Freshwater and airborne textile fibre populations are dominated by 'natural', not microplastic, fibres

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Abstract

The potential role of natural textile fibres as environmental pollutants has been speculated upon by some environmental scientists, however, there is a general consensus that their biodegradability reduces their environmental threat. Whilst the risks that they pose remain poorly understood, their environmental prevalence has been noted in several recent microplastic pollution manuscripts. Here we highlight the extent to which natural textile fibres dominate fibre populations of upstream reaches of the River Trent, UK, as well as the atmospheric deposition within its catchment, over a twelve month microplastic sampling campaign. Across 223 samples, natural textile fibres represented 93.8% of the textile fibre population quantified. Moreover, though microplastic particles including synthetic fibres are known to be pervasive environmental pollutants, extruded textile fibres were absent from 82.8% of samples. Natural textile fibres were absent from just 9.7% of samples.

Highlights

- Natural textile fibres dominate freshwater and atmospheric fibre populations
- Environmental concentrations, of textile fibres vary greatly through time and space
- Upstream textile fibre concentrations can exceed that of the Marne River in Paris
- Atmospheric deposition is a potential source of textile fibres in remote locations
- Atmospheric deposition of textile fibres is not correlated to precipitation

Keywords

Textile fibres, Microplastic, Temporal variation, Atmospheric deposition, Surface water, Wastewater

1. Introduction

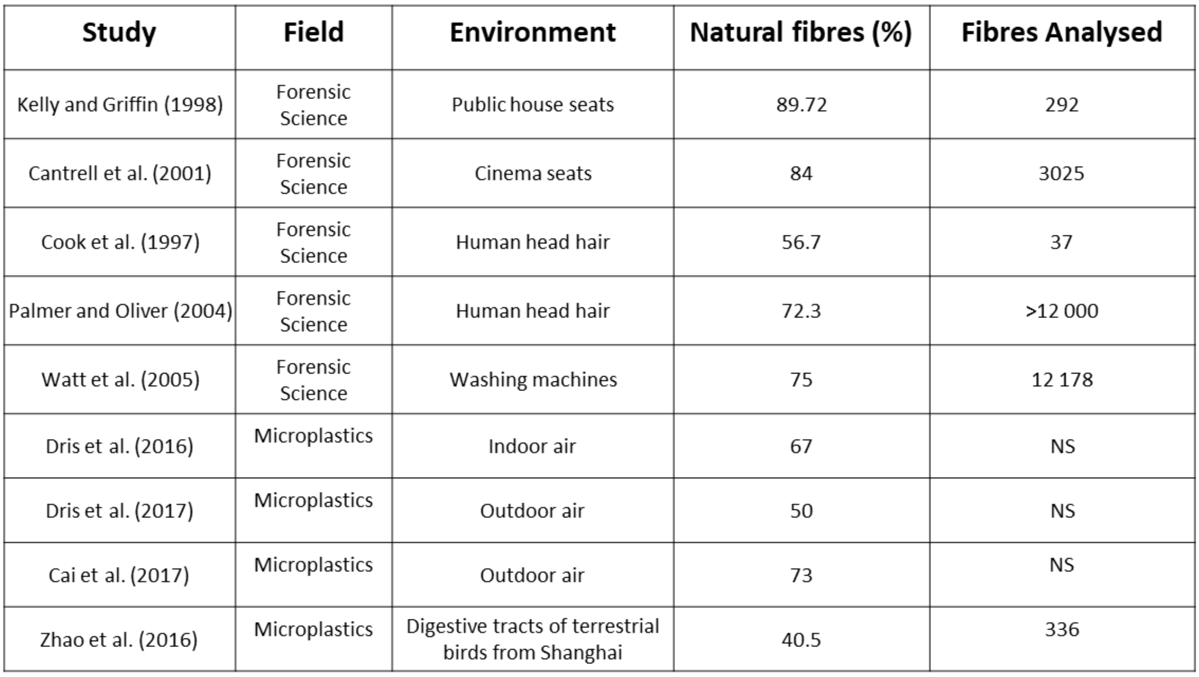
Mismanaged plastic waste is known to exert a variety of pressures on the environment. As awareness of these pressures has grown, efforts have been made to reduce plastic consumption by industry, governments and the general public, including the increased use of plastic alternatives. However, the potential environmental impacts of plastic alternatives are seldom considered in an environmental discourse that is currently so concerned with plastic waste. In 2015, Ladewig et al. (2015) highlighted the potential environmental threat of one such alternative for plastic textile fibres: natural textile fibres. Natural textile fibres, such as cotton and wool, are the product of multiple environmentally hazardous anthropogenic processes and are, therefore, inherently unnatural. For example, the commercial production of cotton fibres requires large volumes of water, pesticides and herbicides (Suran, 2018). The wastewaters of the textile industry have also long been recognised as point sources of chemical pollutants (Correia et al. 1994).

Unlike microplastic textile fibres, natural textile fibres have received little environmental attention. Fibres have the potential to entangle the gut contents of organisms that ingest them (Lusher et al. 2013), and any chemical effects of fibres are exacerbated by the relatively large surface area to volume ratio that they possess. The propensity for organic pollutants to adsorb to the surface of microplastic particles has been previously reported (Bakir et al. 2014), however, the extent to which this is true of natural textile fibres is currently poorly understood. Nevertheless, the faster degradation of natural textile fibres in comparison to microplastic fibres is a potential route for the release of toxic compounds, including dyes, into the environment (Ladewig et al. 2015).

The prevalence of natural textile fibres alongside synthetic textile fibres and microplastic fragments in the gastrointestinal tract of terrestrial birds was reported by Zhao et al. (2016), and in invertebrates by Remy et al. (2015). Dris et al. also acknowledge the presence of natural and synthetic textile fibres in atmospheric fallout (Dris et al. 2016; 2017), as well as the River Seine and one of its tributaries (Dris et al. 2018). However, since Ladewig et al. (2015), few other publications have acknowledged the potential environmental significance of natural textile fibres.

Though natural textile fibres are underrepresented in environmental literature, in the field of forensic science the relative proportions of textile fibres of different type and colour have been reported on a number of anthropogenic surfaces (Table 1). This work consistently records higher abundances of natural textile fibres in comparison to synthetic textile fibres. The findings of these studies are not necessarily representative of environmental matrices, but provide further evidence of the environmental prevalence of natural textile fibres.

Table 1: Prevalence of natural textile fibres in some forensic science and microplastic publications. NS corresponds to information that is not stated.



Study	Field	Environment	Natural fibres (%)	Fibres Analysed
Kelly and Griffin (1998)	Forensic Science	Public house seats	89.72	292
Cantrell et al. (2001)	Forensic Science	Cinema seats	84	3025
Cook et al. (1997)	Forensic Science	Human head hair	56.7	37
Palmer and Oliver (2004)	Forensic Science	Human head hair	72.3	>12 000
Watt et al. (2005)	Forensic Science	Washing machines	75	12 178
Dris et al. (2016)	Microplastics	Indoor air	67	NS
Dris et al. (2017)	Microplastics	Outdoor air	50	NS
Cai et al. (2017)	Microplastics	Outdoor air	73	NS
Zhao et al. (2016)	Microplastics	Digestive tracts of terrestrial birds from Shanghai	40.5	336

In the study of microplastic pollution, determining the proportion of a fibre population that is synthetic has been restricted by the methodologies used to analyse textile fibres. The visual identification of microplastic particles is a widely used and acceptable technique when supported by the chemical analysis of a subsample of particles (Lusher et al. 2017). However, within the study of microplastic pollution, the visual identification of synthetic textile fibres in particular has been criticised for its susceptibility to human error (Remy et al. 2015), despite being recognised as an important initial step in the classification of textile fibres to their main groups (Greaves and Saville, 1995; Nayak et al. 2012).

One common method of determining the chemical composition of microplastic particles, possessing the capability to conclusively identify synthetic polymers, is Fourier Transform Infrared (FTIR) spectroscopy. However, the difficulties of obtaining clear FTIR spectra from the small, often curved, surfaces of textile fibres is a limitation of FTIR spectroscopy that some have not been able to overcome. Microplastic surveys have, in the past, chosen to omit textile fibres from their study entirely (e.g. Foekema et al., 2013; Van Cauwenberghe et al., 2015), or to apply an analytical technique, such as FTIR spectroscopy, to only a small subsample of observed fibres and extrapolate from the identities of the fibres that could be chemically analysed (e.g. Dris et al. 2016).

Understanding the relative environmental concentrations of different types of textile fibre will facilitate a more critical consideration of the environmental impacts of textile fibres as a whole. Three broad categories of textile fibre are commonly used in the textile industry: 1. natural fibres derived from the processing of plant fibres, such as cotton, and animal fibres, such as wool; 2. regenerated fibres, such as rayon, which are reconstituted from the dissolved cellulose of plant materials and shaped into fibres by extrusion; and 3. synthetic fibres, formed by the extrusion of petrochemical based compounds. There are key visual distinctions, beyond those frequently used in the study of microplastic pollution, that differentiate between natural textile fibres and those formed by extrusion. The structures and formation of fibres in each of these three categories, including the extrusion process, are described in detail in Greaves and Saville (1995) and Hearle (2009).

By exploiting these visual characteristics in the study of microplastic pollution it is possible to categorise textile fibres as either natural or extruded using simple stereomicroscopy. This more accurate preliminary characterisation of textile fibres will enable the consideration of textile fibres in microplastic studies where suitable analytical techniques are not available, and will reduce the number of fibres in need of subsequent spectroscopic identification where they are.

Synthetic textile fibres have received considerable negative press in the reporting of microplastic pollution. Therefore, though textile fibres represent only one type of fibre use, a thorough and accurate understanding of the threats that textile fibres of all types pose, which is not reliant on extensive extrapolation, is of great social and environmental importance. With the aim of placing synthetic textile fibre pollution into a broader environmental context of anthropogenic particulate pollutants, this study expands existing criteria for the visual categorisation of textile fibres used in microplastic quantification to quantify the textile fibre population of 223 samples of river water and atmospheric deposition from 14 sites across the River Trent Catchment, UK. These findings provide strong support for the concerns detailed by Ladewig et al. (2015).

