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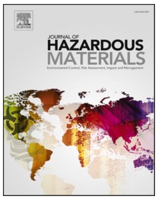

Airborne microplastic monitoring: Developing a simplified outdoor sampling approach using pollen monitoring equipment

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Abbreviations: A/CO/PS, alkyd, castor oil, styrene blend; ABS, acrylonitrile butadiene styrene terpolymer; ABS/PC, acrylonitrile butadiene styrene/polycarbonate; EEA, ethylene/ethyl acrylate copolymer; EPDM, poly(ethylene:propylene:diene); EVA, ethyl vinyl acetate; EVOH/EVA, ethylene vinyl alcohol/ethylene vinyl acetate tie layer; HDPE, high-density polyethylene, LDPE, low-density polyethylene; LOD, limit of detection; LOQ, limit of quantification; MCT, mercury cadmium telluride; MP, microplastic; PA, nylon 6; PAC/PA, poly(acrylate:styrene); PBA, polybutyl acrylate; PBA/PAN, poly(butyl acrylate), poly(acrylonitrile), PBAT, poly(1,3-butanediyl adipate); PBMA, poly(11-bromoundecyl methacrylate); PBT, poly(butylene terephthalate); PCS/PS, Poly(4-chlorostyrene:styrene); PE, polyethylene; PE/EEA, polyethylene/ethyl acrylate copolymer; PE/TiO_2, polyethylene white layer (titanium dioxide); PEA, poly(ester amide); PESTI, polyester tere & iso-phthalate; PEA/PS/PAM, poly(ethyl acrylate:styrene:acrylamide); PEMA, poly(ethyl acrylate:methacrylate); PET, polyethylene teraphalate; PEUR, polyetherurethane; PMA, poly(methacrylate); PMMA, poly(methyl methacrylate); PMP, poly(4-methyl-1-penten-3-one) and poly(4-methyl-1-hexen-3-one:4-methyl-1-penten-3-one); PMA/PS, Poly(methacrolein:styrene); PO, polyolefin; PP, polypropylene; PP/EPDM, polypropylene/ethylene propylene diene rubber copolymer; PP/PE, polypropylene-polyethylene copolymer; PPA, polyphthalamide; PPI, poly(pentadecyl isocyanate); PS, poly(styrene); PS/BMA, styrene/butyl methacrylate copolymer; PS/PMMA, poly(styrene:methyl methacrylate); PTFE, polytetrafluoroethylene; PU, polyurethane; PVA, polyvinyl alcohol; PVC, polyvinyl chloride; SAE, poly (styrene:acrylate ester); SAN, poly(styrene:acrylonitrile); SBR, styrene/butadiene copolymer; SEB, styrene/ethylene-butylene, ABA block copolymer; TPE, thermoplastic elastomers; TSP, total suspended particulates; μFTIR, micro-Fourier transform infrared.

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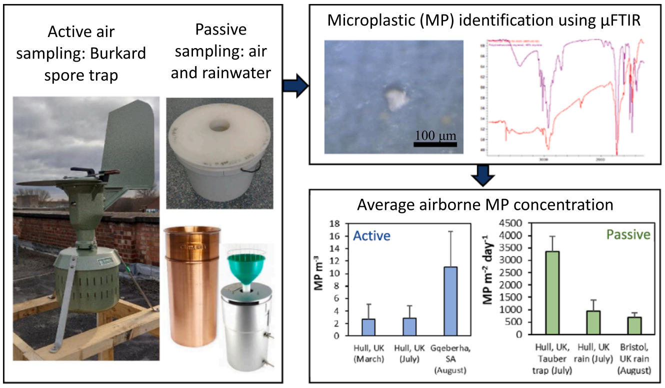
HIGHLIGHTS

- Pollen monitoring equipment is effective at capturing airborne microplastics.
- MPs were detected in 95 % of daily samples from sites in the U.K. and South Africa.
- Up to 29 polymer types were detected, with nylon as the most abundant.

GRAPHICAL ABSTRACT



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ABSTRACT

A novel, yet simple, airborne microplastic (MP) sampling approach using global pollen monitoring equipment was applied to identify, characterise and quantify outdoor airborne MPs for the first time. Modification of Burkard spore trap tape adhesive provided particle capture and facilitated downstream spectroscopy analysis. 36 polymer types were identified from a total of 21 days sampling using Burkard spore traps at two locations (United Kingdom and South Africa). MPs were detected in 95 % of daily samples. Mean MP particle levels were 2.0 ± 0.9 MP m^{-3} (11 polymer types) in Hull (U.K.), during March, 2.9 ± 2.0 MP m^{-3} (16 types) in Hull in July, and 11.0 ± 5.7 MP m^{-3} (29 types) in Gqeberha, (S.A.) in August 2023. The most abundant polymer type was nylon (Gqeberha). The approach was compared with two passive sampling methods whereby 27 polymer types were identified and of these, 6 types were above the limit of quantification (LOQ), with poly(methacrolein:styrene) (PMA/PS) the most abundant. Irregularly shaped MPs < 100 μm in length were predominant from all sampling approaches. For the first time, airborne MPs were chemically characterised and quantified using volumetric pollen sampling equipment, representing a viable approach for future airborne MP monitoring.

1. Introduction

Microplastics (MPs), produced by primary manufacturing or secondary degradation, are synthetic polymer particles ranging in size from 1 μm to 5 mm [1]. MPs are considered ubiquitous environmental contaminants [2,3]. Airborne microplastics (AMPs) have been detected outdoors from head height up to the planetary boundary layer [4-7]. The atmosphere is considered a sink of MPs that contaminate the land and oceans as AMPs can be transported hundreds of kilometres [4,8]. AMPs have also been quantified in indoor settings [9-12] resulting in unavoidable exposure. Indeed, MPs have been detected in human lung and respiratory tract [13,14]. Experimental exposures of human cells to MPs cause cytotoxic effects [15] and the leaching of additives or sorbed contaminants is of concern to both the environment and public health [16]. While the clinical implications of MPs have yet to be determined, there is a pressing need for standardised protocols for comprehensive risk assessments [17].

AMPs are not currently monitored though have been reported from ad hoc studies with concentrations ranging from > 1 to < 1000 MP m^{-3} (Table S1, S2). Fibres and fragments are the dominant MP shape depending on the study involved (Table S1, S2). Predominant polymer types also differ (Table S1, S2), though polyethylene terephthalate (PET) and polypropylene (PP) have been reported as most common [18].

AMPs can be collected using passive or active sampling methods. Passive sampling collects particle fallout with concentrations reported in MP m^{-2} day^{-1} (Table S1). Active sampling involves the use of a pump enabling MP m^{-3} to be quantified (Table S2); equipment includes total suspended particulate (TSP) samplers (Table S2), gravimetric samplers [19] and purpose-built equipment [4,20]. Sampling equipment and methodologies impact upon the MP concentrations quantified, highlighting the need for a standardised approach [21-23]. Burkard traps are Hirst-type vacuum powered particle impaction samplers used for global pollen monitoring at hundreds of sites worldwide [24,25], and have also been used to capture inorganic airborne particulates [26,27]. This approach allows all fine particulates to be sampled from a consistent volume of air during continual 7-day sampling without the presence of an operator, providing sample resolution down to hourly intervals [28,29]. Recently, AMPs have been visually identified and quantified from Burkard trap samples [30], though they are yet to be chemically characterised due to incompatibility between the commonly used trap tape/adhesives and downstream spectroscopy techniques, requiring further method development [30].

This study aims to establish a simplified approach for outdoor AMP sampling from air samplers already in operation for global pollen monitoring. A protocol was developed to capture outdoor AMPs with Burkard traps, separate the particles from the sampling media, and chemically identify MP polymers. AMPs were characterised and quantified from three approaches of sampled air as well as from field and laboratory blanks, with limits of detection and quantification (LOD/LOQ) applied to the polymers detected. This work provides a necessary step towards developing a standardised, accessible means of AMP monitoring using a robust and reproducible methodology.

