal mucus and alterations in metabolism (Rahman et al. 2021). Studies have shown that certain types of MPs, such as polyethylene (PE) particles with sizes ranging from 3 to 16 µm and polystyrene (PS) particles with a size of 10 µm, can have cytotoxic effects by generating reactive oxygen species (ROS) in human glioblastoma (T98G) and human cervical adenocarcinoma cells (Schirinzi et al. 2017). PS particles with sizes of 0.1 and 5 µm have also been found to inhibit the activity of plasma membrane ATP-binding cassette (ABC) transporters and disrupt the mitochondrial membrane potential in human epithelial colorectal adenocarcinoma cells (Wu et al. 2019). Some studies also show an increase in histamine release, disruption of the epithelial layer, oxidative stress, inflammatory responses and DNA damage (Dong et al. 2020; Hwang et al. 2019; Poma et al. 2019; Prietl et al. 2014). These studies emphasize the necessity of identifying their sources and the possible co-occurrence of other contaminants in indoor environments.

MPs as source and sink for micro-pollutants

Biomass burning is an important source of PAH in residential settlements. MPs have a high affinity for these hazardous hydrophobic compounds (Amelia et al. 2021), and thus can act as vectors for their transport. The Raman spectra of MPs in dust, which were not treated with wet peroxide oxidation, showed presence of PAH (pyrene)-bound PS MPs (Fig. 3). These samples were collected from two settlements (H4 and H7). The occurrence of soot particulate on the PS MP was confirmed by their characteristic D and G band vibrations at ~ 1300 and 1600 cm^{-1}, respectively (Tommasini and Zerbi 2010). Soot particulates are efficient adsorbers of PAHs (Jonker and Koelmans 2002). We observed a Raman band at 1240 cm^{-1}; this specific vibrational mode is highly characteristic of pyrene and is often used as a diagnostic peak for its identification in Raman spectroscopy (Gu et al. 2013; Hahm et al. 2016). The residents in H4 and H7 were using both LPG and firewood as cooking fuel. We also noticed that the residents often introduced single-use plastics (disposable plates, cups, packaging covers) into the stoves (supplementary material: Figure S5) to speed up the firing of wood. González-Rodríguez et al. highlighted the co-occurrence of PAHs in burned polymer (polystyrene, polypropylene and nylon) samples (González-Rodríguez et al. 2011). Thus, the plastic burning activities for cooking activities may act as a prime source for PAH-bound MPs in the dust in the study locations.

Of late, studies have highlighted the strong interaction between the PAHs and MPs in various aquatic environments in China, Taiwan, Spain and Brazil (Camacho et al. 2019; Chen et al. 2020; Gorman et al. 2019; Jingchun et al. 2020;