2. Materials and Methods

2.1. Site descriptions

Surface water samples were collected from 10 sites in the Trent catchment; three on the River Trent (RT), three on the River Leen (RL) and four on the River Soar (RS) (Figure 1). The location of these sites enabled the consideration of textile fibre concentrations near the sources of each river (RT1, RL1, RS1), immediately upstream (RT2, RL2) and immediately downstream (RT3, RL3, RS2, RS3) of urban population centres, and at sites that do (RS2-44) and do not (RT 1-3, RL 1-3, and RS1) receive wastewater treatment plant effluent, a known source of synthetic fibres (Leslie et al. 2017).

Atmospheric fallout was collected from the roofs of four buildings across the University of Nottingham's (UoN's) three UK teaching campuses (Figure 1). The UoN's University Park (UP) Campus is a 300 acre plot bordered on its south side by the approximately 50 acre Highfields Park and on its north by the approximately 500 acre Wollaton Hall Deer Park. Sites A and B are located on UP. Site A is located on the roof of one of University of Nottingham's main teaching buildings, which neighbours the central administrative building. It also spans a primary thoroughfare across the University Park Campus. The location of site B, on the roof of the University Of Nottingham's main gymnasium, is surrounded by student accommodation.

Approximately 650 m from UP, the UoN's 65 acre Jubilee Campus (JC) is surrounded by residential housing. Site C is located on JC. Site D is located on the UoN's 100 acre Sutton Bonington Campus. Approximately 12 km south of UP, Sutton Bonnington lies close to the rural border of Nottinghamshire and Leicestershire.

Access to the roofs is restricted to maintenance staff only, and clear signage stressed the importance to maintenance staff of staying clear of sampling apparatus at each site.

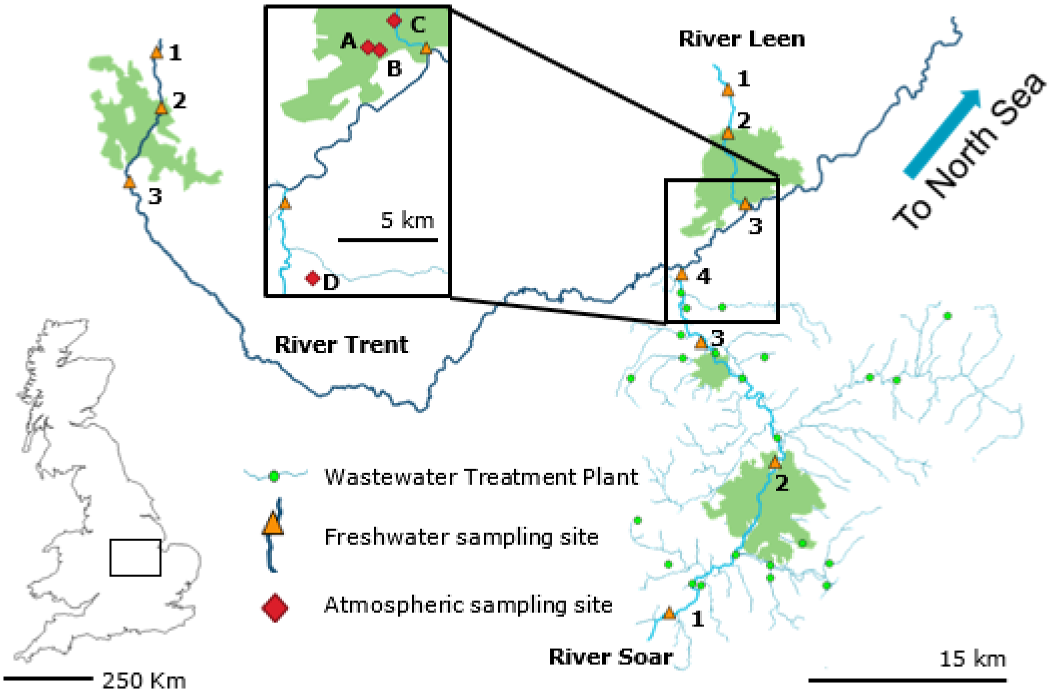


Figure 1: Locations of freshwater (numbered) and atmospheric (lettered) sampling sites within the Trent Catchment, UK. Green areas represent the urban areas of Stoke-on-Trent (River Trent), Nottingham (River Leen), Leicester (River Soar upstream) and Loughborough (River Soar downstream).

2.2. Sample collection

From the bank of the river at each freshwater sampling site, a 2 L paint kettle attached to a 5 m telescopic metal pole was used to retrieve 30 L of surface water. The water was concentrated in the field by passing it through a 63 μm sieve, removing the suspended silt and clay fractions of the suspended solids within the sample. The residue retained on the sieve was washed into a 200 ml glass bottle using distilled water. As the lids of the bottles were plastic, each lid was lined with aluminium foil that was replaced for each sampling occasion. Samples were collected every four weeks over a 12 month period.

Samples of atmospheric fallout were collected fortnightly using an approach similar to that of Dris et al. (2016). The sampling apparatus consisted of a 2.5 L amber glass bottle, into which fallout was funnelled by a 12 cm diameter (0.0113 m^2) glass funnel. Each fortnight the glass funnel was thoroughly rinsed with distilled water, ensuring its entire surface was rinsed, before replacing the amber glass bottle. Samples were collected over the same 12 month period as freshwater samples.

2.3. Sample processing

All freshwater samples were treated with hydrogen peroxide (H_2O_2) in order to digest organic material within the sample. The use of varying concentrations of H_2O_2 in the digestion of organic matter is common in the study of microplastic pollution, including 15% (Zhao et al. 2016) 30% (Liebezeit and Dubaish, 2012; Mathalon and Hill, 2014; Tagg et al. 2015) and 35% (Mintenig et al. 2017), and its effect on the appearance of plastic particles has been documented by Nuelle et al. (2014). In this study, 30% H_2O_2 was added to each aqueous sample. The sample was heated to 75ºC for 4-5 hours. Initially, 100 ml of H_2O_2 was added to each sample, however, during this stage of the third sampling occasion (week commencing 15/01/2018), the lids of the sample bottles perished exposing six of the samples to laboratory contamination. These six samples were therefore discarded. It is thought that the volume of H_2O_2 used and the unusually high organic matter content of these samples, collected during a period of heavy rain, contributed to this. As a result, after sample occasion three the volume of H_2O_2 added to each sample was reduced to 50 ml, and the lids of the glass jars were lined with two layers of aluminium foil. Moreover, just 15 L was collected during the fifth sampling occasion (week commencing 12/03/2018), falling during another period of heavy rain, to minimise the likelihood of sample bottles perishing.

Following H_2O_2 digestion samples were, where necessary, once again passed through a 63 µm sieve in order to remove any particles of silt and clay derived from the disaggregation of sedimentary agglomerations during the H_2O_2 digestion. The retained residue was washed back into its respective sample bottle in the same manner as in the field. Millipore filtration apparatus was then used to vacuum filter samples through 0.45 µm mixed cellulose ester gridded filter papers (Whatman ME 25/41) following the standard vacuum filtration procedures for H_2O_2 of three distilled water washes. The sample bottle and the sides of the vacuum filtration glassware were then rinsed using a distilled water wash bottle to ensure no particles remained adhered to the glassware, and the filter paper was immediately sealed in a plastic Petri dish.

In order to reduce the volume of collected rainfall, samples of atmospheric deposition were first shaken vigorously to ensure all particles were in suspension before being passed through a 38 µm sieve (step one). 300 ml of distilled water was then added to the empty amber glass bottle, which was shaken vigorously to entrain any remaining particles, after which I was passed through the same 38 µm sieve. This was done three times in succession (step two). The residue retained on the sieve from steps one and two was then washed into a 50 ml glass beaker using distilled water. Each sample was then vacuum filtered through the same mixed cellulose filter papers used for the freshwater samples, with the 50 ml beaker and sides of the vacuum filtration apparatus being rinsed using a distilled water wash bottle before the filter paper was sealed in a plastic petri dish.

The significance of the relative proportions of natural and synthetic textile fibres was assessed using a Wilcoxon test.

2.4. Contamination control

At each freshwater site, prior to freshwater sample collection, the paint kettle was submerged and emptied three times. It was then used to reverse wash the 63 μm sieve three times. The inside of the sieve was then rinsed with distilled water from a wash bottle ensuring the entire mesh and sides had been rinsed. On two occasions procedural blanks were collected for which this wash bottle rinse was collected and treated in the same manner as the environmental samples to assess the efficacy of these three steps.

A total of 8 amber glass bottles were used in the collection of atmospheric deposition, four of the bottles being rotated each fortnight. In addition to the three 300 ml distilled water washes that each sample received during sample processing, a further three distilled water washes were performed before a bottle was placed on the roof. To assess the efficacy of these steps, procedural blanks were collected for which the surfaces of the 38 μm sieve were washed into a glass beaker and processed in the same manner as the environmental samples. This was done in triplicate.

Unlike in the study of microplastics, wearing only natural textile fibred clothing was not a sufficient measure to limit contamination during sample collection. Instead, the type of fibre and colour of the garments worn during sample collection was recorded so that it could be considered during sample analysis. During sample collection tightly woven synthetic waterproof garments were worn. During sample processing a PVC apron was worn over a polyester / cotton blend laboratory coat to minimise the contamination of samples by fibres that had settled on the laboratory coat, and white / translucent fibres were excluded from analysis as these were assumed to have been sourced from the laboratory coat during sample processing.

Prior to sample processing, all laboratory surfaces were wiped down with dampened paper towels to remove surface dust. All glassware, including the freshwater sample bottles, and the sieves, were rinsed with distilled water prior to coming into contact with the samples. Sample bottles and beakers were triple washed with distilled water before being rinsed with distilled water using a wash bottle. All glassware components of the vacuum filtration apparatus, except for the conical flask into which the liquid fraction of the sample is sucked, were thoroughly rinsed with distilled water using a wash bottle, ensuring the entire surface was disturbed by the distilled water. The sieves were reverse rinsed with distilled water and the mesh and sides were then rinsed with distilled water using a wash bottle as done in the field. The filter papers were also rinsed with distilled water prior to being used.

