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Quantification of microplastic by particle size down to 1.1 μm in surface road dust in an urban city, Japan☆

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ABSTRACT

The impact of microplastics (MPs, plastic particles ≤5 mm) on ecosystems is of great concern. Road surfaces represent a significant source of MPs where plastic fragments are physically and chemically reduced to MPs. However, the literature lacks information on fragmentation tendencies below 11 μm. This study aimed to characterize the occurrence of MPs in road dust in different size fractions down to 1.1 μm. Road dust was collected at five sites near a major road in Kusatsu city, Japan, and partitioned by size into 13 fractions (1.1–850 μm). The coarser fractions accounted for a greater proportion of the dust. The percentage of organic matter, determined by loss on ignition, increased as the fractions became finer. Pyrolysis-gas chromatography-mass spectrometry was used to quantify 12 types of polymers in each fraction. The dust was found to contain nine types of MP, namely, polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC), polystyrene (PS), styrene/butadiene rubber (SBR), acrylonitrile/butadiene/styrene resin (ABS), polycarbonate (PC), polymethylmethacrylate (PMMA), and polyamide 66 (PA66). The total MP concentration in road dust particles by particle size fraction (concentration$_f$) began to increase from the 125–250 μm fraction and remained elevated in finer fractions down to 1.1 μm, indicating that MPs in the road dust micronized to at least 1.1 μm. However, for individual polymer types, the tendency for concentration$_f$ to increase or decrease with particle size fraction varied: the concentration$_f$ of some polymers, such as PE and PVC, remained elevated in fractions down to 1.1 μm; the concentration$_f$ of SBR, a rubber-MP, showed a stable or decreasing trend in fractions of 7.0–11 μm and finer. Particles of PE, PVC, and some other plastics might become increasingly finer, even down to 1.1 μm. Further research is needed to understand the comminution limits of these polymers under pertinent environmental conditions.

1. Introduction

Microplastics (MPs) are fragments of plastic, and there are concerns about the impact of MPs on water and terrestrial ecosystems (Barnes et al., 2009). MPs are often defined as fragments of plastic with a particle size of ≤5 mm, while particles between 1 and 100 nm are classified as nanoplastics, following the general agreement in the field of nanomaterials (Hartmann et al., 2019). In this study, MPs are defined as plastic particles between 1 and 5000 μm in particle size; this definition includes rubber particles (i.e., from tires), though they are not plastic according to some polymer chemistry definitions (Hartmann et al., 2019). In the environment, MPs are subjected to physical, chemical, and biological effects, such as ultraviolet light, waves, abrasion in the road, and microbial degradation, and are broken down into even finer particles (Cole et al., 2011). Many MPs exist in the marine environment (Blettler et al., 2018), and MPs have harmful effects on marine organisms such as fish and shellfish when ingested (MacLeod et al., 2021). In recent years, in addition to the effects of MPs themselves, their effects as a vector transporting organic pollutants in the environment have also been studied (Dai et al., 2022; Yukioka et al., 2018), and it is clear that the adsorption amounts of organic pollutants differ depending on polymer type (Yukioka et al., 2018).

MPs generated on land might accumulate into marine environments, mainly through washing away by rainfall and river transport or through direct discharge (Wang et al., 2021; Yano et al., 2021). The path of MPs from land to water includes point sources such as sewage treatment

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plants and non-point sources, including flow directly into the water by surface runoff (He et al., 2022). In addition, the atmospheric environment has been identified to represent as a transporter and reservoir of microplastic particles and fiber (Brahney et al., 2021). It has been reported that the concentration of MPs from non-point sources is higher than that of sewage treatment plants (Yano et al., 2021), and MPs generated on road surfaces may flow directly into rivers as a result of rainfall and other factors, increasing the burden on the aquatic environment.

MP in road dust is reported throughout the world: Australia (Monira et al., 2022; O'Brien et al., 2021; Su et al., 2020), Japan, Nepal, Vietnam (Yukioka et al., 2020), China (Zhang et al., 2022), Iran (Abbasi et al., 2019, 2017), India (Patchaiyappan et al., 2021), Germany (Dierkes et al., 2019), and Sweden (Järlskog et al., 2020). Count-based or mass-based identification and quantification methods were used for the analysis of MPs in those studies. In most of those studies, MPs were identified by count-based identification using various measurement techniques, including Fourier Transform Infrared Spectroscopy (FTIR) (Monira et al., 2022; Yukioka et al., 2020; Zhang et al., 2022), Raman microscopy (Patchaiyappan et al., 2021), or stereo microscopy (Järlskog et al., 2020; Su et al., 2020). A few of those studies identified and quantified MPs by mass-based identification using pyrolysis–gas chromatography–mass spectrometry (a GC/MS unit with a pyrolysis device attached) to analyze pyrolysis products with the characteristics of each polymer type (Dierkes et al., 2019; O'Brien et al., 2021). Most of the previous studies detected generic plastics, such as polyethylene, polypropylene, polystyrene, polyethylene terephthalate, and polyvinyl chloride, but a few studies also detected tire-derived MPs such as styrene-butadiene rubber and synthetic rubber in road dust (Yukioka et al., 2020; Zhang et al., 2022). The minimum size of MP particles in road dust targeted in above-mentioned studies has so far been 11 μm (Patchaiyappan et al., 2021), therefore, the occurrence and characteristics of MPs in road dust of finer particle size remains unknown. Previous studies have also focused on tire wear particles and brake wear particles in road dust as non-exhaust traffic-related particles (Grigoratos and Martini, 2014). Tire wear particles (TWP) included rubber MPs such as styrene/butadiene rubber (SBR), natural rubber (NR), and butadiene rubber (BR), occurring as the mixed particles of rubber and minerals originating from the combination of tire and road wear (Adachi and Tainosho, 2005; Rausch et al., 2022). In studies investigating TWP in road dust, an automated Scanning Electron Microscopy/Energy Dispersive X-ray spectroscopy (SEM/EDX) (Järlskog et al., 2022), and pyr-GC/MS (Rødland et al., 2022) have been used to identify and quantify TWP down to several μm in particle size. However, analytical uncertainties have also been noted for finer particles. Elemental composition analysis using SEM/EDX revealed TWPs down to 2 μm, but the 2–20 μm fraction could not be distinguished from bitumen particles (Järlskog et al., 2022). Rødland et al. (2022) also used pyr-GC/MS to determine tire concentrations using the combined peak of SBR, BR, and SBS in road surface dust at >1.6 μm. However, since no fractionation was performed in that paper, it was not known at what particle size TWP were present. Due to the limitations of the analytical methods described above for both generic and rubber MPs, little is known about the size-based occurrence of each polymer in actual road environments due to degradation and fragmentation (i.e., micronization tendency).

