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The contamination of *in situ* archaeological remains: A pilot analysis of microplastics in sediment samples using μ FTIR

Jeanette M. Rotchell^{a,b,*}, Freija Mendrik^{a,c,1,2}, Emma Chapman^a, Paul Flintoft^d, Ian Panter^d, Giulia Gallio^d, Christine McDonnell^a, Catriona R. Liddle^a, David Jennings^d, John Schofield^e

^a School of Natural Sciences, University of Hull, Kingston-upon-Hull HU6 7RX, United Kingdom

^b College of Health and Science, University of Lincoln, Brayford Pool, Lincoln LN6 7TS, United Kingdom

^c Energy and Environment Institute, University of Hull, Kingston-upon-Hull HU6 7RX, United Kingdom

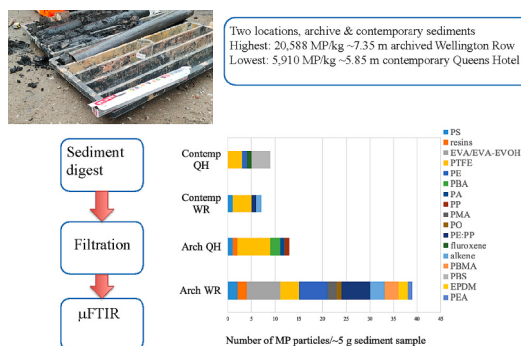
^d York Archaeology, (York), 47 Aldwark, York, North Yorkshire YO1 7BX, United Kingdom

^e Department of Archaeology, University of York, King's Manor, Exhibition Square, York YO17EP, United Kingdom

HIGHLIGHTS

- Microplastic (MP) particles are present in archaeological sediment samples.
- MP particles, of 16 polymers, were found in archived and contemporary samples.
- MP levels varied from 0 to 20,588 MP/kg dependant on location and depth.
- MPs may impact scientific value and preservation of archaeological deposits.

GRAPHICAL ABSTRACT



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ABSTRACT

Background: Microplastics (MPs) are found in all environments: aquatic, airborne, and terrestrial. While their presence is not disputed, their potential impacts are not yet known.

Objective: To undertake a pilot analysis of MP contamination in archaeological sediment samples, taken in the late 1980s from two archaeological excavation sites in the historic city of York (UK) as well as contemporary sources close to the same sites, with respect to the presence (if any), levels, and characteristics of any particles identified.

Methods: This study analysed pre-digested sediment samples as follows: $n = 3$ from Queens Hotel (QH) site and $n = 3$ Wellington Row (WR) contemporary core-source, and $n = 3$ QH and $n = 3$ WR archival-source samples, alongside procedural controls ($n = 8$), using μ FTIR spectroscopy (size limitation of 5 μ m) to detect and characterise any MPs present.

* Corresponding author at: School of Natural Sciences, University of Hull, Kingston-upon-Hull HU6 7RX, United Kingdom.

E-mail address: jrotchell@lincoln.ac.uk (J.M. Rotchell).

¹ Current address: International Marine Litter Research Unit, Drake Circus, Plymouth, PL4 8AA, United Kingdom

² Current address: School of Biological and Marine Sciences, Drake Circus, Plymouth, PL4 8AA, United Kingdom.

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Results: In total, 66 MP particles consisting of 16 MP polymer types were identified across both site and contemporary/archived samples. The highest levels of MP particles, 20,588 MP/kg was identified at the lowest sample depth (~7.35 m) at archived WR, 5910 MP/kg in the mid depth layer (~5.85 m) at the contemporary QH site. Of the MPs detected in sediment samples overall, polytetrafluoroethylene (PTFE), polybutylene sulfone (PSU), and polypropylene: polyethylene (PE:PP) copolymer polymer types were most abundant; mainly fragmented and irregular shape.

Conclusions: This is believed to be the first evidence of MP contamination in archaeological sediment (or soil) samples with polymers and size ranges measured and while accounting for procedural blanks. These results support the phenomenon of transport of MPs within archaeological stratigraphy, and the characterisation of types, shapes and size ranges identified therein. Through contamination, MPs may compromise the scientific value of archaeological deposits, and environmental proxies suspended within significant sediment, and as such represent a new consideration in the dynamism of, as well as arguments for preserving, archaeological deposits *in situ*.

1. Introduction

Microplastics (MPs) are plastic particles with a size range typically agreed to be between 1 µm and 5 mm (Hartmann et al., 2019). One of the more recent locations in which MPs have been detected is soil to the extent that MPs are now regarded as ubiquitous in soil matrices globally (Yang et al., 2021) (summarised in Supplemental Table S1). A recent study identified MPs in soils from Europe, North America, South America, China, and Australia, with levels as high as 62.5 MP items/kg within deep soil in Chinese farmland (Guo et al., 2020). Furthermore, 90 % of Swiss floodplain soils contained MPs with a mean of 5 mg/kg (Guo et al., 2020). Potential sources for MPs in soil include landfill (Guo et al., 2020), tyre debris in road runoff, compost, flooding of waste waters, irrigation, and/or atmospheric deposition (Li et al., 2020) (Table S1). Such contamination by MPs in the environment has been established as an emerging threat to biodiversity and ecosystem function (Boots et al., 2019).

MPs in soil may have various impacts on both the biotic and abiotic environment although research into these effects is limited (Büks et al., 2020) largely due to the restrictions and inconsistencies of technology, meaning complex matrices are difficult to analyse (Yang et al., 2021). A recent study into the effect of MPs on soil properties in Germany and Switzerland indicated that MPs, particularly those of a large and intrusive shape, altered soil microbial activities and soil chemistry, leading to the degradation of organic matter at an abnormal rate (de Souza Machado et al., 2019). Furthermore, a similar study into the effects of MPs in soil concluded there is a significant negative difference in bulk density, fertility, and water-stable aggregates in soils with high MP levels (Yang et al., 2021). In terms of biotic factors, soil-living organisms suffer from the high levels of soil MPs as they are easily ingested, ultimately affecting their survival rate. A study into the effects of MP exposure on earthworms (*Lumbricus terrestris*), for example, concluded that exposure to MPs at a concentration (in the soil matrix) ranging from 0.2 to 1.2 % over 60 days reduced their growth rate (Boots et al., 2019).

A recent review reports that MPs in accumulated sediment can be conserved to the same extent as fossils and therefore that, as ‘technofossils’, MPs can be considered indicators of recent time periods (Campanale et al., 2020). Even biodegradable polymers (such as polylactic acid) have proven to have degradation that is incomplete and/or lengthy (Boots et al., 2019). Of an approximate 170 publications from 1950 to 2020, the distribution of studies into non-biodegradable MPs in soil were globally uneven: 60 % of studies were conducted in Asia and 23.3 % were from Europe, of which 68.5 % of publications regarded agricultural soils (Zhang et al., 2020a). Notably, Africa and India had no studies of MPs in soil (Büks et al., 2020) while South America and Oceania had relatively few (Yang et al., 2021) (Table S1).

