



## Short Communication

# Microplastic pollution in a stormwater floating treatment wetland: Detection of tyre particles in sediment



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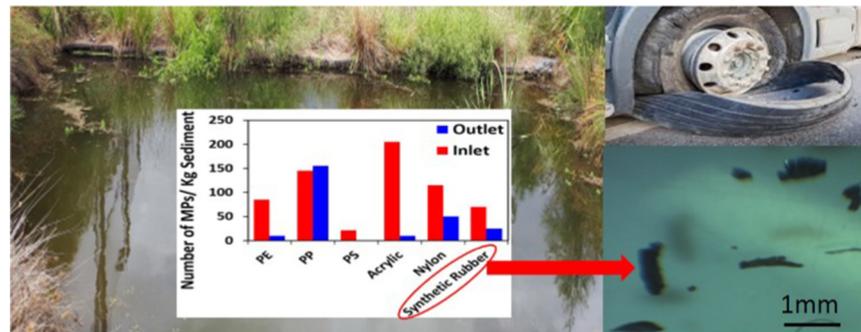
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## HIGHLIGHTS

- Microplastics detected in water and sediment of a stormwater treatment wetland
- More microplastics detected in sediment at inlet compared to outlet
- More microplastics detected in water at outlet compared to inlet
- Synthetic rubber particles with possible origin of car tyre found in sediment
- Microplastics from wetland's plastic construction material were not observed.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Synthetic rubber particles released from car tyres are expected to be an important type of microplastics in the environment, with road runoff and stormwater likely to transport tyre particles to the aquatic environment. Stormwater treatment wetlands are one of the key methods for treating road runoff and stormwater, but the presence and concentration of synthetic rubber microplastics from tyre particles in wetlands are largely unknown. In addition, constructed floating wetlands can be built using recycled PET plastic bottles, raising concerns about potential release of microplastics to the environment. In this study, we measured the concentrations of microplastics in water and sediment from the inlet and outlet of a stormwater floating treatment wetland on Queensland's Gold Coast. An average of  $0.9 \pm 0.3$  and  $4.0 \pm 2.4$  microplastic particles/L were detected in the water phase in the inlet and outlet samples, respectively. The sediment contained an average of  $595 \pm 120$  and  $320 \pm 42$  microplastic particles/kg dry sediment in inlet and outlet sediments, respectively. Between 15 and 38% of microplastics in the sediment were identified by FTIR as synthetic rubber-carbon filled particles, most likely derived from car tyres. The presence of synthetic rubber microplastics confirms that tyres can contribute to microplastic pollution in stormwater, with road runoff likely to be an important pathway. No microplastics with the same characteristics and polymer composition as the floating wetland construction material were detected in the water and sediment samples, indicating that the microplastics in the water and sediment detected here did not originate from the floating wetland's material. However, further investigation of older treatment wetlands is required to better understand the potential role of floating treatment wetlands as a source of microplastics.

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## 1. Introduction

Microplastic pollution of the aquatic and terrestrial environment is a serious environmental issue, with particles detected in oceans, rivers, lakes and estuaries (Ivar do Sul and Costa, 2014; Wagner et al., 2014), as well as in soil and sediment (Castañeda et al., 2014; Peng et al., 2018; Zhang and Liu, 2018). Microplastic particles (1  $\mu\text{m}$ –5 mm) are potentially bioavailable and may pose a risk to exposed biota (Besseling et al., 2014; von Moos et al., 2012; Ziajahromi et al., 2017a) and human health (Smith et al., 2018).

Microplastic particles from car tyres (e.g. synthetic rubber) are expected to be one of the major contributors to microplastic pollution in the environment (Kole et al., 2017; Leads and Weinstein, 2019; Unice et al., 2019; Wagner et al., 2018). For example, Kole et al. (2017) suggested that particles from tyres may contribute 5 to 10% of the total microplastics in the marine environment. It is estimated that the emission of car tyre rubber in the US and Europe was 1,524,740 and 1,300,000 t/year (Kole et al., 2017; Wagner et al., 2018). In Australia, the total amount of tyre wear was calculated to be 20,000 t/year (Kole et al., 2017). However, to date, only few studies have detected microplastics from car tyres in the environment (Leads and Weinstein, 2019). Tyre particles are expected to be transported to aquatic systems via road runoff (Vogelsang et al., 2019), meaning they are likely to be present in wetlands designed to treat stormwater and urban runoff. Numerous studies have focused on the presence of microplastics in marine and freshwater ecosystems as well as wastewater treatment plant effluent, but less attention has been paid to treatment wetlands receiving road runoff and stormwater (Horton et al., 2017; Kole et al., 2017; Liu et al., 2019; Sutton et al., 2016). In a recent study, Liu et al. (2019) reported the presence of microplastics in water samples collected from urban and highway stormwater treatment wetlands in Denmark, but due to the lack of an appropriate reference standard to determine tyre material (carbon black added) using FTIR, no car tyre particle was detected.