2. Methods

2.1. Active and passive air sampling

Active air sampling was conducted using Hirst-type 7-day recording volumetric spore trap known as a Burkard spore trap, consisting of a 14 × 2 mm orifice through which air is drawn into a chamber at a rate of 10 L/min (Fig. S1a, b) (Burkard Manufacturing Ltd., U.K.). Airborne particles impact upon adhesive tape (Fig. S1e) wound around a clockwork drum (circumference: 345 mm) which rotates at 2 mm/hr allowing continuous volumetric sampling for 7 days. Airflow is created by a pump powered by a 12 v battery charged by a solar panel. The trap has a wind vane which allows the upper section to rotate so the sampling orifice faces into the wind.

A Burkard trap was used to continuously sample air for 7 days from the roof of Hardy Building at the University of Hull (U.K.) (53° 46' 16.87" N; 0° 22' 2.64" W) commencing at 10:00 am on 10/03/2023 and 11:00 am on 18/07/2023 respectively [31]. A second Burkard trap was used for 7 days of sampling at Nelson Mandela University's South Campus in Summerstrand, Gqeberha, S.A. 34° 0'4.66"S; 25°40'2.40"E commencing at 13:00 pm on 03/08/2023. The traps were positioned on flat rooftops with the sampling orifice at least 1 m above the roof surface and at least 2 m away from the edge of the building (Fig. S2). Glycerine jelly, a commonly used Burkard trap adhesive, was selected due to its water solubility allowing its downstream removal from samples for subsequent μFTIR analysis; alternate commonly used adhesives including Mowiol (poly vinyl alcohol based), petroleum jelly and silicone were not suitable for this purpose. Its preparation for use on tape and trap loading is detailed in Supplementary Method SM1.

For comparison, passive air sampling was conducted in Hull alongside the July Burkard trap sampling run at the same rooftop location on the same dates. Samples were collected using Tauber traps ($n = 2$) and Palmex RS1 Rain Samplers (Palmex Ltd.) ($n = 2$) for 7 days (Fig. S1c, d). Tauber traps are used to measure passive deposition of pollen and consist of a bucket-style container with a tightly fitted sloping collared lid containing a 5 cm diameter hole with an internal 5 mm mesh to prevent collection of large debris [32,33]. Palmex rain samplers have a 12 cm diameter funnel and are tube-dip-in-water collectors with pressure equilibration to ensure flow and have a design that prevents evaporation. Passive air sampling was also conducted at the University of the West of England Bristol (UWE) Frenchay Campus, Bristol, U.K. (51° 30' 1.38" N; −2° 32' 53.36" W) using rooftop copper rain gauges (ClimeMET, U.K.) ($n = 3$) consisting of a 12.3 cm diameter copper and brass funnel housed within a copper body. Sampling commenced at 11:00 am on 04/08/2023 for 7 days. All rainwater samples were collected into pre-cleaned glass bottles/flasks.

2.2. Sample processing and filtration

At the end of the 7-day sampling period, a needle was used to score the stop position on the tape inside the Burkard trap through the sampling inlet. The sample drum was transported to the lab in a dust-free lidded container. The tape was cut into 1-day (48 mm) sections with stainless steel scissors using a graduated cutting block. Each day section was then cut along the parallel direction of the middle of the tape, and the top half was mounted on a slide using glycerine jelly for microscopy and the bottom half underwent oxidative digest using pre-filtered 30 % hydrogen peroxide solution to remove organic material for subsequent μFTIR analysis. All sample digests were performed at the University of Hull which involved placing the ½ day section of tape whether unmounted (U.K. samples) or mounted on slides with glycerine jelly (S.A. samples prepared this way to enable shipping) into a beaker with 50 mL filtered water and incubating at 60 °C overnight. All slides were thoroughly pre-rinsed with filtered water beforehand. The following day, the tape, slides and cover slips were removed with forceps and rinsed with a total of 200 mL filtered hydrogen peroxide into the beaker.

For the Tauber traps, after sampling the trap lids were covered with tin foil, transported to the lab and the collection buckets of the traps were rinsed with 200 mL filtered hydrogen peroxide into a 1 L glass flask. Glass flasks/bottles from rainwater samplers were also covered with tin foil, transported to the lab and weighed before and after an incubation at 60 °C for approx. 1 week to evaporate all water and to calculate rainwater volume. 200 mL filtered 30 % (w/v) hydrogen peroxide solution (Fisher Scientific, Loughborough, U.K.) was then added to each sample. The evaporation step avoided the dilution effect of adding hydrogen peroxide directly to the water. Tauber traps did not contain rainwater so this step was not required.

Sample beakers/flasks from all sampling methods were covered with tin foil and incubated at 60 °C at 80 rpm for 5 days. Samples were filtered using glass vacuum filtration equipment onto Anodisc 47 mm aluminium oxide filters with a 0.2 μm pore size (Anodisc, Watford, U.K.). 200 mL of filtered water was used to rinse the sample beaker/flask and filter through the anodisc to ensure maximum particle recovery. Anodiscs were placed in a petri dish and left to dry for at least 24 hr before μFTIR analysis. Procedural blanks were processed and filtered the same way as samples (Supplementary methods SM1).

2.3. Chemical characterisation of particles using μFTIR

All particles > 10 μm were analysed directly from ¼ of each anodisc using a Nicolet iN10 μFTIR microscope in cooled transmission mode using liquid nitrogen to cool the mercury cadmium telluride (MCT) detector (ThermoFisher, Waltham MA, USA). The Nicolet iN10 instrument has a 15 × 0.7 numerical aperture high efficiency objective and condenser, and independent reflection and transmission illuminations. The following parameters were used for μFTIR analysis: a spectral range of 4000–1250 cm^{-1}, a high spectral resolution of 8 cm^{-1}, and a scan number of 64. A background spectrum was obtained before each sample spectrum. Sample spectra were compared to a database of spectra (Omnic Picta, Omnic Polymer Libraries). Sample spectra were not transformed, smoothed or baseline corrected. Only particles with a ≥ 70 % match were recorded and only those that were MPs were included in the results presented [34]. Particle length and width (longest and 90° to longest dimension) were measured using the aperture size tool in the Omnic Picta software (ThermoFisher, Waltham MA, USA) at a standard 123 × magnification. Particle shapes were recorded as 'irregular' in the case of fragments and films [35] or as 'fibre' where the length to width ratio was > 3 [36]. The Burkard trap spectra dataset has been made available at the following DOI: 10.6084/m9.figshare.25460650.

A number of quality assurance, strict control measures (including procedural blanks) and spiking experiments to test the laboratory processes were used to reduce the impact of the ubiquitous nature of AMPs and to quantify and characterise any unavoidable background contamination (Supplementary Methods SM1 and SM2).

2.4. Data analysis

MP data are presented herein as follows: unadjusted, blank-subtracted (using the mean of the procedural blank values regardless of polymer type), and a LOD/LOQ method [37]. Unadjusted values for MPs in the original whole sample are calculated from raw count data from subsamples i.e. ½ of 1-day Burkard trap tape analysed and ¼ of anodisc analysed by multiplication assuming even particle distribution on the Burkard trap tape and anodisc filters. Blank-subtracted values were calculated as the average MPs in the original whole relevant blanks subtracted from the MPs in the original whole samples. LOD/LOQ values were calculated for each individual MP polymer type which were individually blank-subtracted (Supplementary Method SM2).