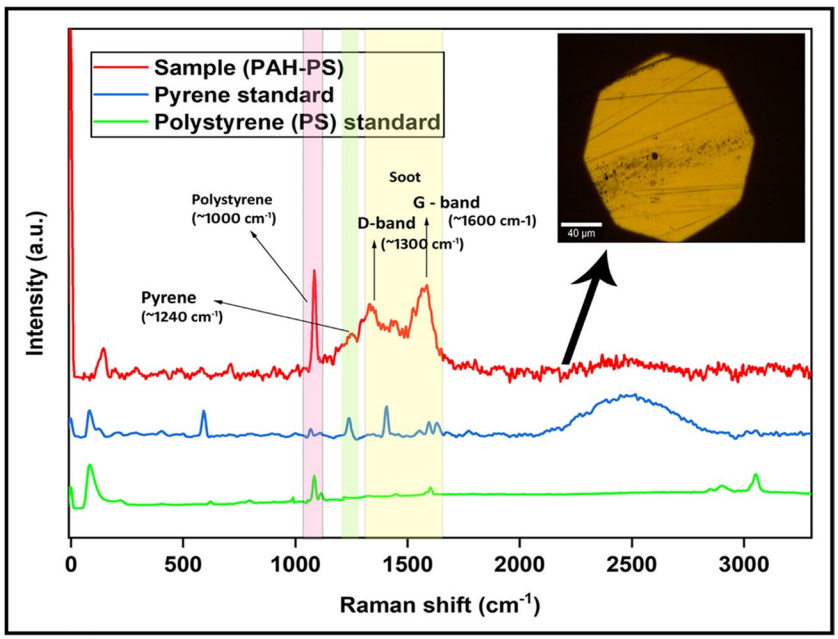


Fig. 3 Raman spectrum of PAH (pyrene)-bound PS (polystyrene) microplastic (MP) in dust sample (H4). The inset figure given on top of the PAH-PS spectrum shows the microscopic image (20×) of soot particle attached on to the surface of PS MP

Mai et al. 2018; Tan et al. 2019). However, the information on PAH-bound MPs in indoor and outdoor air/dust is not well documented. There are no reports regarding the PAH-bound MPs in indoor dust to compare with our findings. Identifying PAHs associated with MPs is usually carried out using a mass spectrometric technique such as GC–MS. This is often a time-consuming and requires a solvent extraction process. In this aspect, our technique has the edge for rapid non-destructive screening of PAH-bound MPs in environmental matrices.

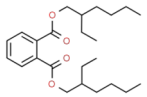
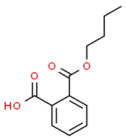
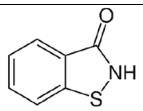
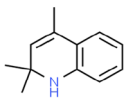
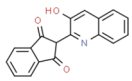
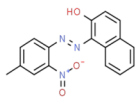
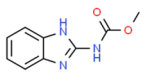
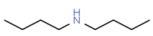
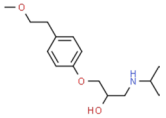
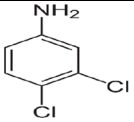
We also performed non-target analysis to identify plastic additives in MPs and dust samples. This technique is useful when there are no standards available to confirm unknown compounds.

In the positive ionization mode, we detected a parent ion with an m/z value of 192.0767 (as shown in Figure S12). This parent ion exhibited two fragments: a major fragment with an m/z value of 160.0511 and a less intense fragment with an m/z value of 132.0554 (supplementary material). The elemental composition of the parent ion and fragments was determined to be $C_9H_{10}N_3O_2$, $C_8H_6N_3O$, and $C_7H_6N_3$, respectively. Notably, the neutral loss observed between the parent ion and the major fragment corresponded to CH_3OH (methanol). So, an ether linkage in the structure is possible where one end of the ether was attached to a methyl group. The neutral loss between the parent ion and the less intense fragment with an m/z value of 132.0554 was $C_2H_4O_2$. The loss between the major fragments was aldehyde (CHO). This aldehyde group is not free in the structure. If it were free or not sandwiched between the hydrocarbon chain, then the intensity of this fragment would be more. Since the intensity or the relative abundance of this fragment was low, we suspect that this carbonyl group is sandwiched in between the hydrocarbon chains, possibly next to the ether link, or the structure of the parent ion as *R*-CO–O–CH_3. Thus, the structure contains a carboxylate ester link. Furthermore, one end of the carboxylate ester group is attached to a methyl group. With this information, we have looked at the structures (supplementary material) proposed in ChemSpider. From the proposed structures, it was found that, except for structure A, all others contained a free amino group (NH_2). Since we did not get any fragment which can result from the loss of the amino group from the parent ion, we suspect the structure of the parent ion to be that of structure A, which is carbendazim. We followed a similar protocol for the identification of other compounds. We identified seven organic compounds in dust having a link with plastics (the MS/MS fragmentation pattern of identified compounds is given in supplementary material). Furthermore, the presence of MBP and metoprolol in the samples was confirmed using analytical standards (the MS/MS fragmentation pattern of identified compounds and analytical standards is given in Figure S6 and S7 in supplementary material). The identified compounds belonged to different classes, such as pesticides, plasticizers, dyes and antioxidants. Figure S8 shows the total ion chromatogram (TIC) in the positive ionisation mode. We identified seven compounds from MPs isolated from the dust. The list of compounds identified from both the dust and alcoholic extracts of MP samples is presented in Table 3. The details on the PPM error and the fragmentation pattern of the tentatively identified compounds in this study are provided in the supplementary material (Table S2 and S3; Figure S9-S18).