If MPs on roads reach a particle size of a few μm, it is thought that they would be able to migrate not only to aquatic environments but also into the atmosphere, where they raise concerns about their effects on the human body via the respiratory system (Munyaneza et al., 2022). Furthermore, the amounts of adsorbed organic pollutants may increase as the particles become finer and so become more harmful to living organisms. Therefore, it is necessary to clarify the existence of MPs by particle size, including particles of only a few μm or less.

Therefore, the main objective of this study was to quantify and characterize the occurrence of MPs in surface road dust, with particular new insights into the ultra-fine fractions of the road dust over a broad size range of 850 μm down to 1.1 μm. In this study, MPs included one major tire material, SBR, and 11 other components, including common plastics such as polyethylene (PE), polypropylene (PP), and polystyrene (PS).

2. Materials and methods

2.1. Sample collection, sieving procedure, and pretreatment

Road dust samples were collected at five sites (R1–R5) on August 31, 2021, near Prefectural Road 141 in Kusatsu, Shiga, Japan (Fig. S1, Table S1). Eight preceding clear days were allowed before sample collection. Kusatsu is in the southeastern part of Shiga prefecture, with a population of 137,200 (Kusatsu city, 2021). Intersections of major and sub-arterial roads and city roads in this city were selected as sampling sites. Traffic volume was 13,800 vehicles/daytime on R1, 14,400 vehicles/daytime on R2 and R3, 2800 vehicles/daytime on R4, and 8000 vehicles/daytime on R5 (Ministry of Land, Infrastructure, Transport and Tourism, Japan, 2015). The speed limit on major arterial roads (prefectural roads) and sub-arterial roads (city roads) was 50 km/h, and 40 km/h on other city roads. Although cleaning activities such as trash pickup and sweeping were regularly conducted, detailed information on these activities was unavailable. In this study, road dust was collected at each sampling site, directly on the road surfaces and curbsides, ensuring the large sample volume to investigate particle size distributions down to finer size with suitable analytical reliability. Dust was collected by using a cordless stick vacuum (Dyson v15 Detect; Dyson Limited, Singapore). The amount of road dust collected at each site was 10.1 kg for R1, 7.3 kg for R2, 7.6 kg for R3, 7.9 kg for R4, and 7.9 kg for R5 (Table S1). Samples were stored in a disposable freezer bag, taken back to the laboratory under light-shielded conditions, and stored in a cool, dark room (4 °C) until analysis.

The road dust sample from each site was divided into two groups: one for particle size distribution analysis and the other for fractionation. Samples for fractionation were fractionated according to particle size using a sieve and fractionator. First, samples were divided into seven fractions (850–500 μm, 500–250 μm, 250–125 μm, 125–75 μm, 75–53 μm, 53–38 μm, and 38–20 μm) by sieving through stainless-steel sieves. Then, for samples finer than 20 μm, the sample was further divided into six fractions (20–11 μm, 11–7.0 μm, 7.0–4.7 μm, 4.7–3.3 μm, 3.3–2.1 μm, and 2.1–1.1 μm) by using a fractionator (Fig. S2). The fractionator used an Andersen-type -volume air sampler capable of collecting aerosol fractions (AN-200; Sibata Scientific Technology, Ltd, Japan); airborne dust that blew up to the sampler was aspirated into the air sampler for fractionation and collection. The fractionated sample was transferred to a stainless-steel container and stored for analysis.

2.2. Analysis of size distribution, loss on ignition, and Pyr-GC/MS

2.2.1. Particle size distribution

A laser diffraction particle size analyzer (SALD-2200; Shimadzu Corporation, Japan) was used to measure the volume-based particle size distribution of the road dust samples to determine the percentage of surface road dust by particle size. The size range of the particle size analyzer was 0.03–1000 μm. The particle size distribution of sample fractions finer than 20 μm was also measured to confirm the effectiveness of the fractionator (Fig. S3).

2.2.2. Loss on ignition

To determine the percentage of organic matter in the surface road dust, we measured the loss on ignition (LI) by particle size. For each fraction of 20–850 μm particle size, 1000 mg per sample was weighed out, and for each fraction of 1.1–20 μm particle size, 20 mg per sample was weighed out. The samples were heated in a muffle furnace at 600 °C for 2 h and weighed again. The LI was calculated using Eq. (1):

Table 1
Characteristic pyrolyzates, indicator ions, IDL, and IQL of 12 polymers.

