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The application of tape lifting for microplastic pollution monitoring

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ABSTRACT

Microplastics (MPs) are man-made polymer particles in the size range 1 μm to 5 mm. They have been proven to be present in all of Earth's environments through extensive global studies. Such studies regularly involve the isolation of MPs from water or other media using a filtration method. MPs are then commonly analysed for size and polymer type, either in situ on the filter or after removal from it by hand picking. These approaches provide the opportunity for the accidental loss of such particles and do nothing to protect the sample from contamination, whilst hand-picking from filter papers is also time consuming. The analysis frequently focusses solely on one technique and rarely facilitates the full characterisation of the MPs.

This paper sets out a workflow that addresses these shortcomings. Tape lifting (a forensic approach to particulate recovery) is at the heart of this workflow. This technique uses self-adhesive tape to recover particles of interest and results in a tape lift in which those particles are held between the tape and a sheet of suitable material. In the proposed workflow, the tape is Easylift® and the sheet is glass. Tape lifting offers significant time saving in the field, allowing more samples to be taken. It also creates a secure environment for the particles of interest and facilitates reproducible research by preserving samples for future study.

To investigate the recovery rate of MPs from filter papers using Easylift®, a simulation experiment was conducted, which tested glass fibre and cellulose fibre filter papers and ceramic and glass-frit Büchner funnels. It found that the rate of recovery of MPs from filters onto the tape had a mean of 96.4% ($s_{n-1} = 3.5$ percentage points, $n = 12$) with evidence that both filter type and funnel type effect that rate and that there is an interaction effect between these factors. In addition, the recovery rate from water onto the filter papers was investigated; this had a mean of 92.1% ($s_{n-1} = 4.1$ percentage points, $n = 12$) with no evidence that the filter type or funnel type used influenced that rate.

This paper also explores Easylift®'s attributes that facilitate the proposed workflow by enabling analysis of MPs whilst they are held within the tape lift. Easylift® is compatible with a wide range of non-destructive analytical techniques including polarized light microscopy (PLM), confocal Raman spectroscopy, fluorescence microscopy, microspectrophotometry (MSP) and hyperspectral microscopy, and this compatibility is explored in this paper. The compatibility with these techniques allows samples to be fully characterised for their morphological, optical and chemical properties, providing further information about the samples that can aid future studies that investigate source identification and the detection of MP features that may affect ecotoxicological effects.

1. Introduction

Microplastics (MPs), defined as "any synthetic solid particle or polymeric matrix, with regular or irregular shape and with size ranging from 1 μm to 5 mm, of either primary or secondary manufacturing origin, which are insoluble in water" by Frias and Nash (2019) are recognised as a global pollutant. Microplastics are regularly categorised by their form in studies; this commonly includes, pellets, fragments, and fibres, as well as sometimes films, filaments, sponges, foams and microbeads also being reported (Frias and Nash, 2019).

It is now clear that microplastic pollution is widespread (Eriksen et al., 2014) and has been found in many natural and man-made environments, including the Arctic (Bergmann et al., 2019, Peeken et al., 2018), the Alps (Bergmann et al., 2019), the Amazon river (De Souza e Silva Pegada et al., 2018) and even in commercially-produced bottles of drinking water (Mason, Welch & Neratko, 2018). It is also

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acknowledged that microplastics are present in all Earth's systems, including the hydrosphere (Zhang, Z., Mamat, Z., Chen, Y., 2020), atmosphere (Dris et al., 2016), lithosphere (Rillig and Lehman.,2020; Koutnik et al., 2021) and biosphere (Zantis et al., 2021).

A significant number of the MP studies conducted thus far have involved the isolation of MPs from water in which they are suspended. In many cases, this is because the MPs of interest have been found in natural waterbodies such as rivers or seas. There are several methods available for extracting MPs from such waterbodies, the choice of which is dependent on the focus of the study in question (Fu et al., 2020). For instance, in studies of MP pollution when large volumes of surface water are being sampled, nets are used. A neuston net, as used by Law et al (2014), may be employed as may similar nets such as plankton or Manta trawls (Bergmann et al., 2015). Alternatively, to capture all MPs and not filter by net mesh size, a grab sample, where a sample of water is taken and then filtered either in the field or a laboratory setting, is preferable (Miller et al., 2017).

The majority of microplastic research papers that report the taking of grab samples also report the use of vacuum filtration to recover microplastics from water samples in the laboratory (Di & Wang, 2018; Nel, Dalu & Wasserman, 2018; Murphy et al., 2016; Prata et al, 2019). The main aim of such filtration is to separate microplastics from the sample matrix to simplify the subsequent analysis (Xu et al., 2019). There is currently no accepted standardised method for doing this. Also, in any one study, the filtration system used (filter type, funnel type etc) may not have been optimised to maximise the capture of microplastics present in the sample. Furthermore, most papers do not provide exact details of the filtration method used, although, for example, they may state that a Büchner funnel (Barrows et al., 2017) or a glass frit (Wolff et al., 2019) was employed. It is, however, clear that several different types of filter have been used in studies of microplastic pollution, including mixed cellulose ester membrane filters (Stanton et al., 2019), glass fibre filter papers (Lahens et al., 2018) and cellulose fibre filter papers (Cordova, Hadi & Prayudhu, 2018); for a more extensive list, please see Table A.1 in the Appendices. In addition to water samples, filter papers are also used in air sampling for MPs when utilising an air pump (Prata et al 2020). To the best of our knowledge, there have been no studies conducted to evaluate the effect of filter funnel design and filter type on the proportion of microplastics present in the water that are isolated by the filtration process.

After filtration, it is common practice to individually hand pick the MP particles from the filter using tweezers (for example, see Kutralam-Munissamy et al., 2020; Saeed et al., 2020; Amin et al., 2020; Qiu et al., 2016; Qiu et al., 2015; Woodall et al., 2015). This is time consuming, provides the opportunity for the accidental loss of such particles and does nothing to protect the sample from contamination by, for example, airborne MPs. The recovery of particulates from surfaces using a quick and effective method that reduces the opportunity for loss and contamination is a well-established process in forensic science, specifically in forensic fibre examination. The method of choice for recovering particulates is tape-lifting (Pounds, 1975; Schotman and van der Weerd, 2015; Robertson and Roux, 2018). Tape lifting involves the application of transparent, colourless self-adhesive plastic film (the tape) to the surface to be sampled. The tape is then removed from the surface and it, plus any trace particulates that are adhered to it, is then secured to a suitable backing material. That material is commonly an acetate sheet. The combination of the tape, its backing and the trace particulates held between them is known as a tape lift. (Jackson and Jackson, 2017; Jones, Gwinnett and Jackson, 2018; Robertson and Roux, 2018). Tape lifts are subsequently then searched by eye using a low-power stereo microscope to locate any particulates of interest, such as fibres. These particulates are labelled by circling around them using indelible pen so that they can be returned to after screening (Schotman and van der Weerd, 2015). The next stage is to thoroughly compare and characterise these fibres in order to classify all of the fibres according to their colour, shape, dimensions and what they are made of. For this analysis to occur, fibres normally need to be dissected from the tape lift as both the tape and backing are composed of materials that interfere with analysis of the optical and chemical properties of the samples. This has now been mostly overcome with the development of the Easylift® tape lifting system by two of the authors of this paper (CG and AJ) (Jackson and Gwinnett, 2013; Jackson and Gwinnett, 2014; Jackson and Gwinnett, 2015; Jackson and Gwinnett, 2017). The characteristics of that system are such that *in situ* analysis of fibres using polarised light microscopy (PLM), fluorescence microscopy, confocal Raman spectroscopy and microspectrophotometry (MSP) can occur without the need for dissection (Jackson and Gwinnett, 2013). Easylift® was first developed for the recovery and examination of fibres for the forensic industry and as such has not previously been tested for recovering fibres from filter papers. There are many reasons why tape lifting generally is the method of choice in forensic science. These include its speed and convenience, its cost-effectiveness and the fact tape lifts provide an environment that is resistant to the contamination and loss of trace particulates (Keutenius, O'Keefe and Allen, 2013). Furthermore, tape lifts can be kept for protracted periods of time allowing easy transportation, storage and later analysis. Tape-lifting with Easylift® has the added advantages over standard tape lifting of allowing *in situ* analysis of fibres and other particulates which further reduces the risk of contamination and loss and speeds up sample preparation. The authors believe that tape lifting with Easylift® could offer similar benefits to the field of MP recovery.