To date, and to our knowledge, there have been no previous investigations into MP contaminations of archaeological sediments. These sediments are collected from most archaeological excavations conducted across the world, both rural and urban, to assist with laboratory-

based investigations into past environmental conditions at the site, its geomorphology, and the cultural and natural post-depositional processes which might have affected the survival of archaeological remains. These ‘soil samples’ (as they are usually described) also typically form part of the site archives, being stored indefinitely under controlled conditions, for future research and as reference material. Such soil samples have been routinely taken on archaeological excavations since the 1970s, and often earlier. However, those which remain in site archives are usually from this period onwards.

Through contamination, MPs may compromise the scientific value of archaeological deposits and environmental proxies suspended within significant sediment. Porous biological remains such as plant, animal, and insect remains are likely at greater risk and, in these cases, contamination may prevent or impede the chemical analysis of such items. If revealed to compromise archaeological remains, MP contamination could influence future excavation and sampling strategies, as well as impacting arguments for the long-term preservation of archaeological deposits. For buried archaeological remains in England, the current planning system places an emphasis on the *in situ* preservation of any such remains that are deemed to have national importance determined either locally, by local authority archaeologists or nationally by the Department for Digital, Culture, Media and Sport, DCMS (advised by Historic England, the UK Government’s statutory adviser on heritage matters in England). Internationally, this position varies by country and state. While the argument for prioritising the preservation of buried structural remains is widely accepted (as they form visible features which the public can enjoy, and which can clearly enhance understandings of the past), this paper has direct implications for the preservation of buried archaeological deposits occurring where associated structural remains are absent. As Historic England (2016) states: under some circumstances, ‘it may be clear that the harm to archaeological remains is too great to ensure their continued conservation.’ This investigation explores if this might be the case with MP contamination.

This study aims to question whether and to what extent MPs exist within archaeological soil samples (although correctly these are *sediment* samples, as the deposits are the direct result of human activity). These samples are all recent with most dating to the period since the early 1970s when the potential of environmental sampling was first realised. The number of samples increased rapidly with the growth in developer-funded commercial archaeology from the mid 1980s onwards. This post-war period coincides with the exponential rise in the use of plastics within society. Samples from the late 1980s and from the present day are included in this study, based in the historic city of York, with a long history of archaeological excavations, revealing exceptionally well-preserved archaeological remains from the Roman period onwards (Kenward et al., 1986; Hall, 1988). The sediment samples investigated came from two earlier excavations in the city, with additional cores taken from nearby the original excavation sites in May 2023, for direct comparison.

2. Methods

2.1. Sediment sample acquisition

Both selected sites, at Wellington Row and the Queen’s Hotel in York, represent two thousand years of near-continuous urban occupation (Ottaway, 1993) (Table 1). The formation of archaeological sites occurs through a blend of cultural activities and natural processes, such as the repeated construction/demolition of structures and refuse dumping, alongside flood events and erosion (Schiffer, 1995; Bohn, 2022). These activities and processes are present within the archaeological record as archaeological sediments or ‘contexts’ (Walkington, 2010; Carver, 2013). Two sites with archived samples and the evidence for deeply stratified archaeological sediments were considered necessary for the borehole survey to reveal geographic and diachronic changes in MP contaminations. The Queen’s Hotel site (NGR SE 60111 51,619), excavated in 1989 and 1990, was revealed to have 5.33 m of archaeological sediment, dating from the late first century CE to the 20th century (Ottaway, 1993) (Fig. 1.). The exact location of the Queen’s Street excavation could not be targeted in the borehole survey, as the site is occupied by a modern development. As the site itself could not be subjected to drilling, the closest available undeveloped area was identified as 40 m to the north of the excavation. Located approximately 65 m from the River Ouse, the borehole revealed waterlogged sediments 1.45 m below the ground surface.

The Wellington Row site (NGR SE 6001 5181) was excavated in 1988 and 1989 (Ottaway, 1993) (Fig. 1). Archaeological sediments at Wellington Row were measured as 2.91 m in depth, although the base of the strata was never satisfactorily identified during the excavation. The earliest deposits were revealed as being late first or early second century and extended to the 19th and 20th centuries. Similar to the Queen’s Hotel site, the Wellington Row borehole could not be sited on the original excavation as the site had subsequently been developed. Consequently, the borehole was situated approximately 12 m from the original excavation. The location of the Wellington Row borehole is located 40 m from the River Ouse and waterlogging was observed 2.2 m

Table 1
Sampled sediment description from Wellington Row (WR) and Queen’s Hotel (QH).

Site	Context Number	Ordnance Datum	Description
Wellington Row	7060	7.35 m–7.36 m	Light brown, friable, slightly clayey silt. Organic content c.20 %. Occasional inclusions of small sub-rounded stones and large pieces of animal bone.
Wellington Row	72,146	5.98 m–6.03 m	Mid-brown, friable, slightly clayey silt. Organic content c.15 %. Occasional inclusions of small sub-rounded stones and very occasional inclusions of large sub-rounded stones.
Wellington Row	4230	4.87 m–4.96 m	Dark brown, friable, friable clayey silt. Organic content c.20 %. Frequent inclusions of wood.
Queen’s Hotel	6006	8.47 m–8.49 m	Dark brown, friable, slightly clayey silt. Organic content c.40 %. Moderate inclusions of rounded and subangular stones and occasional inclusions of animal bone.
Queen’s Hotel	5115	5.84 m–5.87 m	Very dark brown/black, sticky silty clay. Organic content c.10 %. Moderate inclusions of large sand particles and angular stones.
Queen’s Hotel	5205	5.84 m–5.87 m	Light brown, sticky, slightly silty clay. Organic content c.5 %. Occasional inclusions of sub-rounded stones.

from the ground surface. Recent and ongoing groundwater monitoring schemes suggest that the waterlogged deposits in this part of the city are recharged primarily by the River Ouse. Proximity to the river could be transporting MPs, rather than being illuviated vertically through the sediment, especially as much of the urban streetscape is concrete and tarmac.

Boreholes were located using an RTK-rectified Geomax GPS. Boreholing was undertaken using a tracked window-sampling rig operated by geotechnical engineers to the maximum achievable depth, which was 7.00 m BGL (below present ground level) for the North Street borehole (460,072.8, 451,690.7 - OD: 10.64), and 4.00 m BGL for the Wellington Row borehole (460,002.0, 451,800.4. - OD: 10.08). The uppermost metre of each borehole was excavated by hand to mitigate the possibility of sub-surface services. The lithology of the geoarchaeologically-monitored boreholes was recorded using the Troels-Smith system of sediment classification (1955). This system breaks down a sediment sample into five main components and allows the inclusion of extra components that are also present but not dominant. Key physical properties of the sediment layers are darkness (Da), stratification (St), elasticity (El), dryness of the sediment (Sicc) and the sharpness of the upper sediment boundary (UB). A summary of the sedimentary and physical properties classified by Troels-Smith (1955) and a stratigraphic breakdown of the deposits were recorded on proforma log sheets. The logs were supplemented by digital photography.