Polymers	Characteristic pyrolyzates	Abbr.	RI	Indicator ions (m/z)	IDL (μg)	IQL (μg)
Polyethylene (PE)	1,20-Heneicosadiene	C21″	2089	82	0.33	1.11
Polypropylene (PP)	2,4-Dimethyl-1-heptene (propylene trimer)	C9′	846	126	0.06	0.20
Polystyrene (PS)	2,4,6-Triphenyl-1-hexene (styrene trimer)	SSS	2543	91	0.01	0.03
Acrylonitrile/butadiene/styrene resin (ABS)	2-Phenethyl-4-phenylpent-4-enenitrile	SAS	2282	170	0.01	0.05
Styrene/butadiene rubber (SBR)	4-Phenylcyclohexene (Styrene butadiene hybrid dimer)	SB	1400	104	0.05	0.16
Polymethylmethacrylate (PMMA)	Methyl methacrylate	MMA	727	100	0.01	0.02
Polycarbonate (PC)	4-Isopropenylphenol	IPP	1336	134	0.17	0.56
Polyvinyl chloride (PVC)	Naphthalene	Nap	1247	128	0.03	0.10
Polyurethane (PU)	4,4′-Methylenedianiline	MDA	2265	198	0.06	0.22
Polyethylene terephthalate (PET)	Benzophenone	BP	1730	182	0.80	2.65
Polyamide 6 (PA6)	ε-Caprolactam	Capro	1331	113	0.14	0.48
Polyamide 66 (PA66)	Cyclopentanone	CP	826	84	0.02	0.07

RI: Retention index, IDL: Instrument detection limit, IQL: Instrument quantification limit. The characteristic pyrolyzate of PVC (i.e., naphthalene) may also indicate the presence of bitumen.

$$LI = \frac{m_a - m_b}{m_a} \times 100 \quad (1)$$

LI: loss on ignition (%)
m_a: Sample before loss on ignition (mg)
m_b: Sample after loss on ignition (mg)

2.2.3. Pyr-GC/MS

Single-shot Pyr-GC/MS was used to analyze MPs in road dust. An advantage of pyr-GC/MS is that it does not require complex pretreatments such as organic decomposition with hydrogen peroxide or specific gravity separation for analysis (Lv et al., 2021). Fine plastics are considered chemically more unstable and sensitive to strong organic decomposition (Fischer and Scholz-Böttcher, 2017). Therefore, this study analyzed MP by pyr-GC/MS without complex pretreatment. From each fractional sample, 30 mg was dissolved in ethanol to make a 10 mg/mL solution. After sufficiently homogenizing the solution, 100 μL of the solution was transferred to a pyrolysis cup (Eco-Cup LF; Frontier Laboratories Ltd, Japan). The representativity of the analytical procedure was verified in advance using standard polymer samples (coefficient of variation<15%, $n = 7$). Ethanol in the cup was vaporized in an incubator at 55 °C, and the cup was then introduced into the Pyr-GC/MS unit. Analysis was performed with a gas chromatograph–mass spectrometer (6890A-5973N; Agilent, United States) equipped with a multi-shot pyrolyzer (EGA/Py-3030D; Frontier Laboratories Ltd), an auto-sampler (AS-1020E; Frontier Laboratories Ltd), and Ultra ALLOY-MP capillary separation column (30 m column length, 0.25 mm I.D., 0.5 μm film thickness; Frontier Laboratories Ltd) The pyrolysis was performed at 600 °C, utilizing detailed analytical conditions shown in Table S2.

The samples were analyzed for 12 types of polymer, namely, polyethylene (PE), polypropylene (PP), polystyrene (PS), acrylonitrile/butadiene/styrene resin (ABS), styrene/butadiene rubber (SBR), polymethylmethacrylate (PMMA), polycarbonate (PC), polyvinyl chloride (PVC), polyurethane (PU), polyethylene terephthalate (PET), polyamide 6 (PA6), and polyamide 66 (PA66) (Table 1). The pyrolysis products of each polymer were those reported by Ishimura et al. (2021). In line with this previous study, $CaCO_3$ was added to each sample to obtain stable pyrolysis products. Calibration standards were prepared for the 12 types of polymers using plastic reference standards diluted with $CaCO_3$ (PY1-4940; Frontier Laboratories Ltd), with five calibration points ranging from 0.04 to 1.00 mg/cup and reaching an $R^2 > 0.95$ for each polymer. By repeated measurements of the plastic reference standards at a concentration of about 10 times the signal-to-noise (S/N) ratio ($n = 7$),