In addition to the potential benefits to the recovery of MPs from filter papers that tape lifting may have over standard hand-picking, there are possible improvements to the analysis workflow of MPs that can be taken from forensic fibre examinations using Easylift® tape. Microplastic pollutants may be classified by various properties, but currently the most popular is to identify size and polymer type (Bergmann et al., 2019). In addition to these properties, other features have also been utilised including surface area (Rivers, Gwinnett and Woodall, 2019), surface morphology (e.g. surface texture) and colour (Wang et al., 2020). Semi-automated approaches have been used including those linking Fourier Transform Infrared (FTIR) microscopy and image analysis (Primpke et al, 2017) and Raman micro-spectroscopy for both morphological and chemical characterisation (Frère et al, 2016). Although there is a steady increase in the range of the types of characteristics being quantified and observed in microplastic studies, there are no known current MP analysis workflows that fully characterise the morphological, optical and chemical properties of the MPs without the potential for loss or contamination when applying sequential techniques.

The techniques used in the forensic characterisation of fibres are many and various (Robertson, Roux and Wiggins, 2018). They include microspectrophotometry (MSP) (Palenik, Beckert and Palenik, 2016), infrared and Raman spectroscopy, fluorescence microscopy, and polarised light microscopy (PLM). The last of these has a number of valuable attributes, principal amongst which are that once a fibre is ready for inspection by this technique, it is fast, non-destructive and can be highly discriminating. To a significant degree, this discriminating power is borne of the fact that very nearly all fibres are birefringent. This is a property that very nearly all MPs, whether fibres or not, have too. Birefringence determination has been used to help identify polymer type in forensic analysis and the textile industry for decades (Sieminski, 1975; Johri and Jatar, 1979; Fong, 1982; Gorski and McCrone, 1998; Wilding, 2009). This is particularly useful for samples which are bio-fouled and/or very small that are difficult to identify using Fourier Transform Infrared (FTIR) spectroscopy. With reference to micro-Fourier Transformed Infrared (μ-FTIR) spectroscopy, it has been stated that the "current potential size limit for identification ranges between 20 and 100 μm" (Frias and Nash., 2019). Samples smaller than 20 μm are still able to be analysed and identified using PLM with a suitable objective lens. Currently, the use of PLM for characterising fibres from environmental samples is rare with the first use of this seen in the analysis of fibres found in deep sea sediment (Woodall et al., 2015).

In forensic examinations, fibres are subjected to a series of techniques to fully characterise the samples beyond just size and polymer type. An enhanced workflow analysing the breadth of characteristics of these polymers can allow, for example, the sub-classifications of MPs which share the same polymer type, but which have different morphological, optical and chemical properties. This more granular characterisation of MPs provides evidence that could further help understand factors that may contribute to certain ecotoxicological effects (Wright, Thompson and Galloway, 2013) and inform the inference of source.

This study investigates the use of a forensic tape, Easylift® for the retrieval of MPs from filter papers and suggests an improved workflow of MP analysis (summarised in Section 2.1), enabled by the chosen tape, that allows greater characterisation of these pollutants by facilitating a multi analysis approach. This paper provides an initial evaluation of the benefits and limitations of using this tape for MP work. In addition, this study evaluates the effect of filter funnel design and filter type on the proportion of microplastics that are isolated both from water by the filtration process and from filters by tape lifting.

The study achieves the above via:

1) A presentation of the findings of a simulation experiment conducted using Easylift® whose aims were to:
   a) establish the ability of:
      - Easylift® tape to recover MPs from damp filter papers;
      - vacuum filtration to recover MPs from water;
   b) study the effect of filter type and funnel type on the percentage recovery rate of target MP fibres from:
      - filters by tape lifting with Easylift®;
      - water by vacuum filtration.
2) A largely qualitative exploration of many of Easylift®'s key attributes which facilitates the use of multiple analytical techniques.

2. Materials and methods

2.1. The new workflow

A proposed workflow has been developed for the processing of particles of interest that have been recovered from water or air by filtration for the purposes of MP pollution monitoring. That workflow consists of seven Steps, occur across two Stages. The workflow is described in Table 1. Stage 1 (Steps 1-4) outlines the recovery of microplastics from filter papers using Easylift® tape. Stage 2 (Steps 5-7) outlines the searching for and subsequent analysis of any MPs, the latter allowing the use of multiple analytical techniques. Steps 6 and 7 specifically facilitate the sequential analysis of MPs in order to fully characterise their morphological, optical and chemical properties; this is important for identification of the source of the MPs.

The four Steps that make up Stage 1 must be completed in quick succession with the minimum of delay. This is to minimise the opportunity for the contamination of the sample with airborne MPs and to avoid the filter drying out between Steps 1 and 2. However, after Stage 1 has been completed, the resultant tape lift may be stored for as long as needs be in a cool, dry, dark place such as a laboratory cupboard. It will therefore be common practice amongst those using this workflow for Stage 1 to be completed in the field and for Stage 2 to be undertaken at a later date in the laboratory.

Those wishing to adopt the proposed workflow may need to adapt it to their own needs. For example, in a given study, it may be known that, for operational reasons, there will be unavoidable but nonetheless undesirable delays during the completion of Stage 1. The negative impact of such delays can be mitigated by the use of suitable covers and/or containers in addition to those indicated in Table 1.

During the development of the workflow described in Table 1, it was necessary to establish the ability of Easylift® to recover MPs from damp filter papers as this is crucial to its overall success. We therefore conducted the simulation experiment described in Section 2.2. As detailed in Sections 3.1.3 and 3.1.4, this experiment has also allowed us to explore the effect of filter type and funnel type on that ability and on the efficacy of the filtration process itself. The compatibility of Easylift® with a wide range of non-destructive techniques had already been established before we started work to develop this workflow (Jackson and Gwinnett, 2013). However, we have since expanded that work, with the results given in Section 3.2.

In addition, as part of an expedition in 2019 to map the MP pollution of the Hudson River in the USA ('*Mountains to Sea, Sky to Seafloor,*

Table 1
The proposed workflow.

Stage	Step	Procedure
1	1	Immediately after filtration[1] has been completed, the filter paper is removed from its funnel or holder and is placed onto a clean ceramic plate. If the sample has been extracted from air, a few drops of distilled water are placed onto that plate. This is done immediately before the paper is placed onto that plate and the paper is then placed onto those drops[2]. Without delay, to minimise the possibility of contamination[3] with airborne MPs, the paper is covered with a suitable object, such as a clean, glass Petri dish lid.
	2	Without delay, the backing paper is removed from a new piece of Easylift® tape (Figure A.1)[4] and the adhesive surface of that tape is then gently contacted with the inside of the filter funnel/holder in the region where the filter paper's edges had previously been located[5]. Immediately, the funnel/filter holder is covered with a suitable, clean object. The cover is removed from the ceramic plate referred to in Step 1. The adhesive side of that same piece of Easylift® is then immediately brought into repeated contact with the filter on that plate whilst the filter is damp[2]. This is done such that the whole surface of that filter on which MPs may reside is tape lifted (Figure A.2)[4]. For samples that contain substantial amounts of debris, the taping of the filter is repeated twice using the same tape.
	3	The Easylift® tape used in Step 2 is then adhered to a clean, glass microscope slide without delay, the tape being held in place by its adhesive. This makes a tape lift, which is then labelled with a unique reference using an indelible marker on one of the Easylift® tape's two blue handles (Figure A.1)[4].
	4	The filter paper and the interior of the funnel/holder from which it has been taken is then immediately and carefully examined using a magnifying lens. Any particles of interest seen are removed using tweezers. A corner of the Easylift® tape of the above-mentioned tape lift is then peeled from its microscope slide and any such particles are sandwiched between that portion of the tape and its slide.
2	5	The tape lift from Stage 1 is examined using a stereomicroscope and circles are drawn on the tape around any particles of interest. These circles are numbered to allow each such particle to be uniquely identified (Figure A.1)[4].
	6	The particles of interest are then characterised *in situ* in the tape lift using methods such as polarised light microscopy, confocal Raman spectroscopy, microspectrophotometry, hyperspectral microscopy and/or fluorescence microscopy. This allows the classification of these particles, which are quantified by counting.[6]
	7	If wished, particles of interest are then removed from the tape lift by dissection (Figure A.3)[4], allowing further testing using techniques, such as Fourier Transform Infrared spectroscopy, that are incompatible with the presence of tape.