Constructed wetlands have been used worldwide in the U.S, Europe and Australia to treat stormwater and wastewater (Vymazal, 2010). Some constructed wetlands, such as floating treatment wetlands, are built from plastic materials (e.g. polyvinyl chloride (PVC), polyethylene, polystyrene and polyethylene terephthalate (PET)) (Pavlineri et al., 2017), and thus there are concerns about the potential contribution of the construction material to microplastic pollution. Therefore, floating treatment wetlands, which are designed to treat stormwater as well as provide a habitat for various organisms (e.g. plants, birds, fish) and

microorganisms (Chen et al., 2017; Sandoval-Herazo et al., 2018), may act as both a sink and source of microplastics.

In this preliminary study, we investigated the presence of microplastics in a floating stormwater treatment wetland constructed from PET with the specific aims of determining i) the concentrations of microplastics, including particles from car tyre in a constructed wetland for stormwater treatment, and ii) whether the floating treatment wetland construction materials are a source of microplastics. This was achieved by collecting water and sediment samples from both the inlet and outlet of a floating treatment wetland.

## 2. Materials and methods

### 2.1. Sampling

The sampling of sediment and water was conducted on 2th and 9th December 2018, respectively, from a floating treatment freshwater wetland located on the Gold Coast, Australia (Fig. S1). The wetland was constructed from recycled PET (a matrix of fibres) (SPEL.Stormwater, 2018) and installed in May 2016.

Samples of the top 5 cm of the bottom sediment were collected with a stainless steel shovel from area of approximately 20  $\times$  30 cm and transferred to pre-cleaned glass jars (Jiang et al., 2018). The sediment sampling was performed at the wetland inlet and outlet with two replicates. Approximately 1 kg (wet weight) of sediment sample was collected from each sampling point. To minimize the background contamination during sampling, the sample jars were kept closed as much as possible and no plastic materials were used to collect the samples. A field control sample (an open glass jar) was also set up during sampling to monitor the possible microplastic contamination from the atmosphere (e.g. air-borne microplastics) and other sampling procedures.

Water samples were collected from each sampling point (inlet and outlet) with two replicates. The water samples were collected after a heavy rain event (~34 mm/d) (Australian Bureau of Meteorology, 2018) from the middle of water body (approximately 50 cm below the surface) and passed through a validated sampling device developed by Ziajahromi et al. (2017b). The size ranges of stainless steel mesh screens were 25, 100, 190 and 500  $\mu\text{m}$ . A field control sample (an open 1 L glass jar) was also included during sampling.

To avoid disturbed sediment contributing to microplastic concentration in the water phase, the sediment and water samples were collected on different sampling occasions.