Tests for normality, homogeneity and significance were performed on unadjusted MP values using GraphPad InStat v3 (GraphPad Software Inc., La Jolla, U.S.A.). The non-parametric Kruskal-Wallis test, followed by Dunn's multiple comparisons post-hoc tests, were applied to unadjusted AMP concentration data and to AMP length and width data. A significance level of $p < 0.05$ was applied.

3. Results

3.1. MP abundance

The total number of particles, both MPs and non-MPs, identified from all active and passive air sampling approaches and locations combined was 1065, of which 299 (28 %) were MPs. 199 MPs were detected from a total of 21 days of active air sampling using Burkard traps (analysing a 1/8 subsample of each day) from 3 sampling runs at 2 locations (Hull-March, Hull-July and Gqeberha-August). MPs were detected in 20/21 (95 %) samples, and only absent from Hull-July from day 4. On average (unadjusted average ± standard deviation), 2.0 ± 0.9 MP m^{-3} (range 1.1–3.3) were detected from Hull-March, 2.9 ± 2.0 MP m^{-3} (range 0–5.6) from Hull-July and 11.0 ± 5.7 (range 2.8–21.1) from Gqeberha-August (Table 1). There was a significant difference in unadjusted MP levels between sampling run ($p = 0.0087$), with significantly higher MP levels in Gqeberha-August compared to Hull-March ($p < 0.05$). Unadjusted MP levels in Gqeberha-August were significantly higher than in the combined blanks ($p < 0.05$). There was no significant difference in MP levels between Gqeberha-August and Hull-July or between the two Hull (March v July) sampling runs ($p > 0.05$).

Using passive air sampling approaches, 100 MPs were detected in total from 7 days deposition into Tauber traps (Hull-July, n = 2), Palmex rain samplers (Hull-July, n = 2) and copper rain gauges (Bristol-August, n = 3) with a 1/4 subsample of each analysed. MPs were detected in all samples and unadjusted averages were 3344 ± 617 MP m^{-2} day^{-1} for Tauber traps, 935 ± 464 MP m^{-2} day^{-1} for Hull rainwater and 696 ± 174 MP m^{-2} day^{-1} for Bristol rainwater (Table 2).

Table 1
Number of MPs identified per m^3 of air in the Burkard trap samples by µFTIR spectroscopy. Three different contamination adjustments are used to display results: † unadjusted, †† blank subtracted average, ††† LOD/LOQ adjusted values >LOQ. Abbreviations: EPDM, Poly(ethylene:propylene:diene); LDPE, low-density polyethylene; PA, nylon 6; PET, polyethylene terephthalate; PO, polyolefin; PTFE, polytetrafluoroethylene; SD, standard deviation.

Sample	MP m^{-3} †	MP m^{-3} ††	MP m^{-3} †††
Hull-March			
Day 1	1.1	0.6	0
Day 2	3.3	2.8	PET 1.67
Day 3	1.1	0.6	0
Day 4	2.2	1.7	0
Day 5	2.8	2.2	PTFE 1.67
Day 6	1.1	0.6	0
Day 7	2.2	1.7	0
Mean	**2.0**	**1.5**	**0.5**
SD	0.9	0.8	0.8
Hull-July			
Day 1	0.6	0	0
Day 2	5.6	4.4	LDPE 2.22
Day 3	3.9	2.8	LDPE 1.67
Day 4	0	0	0
Day 5	4.4	3.3	0
Day 6	2.8	1.7	0
Day 7	2.8	1.7	0
Mean	**2.9**	**2.0**	**0.6**
SD	1.9	**1.5**	0.9
Gqeberha-August			
Day 1	12.2	5.7	0
Day 2	2.7	0	0
Day 3	6.7	0.2	0
Day 4	21.1	14.6	Nylon 8.14; PA 6.66
Day 5	12.8	6.3	EPDM 4.81
Day 6	9.4	7.0	PO 1.67
Day 7	11.7	5.2	0
Mean	**11.0**	**5.6**	**3.0**
SD	5.7	4.5	5.1

Table 2
Number of MPs identified from Tauber traps and rain sampler by µFTIR spectroscopy. Three different contamination adjustments are used to display results: † unadjusted, †† blank subtracted average, ††† LOD/LOQ adjusted values >LOQ. Abbreviations: EVA, ethyl vinyl acetate; PBMA, poly(11-bromoundecyl methacrylate); PMA/PS, poly(methacrolein/styrene); PP/PE, polypropylene-polyethylene copolymer; PTFE, polytetrafluoroethylene.

Sample	MP $m^{-2}day^{-1}$†	MP $m^{-2}day^{-1}$ ††	MP $m^{-2}day^{-1}$ †††
Hull-July Tauber traps			
1	2910	1455	0
2	3783	2328	PMA/PS 1164.09
Mean	**3347**	**1892**	**582**
SD	617	617	823
Hull-July Rain samplers			
2	606	429	PTFE 151.57
3	1263	1086	PP/PE 202.10; EVA 202.10; PBMA 151.57
Mean	**935**	**758**	**354**
SD	464	464	286
Bristol-August Rain samplers			
1	507	91	0
2	850	452	0
3	732	732	Alkyd resin 144.26
Mean	**696**	**311**	**48**
SD	174	193	83

3.1.1. MP polymer types

36 MP polymer types were identified in total from Burkard traps: 11 MP types from Hull-March, 16 types from Hull-July, and 29 types from Gqeberha-August (Fig. 1A). Concentrations of the various AMP polymer types are shown in Fig. 1A. The most abundant MP types in Hull-March were polytetrafluoroethylene (PTFE, 24 %), polystyrene (PS, 16 %) and PET (16 %) whereas low-density polyethylene (LDPE, 33 %), poly(ethylene:propylene:diene) (EPDM, 14 %) and PS (8 %) were most abundant in Hull-July (Fig. 2a, b). The most abundant MP types in Gqeberha-August were nylon (6/9, 6/12) (19 %), PS (12 %) and PTFE (11 %) (Fig. 2c). The Burkard blanks contained a total 20 MP polymer types: 1 MP type in Hull-March, 6 types in Hull-July and 14 types in Gqeberha-August with the most abundant from each sample run being ethylene/propylene copolymer (EPR, 100 %), poly(ethyl acrylate:styrene:acrylamide) (PEA/PS/PAM, 37 %) and styrene/butadiene copolymer (SBR, 26 %) respectively (Fig. S4). 10/20 (50 %) of the polymer types found in the blanks were also detected in the corresponding samples (Table S4, S5, S6). After LOD/LOQ calculations, 6 MP polymers were detected over the LOQ limit in Burkard traps: PTFE, PET (Hull-March), LDPE (Hull-July), nylon (6, 6/9, 6/12), EPDM and polyolefin (PO) (Gqeberha-August) (Table 1). Common MPs detected herein from both active and passive air sampling approaches are pictured in Fig. S3 alongside their µFTIR spectra.

The total and variety of MP polymers differed depending on the passive sampling method adopted with 27 MP polymer types detected in total: 11 types in Hull-July Tauber traps, 16 in Hull-July rainwater samples and 12 in Bristol-August rainwater (Fig. 1B). The most abundant MP types also differ depending on the sampling method adopted (Fig. 1B). For the Hull sampling sites, Tauber traps captured polypropylene/polyethylene (PE/PP, 26 %), polyurethane (PU, 18 %) and poly(methacrolein/styrene) (PMA/PS, 18 %) (Fig. 2d), while rain samplers also captured PP/PE copolymer (14 %), but also ethyl vinyl acetate (EVA), PP and poly(11-bromoundecyl methacrylate) (PBMA) (each at 11 %) (Fig. 2e). At the Bristol-August rain sampling location, the most abundant MP types differed from the Hull sampling site; with PTFE (52 %) in abundance followed by alkyd resin and poly(methacrolein/styrene) (PMA/PS) (both at 10 %) (Fig. 2f).