[1] The use of cellulose filters is not recommended for studies interested in the presence or prevalence of anthropogenic cotton as cellulose filter fibres are similar in appearance to cotton fibres.

[2] The filter should be damp (not wet) with water when it is contacted with Easylift® in Step 2.

[3] For more on contamination control in microplastic pollution studies, see Woodall et al. (2015).

[4] The Figures referred to in this table are given in the Appendix A.

[5] The tape needs to be removed slowly and with care from smooth surfaces to avoid the tape tearing or adhering to itself.

[6] Easylift® has been specifically designed to allow a wide range of non-destructive techniques to be used for this process of characterisation and classification.

Research and Technology Expedition' with the Rozalia Project for a Clean Ocean (Rozalia Project, 2017), two of the authors (CG and AO) conducted extensive field trials of the workflow given in Table 1. During that expedition, 159 air samples and 224 water samples were collected along that river from the headwaters, Lake Tear of the Clouds (44.17°N, −73.96°W) to the Atlantic Ocean marked by Ambrose Light (40.74°N, −73.96°W); a total of 507 km (315 miles), with samples taken every 4.8 km (3 miles). These were collected using Stage 1 of the workflow set out in Table 1 and are currently being processed according to Stage 2 of that workflow. The intension is to publish that work once that processing has been completed. However, it is worth noting here that the work finished thus far has shown:

- the proposed workflow saves time in the field, therefore allowing more samples to be taken. To illustrate this, in the afore-mentioned 2019 expedition, a total of 383 samples were taken. This contrasts with the total of 142 samples (all of surface water) taken without the aid of the proposed workflow in an expedition in 2016. That earlier expedition also concerned MP pollution mapping (Miller et al., 2017). It was of the same duration as the 2019 expedition and was led by the same team along the same river;
- the proposed workflow works when monitoring either airborne or waterborne MP pollution;
- tape lifting is effective in the post-filtration recovery of particles of interest when organic matter/debris is present. Very few MPs (< 20 in total across all samples) were not recovered via the tape and had to be tweezered from the surface;
- tweezers can be used to recover any particles of interest that are not recovered by tape lifting and that this can be easily achieved in the field;
- the *in-situ* characterisation of MP particles is effective when organic matter/debris is present. Analysis was unhindered when using polarized light microscopy.

2.2. The simulation experiment

During the simulation experiment, as outlined below and detailed in Appendix B.1, target MP fibres were suspended in tap water, then separated from that liquid by Büchner filtration under vacuum and then recovered from the filter paper by tape lifting. The target MP fibres used were fluorescent polyester fibres from a high-visibility vest, the vacuum pump was a Vacuubrand® PC 3012 VARIO and the tape used was Easylift®. Easylift® tape, which is manufactured by Tecman Ltd, is available from Staffordshire University via the corresponding author and is shown in Figure A.1 in the Appendices. In the simulation experiment, for each piece of Easylift®, its backing paper was removed immediately prior to the tape's use. The target MP fibres used were chosen in part because they are readily seen by virtue of their visible fluorescence when viewed under the light from a hand-held LED torch (i.e. flashlight) that emits light at 395 nm (Vansky model). Illumination with such a torch was used in an otherwise darkened room whenever a count of target MP fibres was made.

This experiment has a balanced 2 × 2 factorial design. The independent variables (IVs) are filter type and the type of Büchner funnel used, each with two levels. There are two dependent variables (DVs) of interest. DV_1 is the rate at which tape lifting recovered the target MP fibres from the filter and DV_2 is the rate at which filtration recovered the target MP fibres from the water. Details of how these DVs were calculated are given below.

The two levels of the filter type are denoted Cellulose and Glass fibre, the former being Whatman number 3 cellulose filter papers (Whatman catalogue number 1003 070, pore size of 6 μm) and the latter Whatman glass fibre filters GF/A (Whatman catalogue number 1820 070, pore size of 0.7 μm), both 70 mm in diameter. The two levels of the funnel type are named Ceramic and Glass. The first of these was a ceramic funnel, available from Fisher Scientific (catalogue number 10771752), whilst the other was a glass frit, available from RESTEK (catalogue number KT953825-0000). The filter papers and funnels were chosen as they are commonly used in MP studies as seen in Table A.1 of the Appendices.

There were three repeat procedures for each of the four unique combinations of the levels of the IVs. For each such procedure, a known number (c_1) of between 121 and 394 (inclusive) of the target MP fibres were suspended in 10 L of tap water (this water was checked for the presence of any fluorescent fibres prior to adding the target fibres). That water was filtered under vacuum through a previously unused filter paper. Further tap water was used to wash the surfaces that had been in contact with the water in which the target fibres were suspended and the washings obtained were also passed through the filter. The number of target MP fibres then present on the filter (c_2) was noted. Using the method illustrated in Figure A.2 of Appendix A, the whole surface of the filter on which those target fibres resided was then tape lifted with a single, previously unused, piece of Easylift®. The number of these fibres retrieved by this means (c_3) was also recorded. Also, during this procedure, an accurate estimate of the mass of the water present in each filter at the point of tape lifting was determined. This was done so that this estimate could be included as a covariate during hypothesis testing. It was achieved using an A&D Company Ltd. HR-250A analytical balance. For further details of the experimental procedure described in this paragraph, please see Appendix B.1.

The target MP fibre count data allowed the percentage of such fibres present on the filter that were recovered on the tape [i.e. (c_3/c_2) x 100%] to be calculated for each repeat. This is DV_1. The raw data, means, adjusted means and confidence intervals shown in Part (a) of Fig. 1 were calculated from these percentages.

The percentage of the target MP fibres present in the water that were extracted by filtration prior to tape lifting [i.e. (c_2/c_1) x 100%] was also calculated. This is DV_2. The raw data, means and confidence intervals shown in Part (b) of Fig. 1 were calculated from these percentages.

A blank sample of 10 L of tap water was filtered employing the same procedure as above and using a Cellulose filter and the Ceramic funnel. This sample was found to contain one fibre that was indistinguishable from the target MP fibres. This was considered to be within the likely margin of error in the count data, whether c_1, c_2 or c_3, and so those data were not adjusted to allow for such contamination.

2.2.1. Statistical analysis

Analysis of the data from the simulation experiment was conducted via the three linear models, described below:

- Model 1: A balanced 2 × 2 factorial ANOVA with interaction in which the percentage of target MP fibres present on the filter that were recovered on the tape (i.e. DV_1) was the dependent variable, and the independent variables (IVs) were the filter type and funnel type.
- Model 2: An ANCOVA. The same as Model 1 but with the mass of the total water content of the filter at the point of tape lifting included as a covariate.
- Model 3: A balanced 2 × 2 factorial ANOVA with interaction in which IVs were as in Model 1 and the dependent variable was the percentage of the target MP fibres present in the water that were extracted onto the filter prior to tape lifting (i.e. DV_2).

For all of the tests carried out, a significance threshold of 0.05 (i.e. 95% confidence) was used.