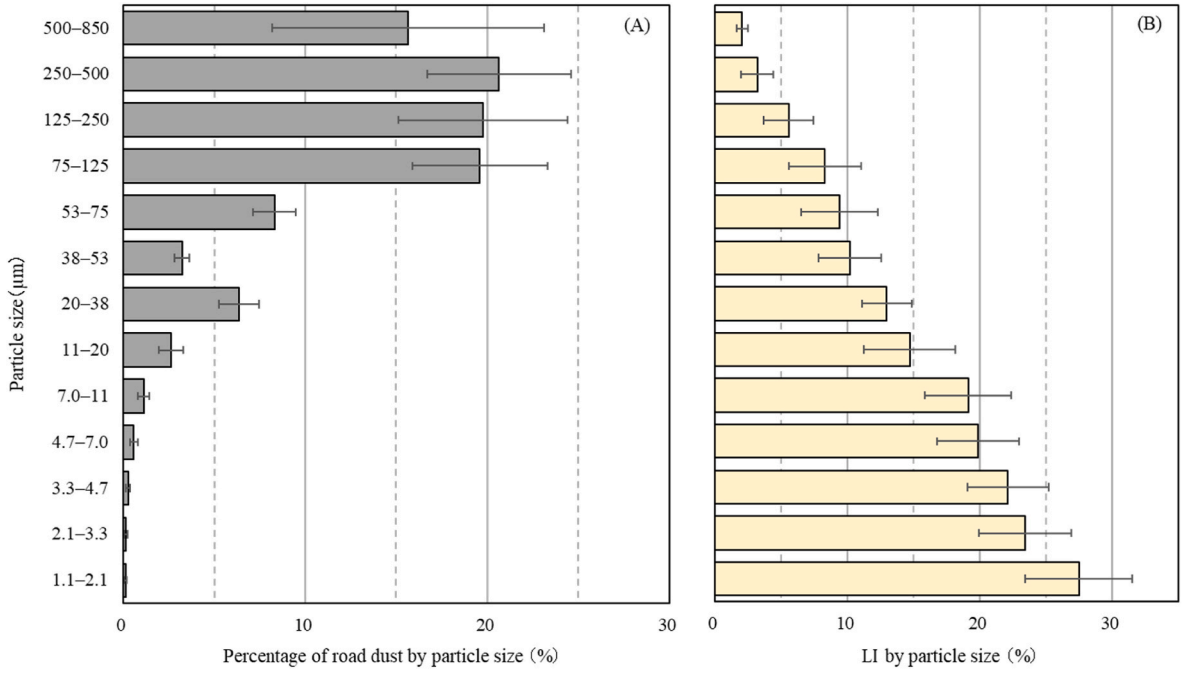

Fig. 1. (A) Percentage by volume of surface road dust and **(B)** loss on ignition (LI), in each fraction. Data are presented as mean and standard deviation, $n = 5$.

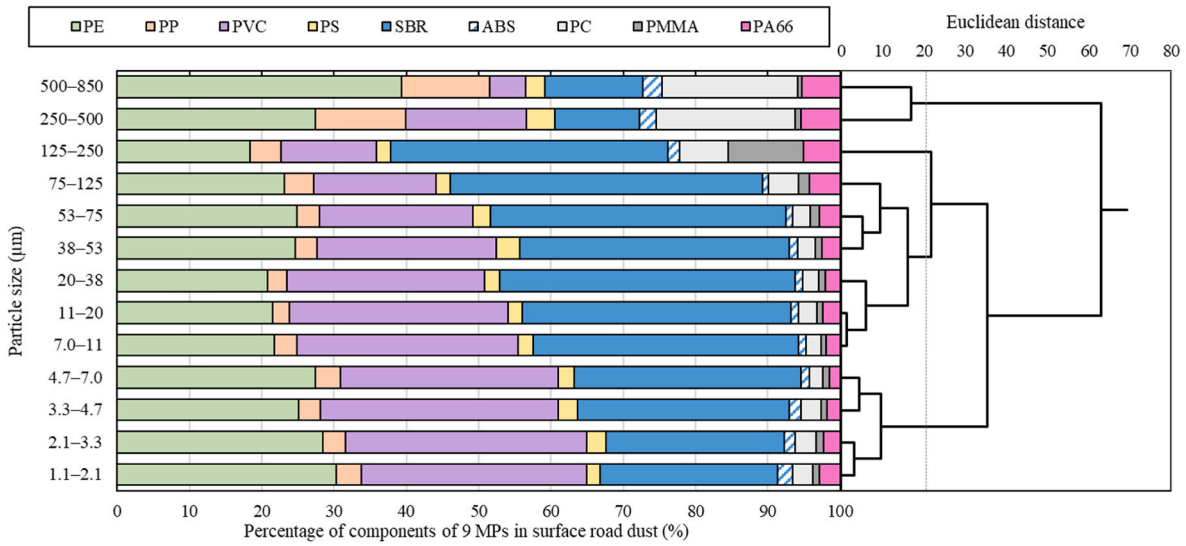

Fig. 2. Composition of polymer types of nine MPs in surface road dust by particle size and the result of cluster analysis by using Euclidean distance and Ward's method. Data are presented as average values, $n = 5$. Polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polystyrene (PS), styrene/butadiene rubber (SBR), acrylonitrile/butadiene/styrene resin (ABS), polycarbonate (PC), polymethylmethacrylate (PMMA), and polyamide 66 (PA66). MPs are shown in order of Japan's domestic production volume (The Japan Plastics Industry Federation, 2020).

the standard deviation is obtained; 3 times the standard deviation was taken to be the instrument detection limit (IDL), and 10 times the standard deviation was taken to be the instrument quantification limit (IQL).

2.3. Quality control

Quality assurance and quality control procedures were followed during all sampling and laboratory analyses. Most of the equipment used was made from metal or glass. Cotton laboratory coats and nitrile gloves were worn throughout the entire analysis. All of the equipment was ultrasonically cleaned with methanol before use. In the field, the Dyson v15 Detect used to collect the road dust was cleaned with an air duster, Milli-Q water, and methanol to avoid cross-contamination between samples. A blank test of this sampling procedure was performed (Table S3), and several polymers were detected at concentrations below IQL. Sample fractionation and pretreatment for analysis were performed in a clean bench with 99.97% interception of 0.3 μm particles to minimize possible contamination. Before and during the operation of pyr-GC/MS, empty pyrolysis cups were inserted to clean the system and to ensure the lack of cross-contamination during analysis. The system was also operated without pyrolysis cups to confirm that there were no MPs contamination of the instrument ($n = 5$). In addition, a procedure blank (purified $CaCO_3$, $n = 1$) was measured as well as the actual samples (Table S3). The concentrations of all polymers were below IDL in the instrument blank, but several polymers were detected in the procedure blank at concentrations below IQL. Sample amounts were determined using a calibration curve corrected with the procedure blank. Any drift in the sensitivity of the instrument was corrected by measuring a known amount of standard. PU, PET, and PA6 were considered not detected (N. D.) since the values of all samples were less than the IDL. For other polymer types, values less than the IDL were calculated as 1/2 IDL and values greater than IDL, but less than IQL were calculated as 1/2 IQL.

2.4. Statistical analysis

Euclidean distance and Ward's method were used for cluster analysis. Three-dimensional ANOVA was also performed for the particle size fraction, polymer type, and sampling point to examine the factors affecting MPs concentration by particle size.