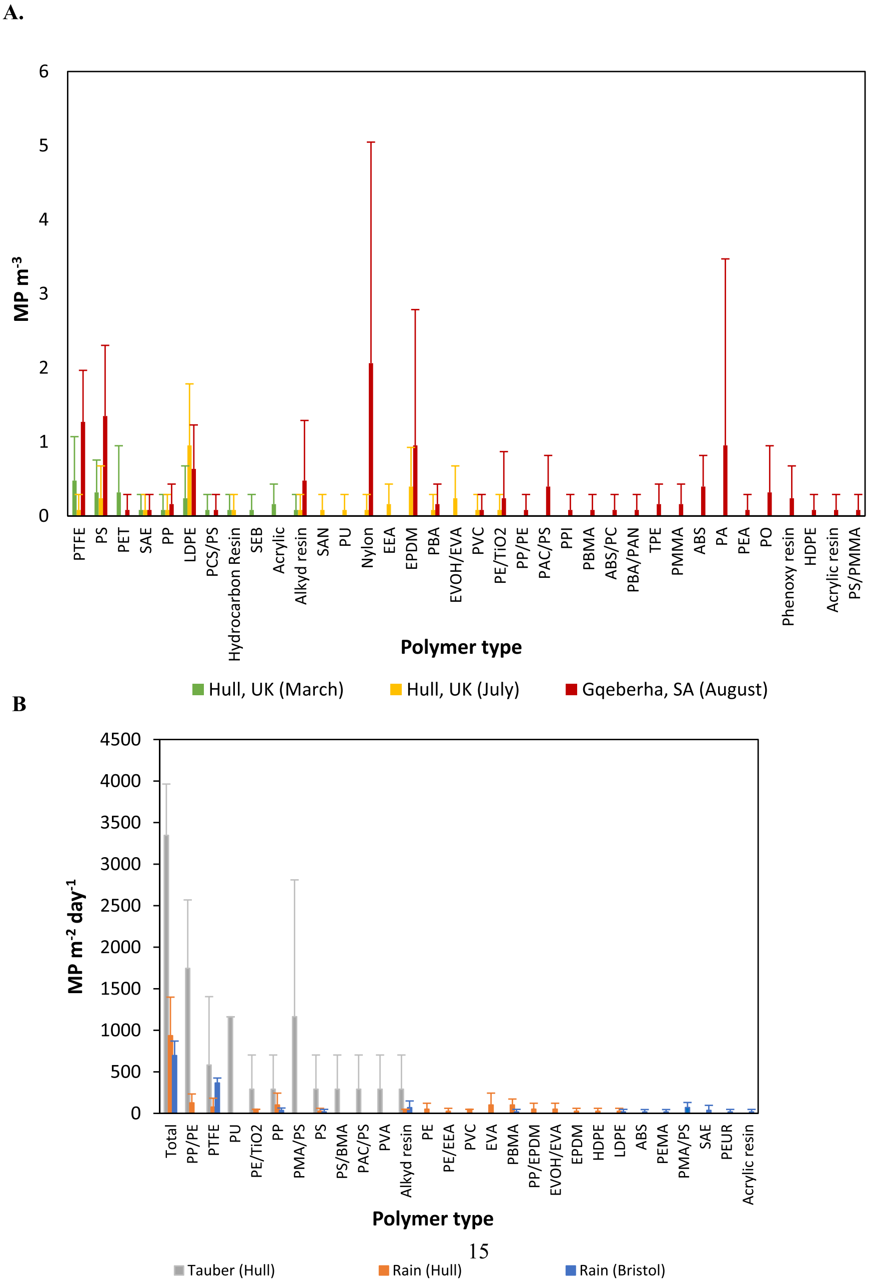
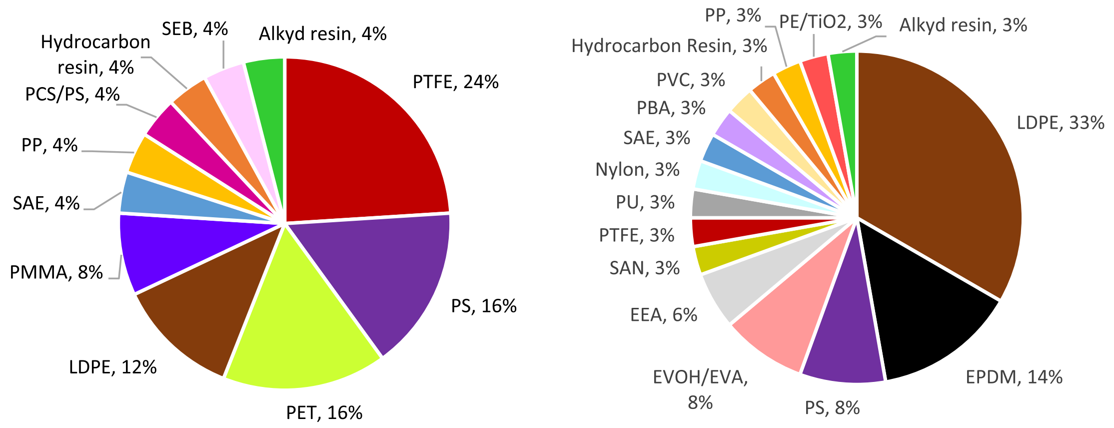


Fig. 1. A. Concentration of airborne MPs (unadjusted) in actively sampled air from Burkard traps (average MP m-^{3} ± standard deviation): Hull March, green; Hull July, yellow; Gqeberha August, red. B. Concentration of airborne MPs (unadjusted) in passively sampled air from Tauber traps and rain samplers (average MP m^{-2} day^{-1} ± standard deviation): Tauber traps, grey; Hull rain, orange; Bristol rain, blue. Abbreviations: ABS, acrylonitrile butadiene styrene terpolymer; ABS/PC, acrylonitrile butadiene styrene terpolymer/polycarbonate; EEA, ethylene/ethyl acrylate copolymer; EPDM, poly(ethylene:propylene:diene); EVA, ethyl vinyl acetate; EVOH/EVA, ethylene vinyl alcohol/ethylene vinyl acetate tie layer; HDPE, high-density polyethylene; LDPE, low-density polyethylene; PA, nylon 6; PAC/PS, poly (acrylate:styrene); PBA, polybutyl acrylate; PBA/PAN, poly(butyl acrylate)/poly(acrylonitrile); PBMA, poly(11-bromoundecyl methacrylate); PCS/PS, poly(4-chlorostyrene:styrene); PE, polyethylene; PEA, poly(ester amide); PE/EEA, polyethylene/ethyl acrylate copolymer; PE/TiO2, polyethylene white layer; PEMA, poly(ethyl acrylate:methacrylate); PET, polyethylene teraphthalate; PEUR, polyetherurethane; PMA/PS, poly(methacrolein/styrene); PMMA, poly(methyl methacrylate); PO, polyolefin; PP, polypropylene PP/PE, polypropylene-polyethylene copolymer; PPI, poly(pentadecyl isocyanate); PS, polystyrene; PP/EPDM, polypropylene/poly(ethylene:propylene:diene); PP/PE, polypropylene-polyethylene copolymer; PS/BMA, styrene/butyl methacrylate copolymer; PS/PMMA; polystyrene/poly (methyl methacrylate); PTFE, polytetrafluoroethylene; PU, polyurethane; PVA, polyvinyl alcohol; PVC, polyvinyl chloride; SAE, poly(styrene: acrylate ester); SAN, poly(styrene:acrylonitrile); SAE, poly(styrene:acrylate ester); SEB, styrene/ethylene-butylene, ABA block copolymer; TPE, thermoplastic elastomers.