For all three Models, the data were checked for deviation from the assumptions that underpin the veracity of the models concerned and no such deviation was found. As a follow up to Model 1, two sets of simple effects tests were carried out with Bonferroni adjustment to control the familywise error rate. One set tested the effect of funnel type at fixed levels of filter type, the other tested the effect of filter type at fixed levels of funnel type. Measures of effect size were calculated for the three Models and for the simple effects tests. For details of these assumption

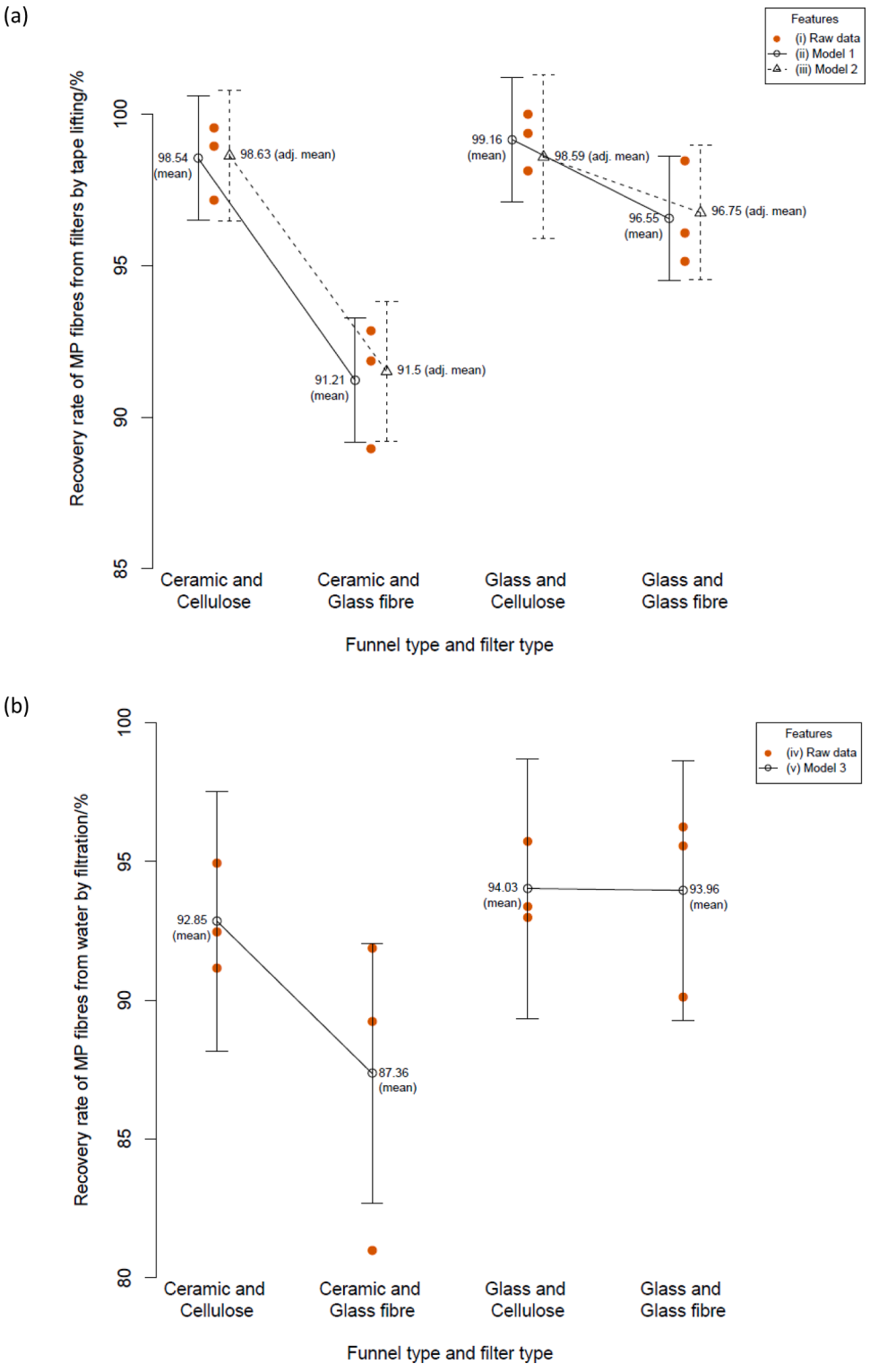

Fig. 1. The percentage rate at which the target MP fibres were recovered (a) from filters by tape lifting with Easylift® and (b) from water by filtration, each grouped by the unique combinations of funnel type and filter type. Features (i) and (iv) show those rates as found in the simulation experiment, these are the raw data. Features (ii) and (v) are respectively from Model 1 and Model 3. They each show the mean values of the relevant rate with 95% confidence intervals as revealed by ANOVA. Feature (iii) is from Model 2. It shows the same as (ii) but adjusted by ANCOVA to control for the effect of the total mass of water in the filter at the point of tape lifting.

deviation checks, simple effects tests, effect size measures and the software used for the statistical analysis, please see Appendix B.2. All of the raw data, the code that was used to analyse it and the output from that code have been published as a data set (Jackson et al., 2021).

2.3. Post-recovery characterisation exploration

For any given sample, the completion of Stage 1 of the workflow (Table 1) produces a tape lift. This tape lift contains the particles of interest sandwiched between the adhesive surface of a piece of Easylift® tape and a glass microscope slide. In Stage 2 of that workflow, this tape lift is searched with the aid of a microscope and any particles of interest are located, characterised, classified and quantified. All of which can be done without the removal of those particles from the tape lift, thereby reducing the opportunity for contamination and loss. This is possible because the optical properties of Easylift® make it compatible with a wide range of non-destructive analytical techniques.

In this part of the study, an exploration of Easylift®'s compatibility

with polarised light microscopy (PLM) (Section 3.2.1), fluorescence and hyperspectral microscopy (Section 3.2.3), confocal Raman spectroscopy (Section 3.2.2), and microspectrophotometry (MSP) (Section 3.2.3) was conducted. In addition, an investigation of MP analysis by FTIR spectroscopy after MP dissection from Easylift® tape was also conducted (Section 3.2.2).

The experimental details of this exploration are given in Appendix C.

3. Results and discussion

3.1. The simulation experiment

The aims of the simulation experiment were to establish both the ability of Easylift® tape and vacuum filtration to recover microfibres from filter papers and water, respectively. This includes an investigation into any effect of filter paper type and funnel type on the percentage recovery rate.

The results are summarised in Fig. 1 and are discussed below in the context of each of these aims in turn.

3.1.1. The ability of Easylift® tape to recover MPs from damp filter papers

In their 2015 paper, Schotman and van der Weerd report the percentage recovery of target fibres achieved by tape lifting a range of fabrics that had been seeded with those target fibres. They tested three target fibre types, three fabric types and eight tape types, resulting in 72 unique combinations of these factors. For each of those combinations, they determined the mean percentage recovery rate (n = 3) and found that all these means were in the range 76.6% to 99.4%, with an overall mean of 94.5%. As can be seen from Fig. 1, all bar one of the mean percentage recovery rates obtained by tape lifting in the simulation experiment reported here are above the overall mean recovery rate that they reported. Furthermore, the one remaining mean in the simulation experiment reported in Part (a) of Fig. 1 (i.e. that found when tape lifting glass fibre filters taken from the ceramic Büchner funnel) is substantially larger than the smallest mean found by Schotman and van der Weerd. Also, the overall mean rate of recovery of MPs from the filters onto the tapes seen in the simulation experiment was 96.4% (with $s_{n-1} = 3.5$ percentage points and n = 12). All this allows us to conclude that the ability of Easylift® to recover target MP fibres from the damp filters used in that simulation experiment are at least as good as might be expected.

Importantly, the very good recovery rates achieved by tape lifting in the simulation experiment led us to forecast that tape lifting with Easylift® would lead to high recovery rates of MP particles in the field. This gave us confidence that sufficiently few of such particles would be left behind by this process that they could be readily retrieved using tweezers. The field trial mentioned in Section 2.1 proved this to be the case. This was so irrespective of whether the samples were taken from the river or the air and irrespective of the presence of organic matter on the filter.