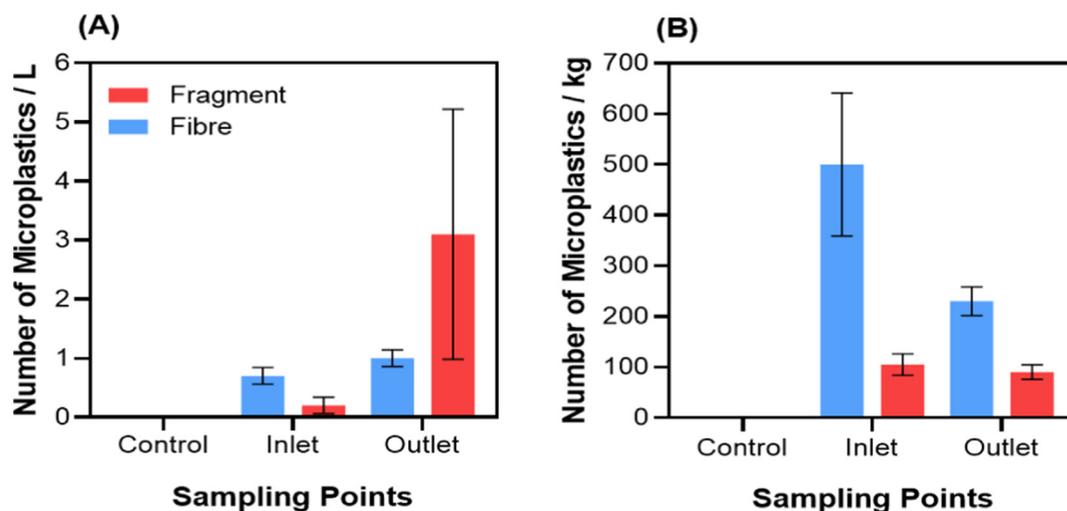
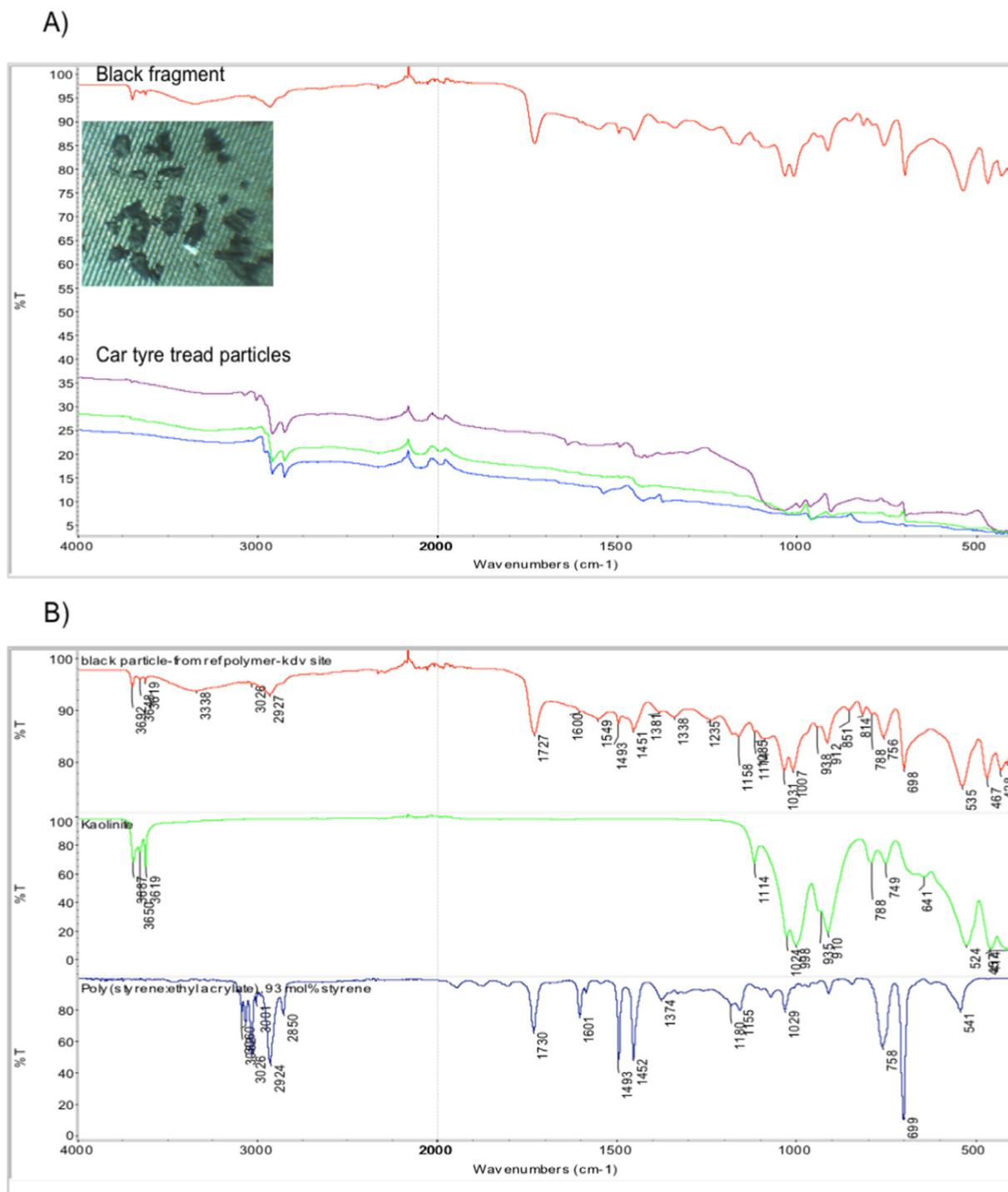


Fig. 1. Number of microplastics A) per litre of water and B) per kg (dry weight) of sediment in the inlet and outlet of the floating treatment wetland according to shape. Data presented as the average of replicates, with error bars indicating standard deviation.

## 2.2. Sample processing

The sediment sample processing was performed based on a method developed for coastal and sea sediment (Besley et al., 2017; Jiang et al., 2018), with modifications. Sediment samples were dried in an oven (60 °C) for 24–48 h until the water content was fully evaporated. The dried sediment (100 g) was weighed and transferred to a 500 mL glass beaker (Besley et al., 2017). Ultrapure water (100 mL) was added to each beaker and stirred using a magnetic stir bar for 30 min to make a homogenous suspension. Fifty millilitres of 30% hydrogen

peroxide (H<sub>2</sub>O<sub>2</sub>) (Chem-Supply, Australia) was added to each beaker to remove the organic matter and stirred at 60 °C (Jiang et al., 2018). The samples were then left for 24 to 48 h on the stirrer at 60 °C until no further reaction was observed. The samples were then transferred to 50 mL centrifuge tubes and 20 mL of sodium iodide (99.9%, Chem-Supply, Australia) with a density of 1.59 g/mL was added to each centrifuge tube for density separation (Ziajahromi et al., 2017b). The tubes were manually shaken for 2 min and then centrifuged for 5 min (3500 ×g) to separate microplastics from the sediment. The buoyant particles were filtered through a 25 µm stainless steel mesh screen