Sediment samples (~5 g semi dry weight) were collected from these two locations *via* contemporary coring and archival sources (Fig. 1; Table 1) between the dates May – June 2023, and in1988–1990, respectively. Sediment samples were collected using a metal spatula and aluminium foil prior to transport to the laboratory for the contemporary cores. For the archive-source samples, approximately 5 g of sample was transferred from the archival container directly into pre-cleaned and pre-weighed conical flasks using a metal spatula. These were derived from within the middle of a stored sample/core to avoid external surface contamination. Digestion of *n* = 23 samples (2 sites of: 6 contemporary, 6 archived, 8 procedural blanks, 3 air environment blanks) in total were conducted in two batches with four procedural blanks per batch. From the point that a sample is taken, there is unavoidable scope for the sample to be open to the outdoor/indoor air environment. To account for this, the procedural controls consisted of *n* = 8 blanks from the point of sampling. A further *n* = 3 environment blanks consisted of a glass beaker left open in the archive room (*n* = 1 for one week duration), plus a beaker open beside the coring machinery during each of coring procedures at WR and QH. These blanks characterise any potential surrounding airborne contaminant types and levels either in the archive facility or at the sampling site where coring took place.

2.2. Sediment sample digestion and filtration

Sediment samples (*n* = 12) were placed in hydrogen peroxide (100 mL of 30 % H₂O₂) alongside the procedural and environmental air blanks (*n* = 11). Flasks were placed in a shaking incubator at 65 °C for approximately 7 days, at 80 rpm. The digest, adapted from previous studies investigating MPs within different environmental and tissue samples (Li et al., 2018; Jenner et al., 2022), removes organic particles yet maintains MP integrity (Munno et al., 2018). Next, a density separation step was conducted whereby 200 mL of pre-filtered hypersaline (NaCl) solution was added. After stirring, the sample was left for 48 h before the top layer was decanted to a pre-cleaned conical flask. Samples were then filtered onto aluminium oxide filters (0.02 µm Anodisc, Watford, U.K.) using a glass vacuum filtration system. Filters were stored in petri dishes before chemical composition analysis.

2.3. Chemical characterisation of particles using µFTIR analysis

Each sediment sample Anodisc filter was placed onto the µFTIR spectroscopy platform, and the length (largest side) and width (next



Fig. 1. Plan of 1980s excavations and boreholes.

largest side) recorded using the aperture height, width, and angle- size selection tool, available with the ThermoScientific Omnic Picta Nicolet iN10 microscopy software. Particles were given a shape category of fibre, film, fragment, foam, or sphere (Free et al., 2014), whereby fibrous particles were characterised with a length to width ratio > 3 (Vianello et al., 2019). μ FTIR analysis was conducted in liquid nitrogen-cooled transmission mode (Nicolet iN10, ThermoFisher, Waltham MA, U.S.A.). The cooled mercury cadmium telluride (MCT) detector allows the analysis of particles accurately down to a size of $5 \mu\text{m}$. The iN10 microscope used has a 15×0.7 N.A. high efficiency objective and condenser. It has a colour CCD digital video camera with an independent transmission illumination mounted, which was used for capturing images of the particles. The model has a standardised $123\times$ magnification with the aperture settings used.

A quarter of each filter, containing the total digested sediment sample, was analysed. A background reference spectrum was first recorded. μ FTIR parameters were: spectral range of $4000\text{--}1250 \text{ cm}^{-1}$ (below this range is not possible with an Anodisc-type filter); high spectral resolution 8 cm^{-1} and a scan number of 64. Features such as smoothing, baseline correction and data transformation were not used. The resulting sample spectra were compared to a combination of polymer libraries (Omnic Picta, Omnic Polymer Libraries) and full spectral ranges were used with a match threshold of $\geq 70\%$. Particles not classified as a plastic, but which did meet the 70% threshold, were recorded but not included in the results shown. The total number of particles (MPs and others) identified across one quarter of all filters analysed was 373, for which 66 (17.7 %) were MPs.

2.4. Quality assurance and control measures

Strict control measures were used to quantify and characterise the nature of any unavoidable background contamination inherent in MP analysis. Due to the ubiquitous presence of MPs in the air, contamination of sediment samples is likely to some extent during collection. To mitigate this risk, each sample was placed immediately in a pre-cleaned conical flask or aluminium foil. In parallel, procedural blanks ($n = 8$) were initiated at the time of sediment collection from the contemporary and archived sampling points. The procedural blanks each mimicked the entire sample processing steps but lacked the sediment sample. Three further procedural blanks contained air from the archive room and from the ambient air beside the coring machinery.

All reagents were pre-filtered and prepared in bulk. MPs identified within procedural blanks represent contamination from indoor atmosphere at the point of sediment collection, contamination from laboratory reagents, equipment, or fallout from the air during the transfer of samples. There are no standardised protocols currently adopted within the MPs research field to account for background contamination, so multiple contamination adjustments were employed in this study for comparison (Table 2). As such, two approaches were used: subtraction, commonly used in the MP research field, and a limit of detection (LOD) and limit of quantification (LOQ) approach (Horton et al., 2021). Using the raw data, subtraction, and LOD/LOQ, adjusted results provide a comparison for each technique (Table 2).

The H_2O_2 used was triple filtered using a glass vacuum filtration set up and 47 mm glass fibre grade 6 filters, pore size $< 1 \mu\text{m}$ (GE Healthcare

Table 2

The number of MPs identified within the sediment samples by μ FTIR spectroscopy. Polymer types are included, and three different contamination adjustments are used to display results in units of MP/kg of sediment. † unadjusted, †† blank subtracted, ††† LOD/LOQ adjusted values. Abbreviations; – Did not meet LOQ criteria. AC, acrylic; EVA, ethylene-vinyl acetate; EVA/EVOH, ethylene-vinyl acetate/ethylene vinyl alcohol; PBDA, poly(11-bromoundecyl acrylate); PE, polyethylene; PMA, poly(*N*-methyl acrylamide); PO, polyolefin; PP, polypropylene; PE:PP, polyethylene:polypropylene copolymer; PE:EAc, polyethylene/ethyl acrylate copolymer; PS, polystyrene; PSU, poly(2,3-butylene sulfone); PVE, poly(2,2,2-trifluoroethyl vinyl ether); hydrocarbon resins.