The dust samples and alcoholic extracts of MPs contained phthalic acid esters such as di(2-ethylhexyl) phthalate (DEHP) and mono butyl phthalate (MBP). DEHP is widely applied as a plasticizer to manufacture various plastic-based products (Liu et al. 2019b). It is a potential human carcinogen (Zarean et al. 2016) and can adversely affect reproductive health (Schierow and Lee 2009). The presence of MBP in the samples could be attributed to the degradation of DIBP (di-isobutyl phthalate) because of microbial activity/photolysis in indoor spaces (Liu et al. 2019b). The humidity levels in indoor spaces could enhance this degradation process (Bope et al. 2019). Our study area is in a tropical environment, and the residential settlements experience elevated humidity levels (> 70%) throughout the year. Our recent study also showed MBP in dust samples collected from different micro-indoor spaces having similar climatic conditions (Sreejith et al. 2021). Floor dust samples from residential settlements in Guangzhou (China) contained various mono-PAEs (Liu et al. 2019b). Among the identified esters, the highest median concentration was noted for MBP (21.54 µg/g). Even though the occurrence of phthalate metabolites in biological matrices (human urine, blood, serum and breast milk) is well documented, there are only few reports regarding the occurrence of mono-PAEs in indoor environments.

Carbendazim and benzisothiazolinone (BIT) were the two pesticides detected in the dust samples. MPs were also found to have BIT. Carbendazim is an antifungal compound used to treat various fungal infestations in crops, vegetables, fruits, paints, leather, fabric and polymeric materials (Salis et al. 2017; Singh et al. 2016). The occurrence of carbendazim was reported in indoor environments in Italy (mean concentration: 0.085 µg/g) and China (median concentration: 35.8 ng/g) (Salis et al. 2017; Wang et al. 2019). It is an endocrine-disruptor (Ferreira et al. 2008) and a human carcinogen (Goodson et al. 2015). Their application is banned in Australia, the USA and in most regions of the European Union (EU). However, it is still in use in countries such as the UK, Portugal, China, Brazil and India. India consumes ~ 1993 metric tonnes of carbendazim every year (Singh et al. 2016). It is reported that carbendazim can have a half-life up to 12 months in bare soil (Singh et al. 2016). Thus, monitoring its level in an indoor environment becomes

Table 3 List of compounds identified in the indoor dust and MP samples by HRMS analysis. *MP* microplastic

	Compound	Chemical formulae	Ionisation mode	Structure	Sample	
					DUST	MP*
1	Di (2- ethylhexyl) phthalate (DEHP)	$C_{24}H_{39}O_4$	$[M-H]^-$		√	√
2	Mono-butylphthalate (MBP)	$C_{12}H_{14}O_4$	$[M-H]^-$		√	√
3	Benzisothiazolinone (BIT)	C_7H_5NOS	$[M-H]^-$		√	√
4	1,2-Dihydro-2,2,4-trimethylquinoline	$C_{12}H_{15}N$	$[M+H]^+$		√	√
5	Disperse Yellow 54	$C_{18}H_{11}NO_3$	$[M-H]^-$		√	-
6	Toluidine Red	$C_{17}H_{13}N_3O_3$	$[M+H]^+$		√	-
7	Carbendazim	$C_9H_9N_3O_2$	$[M+H]^+$		√	-
8	Dibutyl amine (DBA)	$C_8H_{19}N$	$[M+H]^+$		-	√
9	Metoprolol	$C_{15}H_{25}NO_3$	$[M+H]^+$		-	√
10	Dichloroaniline (3,4 – DCA)	$C_6H_5Cl_2N$	$[M+H]^+$		-	√