**Fig. 2.** Representative FTIR spectra of black fragment (red spectra) detected in water samples, A) in comparison with three car tyre tread samples, and B) in comparison with kaolinite (green spectrum) and poly(styrene-co-ethylacrylate) (blue spectrum). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

(plain Dutch weave, Al Metal Mesh Screens, Australia) and rinsed with ultrapure water. Due to the high amount of organic and inorganic matter in the samples, the density separation step was repeated three times for each sample to increase the recovery rate of microplastics in sediment (Besley et al., 2017). After each density separation repetition, the buoyant particles were poured through the same mesh screen.

To further minimize the detection of non-plastic particles such as natural fibre (e.g. natural cotton), a staining method using Rose-Bengal solution (4,5,6,7-tetrachloro-20,40,50,70-tetraiodofluorescein, Sigma-Aldrich, 95% dye content), was applied according to the methodology described in Ziajahromi et al. (2017b). The mesh screens were then transferred to petri dishes and dried in the oven (60 °C).

The water samples were processed according to a methodology previously developed and validated for wastewater samples in Ziajahromi et al. (2017b). In brief, the sampled material was rinsed into the glass beakers with 200 mL ultrapure water. The organic matter was digested using 20 mL of a 30% hydrogen peroxide, and samples were stirred using a magnetic stir bar at 60 °C. Ten millilitres of sodium iodide were added to the beakers for density separation. The samples were transferred to centrifuge tubes and centrifuged for 5 min (3500 ×g). The buoyant particles were then filtered and further processing performed as described above for sediment samples.

### 2.3. Characterisation of microplastics

Suspected microplastics from each sample were initially inspected under a dissecting microscope (Olympus) equipped with a digital camera, and stained particles (i.e., natural fibres and particles) were removed from the mesh screens (Ziajahromi et al., 2017b). The suspected microplastics were then counted, with the average and standard deviation of the replicates reported, and classified based on colour and form, including fibre, fragment and granular (bead). The suspected microplastics from water and sediment samples were further characterised using Fourier-transform infrared (μFTIR) spectroscopy. The FTIR measurement was carried out using a Nicolet iS50 spectrometer that was equipped with both an in-built diamond single bounce sampling accessory and a Continuum Infrared Microscope (Thermo Fisher Scientific, Madison WI, USA). The larger particles (100 to >500 μm) were individually removed from the mesh screens and pressed onto the diamond crystal of the attenuated total reflection (ATR) accessory and a spectrum at 4 cm<sup>-1</sup> resolution and 64 scans were obtained. The smaller particles (25 to 100 μm) were identified using μFTIR. The mesh was directly placed under the infrared (IR) microscope and examined in visual mode to locate the individual particles. FTIR spectra were then recorded in transmittance mode, at 8 cm<sup>-1</sup> resolution and 128 scans after adjusting the aperture of the IR beam to match the shape of the particle. Spectra were subjected to a library search using the search routine of the Nicolet Omnic 9.2 software using a library set that included the Nicolet polymer, forensics and common materials set in addition to the Hummel polymer library. To provide comprehensive information about microplastics, all selected particles in water samples and over 50% of particles in sediment (n = 86) with the same visual properties (e.g. shape and colour) were characterised using FTIR. Where selected microplastics with the same visual characteristics were found to have different polymer composition, FTIR analysis was performed on all particles with those visual characteristics to avoid incorrect characterisation.

### 2.4. Quality control/quality assurance

Negative control samples were processed through all steps of sample processing to determine any potential background plastic contamination from the laboratory (e.g. contamination from air, used chemicals and clothing). The control beakers were rinsed with ultrapure water and processed using the same methodology as described above for the water samples. Additionally, to avoid potential background

contamination in the laboratory, the sample processing was performed under fume hood during the sample analysis process in the laboratory (Wesch et al., 2017), all materials and glassware were rinsed three times with ultrapure water before use and covered with aluminum foil after each step and cotton lab coats were worn during the whole process (Nuelle et al., 2014). The petri dishes were kept covered during microscopy.

## 3. Results and discussion

### 3.1. Quality control/quality assurance

A total of 6 fibres and one fragment were found in the field blank and a total of 4 fibres were found in the laboratory control samples; all were however identified as non-plastic by FTIR analysis (rayon and linen).