It should be noted that the use of tape to recover MPs from either of the Cellulose or Glass fibre filters used in this study also removes some of the filter's fibres onto the resultant tape lift. Differences in morphology and optical properties allow such fibres to be readily distinguished from MPs (see Appendix D for details). However, their presence is not desirable as it increases the sample processing time. Fortunately, as shown in Fig. 2, the addition of water to air-dry filter papers decreases the propensity of tape to retrieve filter fibres. However, we are also aware that this fact suggests that such addition of water has the potential to also suppress the ability of tape lifting to recover particles of interest from filter papers. In our experiment, the filter was damp at the point of tape lifting with an absolute water content ranging from 0.432 g to 0.790 g (with $m = 0.622$ g and $s_{n-1} = 0.117$ g). Model 1 tests the effect of filter type, funnel type and the interaction between them on the percentage recovery of target MP fibres from filters by tape lifting. The only difference between that Model and Model 2 is that the latter includes the above-mentioned absolute water content as a covariate. Surprisingly, when treated as a linear regression, Model 2 shows that, in our experiment, as that water content of the filters increased, so did the percentage recovery of target MP fibres from them onto the tape. The relevant slope is 4.545 percentage points per gram, showing that, in that experiment, this is a noticeable effect. However, that effect would not be large enough over the range of filter water content seen in our experiment to cause concern. More importantly, as detailed in Table A.2 of the Appendices, Model 2 did not find this effect to be significant ($F = 0.691$, $p = 0.433$) and so it may have occurred by chance. We therefore conclude that, within the range given above and with our experimental set up, our data does not support the hypothesis that change in that water content effects the rate at which tape lifting can recover MP particles from the filters used.

With the above findings in mind, the proposed new workflow (Table 1) includes the stipulation that the filter should be damp, but not wet, when it is tape lifted.

3.1.2. The ability of vacuum filtration to recover MPs from water

As exemplified by the papers listed in Table A.1 of the Appendices, studies aimed at monitoring MP pollution frequently employ a filtration step to recover the particles of interest. It is perhaps surprising that, to the best of the authors' knowledge, there are no prior publications that explore the efficiency of this recovery process. We have therefore included such work in the simulation experiment reported here.

The data collected during that experiment has allowed the calculation of the percentage of the target MP fibres present in the water that were extracted onto the filter prior to tape lifting. As illustrated in Part (b) of Fig. 1, these rates range from 81.0% to 96.2%. They have an overall mean of 92.1%, with $s_{n-1} = 4.1$ percentage points and n = 12. Other spiked studies investigating recovery rates of MPs report similar ranges to this study, for example, 92-99.6% when recovering MPs from soil using density flotation (Li et al, 2021) and 94-98% for sediment using a JAMSTEC microplastic sediment separator (JAMSS) unit (Nakajima et al, 2019).

It was noticed during the simulation experiment reported here that, after filtration, a few target MP fibres were found outside the filter's edge at the base of the wall of the funnel. These fibres were therefore not amongst those counted as being recovered on the filter, nor were they subsequently recovered onto the tape. These fibres give a partial explanation for the < 100% recovery rates shown in Fig. 1. In the proposed workflow (Table 1) this loss is mitigated by tape lifting the inside of the funnel as well as the filter.

3.1.3. The effect of filter type and funnel type on target MP recovery by tape lifting

As outlined in Section 3.1 both ANOVA (Model 1) and ANCOVA (Model 2) were used to test the effect of filter type and funnel type on the rate of target MP fibre recovery from filters achieved by tape lifting with Easylift®. As shown in Table A.2 of Appendix A, Models 1 and 2 both reveal that the main effect of each of the IVs (i.e. filter type and funnel type) is significant, as is the effect of the interaction between them (all the relevant p values are < 0.05).

This interaction effect, as revealed by these tests, is illustrated in Features (ii) and (iii) of Fig. 1. These, and Feature (i) of that Figure, all show that for each funnel type, changing the filter type from cellulose to glass fibre was typically accompanied by a decrease in the rate of target MP fibre recovery; however, this effect was much more profound when the ceramic funnel type was used. Also, when cellulose filters were used, this rate was essentially unaffected by funnel type. However, the plots suggest that this is not the case when glass fibre filters were used, for which the rate in question was noticeably reduced when switching from the glass funnel to the ceramic one. To test the significance of this interaction effect, simple effects analysis was carried out based on Model 1, the results from which are shown in Table A.3 of the Appendices.

As might be expected from the patterns seen in Feature (ii) of Figure 1, these tests revealed that tape lifting resulted in a statistically

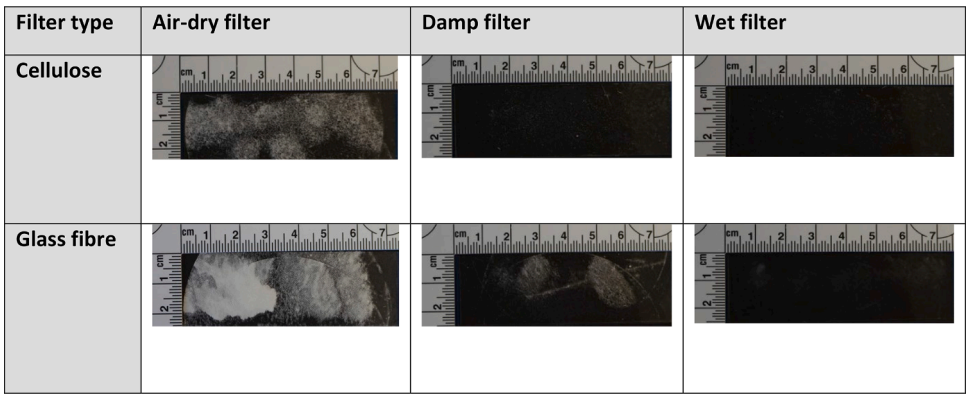

Fig. 2. Images of Easylift® tapes that had been used to tape lift clean filters of varying water content. Please see Appendix B.3 for details of how these images were created.

significantly higher mean target MP fibre recovery rate from the filters when used with the:

1 glass fibre filter and glass funnel combination ($m = 96.55\%$, $s_{n-1} = 1.71\%$ points) than when that filter type was used with the ceramic funnel ($m = 91.21\%$, $s_{n-1} = 2.03\%$ points);
2 ceramic funnel and cellulose filter combination ($m = 98.54\%$, $s_{n-1} = 1.25\%$ points) than when that funnel was used with the glass fibre filter type ($m = 91.21\%$, $s_{n-1} = 2.03\%$ points).

During the simulation experiment it was seen that the glass fibre filters were sufficiently pliable to form clearly visible dimples where the holes in the bed of ceramic funnel occurred. However, this was not the case for the cellulose filters. Furthermore, the MP fibres that resided in those dimples were more difficult to recover using the tape than those found elsewhere on the filter concerned. Also, the dimpling seen in the glass fibre filters when used in the ceramic funnel was not evident when they were used in the glass one. It seems likely that this is a consequence of the even support across its surface that is offered by the frit in the glass funnel. These observations would seem to explain the significant differences detailed above.

The existence of those differences serves to underline the importance of both:

- Step 4 of Stage 1 of the proposed workflow (Table 1) which, in our experience in the field, provides a quick, easy and effective mitigation of the risk of MP loss during that Stage and
- the advisability of the pre-use trialling and testing of the materials and methods to be used in any given field study to optimise the performance of each step of the workflow used.

Finally, it is perhaps worth noting that viewing Model 1 as a linear regression shows that its adjusted R^2 value is 0.808 (Jackson et al. 2021). This suggests that, at least with our experimental set up, approximately 81% of the variance present in the target MP fibre recovery rates achieved by tape lifting is controlled by the choice of filter type and funnel type.

3.1.4. *The effect of filter type and funnel type on target MP recovery by filtration*

The effect of each of filter type and funnel type on the percentage rate at which the target MP fibres were recovered from water by filtration is shown in Part (b) of Fig. 1 and was tested by ANOVA in Model 3. As suggested by that Figure, that test revealed no significant effects, whether main or interaction (see Table A.4 in the Appendices for details). This is not entirely surprising as:

- the target MP fibres were much larger than the pores in both types of filters and
- there was nothing intrinsic to the design of the two funnels that would suggest that one would better serve the extraction of MP particles from water than would the other.

However, it is perhaps noteworthy that neither either of the main effects nor their interaction had a power value of >0.5. From this it can be concluded that had the experiment been carried out with a larger sample size, the ANOVA *may* have detected one or more significant effects.

The limitations of the findings of the simulation experiment are explored in Appendix E.