3. Results and discussion

3.1. Volume-based ratio of surface road dust and loss on ignition, by particle size

Fig. 1A shows the volume-based ratio of surface road dust by particle size. Particles in the 75–850 μm size fractions accounted for about 70%. The proportion was lower in the fractions less than 75 μm, with the proportion diminishing roughly with particle size. These results were similar to trends seen in previous studies which collected road dust using vacuum cleaners with disposable bags: in one study, particles of size ≥53 μm accounted for more than 80% of the total (Deng et al., 2022), in another study, particles of size ≥125 μm accounted for more than 70% with lesser proportions of finer particle sizes (Ha et al., 2012). Furthermore, the use of a cyclone vacuum cleaner in this study enabled the collection of even finer particles compared to vacuum cleaners with disposable bags (the pore size of a normal bag is several 10 μm). On the other hand, previous studies using a wet dust sampler (Järlskog et al., 2022) or a pressure washer in conjunction with a wet vacuum cleaner (Klöckner et al., 2021) showed a peak in the 20–50 μm range, which is different from the particle size distribution in this study. A wet dust sampler was reported to be more efficient at sampling fine particles but less efficient at sampling coarse particles (180–5000 μm, Rødland et al., 2022). Therefore, different sampling methods may result in different particle size distributions. Road dust is considered to have large amounts of coarse particles because it includes roadside soil and decomposing plant material in addition to tire and road-wear particles (Deng et al., 2022). In addition, finer particles generally may migrate to the atmospheric environment, reducing the proportion of particles in finer size fractions.

Fig. 1B shows the percentage of loss on ignition (LI) by particle size fractions of surface road dust. LI is considered an indicator of organic matter content (Ozaki et al., 2015). LI ranged from 1.63% to 33%, and the percentage increased as the particle size decreased. LI of dust from major roads in Japan has been reported to be 0.8%–17% (Ozaki et al., 2015), and the LI of ≥11 μm particle size fractions in this study fell within that range. In addition, a previous study measuring LI by particle size in particles ranging from 40 to 2000 μm found a slight decrease from LI of 15% in fine fractions (<40 μm) to 8% in coarser fractions (250–500 μm) (Gelhardt et al., 2021). Similar to the previous study, LI tended to be