Between 3% to 38% (average of 2 to 23 particles) of the detected suspected microplastics in water and sediment samples were identified by FTIR as non-plastic. The non-plastic particles, which were primarily granular particles, were silica, and therefore were excluded from the results.

### 3.2. Microplastics in water and sediment

An average of 0.9 ± 0.3 and 4.0 ± 2.4 microplastic particles/L were detected in inlet and outlet water samples, respectively. This is consistent with Liu et al. (2019) who reported 0.5 to 23 microplastic particles/L in water samples from stormwater ponds. The total number of microplastic particles in water and sediment samples is provided in Table S1 in the Supporting Information.

A higher amount of microplastics were observed in water from the outlet, compared to the inlet. These were mainly in the form of fragments and mainly black in colour (>75% of the average total microplastics; average of 30 particles) (Fig. 1). A previous study by Coalition Clean Baltic (2017) also reported black fragments as the dominant type of microplastic detected in water inlet and outlet of a stormwater wetland located in Sweden, which was suggested as originating from car tyre wear.

In the present study, FTIR analysis of black fragments using a library search routine showed an initial match with a synthetic rubber, which supported the hypothesis that these particles may have originated from tyres. However, following careful interpretation of the spectrum and comparison with three reference rubber samples from car tyre tread (one from an old tyre and two from new tyres) (Fig. 2A and B), we found that unlike the black fragments detected in the water samples (Fig. 2A), the reference car tyre particles spectra had a very low transmittance across the whole spectrum (Fig. 2B), caused by the absorption of carbon added in tyres to protect them from UV initiated oxidation (Kole et al., 2017; Leads and Weinstein, 2019). Further, a major and significant point of difference was the butadiene signals seen in the tyre spectra (at 950 and 900 cm<sup>-1</sup>; Fig. 2A). These bands are seen in butyl rubbers including styrene butadiene, a common synthetic tyre rubber (Gunasekaran et al., 2007), but were missing from the black fragments spectra. Therefore, even though the FTIR spectra of black fragments had many common bands found in kaolinite as well as bands indicating styrene (bands at 3026, 756 and 698 cm<sup>-1</sup>) and ester groups (bands at 1727, 1158 cm<sup>-1</sup>) (Fig. 2B), which are used in tyre formulations and found in two of the tyre samples, the lack of carbon and a butyl signal spectra makes it questionable that these particles are actually derived from car tyre.

Analysis of microplastics from the environment remains challenging as it is unclear how the chemical properties of particles change over time under different environmental conditions. For example, Selbes et al. (2015) showed a high leaching rate of dissolved organic carbon (DOC) and other inorganic compounds from scrap car tyre particles under acidic environmental condition (pH 3). Thus, if the black fragments were indeed originated from car tyre, the impact that various

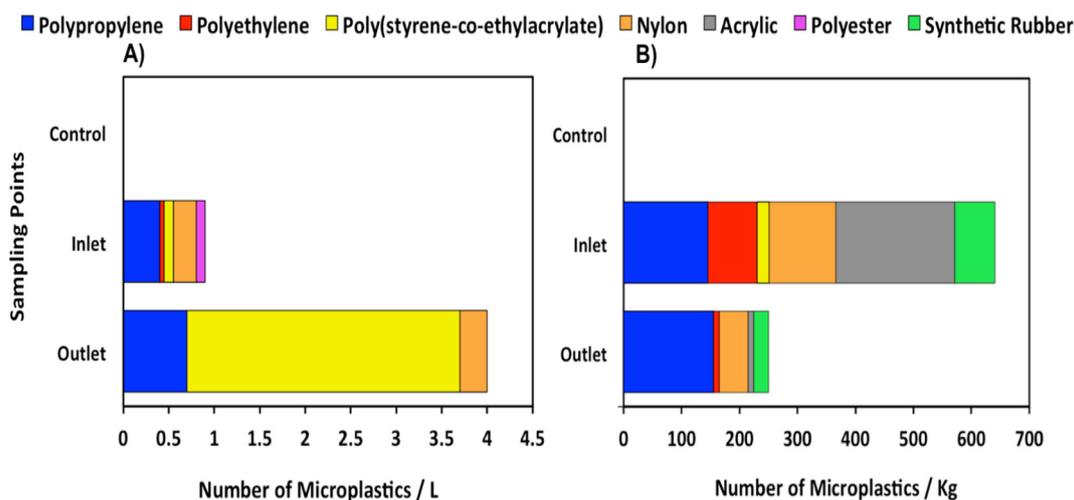


Fig. 3. Number of microplastics A) per litre of water and B) per kg (dry weight) of sediment in the inlet and outlet of floating treatment wetland according to polymer type.

characteristics of the environment (such as pH, salinity, temperature) may have had on the composition of car tyre particles needs to be further investigated. This result highlights the importance of appropriate characterisation of microplastics from environmental samples especially suspected microplastics from car tyres.