10 MP polymer types were detected in passively sampled blanks: 6 MP types in Tauber traps, 5 in Hull rain and 1 type in Bristol rain (Fig. S4). Of these polymers, 9/12 (75 %) were also detected in the corresponding samples. After LOD/LOQ calculations, a total of 6 MP polymer types were detected over the LOD and LOQ as follows: PMA/PS (Tauber-July trap 2), PTFE (Hull-July rain sampler 2), PP/PE, EVA, PBMA (Hull-July rain sampler 3), and alkyd resin (Bristol-August rain sampler 3) (Table 2, S7, S8, S9, Fig. S3). Tauber trap buckets were made of PE/PP and particles of this copolymer were detected in samples and blanks but were found to be <LOQ (Table S3, S7). Similarly, Palmex rain sampler funnels were made of PP which was detected in Hull-July rain samples and blanks but was below the LOQ threshold (Table S3, S8).

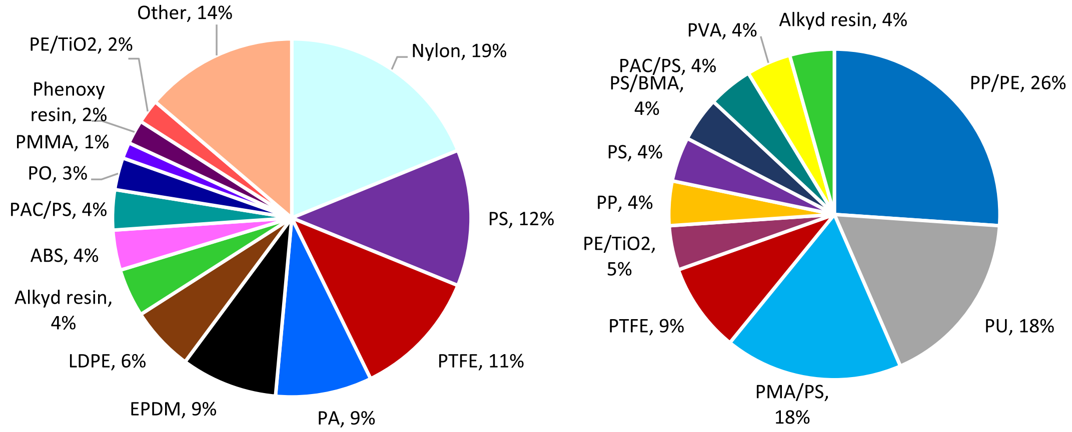
(a) Hull-March Burkard trap

(b) Hull-July Burkard trap



(c) Gqeberha-August Burkard trap

(d) Hull-July Tauber traps



(e) Hull-July rain samplers

(f) Bristol-August rain samplers

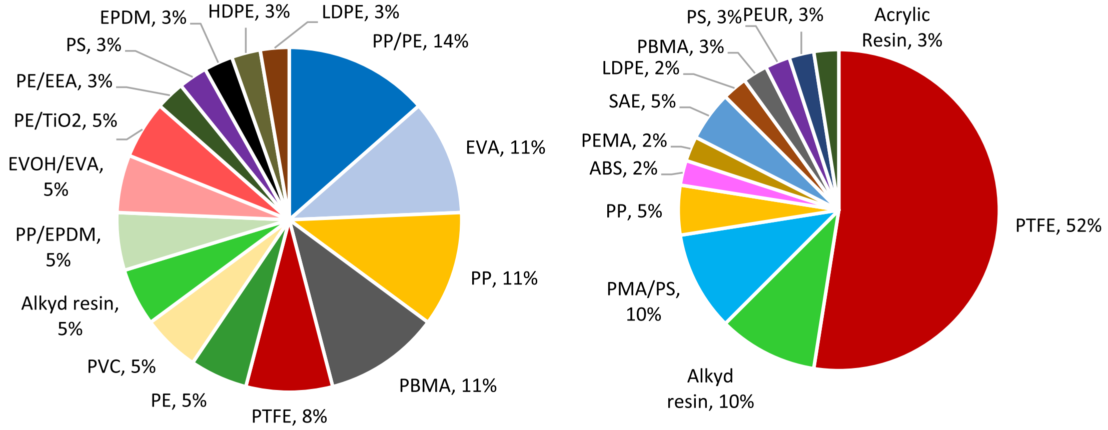


Fig. 2. Microplastic polymer types (unadjusted) identified from 7 days sampling from Burkard traps in (a) Hull-March, (b) Hull-July, (c) Gqeberha-August and (d), Hull-July Tauber traps, (e) Hull-July rain samplers and (f) Bristol-August rain samplers. Abbreviations: ABS, acrylonitrile butadiene styrene terpolymer; EEA, ethylene/ethyl acrylate copolymer; EPDM, poly(ethylene:propylene:diene); EVA, ethyl vinyl acetate; EVOH/EVA, ethylene vinyl alcohol/ethylene vinyl acetate tie layer; HDPE, high-density polyethylene; LDPE, low-density polyethylene; PA, nylon 6; PAC/PS, poly(acrylate:styrene); PBA, polybutyl acrylate; PBMA, poly(11-bromoundecyl methacrylate); PCS/PS, poly(4-chlorostyrene:styrene); PE, polyethylene; PE/EEA, polyethylene/ethyl acrylate copolymer; PE/TiO2, polyethylene white layer; PEMA, poly(ethyl acrylate:methacrylate); PET, polyethylene teraphalate; PEUR, polyetherurethane; PMA/PS, poly(methacrolein/styrene); PMMA, poly (methyl methacrylate); PO, polyolefin; PP, polypropylene; PP/EPDM, polypropylene/poly(ethylene:propylene:diene); PP/PE, polypropylene-polyethylene copolymer; PS, poly(styrene); PS/BMA, styrene/butyl methacrylate copolymer; PTFE, polytetrafluoroethylene; PU, polyurethane; PVA, polyvinyl alcohol; PVC, polyvinyl chloride; SAE, poly(styrene:acrylate ester); SAN, poly(styrene:acrylonitrile); SEB, Styrene/ethylene-butylene, ABA block copolymer.

3.2. MP particle size and shape characteristics

Mean MP particle sizes differed significantly depending on the sampling device used, with the smallest mean MP particle sizes detected in Burkard samples from Hull-July in terms of both length (68.7 ± 46.5 µm, range 26–246 µm), and width (35.6 ± 20.8 µm, range 12–102 µm) (Fig. 3, Table S10). In comparison, rain samples (from Hull-July) contained MPs with the greatest average length (183.0 ± 222.9 µm, range 30–1000 µm) and widths (103.2 ± 87.2 µm, range 20–500 µm). Average MP particle length was 81.6 ± 92.1 µm (range 18–750 µm) from active sampling of airborne particles, 142.0 ± 155.7 µm (range 20–1000 µm) from passive sampling of deposited particles, and 101.8 ± 120.4 µm (range 18–1000 µm) for all samples combined. 77 % of all actively sampled AMPs, and 59 % of passively sampled AMPs, were < 100 µm in length. Comparisons were performed between length and widths respectively for all sample types along with combined blanks for active (Burkard traps) and passive (Tauber traps and rain samplers) sampling approaches. MP lengths significantly differed between sample type ($p < 0.0001$) with significantly greater lengths in Hull-July rain compared to Hull-July Burkard samples ($p < 0.01$) and Gqeberha-August Burkard samples ($p < 0.001$) (Fig. 3). MP widths were also found to differ between groups ($p < 0.001$) with those from Tauber Hull-July traps significantly longer than those from Burkard samples from Hull-March ($p < 0.001$), Hull-July ($p < 0.01$) and Gqeberha ($p < 0.05$) respectively. AMPs from Hull-July rain samples also had a significantly higher average widths than those from all three Burkard traps (all $p < 0.001$), and those from Bristol-August rain were significantly greater than Hull-March Burkard AMPs ($p < 0.05$). Furthermore, MP widths from Hull rainwater samplers were significantly longer than those from the combined passively sampled blanks ($p < 0.01$). Hull-July Burkard average widths were significantly shorter than the combined actively sampled blanks ($p < 0.05$).