3.2. *Post-recovery characterisation exploration*

3.2.1. *Polarised light microscopy*

Fig. 3 shows photomicrographs of a colourless nylon fibre as seen in transmitted light between crossed polars. This fibre's optical path difference (OPD) at any given thickness, its maximum thickness and its shape combine to give it multiple, vivid, interference colours[1] under these conditions. Furthermore, in that fibre, these colours make a clear pattern of bands. This makes it a good choice when trying to detect any changes made to these colours by the introduction of another material into the light path. As is evident from Fig. 3, no such changes are visible on the introduction of Easylift® into that path. Also, the background colour seen in Part (b) of Fig. 3 is uniformly black as far as the human eye can detect. Importantly, it remains so at all times when the slide is rotated through 360° about an axis that runs down the centre of the microscope's light path. This, coupled with the lack of difference between the two Parts of Fig. 3, demonstrates that Easylift® is essentially non-birefringent. This provides confidence that the accuracy with which the eye can be used to establish the birefringence and sign of elongation (SOE) of MP particles by PLM using a first-order red tint plate and/or quartz wedge is unaffected by Easylift® in the light path. Further information about birefringence and SOE can be found in Appendix C.1 and for potential limitations to the use of birefringence in MP pollution studies, please see item 3 of Appendix E. For an in-depth account of fibre characterisation by PLM, see Palenik (2018).

Many coloured birefringent specimens exhibit pleochroism, this is the differential absorption of light that vibrates in different planes and it has two variants, dichroism (as seen in pleochroic fibres) and trichroism. Pleochroism is illustrated in Fig. 4 which shows a dichroic fibre observed in plane polarised light. As that Figure shows, the colour change that

[1] Interference colours seen between crossed polars are used to calculate the birefringence of a given fibre, which is indicative of its polymer type.

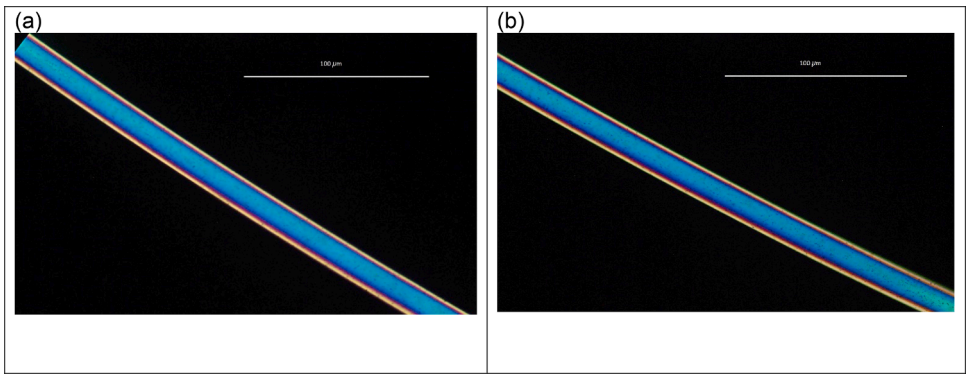

Fig. 3. Interference colours seen in a colourless (i.e. white) nylon fibre when viewed between crossed polars, both without (a) and with (b) Easylift® in the light path. As indicated, the scale bar is 100 μm long in each image. For detail on how these images were made, please see Section C.1 of Appendix C.

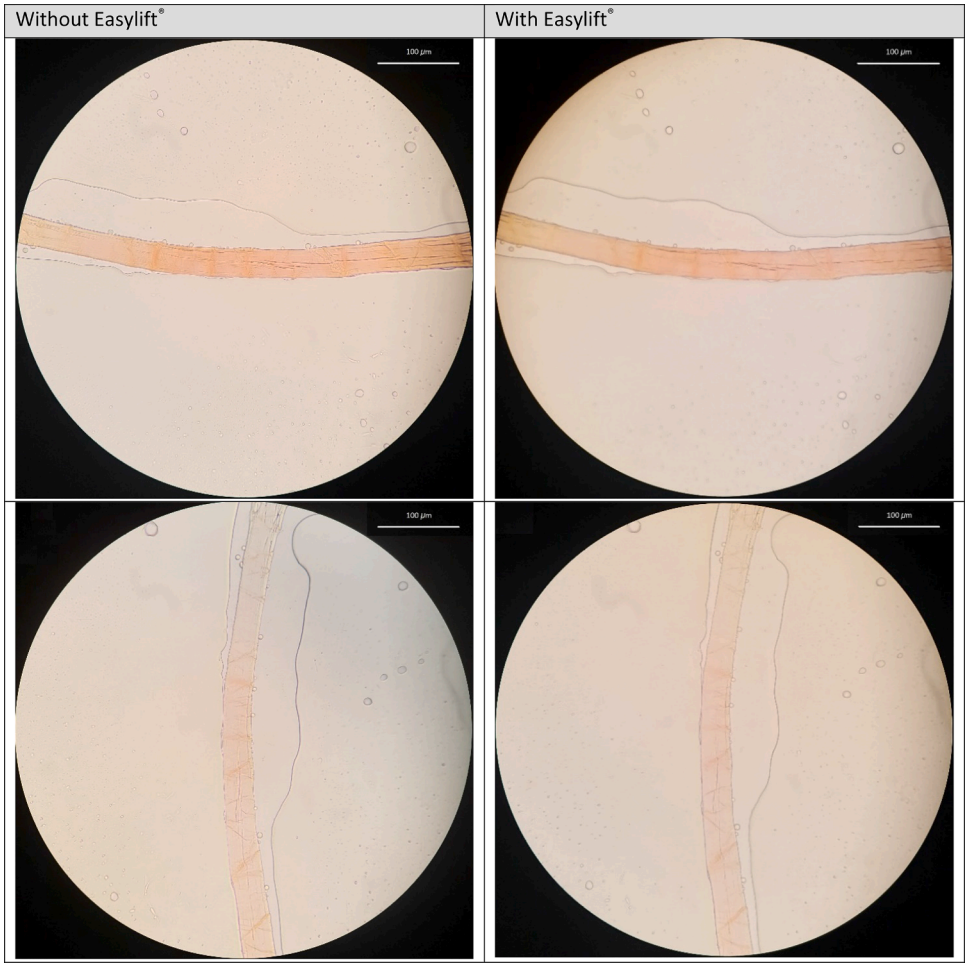

Fig. 4. Photomicrographs of a red fibre in transmitted plane-polarised light showing colour change due to dichroism on rotation about an axis down the centre of the microscope's light path. Note that the fibre is in an air bubble in the mountant. This is unintentional but does not detract from the effect being illustrated. The thin dark lines that can be seen either side of the fibre are the edges of that bubble. As indicated in the images, the scale bars are each 100 μm long. For details of the method used to create this Fig., please see Section C.1 of Appendix C.

occurs due to dichroism on the rotation of the fibre about an axis running down the centre of the microscope's light path is, as far as can be seen, unaltered by the presence of Easylift®.

3.2.2. *Vibrational spectroscopy*

3.2.2.1. Confocal Raman spectroscopy. Raman spectra have been used to differentiate between dyes in the forensic examination of fibres (Lepot et al, 2008) and to identify polymer type in microplastic studies (Araujo et al., 2018).

Fig. 5 shows four spectra obtained by confocal Raman microspectroscopy. Two of these are from a translucent, colourless polyolefin fibre held between Easylift® and a glass slide on the one hand and between that glass slide and a glass coverslip on the other. It also shows two blank spectra, each recoded in the absence of a fibre. One of these blanks was taken from a piece of Easylift® on a glass microscope slide, the other from a glass coverslip on such a slide. The salient peaks of all four spectra are summarised in Table 2.