Sediment sample	MP/kg †	MP/kg ††	MP/kg †††
QH-modern	0		
upper			
mid	5910		PSU 3200
lower	0		
WR-modern	2190		–
upper			
mid	760		–
lower	2190		–
QH-archived	3299		–
upper			
mid	3795		–
lower	3013		–
WR-archived	5589		PE:PP 800
upper			
mid	1370		–
lower	20,588		PE:PP 4800; EVA/EVOH 5600
All sample mean	4058.67	4051.15	1200
SD	5549.51	5177.99	3041.53

Life Sciences, Marlborough MA, U.S.A.). All glassware was manually cleaned, put through a dishwasher cycle using distilled water and then manually rinsed three times with triple-filtered MilliQ water. All equipment and reagents were covered with foil lids. When filtering samples, glassware and the sides of the filtration equipment were rinsed with triple-filtered MilliQ water to avoid any sample particle loss. Each sediment sample was processed individually to prevent cross contamination. A cotton laboratory coat, and fresh nitrile gloves for each sample processing step were used.

2.5. Statistical analysis

Homogeneity and significance tests were performed on unadjusted MP values using SPSS. The data were determined to be not normally distributed with a Shapiro-Wilk test and a Kruskal-Wallis test applied. There are currently no standardised methods for calculating MP

concentrations so two are presented: 1. unadjusted values, and 2. with LOD/LOQ values (Horton et al., 2021).

3. Results

3.1. MP abundance levels detected in sediment samples

In total, 66 MP particles were characterised from all the sediment samples combined, while 16 MP polymer types in total were identified. For the contemporary core-source samples, 9 MP particles of 4 polymer types were identified only within the middle section of the QH sediment samples, with an unadjusted average of 1970 ± 3412 MP/kg (range 0–5190/kg MPs) (Fig. 2; Table 2) and 7 MP particles of 4 polymer types (Fig. 3; Table 2), were identified across each of the WR sediment samples, with an unadjusted average of 1713 ± 826 MP/kg (range 760–2190 MP/kg) (Fig. 2; Table 2). For the archive-source sediment samples, 15 MP polymer types in total were identified, with 6 types within the QH samples, and an unadjusted average of 3369 ± 396 MP/kg (range 3013–3795 MP/kg) (Fig. 2; Table 2). In parallel, 13 polymer types were recorded within archived WR samples, with an unadjusted average of $9182 \pm 10,100$ MP/kg (range 1370–20,588 MP/kg)(Fig. 2; Fig. 3; Table 2). No significant differences in the levels of MPs were detected either within the QH or WR samples.

The MP levels detected in the two archived-source sampling locations did not significantly differ from each other ($p = 0.513$). The combined procedural blanks ($n = 8$) contained 7.52 ± 6.92 MPs per sample (range 0–20 MPs) with 7 MP types that largely differed in terms of the dominant polymer types detected compared with the sediment-source samples (Fig. 4E). The passively-collected outdoor air samples contained $3367 \text{ MP/m}^2/\text{day}$ and $30,305 \text{ MP/m}^2/\text{day}$ at the contemporary WR and QH sites respectively, with PP and PE > PTFE>PS polymers the most abundant (Supplemental Fig. S1. A-B). The passive air sample from the indoor archive room displayed $541 \text{ MP/m}^2/\text{day}$ with PE > nylon>resin/PP polymers present (Supplemental Fig. S1.C). The MP polymer PSU for QH contemporary source sediment, PP:PE, (Fig. 5C), EVA, EVA-EVOH (Fig. 5A) for the WR archive source, were all above the LOD and LOQ (Table 2; S1 Table).

3.2. MP particle characterisation from sediment samples

Of the MPs detected in contemporary core-derived sediment samples, PTFE (57 %) and polypropylene:polyethylene copolymer (PP:PE), polyalkene (PA) and polystyrene (PS) (each representing 13 %) polymer types were detected in WR samples (Fig. 4A), compared with

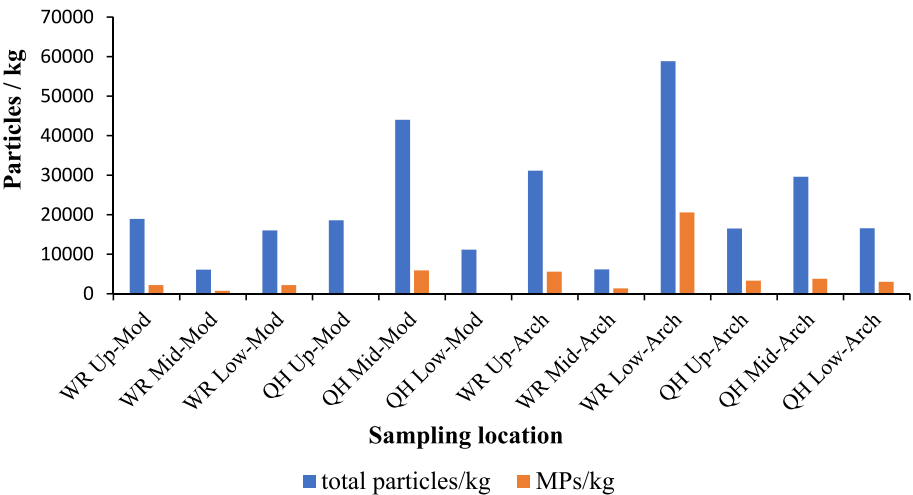


Fig. 2. Total number of particles and MPs in contemporary-source and archive-source sediment samples. Abbreviations: Mod: contemporary samples, Arch: archived samples. No significant differences.