The most frequently detected microplastics across all water samples were polystyrene-co-ethylacrylate (black fragments) followed by polypropylene, nylon and polyester (Fig. 3), which could be attributed to industrial and commercial applications as well as atmospheric depositions (Liu et al., 2019).

An average of  $595 \pm 120$  microplastics/kg dry sediment were detected in sediment samples collected from the inlet, compared to an average of  $320 \pm 42$  microplastics/kg in the outlet sediment. Unlike water samples, a greater amount of fibrous particles (>60% of the total microplastics) were detected in sediment samples (Fig. 1). This is in the range of average microplastics previously reported in freshwater sediment in the UK (539 particles/kg) (Turner et al., 2019) and China (388 to 502 particles/kg) (Jiang et al., 2018). It has been reported that microplastics can accumulate in sediment over time and therefore sediment could be a long-term sink for microplastics (Nakki et al., 2019; Willis et al., 2017).

Black microplastic fragments were also found in both sediment inlet and outlet samples, which comprised 15 to 38% (between 8 and 26 particles) of the total detected microplastics in the sediment samples. Unlike fragments in the water samples, the majority of black fragments in sediment were confirmed by FTIR as synthetic rubber-carbon filled isobutylene (or butyl rubber) (Fig. 4), likely originating from car/truck tyre (Leads and Weinstein, 2019). This is consistent with a recent study that reported fibres and tyre particles (synthetic rubber) as the dominant microplastics detected in the sediment of three tributaries in South California, USA (Leads and Weinstein, 2019).

Butyl rubber has been widely applied in inflatable tubes and tubeless tyres and mainly used as inner layer of the tyre (Sharma and Bhattacharya, 2019), therefore, it is likely that these particles were released to the environment as a result of tyre blowouts (rather than tyre wear which is mainly styrene-butadiene rubber (Sirisinha et al., 2019)), and eventually ended up in the stormwater wetland via road stormwater runoff. However, other road surface materials such as rubberised asphalt and road marking rubber adhesives should not be overlooked as potential sources. Moreover, majority of the black fragments found in the sediment samples had an elongated shape with rubbery surface texture when manipulated with tweezers. The black fragments detected in water samples on the other hand did not have the same morphology or rubbery property.

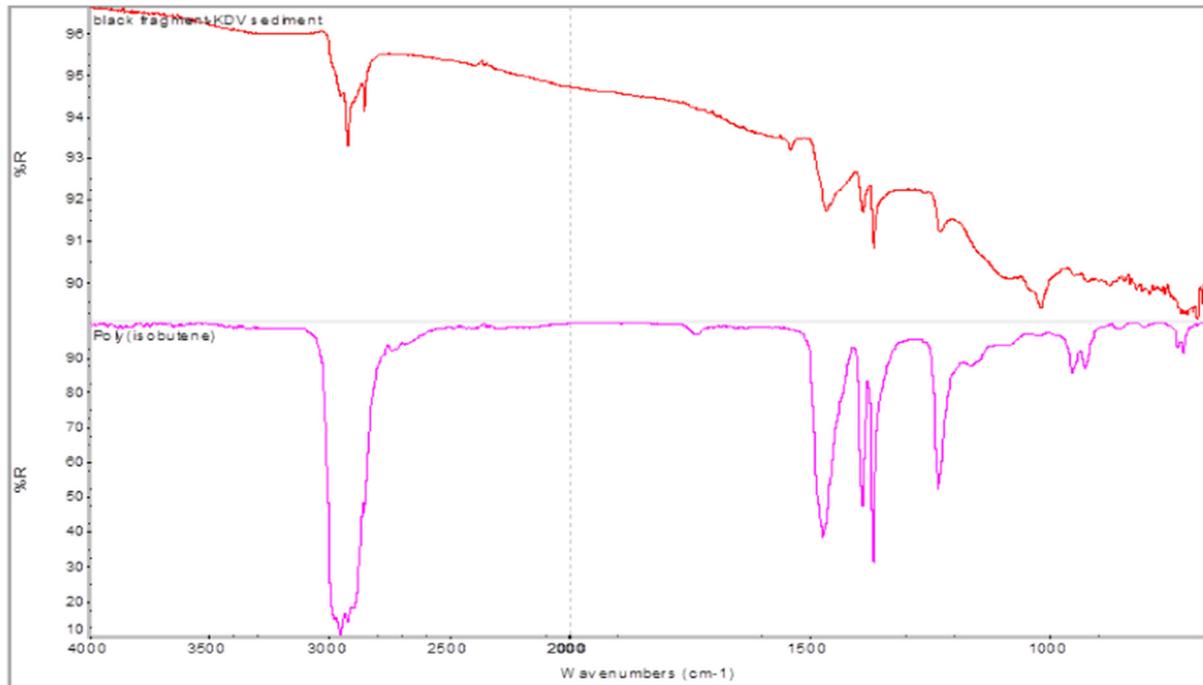
The size of tyre wear particles has been reported to be in the range of 4 to 350  $\mu\text{m}$  (average 50  $\mu\text{m}$ ) (Kreider et al., 2010), however, the majority of black fragments (synthetic rubber) detected in our sediment samples had greater size ranges (mostly >500  $\mu\text{m}$ ). This result suggests that beside microplastics from tyre tread that are reported to be a major source of tyre-based microplastics to the environment (Kole et al., 2017; Liu et al., 2019), tyre blowout could also be considered as another pathway of microplastics to the environment, particularly due to the proximity of the studied wetland to roadside (approximately 250 m).