Of the 14 peaks listed in Table 2, two (those at 1455 and 1738 cm^{-1}) are clearly present in the spectrum of Easylift® and one (the one at 1095 cm^{-1}) is in the spectrum of glass. The remaining 11 peaks can be unambiguously assigned only to the fibre, with seven of these clearly

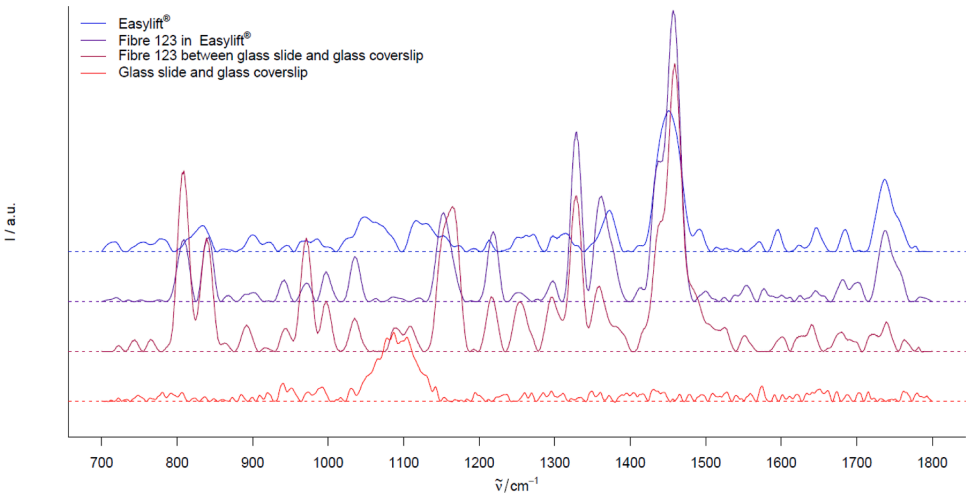

Fig. 5. Raman spectra. For details of the method used to create this Figure, please see Section C.2 of Appendix C.

Table 2
Salient peaks of the Raman spectra shown in Figure 5.

Peak position /cm^{-1} [(s) = sharp (b) =broad]	Easylift®	Fibre 123 in Easylift®	Fibre 123 between glass slide and coverslip	Glass slide and coverslip
808(s)	No	Yes	Yes	No
840(s)	No	Yes	Yes	No
971(s)	No	?	Yes	No
997(s)	No	Yes	Yes	No
1035(s)	No	Yes	Yes	No
~ 1095(b)	No	No	Yes	Yes
~ 1155(b)	No	Yes	Yes	No
1218(s)	No	Yes	Yes	No
1255(s)	?	?	Yes	No
1296(s)	?	?	Yes	No
1328(s)	No	Yes	Yes	No
1360(s)	No	Yes	Yes	No
~1455(b)	Yes	Yes	Yes	No
1738(b)	Yes	Yes	Yes	No

visible in both of the spectra from that particle. Thus, the results shown in Fig. 5 and Table 2 demonstrate that confocal Raman microspectroscopy can successfully obtain Raman spectra from plastic particles held *in situ* in Easylift® tape lifts.

3.2.2.2. Fourier transform infrared spectroscopy. Many MP pollution studies have used Fourier transform infrared (FTIR) spectroscopy for the purposes of polymer identification (e.g. Kedzierski et al., 2019; Lefebvre et al., 2019; González-Pleiter et al., 2020; Corami et al., 2020). All self-adhesive tapes, including Easylift®, have multiple strong absorption bands in the infrared and so *in situ* analysis of particles held on tape lifts by FTIR spectroscopy is not likely to be productive. However, the removal of particles from such lifts is possible by means of dissection. For details, please see Figure A.3 of the Appendices. As shown in that Figure, this process is straightforward with Easylift®. Also, as demonstrated by the spectra given in Fig. 6, such dissection can be used to remove a given particle of interest from an Easylift® tape lift for the purposes of FTIR spectroscopy. The only apparent interference from any remaining adhesive residue on the MP is a small peak at approximately 705 cm^{-1}; therefore, such dissection causes no issues in obtaining a useable spectrum.

3.2.3. Interaction with unpolarised ultraviolet and visible light

As shown in Fig. 7, Easylift® is essentially transparent to visible light (i.e. wavelengths = 400 to 700 nm) and shows transmission of >80% to all ultraviolet light in the wavelength range 300 to 400 nm. Consequently, as illustrated in Fig. 8, microspectrophotometry (MSP) can be used to characterise MP particles held under Easylift®.

As shown by the images given in Fig. 9, the transparency referred to above makes Easylift® compatible with fluorescence microscopy. Those images were captured using a LUMNIA-FLHS modular microscope by means of its hyperspectral camera, thus also illustrating the potential for microplastics held under Easylift® to be characterised using hyperspectral microscopy.

The limitations of Easylift®'s compatibility with the *in-situ* characterisation of MP particles held in tape lifts and our work reported here to

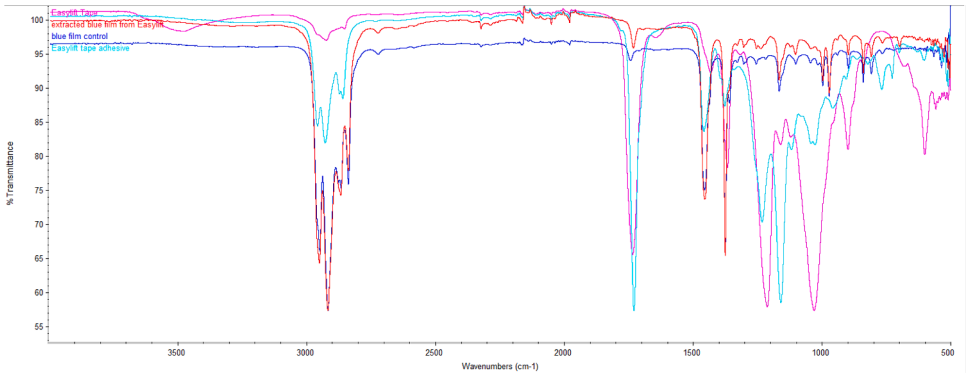

Fig. 6. FTIR spectra of the Easylift® tape (pink) and its adhesive (pale blue), plus those of a fragment of blue-coloured plastic film taken before it was tape lifted with Easylift® (dark blue) and after dissection from the lift so created (red). For details of methods used, please see Section C.3 of Appendix C.

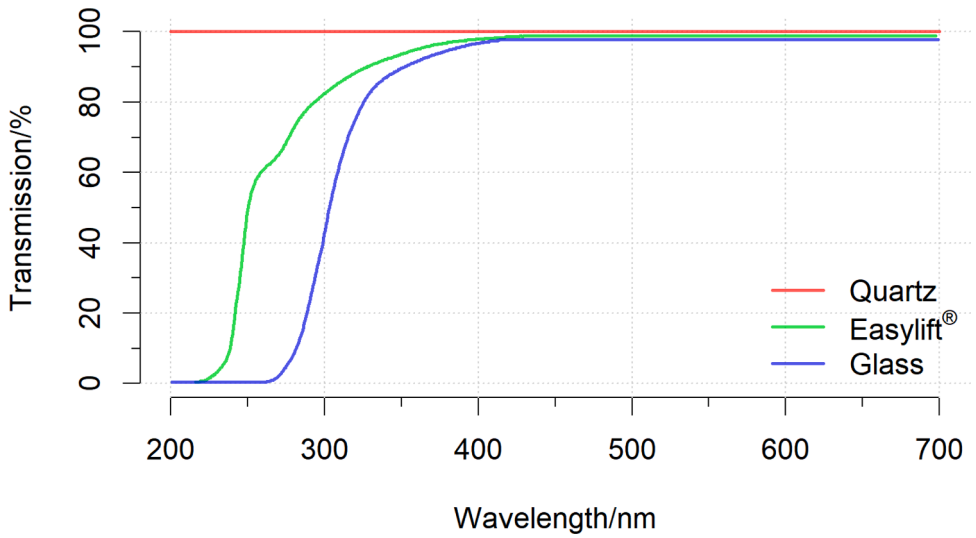

Fig. 7. Ultraviolet-visible transmission spectra (redrawn from spectra provided by Jaap van der Weerd and Linda Alewijnse of the Netherlands Forensic Institute). For methods used, please see Section C.4 of Appendix C.