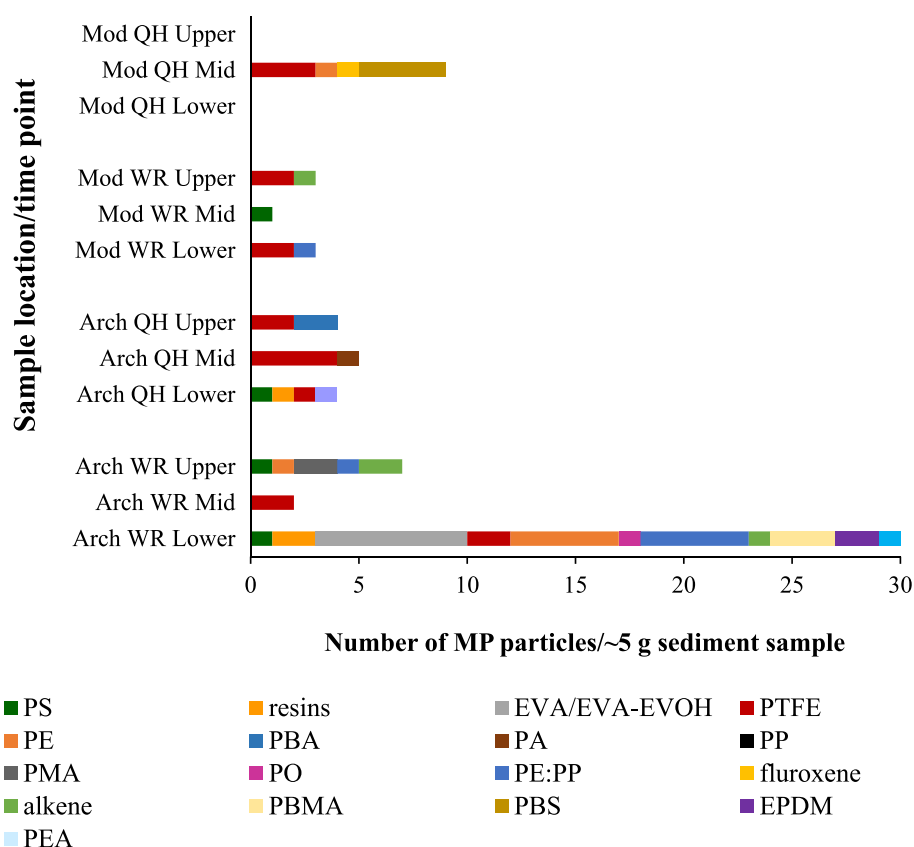


Fig. 3. MP polymer types and numbers identified in contemporary-source and archive-source sediment samples. Abbreviations: Mod: contemporary samples, Arch: archived samples. No significant differences. EDPM, polyethylene:polypropylene:diene; EVA/EVOH, ethylene-vinyl acetate/ethylene vinyl alcohol; PA, polyalkene; PBDA, poly(11-bromoundecyl acrylate); PBMA, poly (11-bromoundecyl methacrylate); PBS, poly(2,3-butylene sulfone); PMA, poly(*N*-methyl acrylamide); PO, polyolefin; PE, polyethylene; PE-E Ac, polyethylene/ethyl acrylate copolymer; PP, polypropylene; PP/PE, polypropylene polyethylene co-polymer; PTFE, polytetrafluoroethylene; PS, polystyrene; resins, hydrocarbon resin.

polybutylene sulfone (PSU)(44 %) and PTFE (33 %) polymer types which were most abundant in QH samples (Fig. 3; Fig. 4B). Of the MPs detected in archive-derived sediment samples, PP:PE (19 %), polyethylene (PE)(16 %), PTFE (11 %), and ethylene-vinyl acetate/ethylene vinyl alcohol (EVA/EVOH) (11 %) polymer types were the most abundant in WR samples (Fig. 4C; Fig. 5C), compared with polytetrafluoroethylene (PTFE) (54 %), and poly 11-bromoundecyl methacrylate (PBMA) (15 %) polymer types most abundant in QH samples (Fig. 4D).

MP particles identified within the contemporary core-source samples had the following mean particle lengths: WR of $68.29 \pm 22.47 \mu\text{m}$ (range 36–103 μm), QH of $37.78 \pm 13.28 \mu\text{m}$ (range 25–60 μm), and a mean particle width of WR: $48.71 \pm 11.61 \mu\text{m}$ (range 29–63 μm) and QH: $28.78 \pm 4.21 \mu\text{m}$ (range 25–35 μm)(Fig. 6). MP particles identified within the archive-source samples had the following mean particle lengths: WR of $113.14 \pm 134.55 \mu\text{m}$ (range 24–482 μm), QH of $58.23 \pm 27.34 \mu\text{m}$ (range 33–126 μm), and a mean particle width of WR: $74.97 \pm 93.13 \mu\text{m}$ (range 23–400 μm), QH: $43.69 \pm 24.97 \mu\text{m}$ (range 30–119 μm). The vast majority of MPs from all contemporary core-source and archive-source samples were fragment shapes, of predominantly clear/white or black/brown colour depending on the site sampled. The MP dimensions from contemporary core-source samples from QH were significantly smaller than those from WR site in both length ($P = 0.009$) and width ($P = 0.002$).

Several non-MP, yet related, as either MP building block polymer monomers or polymer additives, were also observed: the most prevalent such chemical detected was *N*-(2-ethoxyphenyl)-*N*-(2-ethylphenyl)-ethanediamide.

4. Discussion

This pilot project has revealed, we believe for the first time, that archaeological sediment samples, in this case taken from excavations in the centre of the historic city of York in the late 1980s, are contaminated. A variety of MP polymer types, of significantly different sizes, were detected in both contemporary and archive-source sediment samples, indicating that such samples are contaminated specifically by plastics. Procedural blanks taken at the time of sampling, and the samples taken from the plastic storage containers mean that the environment in which the samples were stored were not the source of this contamination and that the sediments were contaminated at the time of excavation. The levels of contamination also appear consistent between the two sites, with no statistically significant differences between the samples. Contemporary samples were taken in mid 2023 from nearby locations and comparable depths to those excavated samples. These samples demonstrate that the same contamination levels remain. There are some interesting discrepancies between the excavated and the contemporary samples which are not fully understood and raise questions that require additional research. These discrepancies include a greater variety of MP polymer types present within the sediment samples from the 1980s relative to the 2023 samples and differing MP polymer type profiles. There is also variability in the extent of plastics contamination at the three depth intervals sampled. However, these variations are not consistent, occurring both between the sites and between the archive and contemporary samples. In short, there is no obvious pattern within the variability, and this is likely due to the highly dynamic nature of the environment in which the samples were taken.