necessary. Benzisothiazolinone (BIT) is used widely in the antimicrobial formulation in water-based paints, cleaning solutions, plastics and air fresheners (Kwon et al. 2013; Law and Maibach 2020). Non-target screening of dust samples from different indoor environments in Sweden revealed the presence of BIT (Wang and Dubocq 2021). BIT exposure can induce dermatitis and is linked to an allergic reaction among industrial workers (Sheehan 2015). Elevated levels of BIT in air fresheners have been found to affect the cell viability, growth and morphology of human lung epithelial cells (Kwon et al. 2013). BIT is an ingredient in hobby and finger paints that children widely use, which is a cause for concern. The incidence of carbendazim and BIT in the samples can be attributed to their migration from sources such as paints, footwear, furniture, fabrics, settled MPs, packaging materials, fruit and vegetable debris. The inhalation, ingestion (food contaminated with dust) or dermal contact of dust contaminated with these pesticides can be an important route for human exposure. However, there is a massive void in the information concerning the status of pesticides in indoor spaces in India. A recent study had highlighted the occurrence of metaldehyde and ethofumesate in classroom dust (Sreejith et al. 2021). Thus, there is an urgent requirement for long-term monitoring of pesticides and their exposure levels in indoor spaces in this context.

1,2-dihydro-2,2,4-trimethylquinoline was detected in both the dust and MP samples. It is applied as an antioxidant in styrene-butadiene rubber (SBR) latex (MartÍN-MartÍNez 2002). The potential sources of this compound at the sampling site include tufted carpets, footwear, wall and vinyl floor tile adhesives (SBR is an important component added during the production stage of these products). A significant number of tufted carpets are polypropylene-based. The abrasive forces on these carpets, such as sweeping, walking and vacuuming, can cause MP emission. Thus, the emitted MPs, when settled on dust, can be a potential source and sink of 1,2-dihydro-2,2,4-trimethylquinoline.

Disperse Yellow 54 and toluidine red, detected in the dust samples, are dyes containing an azo-group. Azo dyes are synthetic organic compounds with an azo group (–N = N–). They are widely applied as colourants in textiles, plastics, leather, personal care products, paper, paints and food (Jagruti 2015). Exposure to this class of dyes can be detrimental to health because of their genotoxic, mutagenic and carcinogenic potential (Chung 2016). Synthetic fibres such as polyamide, polyester, acrylic and acetate fibres are predominantly coloured with disperse yellow 54 (Bode et al. 1985). Toluidine Red is used as a colourant in the manufacture of paints, inks and plastics. We have detected PA and PS MPs in dust samples that may be potential sources of the identified dyes. The transport dynamics of azo-dyes in indoor environments should be given much attention because of their wide application in various consumer products. Children may be the most vulnerable group exposed to these hazardous compounds as they are in close contact with settled dust and toys containing different dye colourants. The abrasions of toys from rough usage of these materials by toddlers may cause MP emission in indoor spaces. The information regarding the occurrence and migration of dyes at indoor spaces is undetermined. Some reports concerning the occurrence of azo-dyes in indoor dust are presented in Table 4.