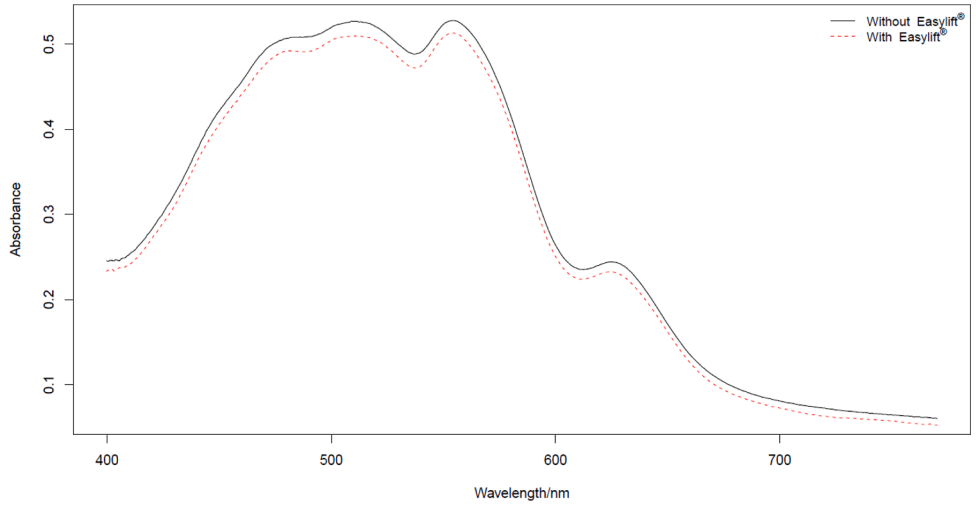

Fig. 8. Visible spectra obtained by MSP from a red nylon fibre. Smooth line = Without Easylift®, Dotted line = With Easylift®. The spectral data were recorded by Chris Hunter of SMCS Ltd. For methods used, please see Section C.4 of Appendix C.

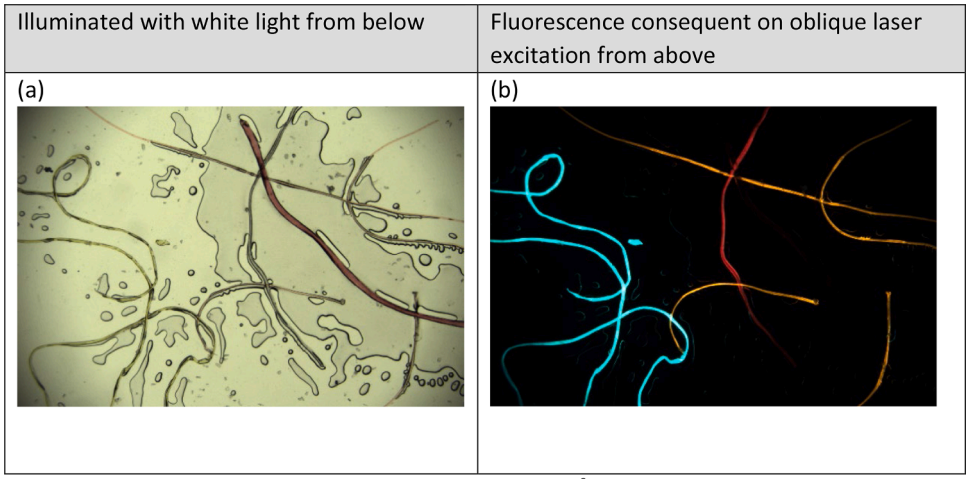

Fig. 9. Images of fibres demonstrating Easylift®'s compatibility with fluorescence microscopy and hyperspectral imaging. Images taken by Nathanail Kortsalioudakis, courtesy of Costas Ballas and Nathanail Kortsalioudakis of SPECTRICON. For methods used, please see Section C.4 of Appendix C.

examine that compatibility are further explored in Appendix E.

4. Future prospects

The suggested workflow in this study focusses on the use of Easylift® for the recovery of MPs from filter papers, mostly seen in water and air sample analysis, yet this approach may be applied to other sample types. The use of Easylift® for recovering MPs from other samples such as soil and sediment has yet to be tested but it is believed that after appropriate digestion and filtration steps, that the tape could be employed in a similar manner to water and air samples, if significant amounts of organic matter do not remain. Direct sampling of surfaces using Easylift® for the presence of particulates is a proven technique in forensic science as most surface types can be tape lifted. This could be extended into MP work, such as sampling road surfaces for tyre particles. Direct sampling of atmospheric MPs using Easylift® has been utilised in the field by upturning the tape and securing it to surfaces of interest, for example, laboratory benches, to detect possible contamination and act as atmospheric controls. After sampling, the tapes are then secured as normal to glass microscope slides and searched. This approach could be further employed for sampling for airborne MPs in areas of interest, for example food displays in stores. Our work reported in this publication uses only filter papers made of either cellulose fibres or glass fibres. Other types of filter paper have yet to be fully tested, although initial investigations indicate that nylon filter papers adhere more readily to the tape and would require further exploration to improve this, whilst steel filters can be very easily taped. As illustrated by Fig. 8, Easylift® is compatible with microspectrophotometry (MSP). Its transparency in the UV range gives Easylift® the potential of being compatible with dyes such as Nile red, Fluorescein isophosphate (FITC) and Safranine T, that have been used to aid the detection of MPs through their subsequent fluorescent properties exhibited once dyed (Lv et al. 2019). We plan to conduct work to test this potential. Furthermore, as MPs are held in place within the same optical plane when under an Easylift® tape, this has the potential to allow for improved automation of the detection, quantification and characterisation of MPs.

5. Conclusions

In MP pollution studies of water or air, it is common for the isolation of MPs from the natural environment to be achieved by filtration followed by either:

- the *in situ* processing of particles of interest on the filter or
- the use of tweezers to remove such particles from the filter for subsequent analysis.

We have devised the workflow detailed in Table 1 to improve on this process.

The tape lifting of filters with Easylift® is at the heart of that workflow. Tape lifting offers significant time saving in the field, allowing more samples to be taken. It also creates a secure environment for the particles of interest. Easylift® tape is used in the workflow because, by design, it is:

1 easy to handle, even when wearing gloves;
2 easy to label;
3 pre-cut so that its transparent portion is the same size as a standard microscope slide;
4 compatible with a wide range of non-destructive analytical techniques such as PLM, MSP, confocal Raman spectroscopy, fluorescence microscopy and hyperspectral microscopy. This allows the characterisation, classification and quantification of particles of interest without the need to expose those particles to the possibility of contamination or loss;
5 readily dissected, allowing the removal of individual particles for further analysis if needs be.

A simulation experiment was conducted during the development of the proposed workflow. It found that the rate of recovery of MPs from:

- water onto the filter papers used had a mean of 92.1% ($s_{n-1} = 4.1$ percentage points, $n = 12$) with no evidence that the filter type or funnel type used influenced that rate;
- the filter papers onto tape lifts had a mean of 96.4% ($s_{n-1} = 3.5$ percentage points, $n = 12$) with evidence that both filter type and funnel type effect that rate and that there is an interaction effect between these factors.

This identifies the potential for loss of particles of interest during each of filtration and tape lifting. The proposed workflow includes steps to minimise the former and eliminate the latter of these losses.

The principal benefits of the proposed workflow are time saving in the field, contamination control and loss prevention. It is also inherently flexible and extensible, allowing it to be tailored by its adopters to meet the needs of their own research, enabling its benefits to be widely available. The workflow also promotes reproducible research as the samples can be preserved after the completion of the study in a form that is easily stored and in which all particles of interest are individually and uniquely labelled. This facilitates sample sharing and analysis of the MPs by others, allowing the external validation of results.

Declaration of competing interest

The intellectual property that underpins Easylift® is owned by Staffordshire University where this work was completed and two of the authors of this paper (AJ and CG) are named as the inventors in the relevant patents. One of the authors (CG) is a professor at Staffordshire University, one (AJ) is an emeritus professor of that institution and one (AO) holds a funded PhD research position paid for by that University. Staffordshire University is interested in licencing the production and sale of Easylift®. Outside the support listed in the Acknowledgements, this research has received no external funding.

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Supplementary materials

Supplementary material (i.e. the Appendices) associated with this article can be found, in the online version, at doi:10.1016/j.envadv.2021.100066.

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