During sample processing, the glassware and sieves were all covered with aluminium foil except for when the samples were being sieved, transferred between receptacles, or when the freshwater sample bottles, glass beakers containing atmospheric samples, or vacuum filtration apparatus were being rinsed. When the samples were not covered with aluminium foil, a dampened filter paper, placed in a petri dish, was exposed to the laboratory air to monitor the deposition of fibres over the same period of time that the samples were exposed. One dampened filter paper was used for each batch of samples processed (1 batch = ten freshwater samples or four atmospheric samples) to measure the total deposition of airborne fibres within the laboratory during the processing of each batch. Furthermore, with only the sample processor was permitted within the laboratory whenever samples were being processed.

2.5. Textile fibre characterisation

2.5.1. FTIR spectroscopy of textiles

In order to assess the suitability of FTIR spectroscopy in the analysis of environmental textile fibre populations, an FTIR library of seven common textile fibres was produced using a combination of Attenuated Total Reflectance (ATR) FTIR and reflectance FTIR spectroscopy. This library consisted of FTIR spectra for: acrylic, cotton, polyamide, polyester, polypropylene, silk and wool textiles.

In the production of this library, garment patches were first analysed by ATR-FTIR spectroscopy using a Bruker Tensor 27 FTIR spectrometer (Bruker Optics, Coventry, UK, equipped with a Graseby-Specac Golden Gate ATR accessory (Orpington, UK). For each spectrum, 16 scans with a 4 cm^{-1} resolution were co-added, providing FTIR spectra of the high fibre density samples. A pill of fibres was then pulled from each garment and analysed using the same technique, producing an ATR spectrum for a sample of lower fibre density.

Reflectance FTIR spectroscopy was then used to produce spectra of an individual fibre from each garment type using a Bruker Hyperion 2000 FTIR microscope (Bruker Optics, Coventry, UK). For each spectrum, 128 scans with a 4 cm^{-1} resolution were co-added. These fibres were too small for analysis by ATR-FTIR spectroscopy.

2.5.2. Visual analysis of textile fibres

Samples were observed under a stereomicroscope (Medline Scientific CETI Varizoom-10, Chalgrove, UK) with a magnification range of 16-160 x and, where greater optical clarity was required, an optical microscope at 100 x magnification (Euromex Bioblue, Arnhem, The Netherlands). Every textile fibre was categorised as either extruded or natural on the understanding that only synthetic textile fibres (e.g. polyester) and regenerated fibres (e.g. rayon) are manufactured by extrusion, whereby a molten polymer is forced through an aperture of fixed – and not necessarily circular – cross-section. The resultant individual fibres therefore have uniform diameter. Prior to sample analysis, the proficiency of the textile fibre analyst was developed through the extensive observation of textile fibres of known origin, aided by a literature-informed expansion of the criteria for visual textile fibre characterisation of the Royal Microscopical Society's *Microscopy of Textile Fibres* Handbook (Greaves and Saville, 1995) (Figure 2). These criteria were applied to the analysis of all environmental samples. Four textile fibres identified using the visual methodology outlined above were analysed by reflectance FTIR spectroscopy and compared to the FTIR library generated from known textile fibres.

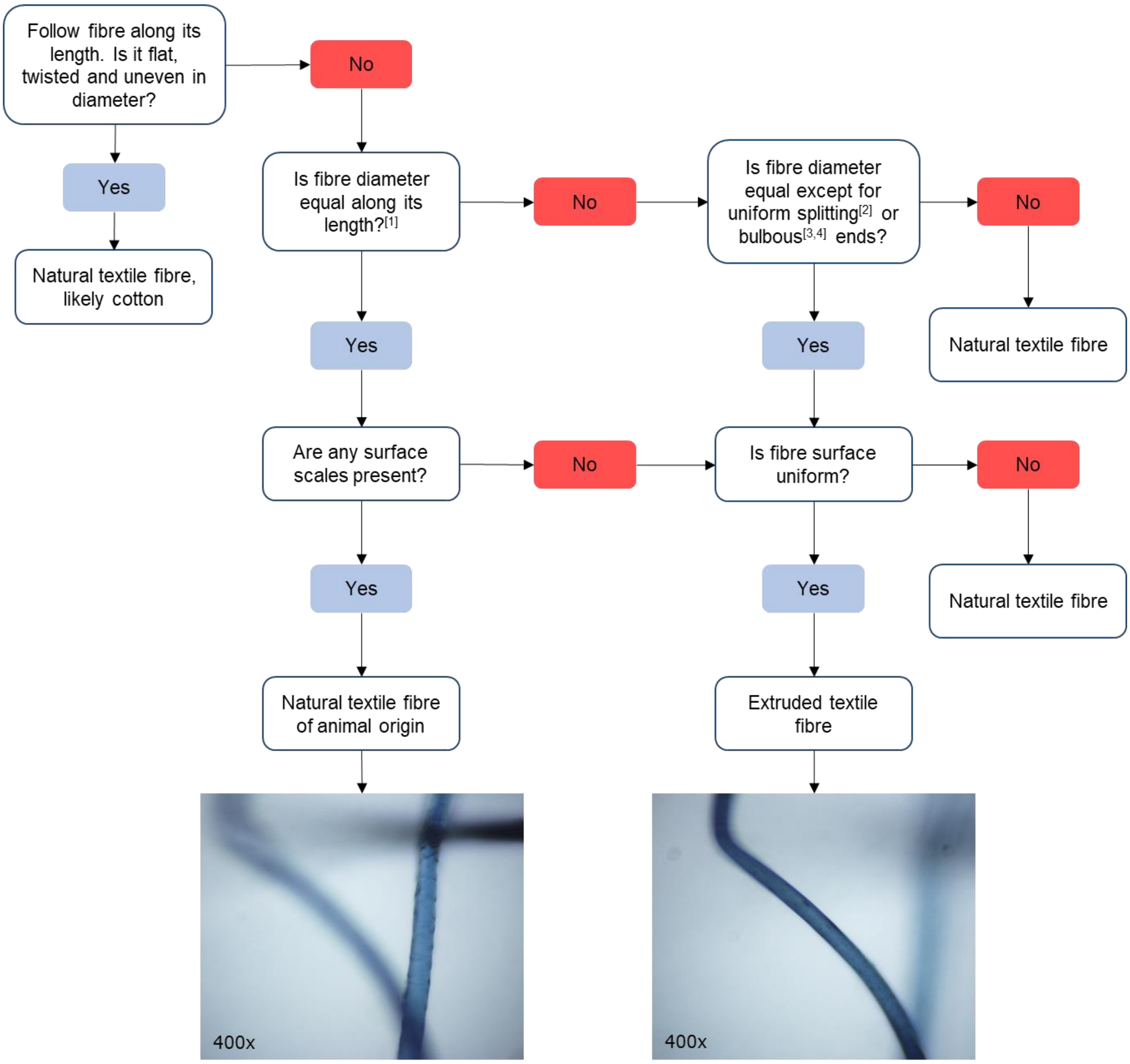


Figure 2: Flowchart used to characterise textile fibres as natural or extruded, with photographs highlighting the subtle differences between an animal (wool) and an extruded (polyester) fibre. Except where stated, these criteria have been adapted from the Royal Microscopical Society's Microscopy of Textile Fibres handbook (Greaves and Saville, 1995).

[1] Norén (2007), [2] MERI (n.d.), [3] Wąs-Gubała and Krauß (2006), [4] Palenik et al. (2013).

3. Results

3.1. FTIR spectroscopy of textile patches, fibre pills and individual fibres

FTIR spectroscopy of textile patches and fibre pills produced clear spectra (Figure 3) that, for the synthetic textile fibres analysed, could be identified by the available Bruker spectral library. However, the library available to this study was limited, being only a demonstration library, and was not able to identify the ATR-FTIR spectra generated from natural textile fibres. In contrast to the ATR-FTIR spectra, the spectra produced by reflectance FTIR spectroscopy were noisy (Figure 3). The identity of the four environmental fibres that were analysed by reflectance-FTIR spectra could not be ascertained from the spectra in Figure 3 due to this noise (Figure 4).