The additional compounds detected in MPs other than the dust include di-butyl amine (DBA), metoprolol and dicholoroaniline (3,4 — DCA). DBA is an important volatile aliphatic amine that is used for the formulation of hindered amine light stabilizer (HALB) (DONG et al. 2005). HALBs are used as UV-stabilizers in polyolefins (polypropylene and polyethylene) (Hahladakis et al. 2018). Because of the extensive consumption of volatile aliphatic amines, they have been detected in indoor and outdoor air samples (Szulejko and Kim 2014). These compounds may interact with nitrites to generate nitrosamines, which are potential carcinogenic compounds. In indoor environments, the room fresheners can be a significant source for the nitrites (Cruz and Bowen 2021). Henceforth, there is an urgent need for studies focusing on the interaction of nitrites with aliphatic amines in indoor spaces. Metoprolol is a pharmaceutical drug (β_1 receptor blocker) used to treat cardiovascular ailments (Clemente-Moragón et al. 2021). The presence of metoprolol in the MPs may have resulted from the adsorption of the drug to the plastic packaging (Palmgrén et al. 2006). Dichloroaniline, a derivative of aniline found in MPs, is widely utilised in the production

Table 4 Reports concerning detection of azo-dyes in indoor dust

Location	Dyes	Reference
USA	C.I. Disperse Blue 373, C.I. Disperse Violet 93, $C_{17}H_{15}O_2N_5Br_2$,$C_{21}H_{23}O_9N_6Br$, $C_{21}H_{21}O_{10}N_6Br$,$C_{23}H_{25}O_{10}N_6Br$	Dhungana et al. (2019)
Canada	C.I. Disperse Violet 28,C.I. Pigment Red 6,Disperse Red 13,Disperse Orange 138, C.I. Pigment Yellow 73, C.I. Pigment Yellow 3, C.I. Pigment Yellow 6, C.I. Disperse Brown 1, C.I. Pigment Red 14, C.I. Disperse Blue 79:2	Kutarna et al. (2021)
Sweden	C.I Disperse Red 60, C.I. Basic Red 9	Wang and Dubocq (2021)
India	C.I. Disperse yellow 54, C.I. Pigment Red 3	This study

of plastics, pesticides and azo-dyes (Ibrahim et al. 2020; Padmini and Miranda 2013). It is an extremely poisonous and endocrine disrupting compound. The toxicity studies on rats have revealed its potential to induce kidney, liver and urinary bladder dysfunctions (Ibrahim et al. 2020).

Conclusion

In this study, the distribution of five common MPs in indoor residential dust and their ingestion related exposure risk was investigated. All the dust samples from the residential settlements were contaminated with MPs. We noted a significant positive correlation between the residential population number and MP levels in the dust samples. PA fibres were the dominant MPs in the samples studied, highlighting the role of synthetic fabrics as a significant source for MPs. The infants were the most vulnerable group to the risk via ingestion of MPs in the dust. Our study showed the scope of using MRS technique for monitoring PAH-associated MPs in settled dust and revealed that MPs can act as a potential sink for PAH compounds in indoor settlements. The comparison of chemical profiles obtained for MPs and dust samples from mass spectrometric analysis revealed that it can be a potential source and sink for SVOCs like di-ethylhexyl phthalate (DEHP), mono-butylphthalate (MBP),1,2-dihydro-2,2,4-trimethylquinoline, and benzisothiozolone. Exposure to these compounds can cause a wide range of human health problems. Some of them are possible human carcinogens, while others are endocrine disruptors or skin or eye irritants. Residential settlements can be hotspots for these particles, especially those with inadequate ventilation systems and improper cleaning regimes. Thus, it becomes crucial to have a continuous monitoring program to check the levels of these particles in indoor spaces and take necessary actions to reduce their abundance. Using single-use plastics for cooking activities should be handled with much attention as they could expose the occupants to noxious gases, particulate matter and MPs enriched with soot and PAHs. Thus, there is an urgent requirement for exposure assessment studies focused on these kinds of cooking practices to quantify their effect on human health.

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Author contribution VSM: conceptualization, methodology, formal analysis, writing — original draft. UKA: writing — review and editing, supervision. CTA: writing — review and editing, supervision.

Data availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication All authors consent to the publication of this study.

Conflict of interest The authors declare no competing interests.

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