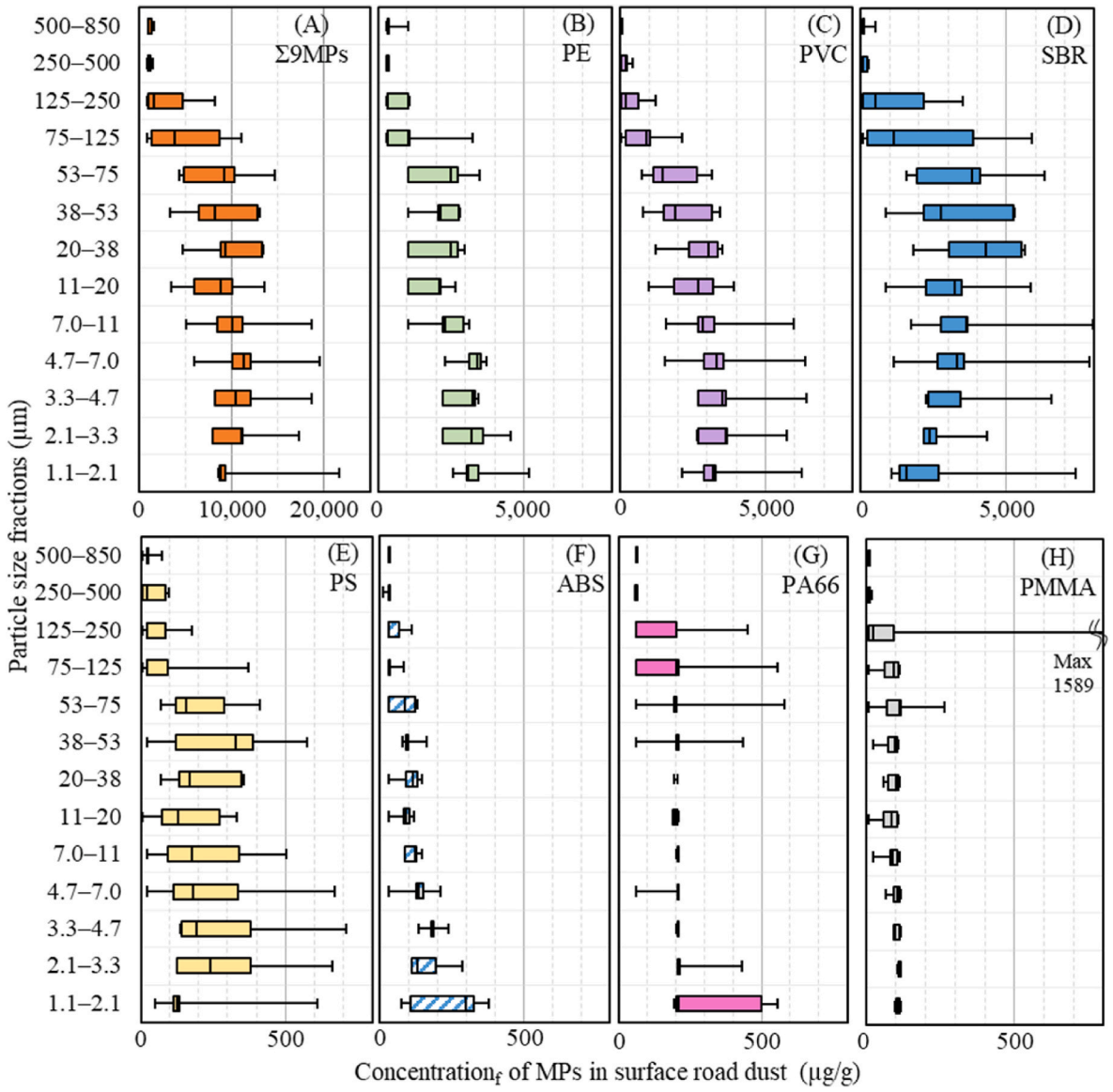

Fig. 3. Concentration of (A) all nine MPs together (Σ9MPs), (B) polyethylene (PE), (C) polyvinyl chloride (PVC), (D) styrene/butadiene rubber (SBR), (E) polystyrene (PS), (F) acrylonitrile/butadiene/styrene resin (ABS), (G) polyamide 66 (PA66), and (H) polymethylmethacrylate (PMMA) in surface road dust by particle size (Concentration$_f$). The unit was μg-MP in the particle size fraction g-dry of road dust in the particle size fraction. Data are presented using boxplots. PP and PC are not included and shown in the supplementary material because all samples were below the IDL (Fig. S5).

finer in the coarser fractions in the current study. The current study also shows that LI increased further in particles of size <40 μm.

3.2. Percentages of polymer types in surface road dust

Fig. 2 shows the composition of MPs by particle size and the result of cluster analysis between particle size fractions. Nine of the 12 types of polymers that we tested for were detected: polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polystyrene (PS), styrene/butadiene rubber (SBR), acrylonitrile/butadiene/styrene resin (ABS), polycarbonate (PC), polymethylmethacrylate (PMMA), and polyamide 66 (PA66). The cluster analysis identified four categories of particle size, i.e., 250–850 μm, 125–250 μm, 7.0–125 μm, and 1.1–7.0 μm, clearly showing that the ratios of polymer types changed as fractions changed from coarse to fine.

In fractions coarser than 250 μm, we detected large quantities of not only PE (27–39%) and PP (12%), which are used in a wide range of inexpensive products such as plastic bags, straws, containers, and pipes (Li et al., 2016), but also PC (19%) and SBR (12–14%). As PC is used for traffic lights, construction materials, and other outdoor products and SBR is used for tires (Jung and Choi, 2022; Li et al., 2016), it is probable that the amounts occurring on road surfaces would be significant.

In the 125–250 μm fractions, the ratio of SBR increased notably (38%), and the ratios of PE, PP, and PC decreased. Yukioka et al. (2020) reported that the ratios of rubber MPs such as PU and SBR in the 100–1000 μm size category were higher and that the ratios of container/packaging MPs such as PE, PP, PS, and PET were lower compared to the 1000–5000 μm size category (Yukioka et al., 2020). A similar trend was observed in this study in the 125–250 μm particle size fraction, although PET and PU were not detected.

In the third cluster (7.0–125 μm cluster), the combined proportion of PE, PVC, and SBR exceeded 80%, with the proportion of PVC increasing with finer particle size from 17% in the 75–125 μm fraction to 31% in the 4.7–7.0 μm size fraction. PVC is often used in outdoor situations such as for plumbing pipes, guttering, and agricultural hothouses (Ikeda et al., 1987; Li et al., 2016), and it increased to 1.1–2.1 μm size fraction

(33%). Similar to this study, O'Brien et al. (2021) showed that the ratios of PVC in particles <250 μm were greater than in the coarser particle size fractions in areas of similar traffic volumes (O'Brien et al., 2021, the traffic volume is 14,000 vehicles per day). Even more interestingly, with the fractions of particle size ≤7.0 μm, the ratio of SBR decreased and the ratio of PE increased. These differences in micronization tendency depending on polymer type are necessary to explain the difference in the MP compositions of the particle size fractions. The following section explains this observation in detail.

3.3. MP concentration in surface road dust, by particle size

Initially, the total concentration of the nine types of MP by each particle size fraction in the total road dust ($concentration_t$) was determined for comparison with a previous study (Fig. S4). The units were μg-MP in particle size fraction/g-dry of total road dust. The total $concentration_t$ showed an increasing trend from the 500–850 μm fraction to the 75–125 μm fraction (Fig. S4). O'Brien et al. (2021) measured MPs in road dust in the size fractions of <250 μm, 250–500 μm, 500–1000 μm, 1000–2000 μm, and 2000–5000 μm in an area with traffic volume similar to that of our study (the traffic volume was 14,000 vehicles per day), with the total MP concentrations of 1813 μg/g-dry, 783 μg/g-dry, 747 μg/g-dry, 732 μg/g-dry, and 828 μg/g-dry, respectively, while detecting PP, PS, PET, PVC, PMMA, PE (O'Brien et al., 2021). In our study, the total $concentration_t$ of the nine types of MPs in the >250 μm fractions was several times lower than in that previous study, with 145 ± 53 μg/g-dry in the 250–500 μm fraction and 108 ± 56 μg/g-dry in the 500–850 μm fraction. In this study, a vacuum cleaner was used, whereas O'Brien et al. (2021) collected samples by sweeping with natural fiber bristle brushes into a metal dustpan, which was the most efficient method for particles >75–100 μm in diameter (O'Brien et al., 2021). Thereby, the concentration of coarse plastics may have been high. In addition, distance from the source may also be related to MPs concentration. The total $concentration_t$ of the 11 size fractions of <250 μm in this study was 2284 ± 1234 μg/g-dry; the values of the <250 μm fraction in the previous study fell within this statistical range. In contrast, the total $concentration_t$ was highest in the 53–75 μm fraction for the finer fractions and decreased with decreasing particle size (Fig. S4). This indicates that MPs in the surface road dust particles have a particle size distribution with a peak of 53–75 μm. The particle size distribution of road dust also showed the percentage of particles to decrease as the fractions became finer, suggesting that the size distribution of MPs reflects the size distribution of all particles in the road dust (Fig. 1A). Another previous study examined the concentration of SBR and TWP as tire origin particles; the authors used thermal extraction desorption-gas chromatography-mass spectrometry (TED-GC/MS) to measure SBR in road dust with particle sizes <20–500 μm and reported the concentrations range of 0.46–0.76 mg/g (Klöckner et al., 2020). A previous analysis using pyr-GC/MS reported tire particle concentrations comprising SBR, BR, and styrene/butadiene/styrene (SBS) within the range of 0.6–258 mg/g in road dust for particle sizes <1.6 μm (Rødland et al., 2022). Moreover, the reported TWP concentration may increase, e.g., to 110–120 mg/g if other inorganic tire particles are included in the analysis (Klöckner et al., 2021). In the present study, the total concentration of SBR in road dust (with particle size 1.1–850 μm) ranged from 0.2 to 1.9 mg/g (Table S5), which is similar to the results of Klöckner et al. (2020). And it was also comparable to lower limits of the concentrations reported by Rødland et al. (2022), although this study contained other tire components. Furthermore, Rødland et al. (2022) and Klöckner et al. (2021) were collected in tunnels, where tire wear particles tend to accumulate (Klöckner et al., 2021), so their concentrations are expected to be higher than in the open road surface such as in this study. Next, to see the micronization tendency of MPs, we determined the concentration of MPs in road dust particles by particle size fraction ($concentration_f$). The units were μg-MP in the particle size fraction/g-dry of road dust in the particle size fraction.