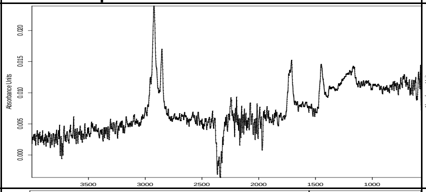
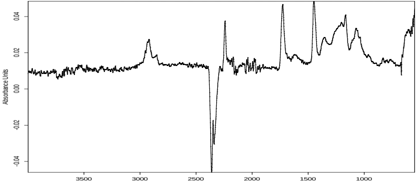
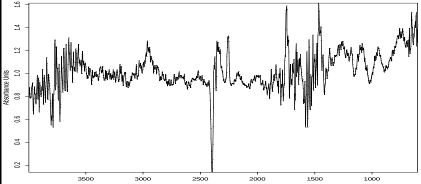
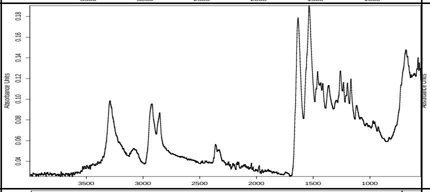
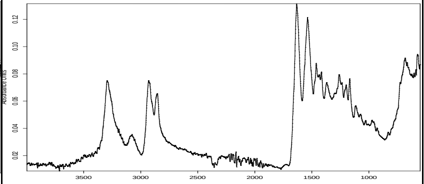
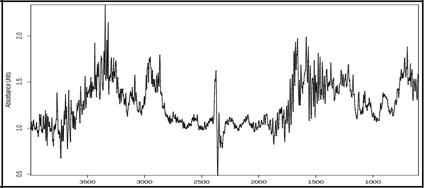
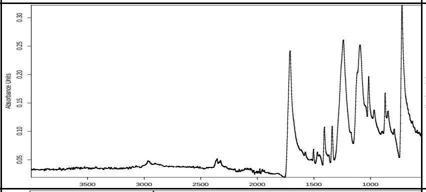
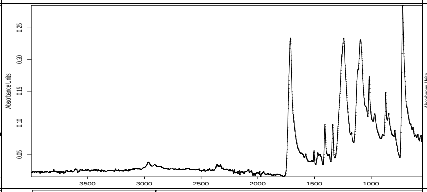
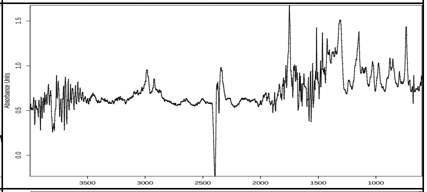
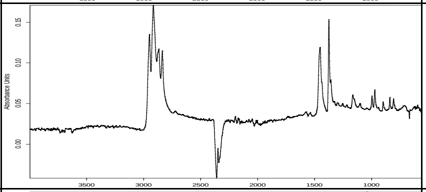
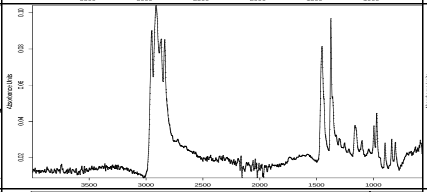
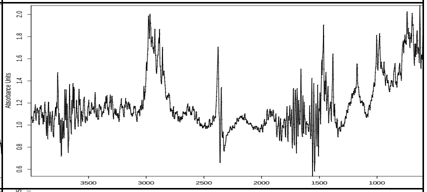
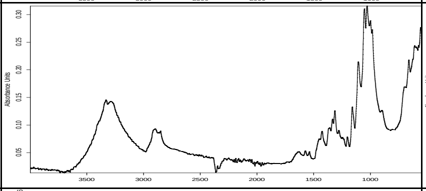
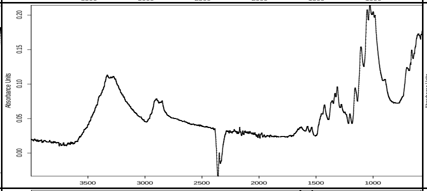
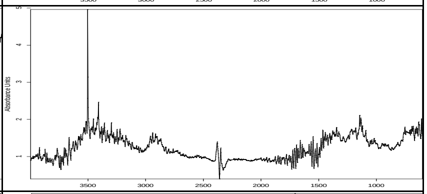
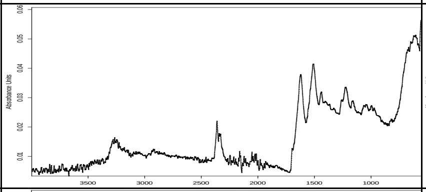
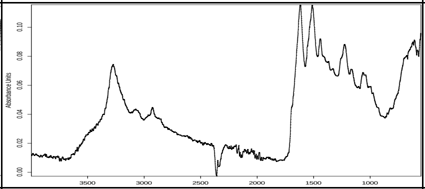
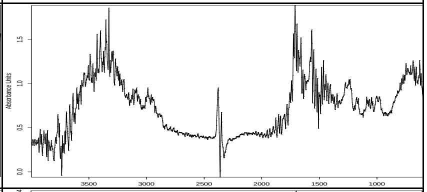
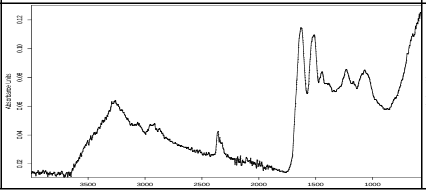
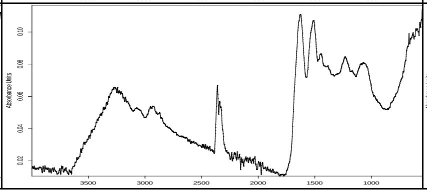
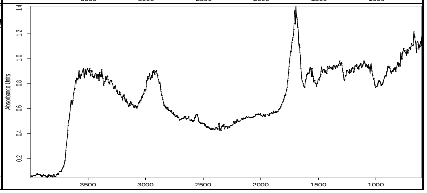
Fibre	Patch spectrum	Pill spectrum	Fibre spectrum
Acrylic			
Poly-amide			
Poly-ester			
Poly-propylene			
Cotton			
Silk			
Wool			

Figure 3: Library produced from the ATR-FTIR spectra of textile patches and pills and reflectance FTIR spectra of individual fibres from garments of known fibre composition.

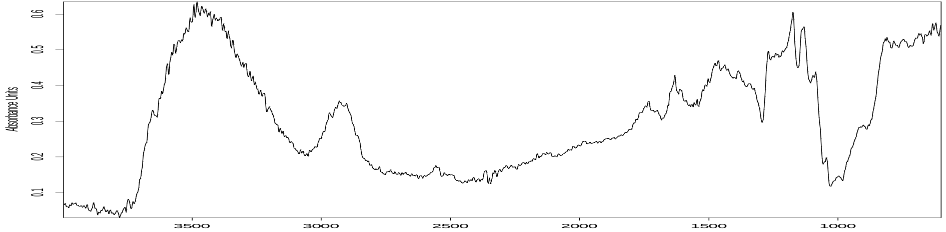
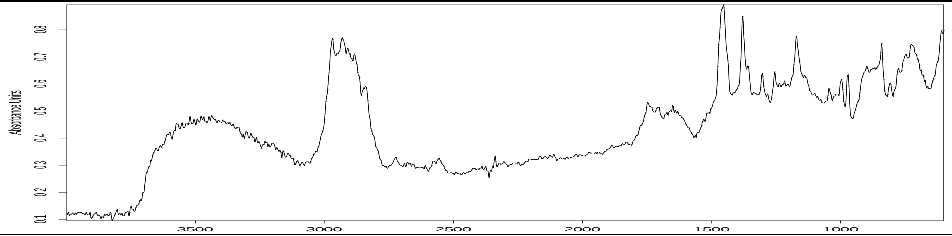
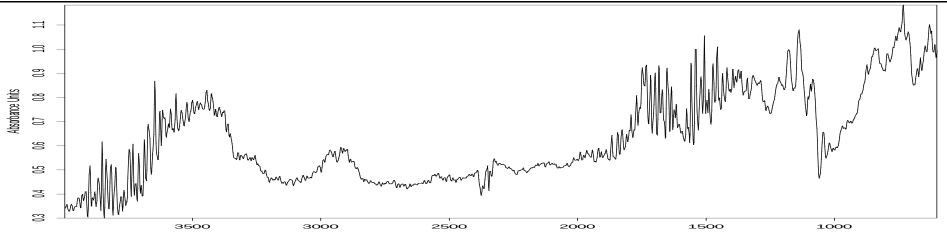
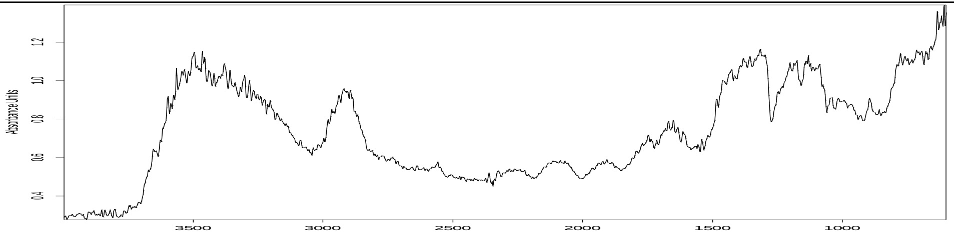
Fibre ID	Visual categorisation	Reflectance spectrum
A	Natural	
B	Natural	
C	Extruded	
D	Extruded	

Figure 4: Reflectance FTIR spectra of four fibres quantified from the 12 month sampling campaign

3.2. Freshwater and atmospheric textile fibre populations

A total of 130 freshwater and 93 atmospheric samples were collected for microplastic and textile fibre analysis between 09/11/2017 and 31/07/2018. 720 fibres were categorised in the freshwater environment, 639 (87.3%) of which were identified as natural (Table S1). In atmospheric fallout, 1100 fibres were categorised of which 1075 (97.7%) were identified as natural (Table S2).

In the context of microplastic pollution, even if all of the extruded textile fibres were petrochemical-based, they made up just 6.2% of the total textile fibre population across all of the atmospheric and freshwater samples. The majority of textile fibres observed were either black/grey (47.09%, n=857) or blue (24.40%, n=444) in colour, as reported in multiple forensic textile fibre population surveys (e.g. Kelly and Griffin, 1998; Cook et al. 1997; Cantrell et al. 2001; Palmer and Oliver 2004; Watt et al. 2005). The absence of extruded textile fibres showing signs of degradation or bleaching support the findings of Nuelle et al. (2014), who demonstrate only limited effects of H_2O_2 on plastic polymers including polyamide, from which nylon is derived, and Polyethylene Terephthalate, a common form of polyester. The effect of H_2O_2 on natural textile fibres is expected to be limited. H_2O_2 is a common bleaching agent used in the textile industry (Carmen and Daniela, 2012), and so the presence of natural textile fibres in such high abundance, of multiple types and in a variety of colours indicates that the H_2O_2 concentrations used were too low to cause even the discolouration of textile fibres.

Textile fibre abundance varied through space and time in both freshwater (Figure 5) and atmospheric samples (Figure 6). Throughout the freshwater sampling campaign, site RL3 had the highest mean freshwater concentration of natural fibres ($\bar{x}$ = 0.29 fibres L^{-1}) (Figure 7), whilst the highest mean extruded fibre concentration was observed at site RT3 ($\bar{x}$ = 0.04 fibres L^{-1}) (Figure 8). Neither of these sites is in receipt of wastewater treatment plant effluent, but they are downstream of the cities of Nottingham and Stoke-on-Trent respectively. In atmospheric fallout, the highest mean natural textile fibre deposition was observed at Site A ($\bar{x}$ = 128.42 fibres $m^{-2}day^{-1}$), with the highest extruded textile fibre deposition observed at Site B ($\bar{x}$ = 2.90 fibres $m^{-2}day^{-1}$) (Figure 9). In contrast to previous surveys of the atmospheric deposition of microplastics, the present study did not observe a correlation between fibre deposition and precipitation (Figure 10). In each of the rivers and across the sites of atmospheric deposition there were significantly more natural textile fibres than extruded fibres. Monte Carlo significance values for the Wilcoxon tests conducted in each of these datasets were all <0.001.

A total of 9 atmospheric and 15 freshwater samples contained no textile fibres at all. These samples in particular are testament to the efficacy of the contamination controls followed (Woodall et al. 2015; Taylor et al. 2016). The abundance of textile fibres from laboratory deposition is detailed in Tables S3 and S4, and across the procedural blanks in Table S5.