Irregularly shaped MP particles (fragments or films) were predominant in all sample types (85 % of all samples combined), with fibres only ranging from 10 % of MPs in Gqeberha-August Burkard trap samples to 28 % in Hull-March Burkard samples (Fig. 3d, Table S10). Foam and

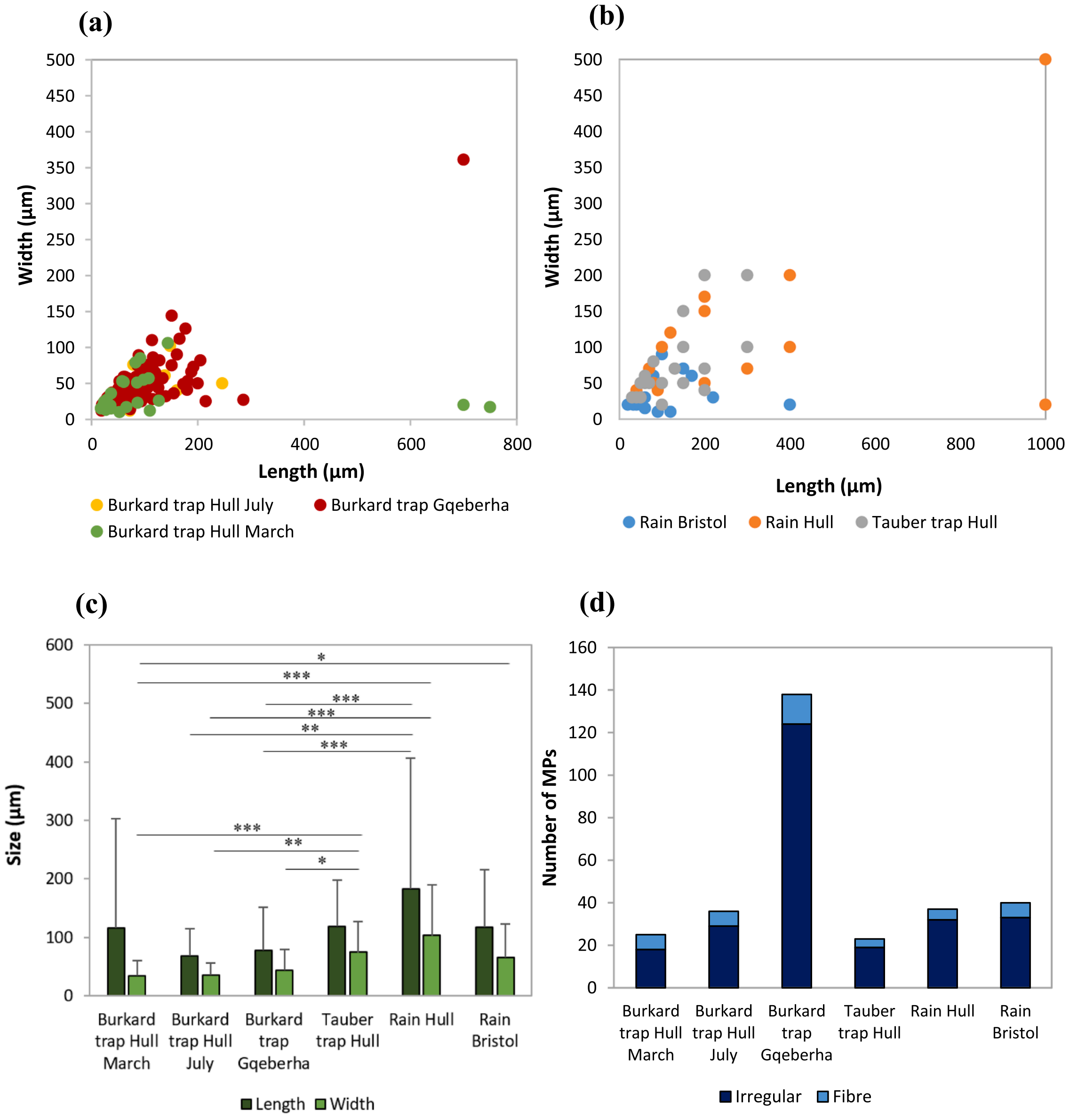


Fig. 3. MP particle sizes obtained from (a) active, (b) passive and (c) all (average ± standard deviation) air sampling techniques and (d) MP shapes; Burkard trap Hull March ($n = 25$), Hull July ($n = 36$), Gqeberha ($n = 138$), Tauber trap Hull ($n = 23$), rain Hull ($n = 37$), and rain Bristol ($n = 40$). Data is from 1 week of sampling and represents a 1/8 subsample for Burkard traps and a ¼ subsample for Tauber trap and rain sampler data. Significance * * $p < 0.01$.

spheres/microbeads were not observed in any sample type or blank. MP colour, though recorded at the time of μFTIR analysis, is not presented as the oxidative digest step using hydrogen peroxide bleaches the colour from some particles. MPs in blanks were also predominantly irregular in shape in all cases (86 % of all samples combined) with the exception of the Burkard trap blank from Hull-March in which only 1 MP was detected and this was a fibre (Table S10).

3.3. MP additives

Several particles were identified as MP polymer additives including 10 types which were detected from Burkard trap samples (>LOD) but were below the LOQ (Table S11). The most abundant additives in individual samples after LOD/LOQ calculations were the flame-retardant chemical barium metaborate and the UV light absorbing chemical additive ethanediamide N-(2-ethoxyphenyl)-N'-(2-ethylphenyl) which were both detected in 9.5 % (2/21) of samples. The additive present in the greatest number of Burkard trap samples was the antistatic agent Tegin 90 present in 23.8 % (5/21) of samples, also detected in 100 % (2/2) of Tauber trap samples (Table S11). In rainwater samples, 8 MP additives were detected, each in 20 % (1/5) of samples, the most numerous being pentaerythritol tetraricinoleate, used as a release agent coating, and ethanediamide N-(2-ethoxyphenyl)-N'-(2-ethylphenyl) (Table S11).

3.4. MP recovery rates

Using MP spikes of high and low-dose PP fibres (low density) and PET fragments (high density) to ascertain recovery rates of the sample processing methods, revealed average recovery rates of 68 % for Burkard trap tape, 70 % for Burkard trap tape mounted on slides with glycerine jelly (simulating the processing of the Gqeberha samples), and 66 % for water extractions (simulating Tauber trap and rainwater sample processing) (Table S12). For all sample types, the lowest recovery rates were obtained for high dose PP, whereas the highest recovery rates were observed for PVC spikes (Table S12). Burkard tape recovery rates ranged from 61 % (high dose PP) to 74 % (low dose PVC). Mounted tape recovery rates ranged from 57 % (high dose PP) to 85 % (high dose PVC). Water recovery rates ranged from 52 % (high dose PP) to 84 % (low dose PVC).

4. Discussion

In this study, outdoor AMPs were captured using Burkard trap active air sampling equipment integral to global pollen monitoring networks. For the first time, AMPs sampled in this way were collected using an easily adapted drum tape media which, in turn, then allowed the downstream chemical characterisation and quantification using any spectroscopy method. The approach used herein was validated using μFTIR spectroscopy for the chemical characterisation element, alongside passively sampled air via particle deposition (Tauber traps) and precipitation collection (rain samplers) for active versus passive sampling comparisons. Using this novel approach, a range of different AMP polymer types, shapes, sizes and levels were detected.