Fig. 3A shows the total $concentration_f$ of the nine types of MP in the surface road dust by particle size. The $concentration_f$ of the nine types of MP in the 250–500 μm and 500–850 μm fractions were 871–1510 μg/g and 928–1659 μg/g, respectively. The $concentration_f$ of the nine types of MP began to increase from the 125–250 μm fraction, and in fractions <75 μm, the $concentration_f$ ranged from a few mg/g up to a few tens of mg/g. Thus, the MP $concentration_f$ was higher in the fine particle fractions of <250 μm than in the coarse particle fractions, and this high $concentration_f$ was maintained down to 1.1–2.1 μm fraction.

The $concentration_f$ of each polymer type in the surface road dust by particle size is shown in Fig. 3B–3H. The $concentration_f$ of PE began to increase from the 53–75 μm fraction and remained elevated until the 1.1–2.1 μm fraction (Fig. 3B). The $concentration_f$ of PVC increased in the 53–75 μm fraction and remained elevated until the finest particle size fraction (Fig. 3C). Particles with an aerodynamic diameter less than 10 μm (PM_{10}) are expected to have the capacity to penetrate the respiratory system (Grigoratos and Martini, 2014; Kim et al., 2015), and MPs in road dust of this size fraction have not been fully investigated. In this study, particle size fractions of <11 μm were considered as PM_{10}. Among fractions of the size that contribute to PM_{10}, there was 1048–5150 μg/g PE and 1553–6390 μg/g PVC. On the other hand, the $concentration_f$ of SBR was highest in the 20–38 μm fraction and then tended to be stable or decrease as the fractions became finer (Fig. 3D). The SBR $concentration_f$ in fractions of the size that contributes to PM_{10} was 1074–7956 μg/g. Although SBR had the highest maximum $concentration_f$ among all of the nine types of MP, the micronization of most SBR particles may have stopped at a particle size of around a few tens of micrometers. The sizes of particles generated by tire friction are said to be in the range of 4–350 μm (Kreider et al., 2010), which is roughly consistent with the micronization trend of SBR in this study. In addition, Rausch et al. (2022), which investigated TWP in particle size of 1–80 μm in the air in the vicinity of a main urban road, found that the 10–80 μm size fraction accounted for 83% of the total TWP, with higher relative contributions in the coarser size fractions compared to other particles such as Minerals and biogenetic/organic particles (Rausch et al., 2022). The results of this study were similar to the trends in the particle size distribution of TWP in the air in Rausch et al. (2022). Furthermore, finer tire wear in PM_{10} may be directly diffused into the atmosphere and not included in road surface dust (Grigoratos and Martini, 2014), and further investigation is needed to determine the extent to which SBR is micronized and present in the environment. In contrast, PE and PVC are even finer and may be contained in dust particles with a particle size of only a few micrometers. The $concentration_f$ of PS tends to start to increase with the size fraction 53–75 μm, and in size fractions of <11 μm, the $concentration_f$ ranged from 23 to 709 μg/g (Fig. 3E). Since PS has a benzene ring and a high affinity for highly hydrophobic PAHs, the adsorption amount of PAHs is reported to be 17 times higher than that of PE (Yukioka et al., 2018). For this reason, although the $concentration_f$ of PS is lower than that of PE, it may be comparable to PE as a carrier of aromatic hydrocarbons. The $concentration_f$ of ABS was 9–377 μg/g and tended to increase as the particle size decreased (Fig. 3F). ABS is used in the internal parts of automobiles and is reported to harden and deteriorate when exposed to ultraviolet light for a long time (Yildirim et al., 2022). Our results suggest that ABS particles may have become even finer than 1 μm, but further investigation is needed to investigate how much finer. The $concentration_f$ of PA66 increased from the 125–250 μm fraction and remained elevated in fractions down to 1.1–2.1 μm (Fig. 3G). The PP and PC $concentration_f$ are shown in the supplementary material as reference values since all samples were below IQL (Fig. S5).

From the above, this study shows the occurrence of MPs in surface road dust of each particle size fraction. The total MP $concentration_f$ began to increase from the 125–250 μm fraction and remained elevated in finer fractions down to 1.1 μm, indicating that MPs in the road dust micronized to at least 1.1 μm. However, the tendency for $concentration_f$ of individual polymer types to increase or decrease with particle size fraction varied. Further research is needed to understand the factors that

Table 2
Results of three-way analysis of variance testing factors affecting MPs in surface road dust by particle size.