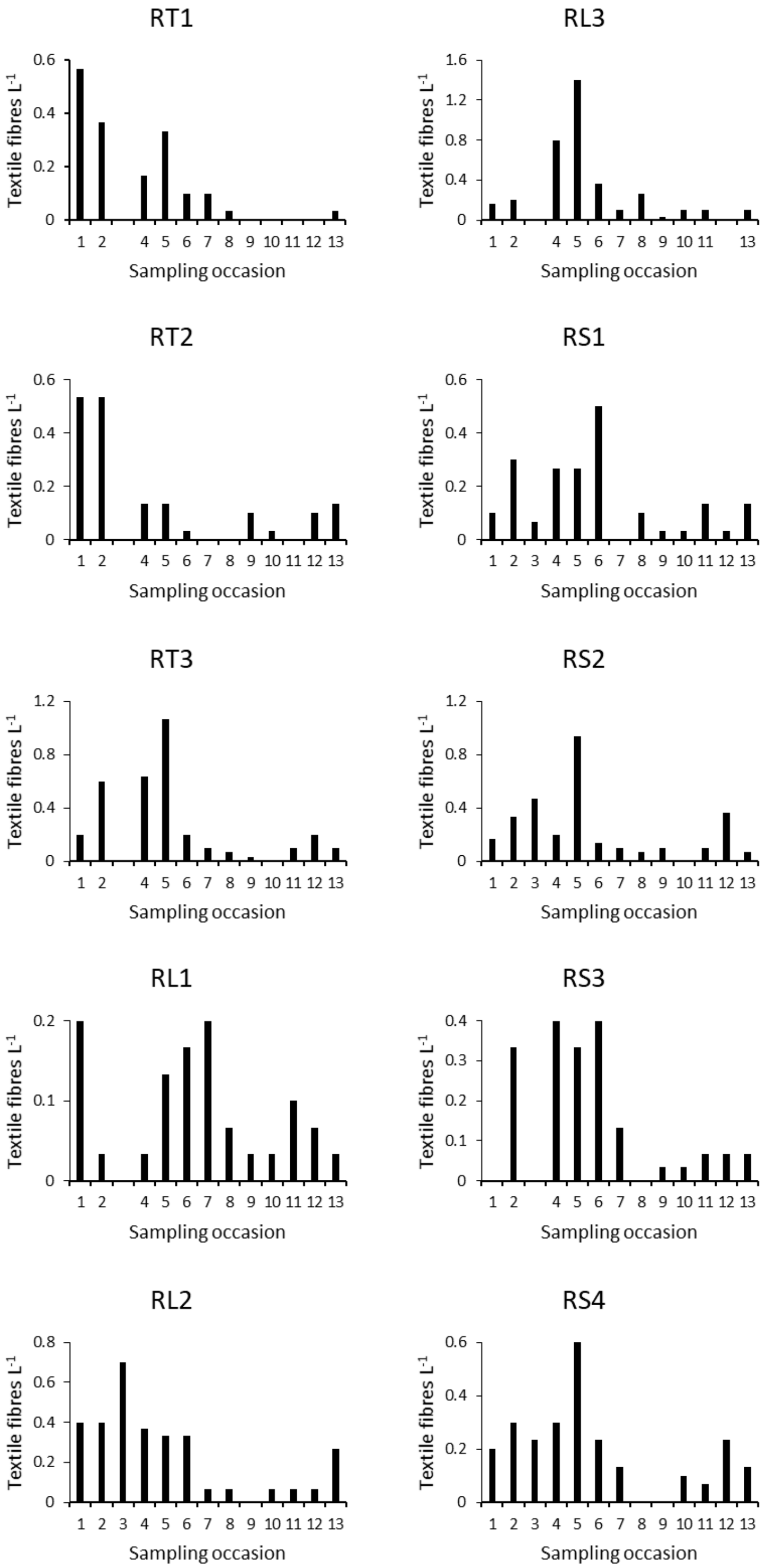


Figure 5: Total fibre concentrations at each freshwater site over the 13 sampling occasions.

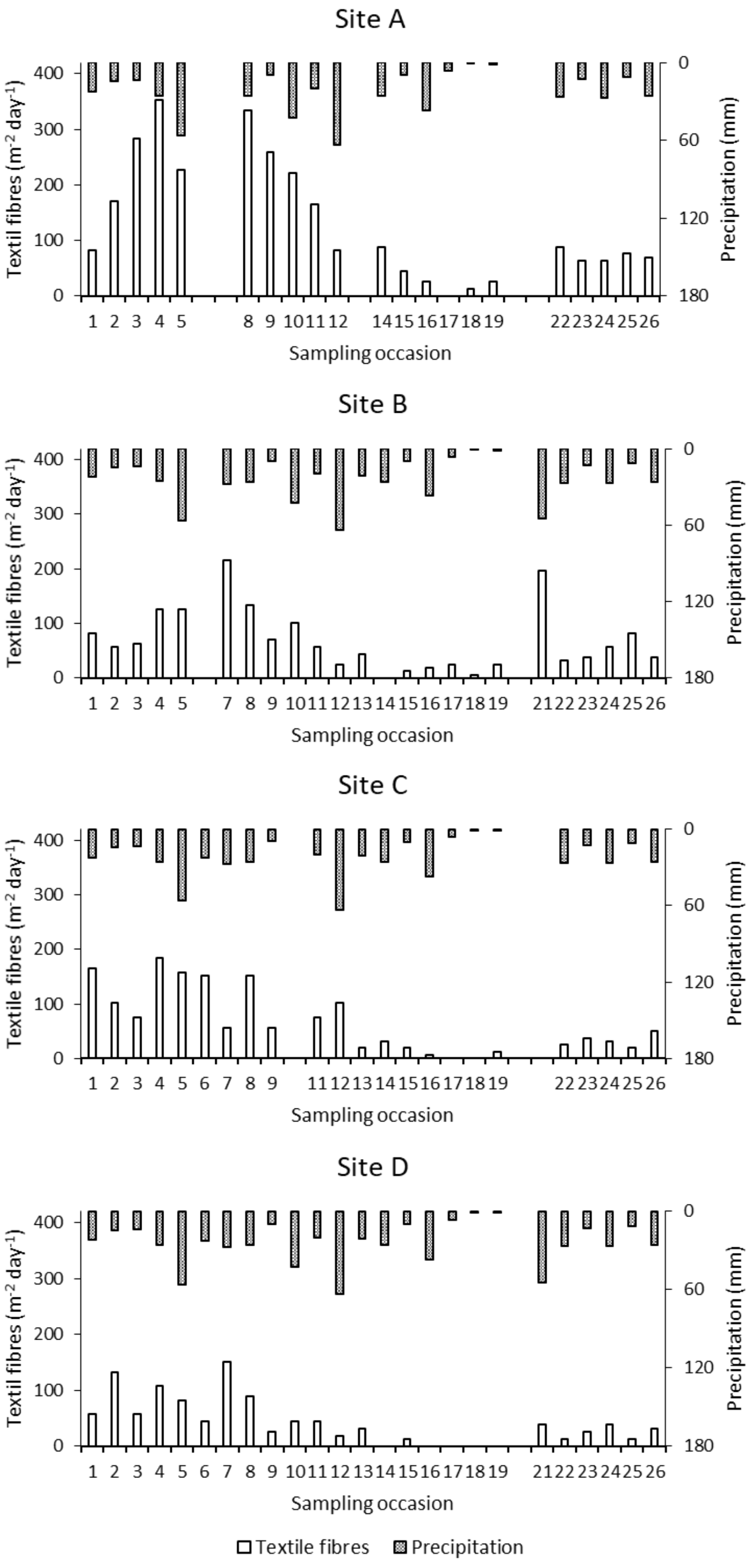
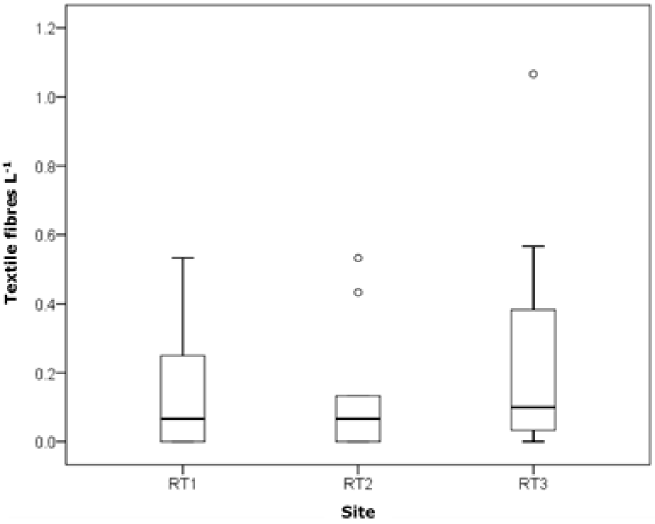
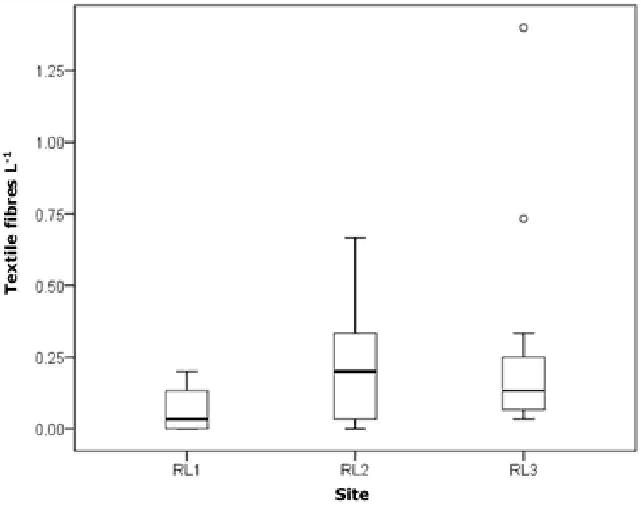


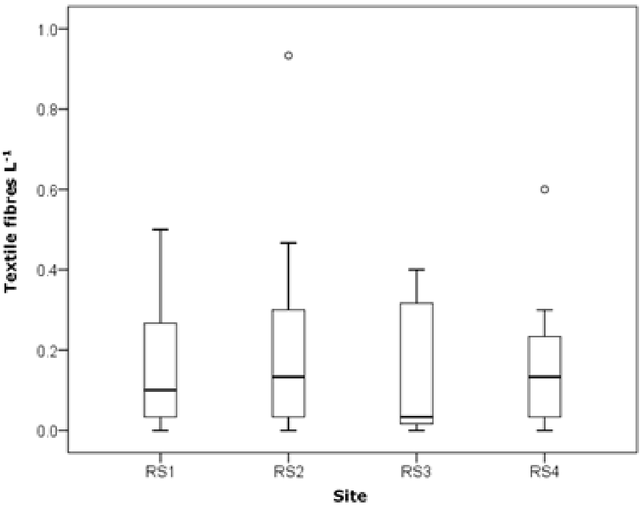
Figure 6: Atmospheric deposition of natural and extruded textile fibres on each sampling occasion for each site. Daily precipitation data was collated from the Met Office's HadUKP dataset for the Central England region (Alexander and Jones, 2001).