The dominant microplastics in sediment samples were polypropylene and nylon (also found in water samples), followed by acrylic and synthetic rubber microplastics. Representative extracted microplastics in water and sediment samples are shown in Figs. 5.

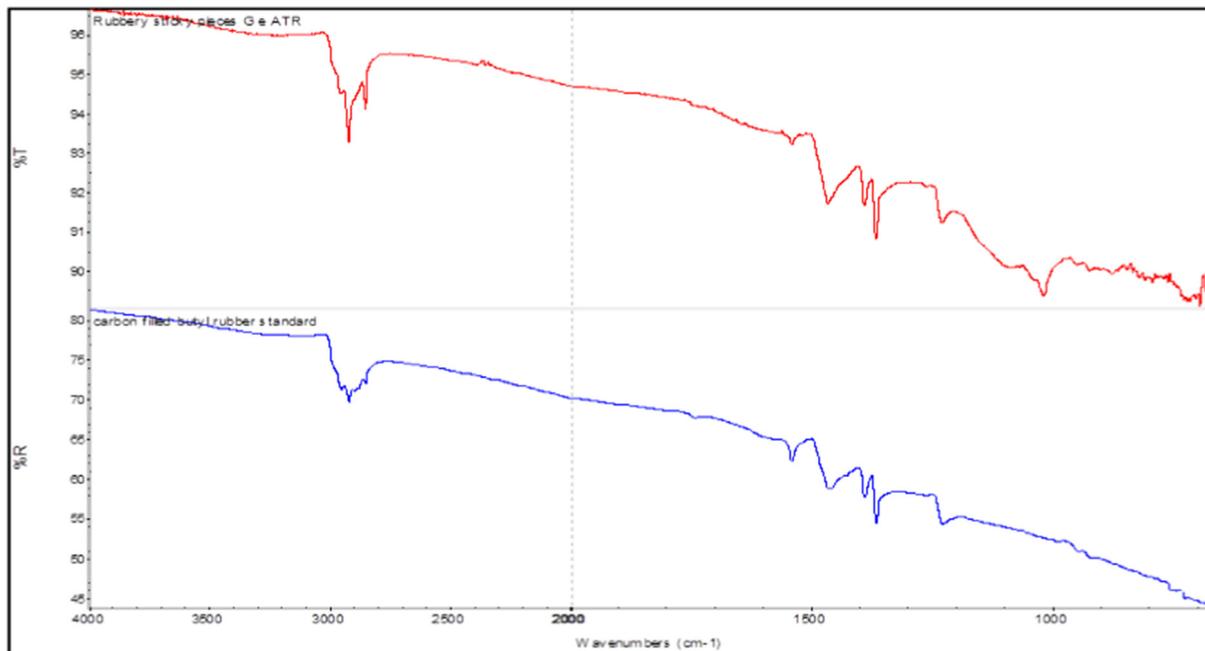
Microplastics in the inlet water samples were mostly in the 190 to >500  $\mu\text{m}$  size range (average 83.7%;  $7.5 \pm 2.1$  particles), while smaller microplastics (average 96.5%;  $39 \pm 22.6$  particles) were found in the outlet samples (primarily 25 to 100  $\mu\text{m}$  size range) (Fig. 6). This suggests that larger particles may be settling out in the wetland. A study by Coalition Clean Baltic (2017) also showed a reduction in the size and abundance of microplastics detected in outlet compared to inlet from constructed wetlands, which was attributed to the potential role of constructed wetlands to retain microplastics received from stormwater. In contrast to the Coalition Clean Baltic (2017) study, we observed an increased concentration of microplastics in the water phase at the outlet. This may be due to the movement of microplastics with the flow of water during a rain event (i.e., higher concentration in first flush) and possibly re-suspension of the settled microplastics with rainfall.