4.1. AMP concentrations and sampler type

Average AMP concentrations detected using the rooftop Burkard traps, of 2.0 ± 0.9 to 11.0 ± 5.7 MP m^{-3} depending on location, were within the lower range of > 1 to < 1000 MP m^{-3} published using various worldwide active and passive air sampling equipment (Table 1, Table S2, [18]. Experiments investigating AMPs sampled from urban rooftops worldwide, report a range of concentrations detected from an average of 0.21 ± 0.06 MP m^{-3} from PM_{10} samplers in Mexico City to 5.2 MP m^{-3} from $PM_{2.5}$ samplers in Bushehr, Iran (Table S2). Concentrations of AMPs were also consistent with those reported (of 1.42 ± 1.42 MP m^{-3}) when sampling at higher altitudes [38]. Significantly higher AMP concentrations were reported in locations around China ranging from 101 ± 47 MP m^{-3} to 282 ± 127 MP m^{-3}, in a number of localities at head height [7,39] with higher concentrations elsewhere (Table S2). Where a Burkard trap has been used previously, AMPs were reported as 23 MP m^{-3} present at head height in rural locations, higher than concentrations detected herein (Table 1), possibly due to visual microscopic identification alone leading to an overestimation, and the lower sampling height [30].

With respect to Burkard trap performance as a means to monitor and compare AMP concentrations at different locations, significantly higher AMP levels were found in Gqeberha-August (S.A.) compared to Hull-March (U.K.) ($p < 0.05$). AMP levels have previously been reported to vary between location, with higher levels detected in urban versus rural locations (Gonzalez-Pleiter, et al., 2021; [39]. Gqeberha (S.A.) has approx. three times the population of Hull (U.K.) which may be a contributing factor though no significant difference was detected between Gqeberha-August and Hull-July. No difference between the Hull time-points were detected, though a greater sampling frequency may be required to detect any annual variation. Comparisons between studies are challenging, with a number of contributing factors known to affect differences in AMP concentrations and characteristics reported, including meteorological conditions [40], location [20], anthropogenic activities [7], season [41], sampling equipment type [21], air sampling volume [42], sample processing methods [23] and data analysis approaches [22].

Using common passive sampling approaches in parallel does not allow a direct comparison with the Burkard trap-derived AMP values (due to the different units involved) but their performances in terms of relevant monitoring data collected can be qualitatively compared. The unadjusted average MP concentrations in Tauber traps were 3347 ± 617 m^{-2} day^{-1} compared to 935 ± 464 m^{-2} day^{-1} in rain samplers at the same location and sampling time suggesting possible differences in equipment. A larger sample size would be needed to test for any statistically significant differences. This difference in AMP concentrations detected between the two passive approaches may relate to design of the Palmex rain samplers preventing evaporation. In which case MPs were extracted from rainwater whereas all precipitation had evaporated from the Tauber trap by the end of the study period. Other studies investigating MP passive deposition report concentrations ranging from 10 to ~3000 MP m^{-2} day^{-1} and the blank-adjusted values from passive sampling obtained herein fall within this range (Table S1).

4.1.1. AMP particle characterisation: polymer type

Using the Burkard trap approach, a wide diversity of AMP polymer types were captured with PET and polyolefins (PO), including LDPE and a PE/PP copolymer, identified as the predominant polymers detected above the LOQ threshold. Such polymer types have wide-ranging applications including in textiles, packaging, moulding, coatings and adhesives (Table S14). This is in keeping with previous findings that the most common polymer types in both air and deposition are PET, PE and PP [18]. Of the other polymer types detected above the LOQ threshold, nylons and PTFE have frequently been detected in outdoor air samples (Table S1, S2; [23]. Of the others, airborne EPDM has been found in indoor industrial settings [10], and EVA and PS have been detected outdoors [38,43]; Table S1, S2). Finally, alkyd resins were detected as airborne contaminants here and from other rooftop air studies [42,44]. Importantly, the Burkard trap approach (with downstream analysis of the particles using μFTIR chemical characterisation) has provided the widest range of polymer detection ($n = 36$ polymers) of all samplers/approaches used and published to date.

In addition to variation in the diversity of polymer type detected, the most abundant polymer types that met the LOQ threshold varied between sampling approach. Using the Tauber and Palmex samplers in parallel, at the same location in Hull (U.K.) during July, highlighted PMA/PS as a new and predominant polymer type (Tables 1, 2). Comparisons have previously been made between pollen capture by Burkard and Tauber traps, revealing a strong correlation between Tauber pollen influx and the sum of average daily Burkard data over an annual timeframe [45]. However, differences in pollen species prevalence over shorter timescales included instances of presence/absence of a taxa in each trap type compared to the other and some monthly differences in dominant taxa type between trap [45]. Similar differences were observed for AMPs in this study, for example, LDPE was the most abundant polymer type in the Hull-July Burkard trap but was absent in Tauber traps. Smaller pollen taxa had lower influx ratios in Tauber traps compared to Burkard traps [45]. Though the average widths of Tauber trap AMPs were significantly greater than those of all of the Burkard traps, average lengths did not significantly differ. Differences in pollen taxa detection rates are influenced not only by particle size and settling velocity differences, but by other factors including meteorological conditions, natural atmospheric variability and variation resulting from subsample analysis [45]. These factors are likely to also influence AMP capture and detection.

4.1.2. AMP particle characterisation: size

Following on from pollen type and size capture efficiencies differing according to the sampling approach used, there were also size-related differences within the AMPs detected. A majority of AMPs detected from Burkard traps (77 %) were < 100 μm in size, consistent with other actively sampled AMP studies [46,4,5,7]. However, size comparisons between different studies are challenging in part due to the different particle size cut off limits applicable to different equipment [21]. For example, samplers with PM_{10} or $PM_{2.5}$ aerodynamic cut offs are limited to detect particle sizes < 10 μm and < 2.5 μm respectively which also impacts upon the reported average AMP concentration [41]. The size limits detectable from Burkard traps in this experiment range from > 10 μm for all sample types based on the lowest limit analysed by μFTIR, to 2 mm as the width of the trap inlet. This is a broader size range than is captured by most other active air sampling equipment which generally have upper size cut offs of < 60 μm [21]. Though no MPs greater than the inlet size in any dimension were detected here, previous studies have reported this phenomenon in Burkard traps and $PM_{10}/PM_{2.5}$ air samplers, likely as a result of particle orientation and/or polymer flexibility (Abbasi et al., 2023; [41]; [30]. For passively collected deposition samples, the internal tubing diameter of the Palmex rain sampler is 4 mm whereas copper rain gauges and Tauber traps are able to collect the largest of microplastics (5 mm). The average size of AMPs from passive samplers was 142.0 ± 155.7 μm and 59 % were < 100 μm in length. This is similar to the average of 164 ± 167 μm for non-fibrous MPs recorded in London at rooftop level [47] and averages of between 29.1 ± 14.9 to 109.4 ± 19.2 μm were found at different sampling sites around Hamburg, Germany using precipitation collectors [43]. The MP particles sampled using the active Burkard traps were significantly smaller in both length and width relative to MP particles identified using the passive sampler devices, providing additional insight into how sampler type impacts upon the representativeness of the AMPs present.

4.1.3. AMP particle characterisation: shape

Using the Burkard trap approach, irregularly shaped MPs (fragment and films) were the most abundant shape detected for all sampling methods (85 %), similar to some studies [48,40,43,7], but contrasting with others [49,41,47]. Some studies report that larger AMPs were predominantly fibres whereas smaller AMPs were predominantly fragments [19,39]. These differences between studies may be at least partially attributed to differences in sampling equipment, sampling heights and analysis methods [21,50,23]. AMPs previously collected from Burkard traps at ground level were predominantly fibres in both indoor and outdoor rural and outdoor urban environments [30]. This raised the question as to whether the equipment had a higher collection efficiency for fibres over other MP shapes, but the greater abundance of non-fibrous MPs from two different Burkard traps herein suggests that this is not the case. It should, however be noted that the Boakes et al. [30] study used Glisseal® vacuum grease as an adhesive whilst this study used glycerine jelly. Numerous other types of capture media are also used for aeroallergen sampling and differences in particle capture and retention efficiencies are seen [51,52]. Further investigation into the efficiencies of different consistencies of glycerine jelly for AMP collection is recommended.