	RT1	RT2	RT3
Mean	0.1361	0.1250	0.2389
Median	0.0667	0.1250	0.2389
Std. Deviation	0.1800	0.1765	0.3284
Minimum	0	0	0
Maximum	0.5300	0.5300	1.0700

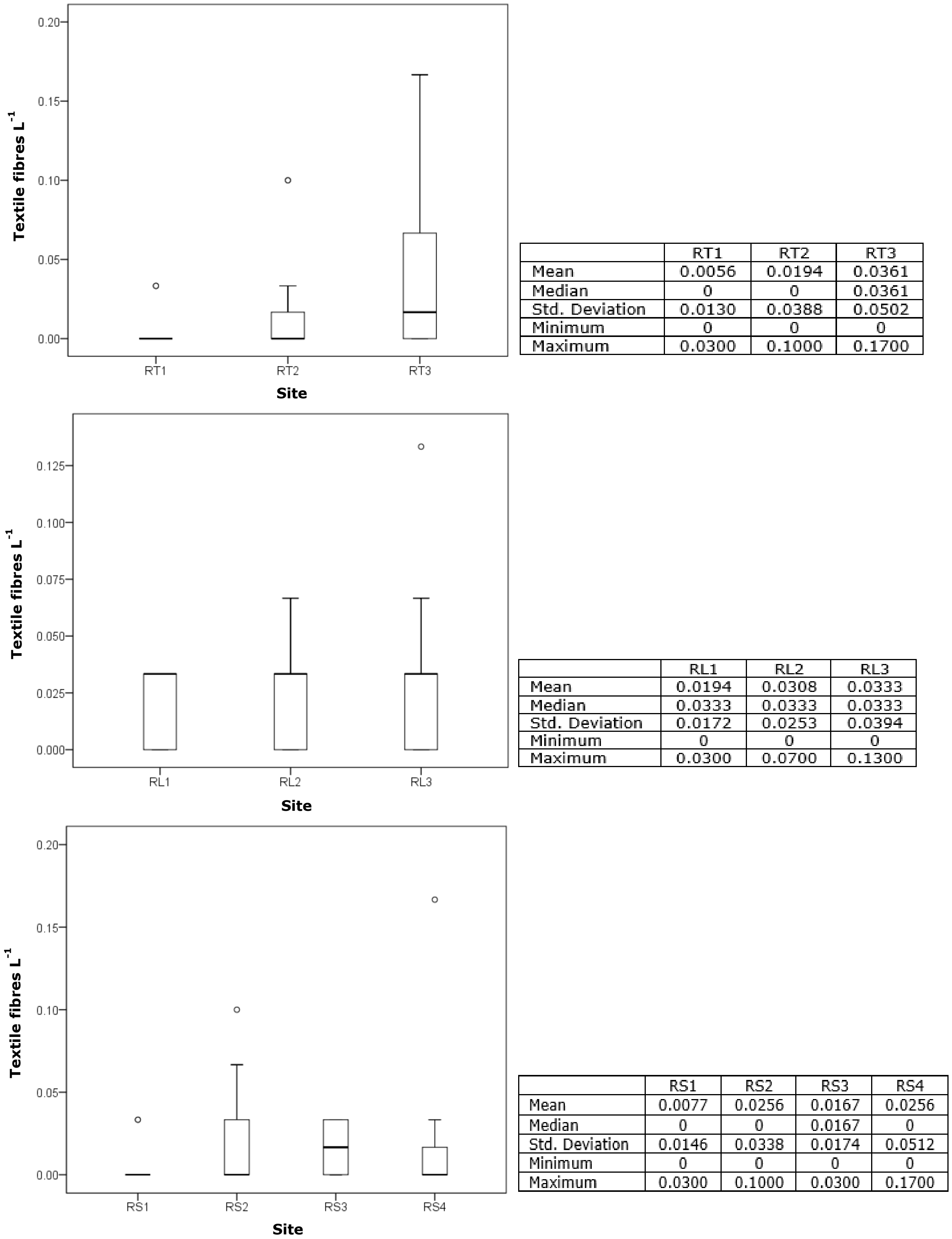


	RL1	RL2	RL3
Mean	0.0722	0.2103	0.2970
Median	0.0333	0.2000	0.1333
Std. Deviation	0.0776	0.2052	0.4167
Minimum	0	0	0
Maximum	0.2000	0.6700	1.4000



	RS1	RS2	RS3	RS4
Mean	0.1436	0.2077	0.1389	0.1692
Median	0.1000	0.1333	0.0333	0.1333
Std. Deviation	0.1462	0.2597	0.1663	0.1697
Minimum	0	0	0	0
Maximum	0.5000	0.9300	0.4000	0.6000

Figure 7: Boxplots illustrating the median and range of natural textile fibre concentrations at each freshwater sampling site.

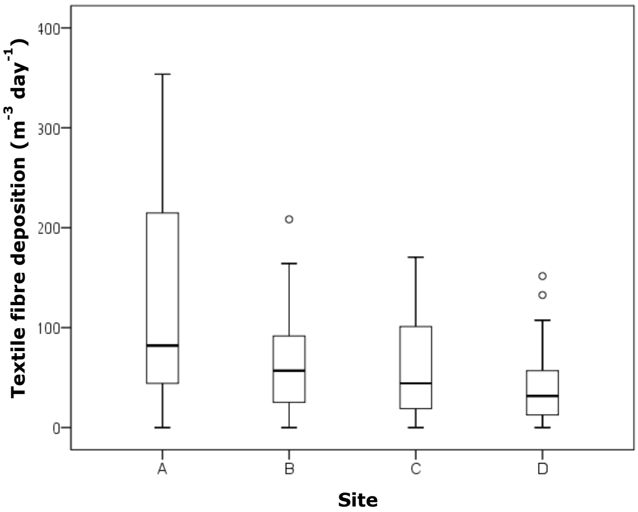


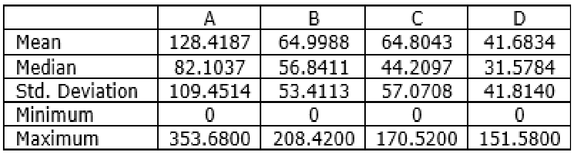
	RT1	RT2	RT3
Mean	0.0056	0.0194	0.0361
Median	0	0	0.0361
Std. Deviation	0.0130	0.0388	0.0502
Minimum	0	0	0
Maximum	0.0300	0.1000	0.1700

	RL1	RL2	RL3
Mean	0.0194	0.0308	0.0333
Median	0.0333	0.0333	0.0333
Std. Deviation	0.0172	0.0253	0.0394
Minimum	0	0	0
Maximum	0.0300	0.0700	0.1300

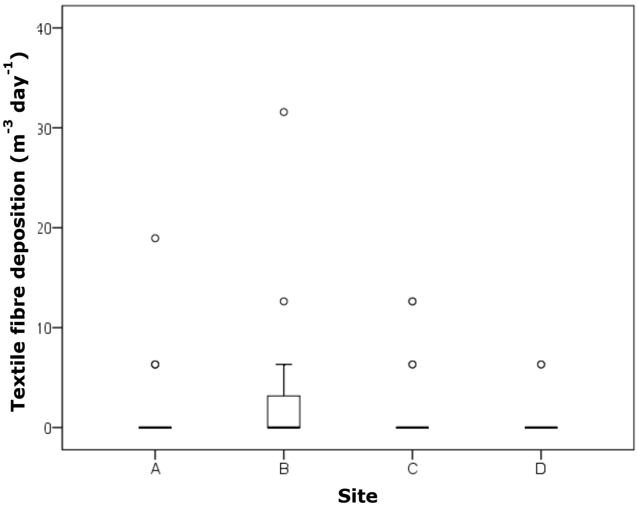
	RS1	RS2	RS3	RS4
Mean	0.0077	0.0256	0.0167	0.0256
Median	0	0	0.0167	0
Std. Deviation	0.0146	0.0338	0.0174	0.0512
Minimum	0	0	0	0
Maximum	0.0300	0.1000	0.0300	0.1700

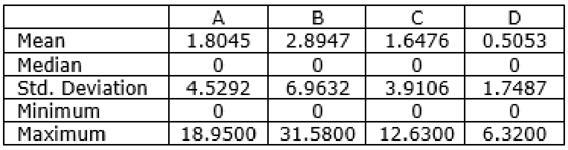
Figure 8: Boxplots illustrating the median and range of extruded textile fibre concentrations at each freshwater sampling site.





	A	B	C	D
Mean	128.4187	64.9988	64.8043	41.6834
Median	82.1037	56.8411	44.2097	31.5784
Std. Deviation	109.4514	53.4113	57.0708	41.8140
Minimum	0	0	0	0
Maximum	353.6800	208.4200	170.5200	151.5800





	A	B	C	D
Mean	1.8045	2.8947	1.6476	0.5053
Median	0	0	0	0
Std. Deviation	4.5292	6.9632	3.9106	1.7487
Minimum	0	0	0	0
Maximum	18.9500	31.5800	12.6300	6.3200

Figure 9: Boxplots illustrating the median and range of natural and extruded textile fibre concentrations at each atmospheric sampling site.