The opposite trend was observed for the sediment samples, with lower concentrations at the outlet. The higher concentrations in sediment at the inlet could be attributed to the faster settling of microplastics with greater density than water such as synthetic rubber ( $1.55 \text{ g/cm}^3$ ) and nylon ( $1.15 \text{ g/cm}^3$ ), as well as the effect of biofouling processes (Kaiser et al., 2017). Further, the size of microplastics is likely to influence the settling behaviour of microplastics as the bigger size microplastics could have greater mass than smaller size microplastics, which would cause deposition of larger microplastics earlier in the inlet sediment. This is in agreement with a modelling study by Besseling et al. (2017) that demonstrated faster retention of bigger size microplastics (1–10 mm) than smaller (<100  $\mu\text{m}$ ) in sediment and consequently higher concentrations in upstream compared to downstream sites. However, further research is recommended to investigate the behaviour and fate of microplastics, particularly synthetic rubber, in wetlands.

A)



B)



**Fig. 4.** FTIR spectra of black fragments (red spectrum) detected in sediment samples, in comparison with A) butyl rubber (pink spectrum) and B) carbon filled butyl rubber (blue spectrum). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

While the floating treatment wetland was constructed from recycled PET (confirmed by FTIR analysis), none of the microplastics detected in the water and sediment samples were PET. This indicates that the microplastics detected in the water and sediment samples did not originate from the treatment wetland itself. However, since the wetland was only installed in 2016, it may be possible that older constructed wetlands could release microplastics from their construction material due to weathering and degradation over a longer time period.

Therefore, more research is recommended to further investigate the sustainability of the constructed wetland material and whether they act as a potential source of microplastics.

#### 4. Conclusions

Microplastics were detected in both water and sediment samples collected from a floating treatment wetland. While black fragments



**Fig. 5.** Microplastics extracted from water and sediment, including poly(styrene-co-ethylacrylate) black fragments (A), butyl rubber black fragments (B), polyethylene fibres (C) and acrylic fibres (D).

with a suspected source of car tyre were detected across both water and sediment samples, the FTIR spectra of the water-based black particles did not perfectly match our car tyre reference samples, and only those found in sediment were confirmed as synthetic rubber-carbon filled microplastics, most likely derived from tyre. However, the presence of elements that are common in tyre formulation in the black fragments from the water samples raises the possibility that these particles originated from tyres but their chemical properties changed as a result of

weathering under environmental conditions. Additional studies are recommended to further investigate the changes in the chemical properties of microplastics, particularly particles from car tyre, under different environmental conditions.

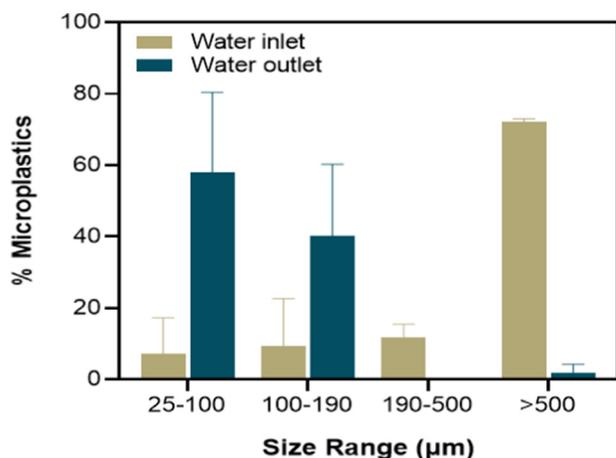
This preliminary study is one of the first to detect the presence of synthetic rubber in sediment with a possible origin of car/truck tyre in stormwater treatment wetland, suggesting that sediment acts as a sink for tyre particles. Higher concentrations of microplastics were observed in sediments compared to the water phase, which raises the possibility that wetlands could potentially play a role in controlling microplastic pollution in surface waters. Higher microplastic concentrations were detected in water from the outlet compared to the inlet after a heavy rainfall event, possibly the result of a first flush effect. Further research is recommended to study the sustainability of wetland for microplastic removal, and temporal profile of microplastic input from stormwater during a rain event. While the current study did not find any evidence of the wetland construction material (PET fibre) acting as a source of microplastics, further studies on older constructed wetlands are needed to more comprehensively determine if the PET material releases microplastics to the environment.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**Fig. 6.** Percentages of different size ranges of microplastics in water inlet and outlet samples. Data presented as percent average of replicates, with error bars indicating standard deviation. Note: the blue bar (190–500 µm) is "0". (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

SPEL treatment floating wetlands". This FTIR measurements were undertaken at the Central Analytical Research Facility hosted by the Institute for Future Environments at QUT. The authors acknowledge Dr. John Colwell at Queensland University of Technology (QUT) for his helpful advice in analysis and spectra interpretation of the synthetic rubber microplastics.

## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2019.136356>.

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