Sources of MPs in soils are mainly through human activity such as

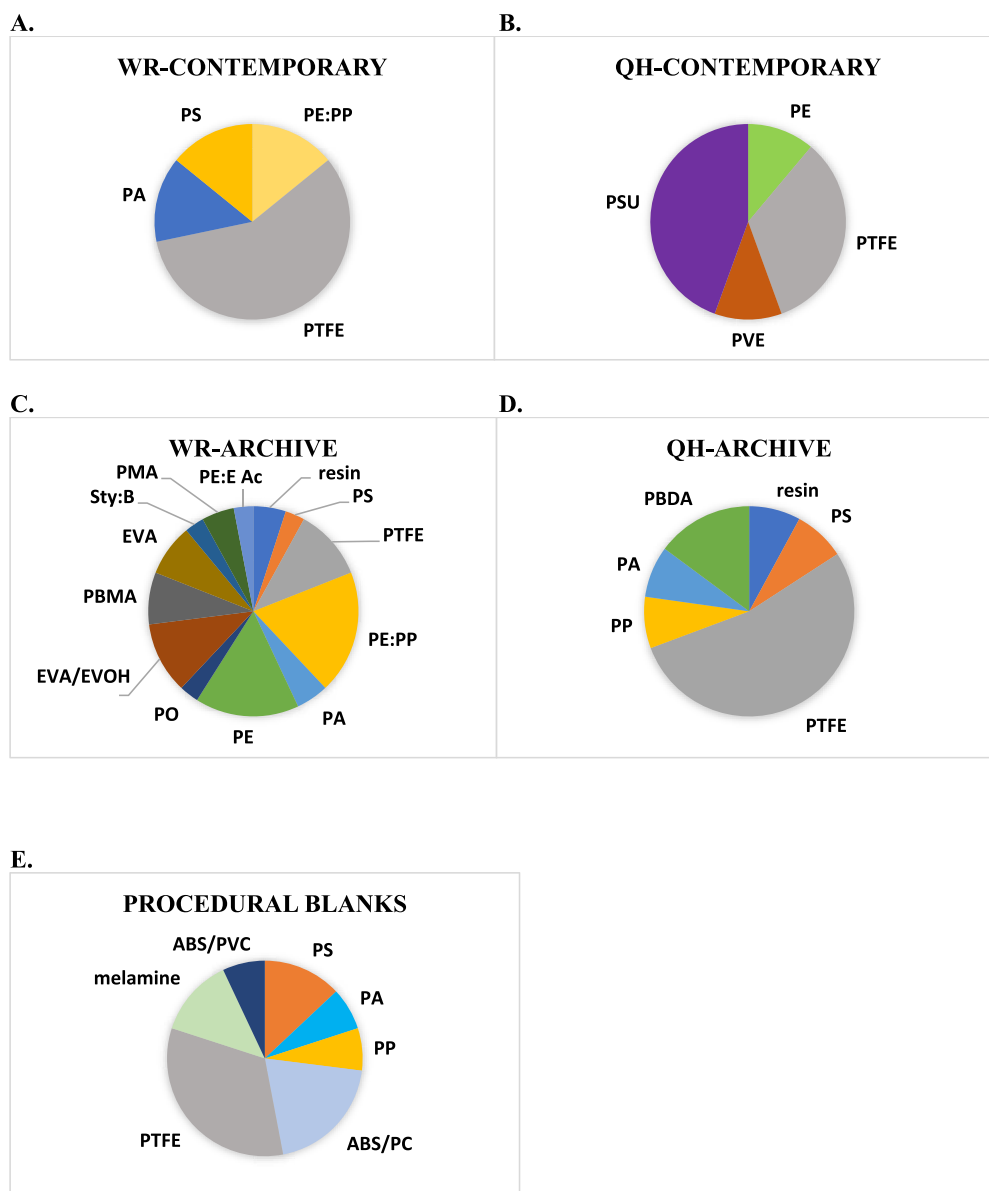


Fig. 4. MP polymer types identified in (A-B) contemporary core-source ($n = 3$) (C-D) archive-source ($n = 3$) sediment samples, and E. procedural blank ($n = 8$) samples. Abbreviations: ABS/PC, acrylonitrile butadiene styrene/polycarbonate; ABS/PVC, acrylonitrile poly vinyl chloride; EVA/EVOH, ethylene-vinyl acetate/ethylene vinyl alcohol; PA, polyalkene; PBDA, poly(11-bromoundecyl acrylate); PBMA, poly(11-bromoundecyl methacrylate); PMA, poly(*N*-methyl acrylamide); PO, polyolefin; PE, polyethylene; PE-E Ac, polyethylene/ethyl acrylate copolymer; PP, polypropylene; PP/PE, polypropylene polyethylene co-polymer; PTFE, polytetrafluoroethylene; PS, polystyrene; resins, hydrocarbon resin; Sty:B, styrene/butadiene copolymer.

industry, agriculture, transportation, and daily life, with the main sources identified as landfill, fertilizer and mulch degradation (Qi et al., 2018; Zhang et al., 2020a; Okoffo et al., 2021). Their fate once in the environment is still a pressing topic for research. Both the WR and the QH sites are located in the floodplain of the river Ouse, and recent groundwater monitoring along Tanner Street (where the 2023 core samples were extracted) demonstrated that, here at least, the below-ground deposits remain connected hydrologically with the river (Boast et al., 2019). Whilst construction of the North Street flood walls in 1992/93 has prevented surface flooding, the position of the water table in the sub-surface deposits is still determined by the height of the river, and the River Ouse is the principal source for recharge of waterlogged deposits in this area of York. Other contributing sources for recharge include rainfall from a wider catchment area, and potentially, leaking water mains, drains and sewers. Very high conductivity values recorded from samples from a dipwell in Tanner Street confirm recharge from river

water containing numerous dissolved chemical species derived from the underlying geology, use of agri-chemicals, and by-products from past industrial activity (Hudson-Edwards et al., 1999).

Concentration hotspots of MP contamination is likely due to impedance of groundwater flow, through a combination of the presence of physical barriers including the remains of ancient masonry walls and timber structures, and the variation in hydraulic conductivity and porosity of the various sediments making up the stratigraphic sequence. For example, clay-based lenses will form an impermeable barrier to the passage of groundwater, in effect creating a perched water table where MPs will accumulate. Interestingly, bentonite, used to seal geotechnical boreholes, was detected within all sediment samples except for the archived samples from QH lower and WR mid. Organic-rich sediments may also act as reservoirs of MPs because of their very low hydraulic conductivity values, which means they act like a sponge with the ability to capture and retain water as the water table falls.