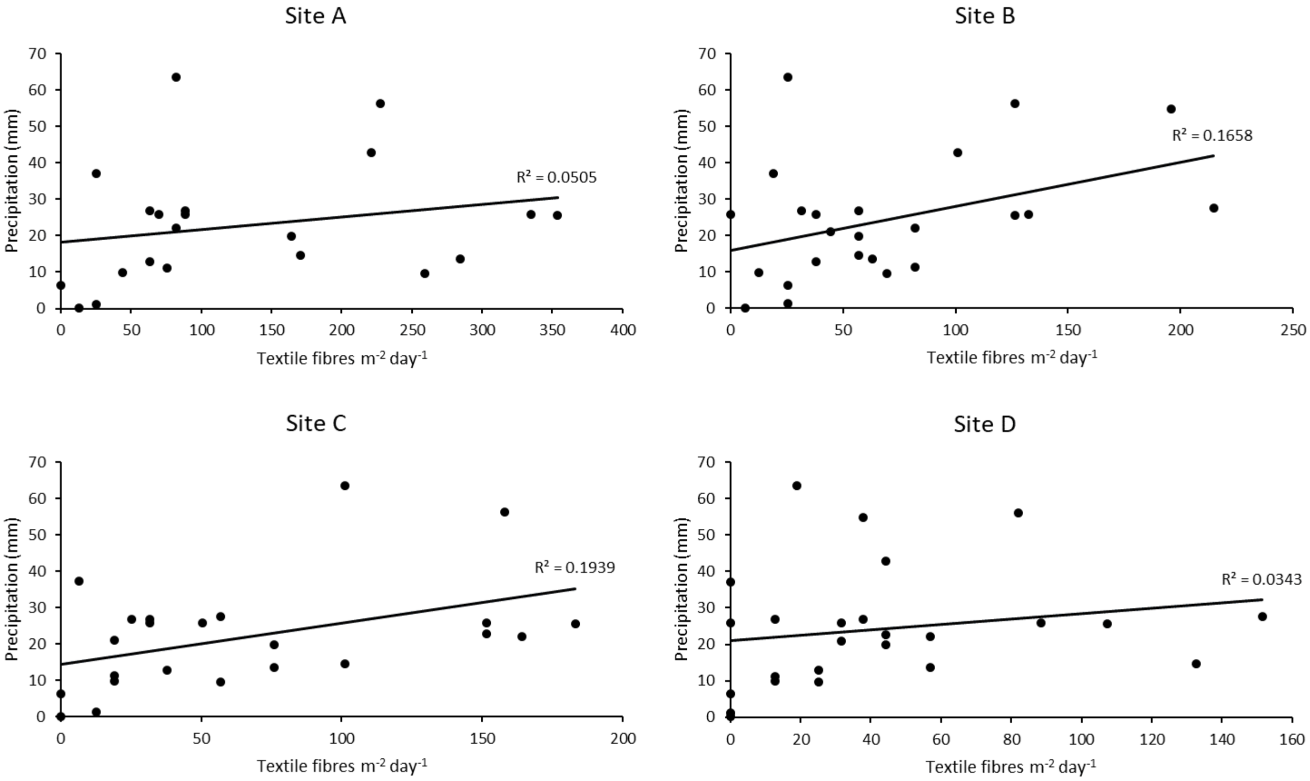


Figure 10: Correlation of number of fibres deposited and precipitation over each fortnightly sampling period.

3.3. Freshwater fluxes of textile fibres

The abundance of freshwater textile fibres at each site are presented here as particles per litre. Using UK National River Flow Archive (NRFA) gauging stations it is, however, possible to consider textile fibre fluxes at various points within the sampled reaches. NRFA gauging stations are located short distances downstream of site RT2, and upstream of sites RL3 and RS4 (Table S6). Flux calculations based on the mean discharges at each of these stations and the mean textile fibre concentrations quantified over this twelve month sampling campaign are detailed in Table 2.

Table 2 Textile fibre flux estimates at sites in close proximity to UK NRFA gauging stations presented to three significant figures. Mean flow data acquired 07/01/2019.

Site	Mean flow (m^3s^{-1})	Mean textile fibre flux (fibres/day)	Minimum textile fibre flux (fibres/day)	Maximum textile fibre flux (fibres/day)
RT2	0.626	7 810 000	0	28 800 000
RL3	0.685	19 500 000	1 970 000	82 900 000
RS4	11.727	197 000 000	0	608 000 000

4. Discussion

4.1. FTIR characterisation of known and environmental textile fibres

Whilst the use of ATR-FTIR is shown here to be an effective method of fibre identification for high fibre density samples such as garment patches and fibre pills, it is not one that can be easily applied to the analysis of individual textile fibres such as those from environmental samples, the majority of which are too small to be handled for ATR-FTIR analysis. Moreover, though the efficacy of reflectance FTIR spectroscopy has been demonstrated for larger (150 µm) microplastic particles (Harrison et al. 2012), its suitability in the analysis of textile fibres has not been assessed.

Of the four fibres from environmental samples analysed by reflectance FTIR spectroscopy (Figure 4), the two natural textile fibres were visually identified as unmercerised cotton. Cotton is the most common natural textile fibre (Ladewig et al. 2015), and in its unmercerised form is easily identifiable as a flat, twisted fibre of uneven diameter (Figure 2). As the identity of these environmental fibres was known, it was possible to make a direct comparison between the reflectance FTIR spectra of the two environmental cotton fibres (Figure 4) and that produced for the FTIR library (Figure 3). The variation between these three spectra not only provides further evidence of the limited value of reflectance FTIR spectroscopy in the analysis of textile fibres, but also highlights the degree of variation in reflectance spectra that can be produced from the analysis of fibres of the same type. It is, however, possible that this variation was influenced by the heterogeneous shape of unmercerised cotton, which will likely lead to a degree of variation in different reflectance FTIR spectra generated from multiple points of the same cotton fibre.

4.2. Visual categorisation of known and environmental textile fibres

The approach applied in the present study cannot conclusively identify the origin of the unknown textile fibres. However, it successfully placed the abundance of extruded textile fibres in the context of environmental textile fibre populations. Categorising textile fibres as natural or extruded affords researchers greater consideration of textile fibres within the study of microplastic pollution in the absence of suitable analytical techniques. Furthermore, where suitable analytical techniques are available, this approach reduces the sample size of textile fibres in need of chemical analysis from the total fibre population to extruded fibres only. Where available, a greater proportion, and therefore more representative subsample, of potentially synthetic textile fibres can then be chemically analysed by means such as FTIR spectroscopy.

4.3. Textile fibre populations in freshwater environments and the atmosphere

The visual characterisation of textile fibres proved to be an effective technique in the analysis of environmental textile populations. Whilst the process outlined in Figure 2 clearly details the steps taken to analyse environmental textile fibres, the authors must stress the importance of fibre analysts developing their proficiency using known textile fibres prior to applying this technique to environmental samples.

The mean total textile fibre concentration observed across the freshwater sites sampled consistently exceeded that reported by Dris et al. (2018) in the Marne River, Paris, who recorded a maximum mean fibre concentration of 0.1 fibres L^{-1}. A number of factors can explain this finding, including the finer mesh size and higher microscope magnification used in the present study and the dilution effect of the much greater volume of water flowing through the Marne River compared to the rivers sampled here.

The entrainment and transport of textile fibres in aquatic environments and the atmosphere might be expected to be influenced by their physical properties – including fibre morphology and density – as well as environmental conditions including rate of flow in rivers, precipitation and wind speed. Determining the extent to which this is the case is beyond the scope of the present study, and so it is not possible to comment on whether or not these factors will influence the environmental prevalence of fibres of different types in highly mobile fluid matrices. However, in relatively motionless fluid environments such as the settling tanks of WWTPs, common natural and extruded textile fibres, that are all denser than water (Table 3), might be expected to settle. However, the emission of microplastic particles, and in particular synthetic textile fibres, from WWTPs has been quantified and is known to vary; across seven WWTPs, Leslie et al. (2017) report microplastic concentrations in WWTP effluent to range from 9-91 particles L^{-1}.

Table 3: Density of common textile fibres, as reported by Morton and Hearle (2008)

Fibre	**Density (g/cm^3)**
Cotton	1.55
Wool	1.30
Silk	1.34
Viscose Rayon	1.52
Polyester	1.39
Nylon 66, nylon 6	1.14
Acrylic	1.19

The sampling of freshwater environments was conducted at three sites that were in receipt of the effluent of wastewater treatment plants (WWTPs) (sites RS2-4) and seven that were not. There was no appreciable increase in textile fibre concentration at sites in receipt of WWTP effluent. This was true even of River Soar site 3, located approximately just 1.7 km downstream of the outflow of a WWTP serving a population equivalent of 72,500. However, whilst the concentration did not increase, the greater flow rates of sites RS2-RS4 will have increased textile fibre abundance. Instead, the highest cumulative abundance of natural and extruded textile fibres on each of the rivers sampled was recorded at sites immediately downstream of urban population centres (Sites RT3 and RL3).

Textile fibres were even observed at the most upstream sites on each of the rivers sampled. Though close to the sources of these rivers, sites RT1 and RL1 are popular recreational sites, whilst site RS1 neighbours a busy haulage yard. The observation of textile fibres at these sites demonstrates the role of localised anthropogenic activity on textile fibre abundance in the freshwater system. The prevalence of textile fibres in atmospheric fallout in both urban and rural sites highlights the role of atmospheric deposition in the transport of textile fibres throughout the environment, including to relatively remote locations. It also raises questions regarding the role of wastewater treatment plants as sources of synthetic textile fibres in aquatic environments.

Wastewater treatment plants are partially open systems, with various stages of the wastewater treatment process exposed to the atmospheric deposition that has been recorded here and elsewhere (Cai et al. 2017; Dris et al. 2016; 2017). The extent to which this deposition contributes to the textile fibre concentrations of final effluent is yet to be quantified.

Though atmospheric deposition of fibres was comparable at sites B-D, the abundance of fibres observed at site A was noted to be much more variable (Figures 6 and 9) (Levene's test p-value <0.001), despite its close proximity to sites B and C (Figure 1). Where previous records of atmospheric textile fibre deposition have extrapolated over large geographical areas from as few as two sample locations (e.g. Dris et al. 2016), the localised variation quantified here between sites that are fewer than 800 m apart, indicates that such extrapolations are likely to be inappropriate.

The recurrent observation of textile fibres at freshwater sites irrespective of rainfall prior to, or during, sample collection also suggests an atmospheric contribution of textile fibres to the freshwater system independent of precipitation and surface run off. Moreover, previous studies have assumed all, or at least the majority, of the fibres present in atmospheric samples were deposited during the rainfall event that prompted the sample collection (e.g. Dris et al. 2016). Here, the identification of textile fibres in atmospheric fallout during periods of no to low precipitation, where shows that such an assumption cannot be relied upon.