Sources of variance	Sum of square	Sum of modified square	Degrees of freedom	Contribution ratio (%)	Proportion of variance	Significant
Particle size (PS)	7.65×10^7	7.43×10^7	12	5.8	33.9	***
Polymer type (PT)	6.99×10^8	6.97×10^8	11	54.2	337.7	***
Sampling point (SP)	4.68×10^7	4.61×10^7	4	3.6	62.2	***
PS*PT	2.19×10^8	1.94×10^8	132	15.1	8.8	***
PS*SP	2.26×10^7	1.35×10^7	48	1.1	2.5	***
PT*SP	1.22×10^8	1.14×10^8	44	8.8	14.7	***
residuals	9.93×10^7	1.47×10^8	528	11.4		

***, $P < 0.001$.

characterize the occurrence of each polymer in road dust of each particle size fraction, including the comminution limits, micronization mechanisms, and transport of each polymer under pertinent environmental conditions.

MPs on the road surfaces may diffuse into other environments along with stormwater runoff or via air (Österlund et al., 2023). The importance of runoff for the transport and occurrence of MPs in urban areas with high impervious surfaces has been noted (Goehler et al., 2022). In particular, the emissions of tire-derived particles, which occur primarily on road surfaces, are influenced by road imperviousness and road length related to vehicle traffic (Goehler et al., 2022), and it is expected to account for a high proportion of MP emissions to rivers (Siegfried et al., 2017) and oceans (Friot and Boucher, 2017). In many areas of the world, MPs in runoff is not fully treated and released into the environment (Bondelind et al., 2020; Siegfried et al., 2017), making a serious impact on the environment and living organisms (Barnes et al., 2009). In addition, the finer the particle size, the more that the particles may migrate into the atmosphere, so it is necessary to clarify the particle size distribution of MPs in the atmosphere to correctly evaluate their effects on humans through the respiratory system.

3.4. Factors affecting the concentration of MPs in road dust, by particle size

To elucidate the effects of factors influencing the concentration of each of the MPs in the surface road dust by particle size ($concentration_f$), a three-dimensional analysis of variance was performed with particle size fractions, the nine types of polymer, and the sampling points. The results are shown in Table 2. The most influential factor was the particle size fraction, which explained 54.2%. The interaction between particle size fraction and polymer type explained 15.1%. These two factors together explained about 70%. This result is considered to reflect our finding that the $concentration_f$ of MPs was higher in the finer fractions than in the coarser fractions and that there was a difference in the micronization tendency for each polymer type. The analysis of variance explained about 90% of the plastic $concentration_f$ by particle size by including the sampling points and interactions.

The contribution of the sampling point was relatively small; however, may it sporadically show a significant effect. Yukioka et al. (2020) compared the sizes and quantities of MPs in road dust with a particle size of 100–5000 μm in Kusatsu (Japan), Da Nang (Vietnam), and Kathmandu (Nepal) and found that the particle sizes of MPs were finer in Da Nang and Kathmandu than in Kusatsu. This may be due to heavier traffic or the fact that in developing countries, the road surface was not cleaned often, and plastics were left to degrade on the road surface for a longer period (Yukioka et al., 2020). O'Brien et al. (2021) and Abbasi et al. (2017) also reported that higher traffic volume and longer residence time result in finer particles in road dust. In addition, studies on tire wear have reported that driving behavior (speed, frequency, and extent of braking), tire characteristics (i.e., size, tread depth, chemical composition), and road surface characteristics (i.e., material, porosity, and condition) influence the generation, composition, and size distribution of tire wear particles (Grigoratos and Martini, 2014; Sommer et al., 2018). Although this study selected sites with different traffic volumes, it is possible that there were no large differences in the frequency of road surface cleaning, tire and road surface characteristics, and driving behavior among the sites because they were geographically close to one another.

4. Conclusion

This study showed the occurrence of MPs of various polymers in surface road dust with particle size fraction in the range of 1.1–850 μm. The finer the particle size of the dust fraction is, the smaller the abundance of surface road dust and the higher the organic matter content. The total concentration of nine MPs in the road dust by particle size ($concentration_f$) began to increase from the 125–250 μm particle size fraction and remained elevated down to finer particle size fractions. In addition to plastics with an extensive production volume (such as PE, PVC, and PS), SBR and ABS, which are used as car parts, had also been micronized and were present in the surface road dust. The tendency for $concentration_f$ of each polymer to increase or decrease with particle size fraction varied: the $concentration_f$ of some polymers, such as PE and PVC, remained elevated in fractions down to 1.1 μm; the $concentration_f$ of SBR, a rubber-MP, showed a stable or decreasing trend in fractions of 7.0–11 μm and finer. Further research is needed to understand the factors that characterize the occurrence of each polymer in road dust, including the comminution limits, micronization mechanisms, and transport of each polymer under pertinent environmental conditions.

MPs on the surface of roads diffuse into other ecosystems along with rainwater or via air, and the finer the particle size, the greater the impact they will have on organisms through feeding and through the exposure pathways of dermal contact and inhalation. To discuss the effects of MPs generated from road surfaces on living organisms, further investigations, such as the amounts of organic pollutants adsorbed onto MPs in road dust and the size distribution of MPs in the atmospheric environment, are needed.

Author statement

Tamaki Morioka: Writing - Original Draft, Formal analysis, Visualization. **Shuhei Tanaka:** Conceptualization, Supervision, Funding acquisition. **Yuta Yamada:** Conceptualization, Methodology, Investigation, Visualization. **Satoru Yukioka:** Investigation, Supervision. **Fumihiro Aiba:** Investigation, Validation.

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

Data will be made available on request.

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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.2023.122198.

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