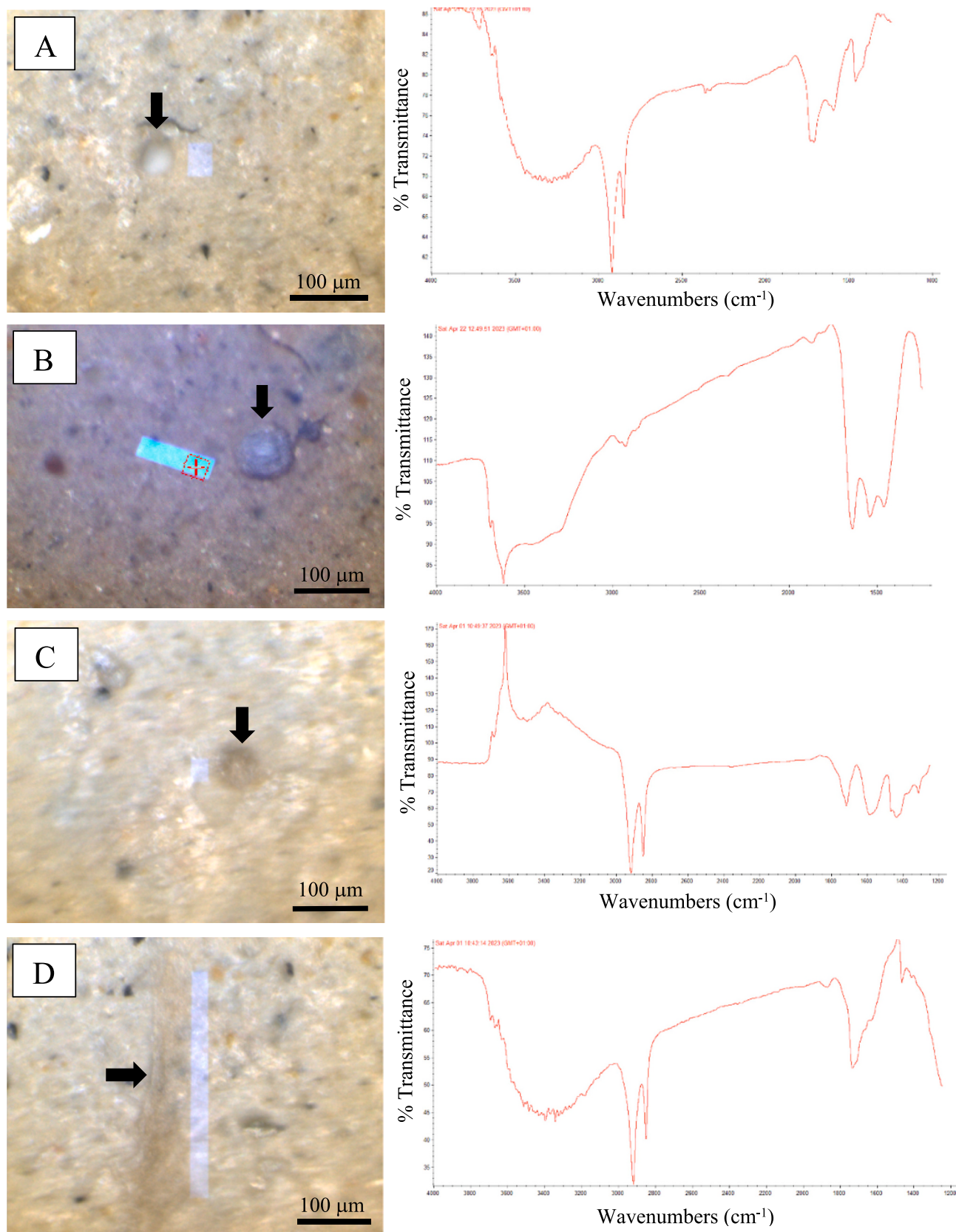


Fig. 5. Selected images of the MPs identified within sediment samples alongside the spectra obtained: (a) EVOH fragment, (b) nylon fragment (c) PE fragment, (d) PO fibre. Scale bar 100 μm . Abbreviations: EVOH, ethylene vinyl alcohol; PE, polyethylene; PO, polyolefin.

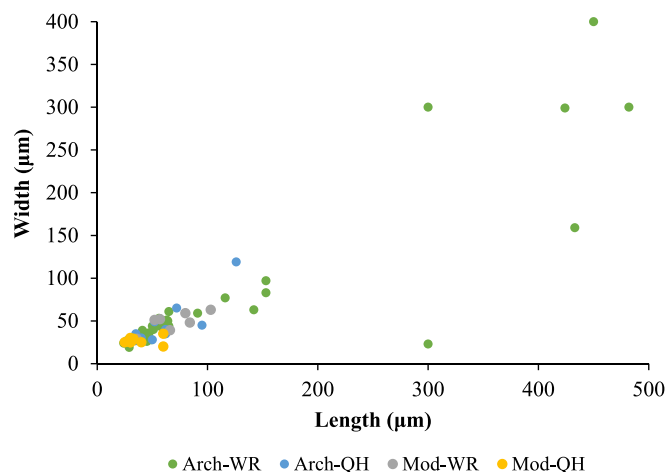


Fig. 6. Size distribution of all MPs analysed. Key: blue, archived QH; green, archived WR; yellow, modern QH; grey, modern WR.

While it is not possible to relate individual MP particles detected within the sediment samples to actual sources of contamination, it is possible to identify the types of industries and products they have been typically used in. Firstly, in general, fragment-shaped particles, which is the main particle shape detected, are thought to originate from the ‘wear and tear’ of larger plastic products such as plastic bottles, leading to the irregular shape. Sediments comprised MP particle polymer types that were predominantly PTFE, EVA/EVOH, PP:PE and PE. The most prevalent polymer type across all the sediment samples, both contemporary and archived sources, and not identified in the various blanks, were EVA/EVA-EVOH and PP:PE copolymer. EVA-EVOH tie layer polymers are used to bond plastic polymers to create flexible packaging materials that are optimised to improve moisture intrusion or tensile qualities (Foldes and Pukanszky, 2005). These may be found in food packaging and lamination type processes. PP:PE copolymer is used in textiles including carpets, as well as in the automotive industry in moulding of car bumpers due to its good impact test results at low temperatures. This copolymer has tougher qualities than PP alone (Zhang et al., 2021).

To add further weight to the suggestion that the 1980s samples were indeed infiltrated by the MP particles present in the environment at the time of sampling, an analysis of potential storage-associated procedural contaminant sources was conducted. The plastic storage buckets (PP), coring tubes (polymethacrylate), as well as a week-long air sample from the archive storage facility identified different predominant polymer types relative to those characterised in the archived sediments. The potential background contamination sources of MP particles comprised PE (which the plastic storage sample bags used for the archived samples were comprised of), nylon, PP (which are common in textiles and frequently reported within indoor air sampling studies), along with PET which was not detected herein (Jenner et al., 2021; Zhang et al., 2020b; O’Brien et al., 2023). PTFE was common in the laboratory analysis procedural blanks, complicating the interpretation of its presence in the sediment samples to the extent that only QH archived samples met the LOD but not the LOQ for inclusion in the total number of MPs present (Table 2).

This pilot project sought to investigate the presence of MP contamination within archaeological deposits. The research, undertaken within the historic city of York, known for its well-preserved waterlogged deposits yielding internationally significant archaeological evidence for the Roman (Parker, 2019) and Viking periods (Hall, 1988), demonstrated that sediment samples taken in the late 1980s and in 2023 were equally contaminated with MPs. These findings align with other archaeological studies currently investigating the archaeology of what is increasingly being referred to across disciplines as a Plastic Age (Schofield, In Press; Thompson et al., 2009), characterised by techno fossils

indicative of an Anthropocene, a new geological era in which human influence overrides natural cycles (Zalasiewicz et al., 2014). These techno fossils will provide chronological markers, just as pottery typologies do for earlier periods. For example: most techno fossils provide very fine dating resolution, not only for identifying the Anthropocene, but also to signal a discrete level within it. Among the modern polymers which are produced and consequently discarded in large amounts around the globe, one might consider, for instance, those under the PE umbrella: there are dozens of polymer types (LDPE, HDPE, and many others) that have been invented within decadal timespans and might represent fine-scale chemostratigraphical markers within Anthropocene strata (Zalasiewicz et al., 2014, 2022; Ivar do Sul and Labrenz, 2021).