4.2. AMP particle concentrations and characterisation: human health implications

The concentrations of AMPs in the outdoor air vary considerably as a result of many factors as discussed above. As a strength, the Burkard trap approach is an active sampling approach that better mimics a human breathing exposure compared with passive/deposition approaches such as the Tauber and rain sampler approaches. These traps are also already commonly deployed as part of the global pollen monitoring networks with associated infrastructure, dissemination and communication facilities. The professionals concerned with public health, respiratory illnesses, and measuring airborne contaminants (in this case pollen) are also end users that would benefit from the AMP datasets. In comparison to the only other active sampler approach; global air quality regulatory-driven particulate matter monitoring infrastructures and network, these are less globally connected, they provide no shape data, and they involve a size cut off that loses the 10 μm to 2 mm AMP dataset.

Of the other polymer types detected above the LOQ threshold, namely nylon, PTFE, and EPDM, these were among the most common MPs detected in studies on the upper respiratory tract, lung tissue, or nasal cavities of humans [13,14,53] with sizes of MP particles as large as 2.48 mm [13,53]. Nylon fibres (of size 12 μm x 30 μm) have also been evidenced to inhibit airway epithelial cell growth in lung organoid studies [54]. Furthermore, of the polymer types detected >LOQ herein, nylon, PTFE, PET, PE, PP, EVA, PS, alkyd resins and EPDM have also been found in a broad range of other human tissue samples [55,56,53]. The routes of exposure from MPs to human tissues are not fully elucidated to date, yet based on AMP ubiquity and their presence in human lung (and other tissues) suggests that inhalation along with dietary exposure are both key routes.

Any AMP monitoring approach must capture size and shape characteristics since these are known to determine the biological impact in terms of toxicity [57]. A meta-regression analysis of published studies investigating biological impacts from MPs, using controlled human cell-based exposures and/or animal model studies, evidenced that both size and shape of MPs influence detrimental outcomes measured as inflammation and oxidative responses as well as changes in cell barrier integrity [57]. Also important to note is that no bead-shaped AMPs were detected using any sampler technique at any location. Current air quality monitoring programmes generally measure pollution levels of nitrogen oxides, sulphur dioxide, ozone and airborne particulates ≤ 10 μm and ≤ 2.5 μm in size [58]. Despite the increasing concern of MPs as emerging environmental contaminants and the possible implications for public health, AMPs are not currently monitored.

In addition to AMPs, a number of particles were detected above the LOD but below the LOQ thresholds containing plastic-associated chemicals which are used as plastic additives, monomers, intermediates or catalysts (Table S11). The migration of such chemicals, many of which are toxic in nature, has environmental as well as human health implications, especially for the phthalate- and bisphenol-type chemicals [59-61]. The detection of these chemicals as airborne environmental contaminants, which can also be found within and detrimentally impacting human tissues [62], highlights the need for their inclusion within any standardised AMP monitoring approach.

4.3. Burkard trap as an AMP monitoring device

A standard operating procedure, based on this investigation, for the use of the Burkard trap as a routine monitoring approach has been prepared. Future sampling using this approach could also be conducted using the tape cut into 4 h sections, rather than daily segments, providing an analysis which could be interpreted along with weather/PM measurements available at the same location investigated. A number of limitations and evidence gaps should be highlighted. First, there is no current consensus on the use of procedural or laboratory blank data correction for MP data; LOD/LOQ approaches have been found to be the most reliable for this purpose [22]. These approaches use data from blanks to account for unavoidable procedural contamination which is identified and quantified by polymer type [63]. Second, the AMP levels reported in this study are likely to be an underrepresentation of the true airborne concentrations. Burkard traps are calibrated with a compatible flowmeter to have an air sampling rate of 14.4 m^3 day^{-1}, but flow efficiency is 70 ± 20 % (Burkard Manufacturing Co. Ltd, UK) and dependant on the individual trap and flowmeter combination used for calibration [64]. Conversion factor calculations are based on this flowrate and assume 100 % trap efficiency [65]. Particle capture and/or retention efficiency may also vary with adhesive, particle type/size and weather conditions including wind speed [51,66,52]. Establishing the relative efficiencies of newly developed MP airborne monitoring approaches is also required, ideally with integrated chemical characterisation of plastic polymer types present. MP spiking experiments demonstrate varied recovery rates of various MP polymers from different sample types (58 – 100 %) [67]. The spiking experiment in this study, which relates to laboratory aspects of the methodologies only, revealed average MP recovery efficiencies of 66–70 % (Table S12). Recovery rates were the lowest on average from the water-based extractions (simulating the methods for Tauber trap and rainwater sample processing) which is in keeping with previous findings [67]. The 70 % recovery rate of Burkard trap tape mounted on glycerine jelly slides demonstrates the feasibility of collecting samples in one location and shipping them to another facility with micro-spectroscopy facilities for digestion, filtration and analysis. Further recovery experiments are required to determine field sampling efficiencies.

In conclusion, outdoor AMPs were sampled using low-cost, robust equipment already in use in well-established airborne particulate monitoring networks. A protocol was developed to allow researchers in different locations to collect Burkard trap air samples and rainwater samples and ship them to another facility with specialist μFTIR equipment for chemical characterisation and quantification, a first for AMPs from Burkard traps. Applying LOD/LOQ thresholds leads to more stringent reporting of MP polymer concentrations. A standard operating procedure for AMP investigations is necessary to standardise the reporting of AMP concentrations and characteristics which can be influenced by differences in sampling equipment, sampled air volume, processing methods and data analysis approaches. This is central in being able to conduct any risk assessment of their potential biological impacts for humans, or other organisms, and their physical/chemical impacts on atmospheric processes.

Environmental implication

Airborne MPs (AMPs) were chemically characterised and quantified using volumetric pollen sampling equipment, representing a viable approach for future airborne MP monitoring. MPs were detected in 95 % of daily samples. Six polymer types were above the limit of quantification, with poly(methacrolein:styrene) the most abundant. Irregularly shaped MPs < 100 μm in length were predominant. Having a viable approach for future airborne MP monitoring is a necessary step in being able to conduct any risk assessment of AMP potential biological impacts for humans, or other organisms, and their physical/chemical impacts on atmospheric processes.

CRediT authorship contribution statement

Charlotte A. Atherall: Formal analysis. **Mark F. Hansen:** Funding acquisition, Conceptualization. **Catriona R. Liddle:** Formal analysis. **Erin Hilmer:** Writing – review & editing, Formal analysis. **Ben Williams:** Writing – review & editing, Funding acquisition, Conceptualization. **Dave White:** Validation. **Diana C. Suárez:** Methodology, Investigation. **Angela G. Garcia:** Methodology, Investigation. **Lynne J. Quick:** Writing – review & editing, Methodology, Conceptualization. **Rob Kinnersley:** Validation. **Emma Chapman:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **Sheen Mclean S. Cabaneros:** Conceptualization. **Jeanette Rotchell:** Writing – review & editing, Writing – original draft, Funding acquisition, Conceptualization. **Paul Walker:** Writing – review & editing, Validation. **M. Jane Bunting:** Writing – review & editing, Conceptualization.

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.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jhazmat.2024.136129.

Data availability

Data will be made available on request.

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