Though extruded textile fibres, a proportion of which may be microplastic, were present in both freshwater and atmospheric samples throughout this sampling campaign, the consistent dominance of natural textile fibres over extruded textile fibres provides strong support for the concerns raised by Ladewig et al (2015) and Zhao et al (2016).

4.4. Freshwater fluxes of textile fibres and difficulties in extrapolating fluxes

The data presented in Table 2 goes some way to highlighting the potential fibre flux of even these smaller freshwater systems. However, the temporal nature of this study has also enabled consideration of the extent to which such extrapolations can vary. The range of fibre fluxes presented in Table 2 illustrates the importance of sample replication in order to account for seasonal variation and the influence of abnormal weather conditions. These extrapolations suggest approximate daily textile fibre fluxes of 19 500 000 and 197 000 000 from the Rivers Leen and Soar respectively, as well as an approximate daily textile fibre flux of 7 810 000 into the Stoke-on-Trent urban area. Despite flux extrapolations being frequently presented in microplastic literature, the large range in values recorded both between sites in a similar geographical area, and at individual sites through time, suggests that little confidence can be given to these values.

5. Conclusion

The findings of the present study show that natural textile fibres constitute a significantly greater proportion of environmental textile fibre populations than extruded textile fibres in all three of the sampled rivers, as well as at all 4 sites of atmospheric deposition. It demonstrates a considerable limitation of the use of FTIR spectroscopy for the analysis of textile fibres in the study of microplastic pollution, and details the subtle differences between natural and extruded textile fibres, advancing the visual characterisation of particles that is still applied to the majority of microplastic pollution studies. Finally, textile fibre concentrations were found to vary greatly through both space and time. This has important implications for the legitimacy of previously extrapolated particle fluxes within the broader study of microplastic pollution, which do not sufficiently account for temporal and spatial variability.

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Supplementary Material

Table S1: Natural and extruded textile fibre concentrations at each freshwater sampling site for each of the 13 sample occasions

Sample occasion	Site	Natural textile fibres / litre	Extruded textile fibres / litre
1	RT1	0.53	0.03
	RT2	0.43	0.10
	RT3	0.20	0.00
	RL1	0.20	0.00
	RL2	0.37	0.03
	RL3	0.17	0.00
	RS1	0.10	0.00
	RS2	0.17	0.00
	RS3	0.00	0.00
	RS4	0.20	0.00
2	RT1	0.37	0.00
	RT2	0.53	0.00
	RT3	0.57	0.03
	RL1	0.03	0.00
	RL2	0.40	0.00
	RL3	0.17	0.03
	RS1	0.30	0.00
	RS2	0.30	0.03
	RS3	0.30	0.03
	RS4	0.30	0.00
3	RT1	Sample lost	
	RT2	Sample lost	
	RT3	Sample lost	
	RL1	Sample lost	
	RL2	0.67	0.03
	RL3	Sample lost	
	RS1	0.07	0.00
	RS2	0.47	0.00
	RS3	Sample lost	
	RS4	0.23	0.00
4	RT1	0.17	0.00
	RT2	0.13	0.00
	RT3	0.57	0.07
	RL1	0.00	0.03
	RL2	0.33	0.03
	RL3	0.73	0.07
	RS1	0.27	0.00
	RS2	0.20	0.00
	RS3	0.40	0.00
	RS4	0.30	0.00
5	RT1	0.33	0.00
	RT2	0.13	0.00
	RT3	1.07	0.00
	RL1	0.13	0.00
	RL2	0.33	0.00
	RL3	1.40	0.00
	RS1	0.27	0.00
	RS2	0.93	0.00
	RS3	0.33	0.00
	RS4	0.60	0.00
6	RT1	0.10	0.00
	RT2	0.03	0.00
	RT3	0.17	0.03
	RL1	0.13	0.03
	RL2	0.27	0.07
	RL3	0.33	0.03
	RS1	0.50	0.00
	RS2	0.13	0.00
	RS3	0.40	0.00
	RS4	0.23	0.00
7	RT1	0.10	0.00
	RT2	0.00	0.00
	RT3	0.10	0.00
	RL1	0.20	0.00
	RL2	0.03	0.03
	RL3	0.10	0.00
	RS1	0.00	0.00
	RS2	0.10	0.00
	RS3	0.10	0.03
	RS4	0.13	0.00
8	RT1	0.00	0.03
	RT2	0.00	0.00
	RT3	0.00	0.07
	RL1	0.03	0.03
	RL2	0.03	0.03
	RL3	0.13	0.13
	RS1	0.07	0.03
	RS2	0.03	0.03
	RS3	0.00	0.00
	RS4	0.00	0.00
9	RT1	0.00	0.00
	RT2	0.10	0.00
	RT3	0.03	0.00
	RL1	0.00	0.03
	RL2	0.00	0.00
	RL3	0.03	0.00
	RS1	0.03	0.00
	RS2	0.00	0.10
	RS3	0.03	0.00
	RS4	0.00	0.00
10	RT1	0.00	0.00
	RT2	0.00	0.03
	RT3	0.00	0.00
	RL1	0.00	0.03
	RL2	0.03	0.03
	RL3	0.07	0.03
	RS1	0.03	0.00
	RS2	0.00	0.00
	RS3	0.00	0.03
	RS4	0.07	0.03
11	RT1	0.00	0.00
	RT2	0.00	0.00
	RT3	0.10	0.00
	RL1	0.10	0.00
	RL2	0.07	0.00
	RL3	0.07	0.03
	RS1	0.10	0.03
	RS2	0.07	0.03
	RS3	0.03	0.03
	RS4	0.03	0.03
12	RT1	0.00	0.00
	RT2	0.10	0.00
	RT3	0.03	0.17
	RL1	0.03	0.03
	RL2	0.00	0.07
	RL3	Sample lost	
	RS1	0.03	0.00
	RS2	0.30	0.07
	RS3	0.03	0.03
	RS4	0.07	0.17
13	RT1	0.03	0.00
	RT2	0.03	0.10
	RT3	0.03	0.07
	RL1	0.00	0.03
	RL2	0.20	0.07
	RL3	0.07	0.03
	RS1	0.10	0.03
	RS2	0.00	0.07
	RS3	0.03	0.03
	RS4	0.03	0.10

Table S2: Natural and extruded textile fibre deposition at each atmospheric sampling site for each of the 26 sample occasions

Sample occasion	Site	Natural textile fibres (fibres $m^2 day^{-1}$)	Extruded textile fibres (fibres $m^2 day^{-1}$)
1	A	75.79	6.32
	B	82.1	0
	C	151.58	12.63
	D	56.84	0
2	A	170.52	0
	B	56.84	0
	C	101.05	0
	D	132.63	0
3	A	284.21	0
	B	56.84	6.32
	C	75.79	0
	D	56.84	0
4	A	353.68	0
	B	120	6.32
	C	170.52	12.63
	D	107.37	0
5	A	227.36	0
	B	126.31	0
	C	157.89	0
	D	82.1	0
6	A	Sample lost	
	B	Sample lost	
	C	151.58	0
	D	44.21	0
7	A	Sample lost	
	B	208.42	6.32
	C	56.84	0
	D	151.58	0
8	A	334.73	0
	B	132.63	0
	C	151.58	0
	D	88.42	0
9	A	258.94	0
	B	69.47	0
	C	56.84	0
	D	25.26	0
10	A	214.73	6.32
	B	101.05	0
	C	Sample lost	
	D	44.21	0
11	A	164.21	0
	B	56.84	0
	C	75.79	0
	D	44.21	0
12	A	82.1	0
	B	25.26	0
	C	101.05	0
	D	18.95	0
13	A	Sample lost	
	B	44.21	0
	C	18.95	0
	D	31.58	0
14	A	88.42	0
	B	0	0
	C	25.26	6.32
	D	0	0
15	A	44.21	0
	B	12.63	0
	C	18.95	0
	D	12.63	0
16	A	25.26	0
	B	18.95	0
	C	6.32	0
	D	0	0
17	A	0	0
	B	25.26	0
	C	0	0
	D	0	0
18	A	12.63	0
	B	6.32	0
	C	0	0
	D	0	0
19	A	25.26	0
	B	18.95	6.32
	C	12.63	0
	D	0	0
20	A	Sample lost	
	B	Sample lost	
	C	Sample lost	
	D	Sample lost	
21	A	Sample lost	
	B	164.21	31.58
	C	Sample lost	
	D	31.58	6.32
22	A	88.42	0
	B	31.58	0
	C	25.26	0
	D	6.32	6.32
23	A	44.21	18.95
	B	37.89	0
	C	37.89	0
	D	25.26	0
24	A	63.16	0
	B	56.84	0
	C	31.58	0
	D	37.89	0
25	A	69.47	6.32
	B	82.1	0
	C	18.95	0
	D	12.63	0
26	A	69.47	0
	B	25.26	12.63
	C	44.21	6.32
	D	31.58	0

Table S3: Textile fibres deposited during the processing of Freshwater samples

Sample occasion	Natural fibres	Extruded fibres
1	0	0
2	0	0
3	0	0
4	0	0
5	1	0
6	3	0
7	0	0
8	1	0
9	1	0
10	0	0
11	1	0
12	0	0
13	0	0

Table S4: Textile fibres deposited during the processing of Atmospheric samples

Sample occasion	Natural fibres	Extruded fibres
1	5	0
2	3	0
3	0	0
4	0	0
5	1	0
6	0	0
7	2	0
8	1	0
9	1	0
10	0	0
11	0	0
12	2	0
13	0	0
14	0	0
15	0	0
16	0	0
17	0	0
18	0	0
19	0	0
20	No samples	
21	0	0
22	0	0
23	1	0
24	1	0
25	0	0
26	0	0

Table S5: Abundance of textile fibres across the 5 procedural blanks

	Natural fibres	Extruded fibres
Freshwater 1	1	0
Freshwater 2	0	0
Atmospheric 1	2	0
Atmospheric 2	0	0
Atmospheric 3	0	0

Table S6: Details of each of the UK National River Flow Archive gauging stations used to estimate microplastic fluxes.

Gauged River	**Nearest Sampling site**	**NRFA Gauging station name**	**Length of operation**
River Trent	RT2	Trent at Stoke-On-Trent	01/1968-present
River Leen	RL3	Leen at Triumph Road Nottingham	01/1968-present
River Soar	RS4	Soar at Kegworth	12/1978-present