Other current projects include the contemporary archaeology of plastic as material culture, with an emphasis on its disruptive impacts on UNESCO-listed World Heritage Sites such as the iconic Galapagos archipelago (Schofield et al., 2020), and its contribution to the archaeological record at Castell Henllys in West Wales, where the archaeological record now includes the evidence of school visits to the site, to view its earlier Iron Age remains (Mytum and Meek, 2021). A broader overview of plastics and archaeology is also now in production (Godin et al., in prep).

In our pilot project, we emphasise that contamination is occurring almost imperceptibly, and has been occurring over many years, seemingly through a diverse range of processes that relate to environmental change, environmental pollution, human behavioural change (e.g. plastics run-off from increased vehicular traffic on road and discard behaviours) and infrastructure (including and arguably notably leaking water and sewage service pipes). This is perhaps not surprising given the extent to which plastic pollution has been increasingly impacting the environment since the Second World War, when plastics entered common usage and single-use plastics became ubiquitous. The finds are also unsurprising when we consider the extent to which we now understand the extent of plastic pollution in the oceans (Harris et al., 2023), in the air (O’Brien et al., 2023), in soils (Table S1) and within human (Jenner et al., 2022; Leslie et al., 2022) and non-human bodies (Li et al., 2018).

Additional to contributing to the wider discourse on plastic pollution, this project will also have implications for cultural heritage management and heritage protection, positioning such conventional and established principles within the context of what is now described as toxic heritage (Kryder-Reid and May, 2023). There is a presumption in the UK, as defined in the National Planning Policy Framework, “that heritage assets are an irreplaceable resource and should be conserved in a manner appropriate to their significance, so they can be enjoyed for their contribution to the quality of life of existing and future generations” (paragraph 189, NPPF, DCLG, 2012). For heritage assets designated as being of national significance, preservation *in situ* is usually the preferred option, aligning with Willems’ observation that, ‘preservation *in situ* has developed into a central dogma of western archaeological heritage management’ (Willems, 2012). Such an approach is often also the preferred mitigation strategy for heritage assets considered of local significance. This accords also with the European Convention on the Protection of the Archaeological Heritage (Council of Europe 1992), commonly known as the Valletta Convention, which promotes the preservation and protection of archaeological heritage. This was a Convention created because ‘archaeologists have become aware that their source material is rapidly disappearing while only a tiny fraction of the information can be recorded’ (Willems, 2008). It did not however take account of the idea that such archaeological remains were becoming contaminated.

The argument for pursuing a policy of preservation *in situ* is that the development of historic urban centres is not stifled by the need to carry out expensive archaeological excavation, whilst ensuring that heritage assets are preserved for future generations to study and enjoy. In essence, and in theory, sustainable development and heritage assets can happily co-exist. However, the possibility that deeply stratified archaeological sediment sequences in York are now contaminated with MPs

raises serious questions about this presumption of *in situ* preservation. If replicated across the UK, then many heritage assets are potentially at risk from increased deterioration and loss of information potential. It has been identified that MPs can persist in soil environments for long periods of time, with degradation being very slow for some types of polymers compared to other environments, due to lack of UV radiation through burial hindering photodegradation (Chamas et al., 2020). As a result, they can have a significant impact on the chemical and physical characteristics of soil, such as soil structure and nutrient cycling, while also causing modifications in the processes of soil enzymes, microbes, animals, and plants (Rillig et al., 2021; de Souza Machado et al., 2019; Zhao et al., 2022).

As of yet, these risks within sediment samples cannot be quantified without further research. One published study (Yao et al., 2019) alludes to a “common observation in nature” that MPs are covered by biofilms of “diverse bacterial communities”. What impact such communities will have on long-term preservation of vulnerable organic archaeological materials has not been assessed. The potential for radiocarbon dating or residue/trace element analysis may be compromised by the presence of MPs, and again requires further investigation to identify whether these are real risks. The loss of information potential may be the greater threat to *in situ* preservation. Furthermore, plastic polymers contain thousands of additives (Hahladakis et al., 2018) which can leach into the surrounding environment and accumulate. The full repercussions of this are also currently unknown. Within soil environments, additives from several different polymer types have been identified to increase acute toxicity in soil, therefore also potentially changing the micro- and macrobiota composition (Kim et al., 2020; Ding et al., 2022).

The position of favouring preservation relates in part to the research potential of buried archaeological deposits, as an archive from which to gain new understandings of the past. The argument is that more information will be available from within these deposits in the future than now through scientific advances that provide new ways of investigating archaeological data. If this potential is compromised, then the argument for retaining archaeological deposits *in situ* comes into question. This study aligns with research that demonstrates our incomplete understanding of soil, which has often been studied predominantly from a biological, physical or chemical perspective, but rarely holistically as a complex ecosystem (Neal et al., 2020). A paradigm that considers soil as an expression of biological process brings us to an appreciation of the potential complexity that may arise from the introduction of MPs in this ecosystem. Archaeology still tends to consider soil as the ‘stuff’ in which artefacts and ecofacts are contained and its appreciation of this complexity remains in its infancy.

In conclusion, this is believed to be the first evidence of MP contamination in archaeological sediment (or soil) samples with polymers and size ranges measured and while accounting for procedural blanks. These results support the phenomenon of transport of MPs within archaeological stratigraphy, and the characterisation of types, shapes and size ranges identified therein. Through contamination, MPs may compromise the scientific value of archaeological deposits, and environmental proxies suspended within significant sediment, and as such represent a new consideration in the dynamism of, as well as arguments for preserving, archaeological deposits *in situ*.

CRedit authorship contribution statement

Jeanette M. Rotchell: Conceptualization, Data curation, Formal analysis, Funding acquisition, Methodology, Project administration, Supervision, Validation, Writing – original draft, Writing – review & editing. **Freija Mendrik:** Data curation, Formal analysis, Investigation, Methodology, Validation, Writing – original draft, Writing – review & editing. **Emma Chapman:** Data curation, Formal analysis, Investigation, Methodology, Validation, Writing – original draft, Writing – review & editing. **Paul Flintoft:** Conceptualization, Data curation, Funding acquisition, Project administration, Supervision, Visualization, Writing

– original draft, Writing – review & editing. **Ian Panter:** Data curation, Visualization, Writing – original draft. **Giulia Gallio:** Data curation, Visualization, Writing – original draft. **Christine McDonnell:** Data curation, Formal analysis, Investigation, Methodology, Validation. **David Jennings:** Conceptualization, Funding acquisition, Project administration, Supervision, Writing – original draft. **John Schofield:** Conceptualization, Funding acquisition, Project administration, Supervision, Writing – original draft, Writing – review & editing.

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.scitotenv.2024.169941>.

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