INTRODUCTION

Tidal creeks are the capillaries that link land and sea in estuarine systems, where materials may be readily exchanged and processed. They can be highly productive ecosystems, with profound value as cultural, recreational, and economical resources. However, land use and land cover change are impacting the integrity of these unique systems by changing hydrology and point and non-point source pollution pressures (Holland et al., 2004; Sanger et al., 2015; Schueler, 2000). Urban land use can lead to release of a variety of pollutants of concern into the environment, including heavy metals, nutrients, fecal coliform bacteria, and organic chemicals. Contaminants released to air or on land may accumulate in soils and on roadways and buildings during dry periods and then be flushed into local waterways with stormwater (Krein & Schorer, 2000; Ngabe et al., 2000; Diamond et al., 2000).

Polycyclic aromatic hydrocarbons (PAHs) are one class of organic contaminants of concern and are a leading threat to aquatic life in urban environments (Van Metre and Mahler, 2005). Both natural and anthropogenic sources of PAHs are observed in the environment, including forest fires, fossil fuels, and coal tar sealants that leach from roofs and roadways (Van Metre and Mahler, 2010). A majority of PAH compounds are known or probable carcinogens, in addition to having other acute and chronic toxic effects to both human and ecological receptors (ATSDR, 1995). Sixteen US EPA priority PAHs are typically monitored in the environment and include compounds with structures containing two to
six fused aromatic carbon rings. This range in molecular size imparts differences in the physicochemical properties among PAH molecules; for instance, water solubility ranges over 3 orders of magnitude, from 30 mg/L for naphthalene to <1 μg/L for perylene (Schwartzbach et al., 2003). Larger PAH compounds with lower water solubility are more hydrophobic and have a stronger tendency to sorb to lipid or organic-rich phases, such as sediments or dissolved organic carbon (DOC). Several studies have found direct associations between DOC or total suspended solids with transport of, for example, PAHs, mercury and other metals (Cai et al., 1999; Foster et al., 2000; Journey et al., 2012; Kirchner et al., 2011; Nasrabadi et al., 2016; Schwientek et al., 2013). High-flow events are major contributors to DOC and suspended sediment loads (Brown et al., 2014; Hinton et al., 1997) and to associated releases and transport of PAHs in rivers (Foster et al., 2000; Schwientek et al., 2013). Previous investigations have found sediments in stormwater ponds and tidal creeks in South Carolina to be impacted by PAHs, including Bull Creek, the location of the present study (Garner et al., 2009; Sanger et al., 1999; Weinstein et al., 2010).

Understanding the chemical properties of the suspended and dissolved material in streams can provide information about sources and system dynamics. For example, the aromaticity of the DOC matrix is indicative of its origin and biogeochemical activity (Weishaar et al., 2003). SUVA$_{254}$ is a simple surrogate indicator of the aromaticity of DOC. Allochthonous materials (with terrestrial origin) have been associated with higher SUVA$_{254}$ values relative to autochthonous material (with in-stream origin), such as algae, in streams (Weishaar et al., 2003). The organic matter content of particulate material may also indicate the source by providing a relative measure of organic and mineral composition. Further, analysis of the relative concentrations of PAH compounds in water samples is an approach used to fingerprint PAH sources (Yunker et al., 2002). Liu et al. (2013) used PAH distribution patterns and diagnostic ratios to differentiate ongoing point source contamination from diffuse background contamination in contrasting river catchments.

The objective of the present study is to characterize the loading of PAHs and carbonaceous matter in Bull Creek, a semi-urbanized tidal creek in Charleston, SC, with a specific look at changes after a historic flood event in October 2015. This flood event affected a large part of South Carolina after historic rainfall fell between October 1 and 5, 2015. Record discharges were recorded at river gages across the state (Feaster et al., 2015). The maximum stage of the Ashley River recorded at a gage site adjacent to Bull Creek (USGS 021720869) following the storm event was 4.3 m (~14 feet), which at that time was the second highest stage recorded at that site in its period of record since 1992. We collected data on PAHs, total suspended solids and their organic matter content, DOC and aromaticity, and stream discharge, in addition to general water quality characteristics, on four other sampling dates over the period September 2015 to April 2016 for comparison. We use the totality of the information provided by these measures to better understand sources to the tidal creek and to work toward improving water quality monitoring approaches.

### METHODS

#### SITE DESCRIPTION

Bull Creek, a small tidal creek tributary watershed (~778 ha) of the Ashley River near Charleston, SC, was chosen as the study site. The Bull Creek watershed was digitized using United States Geological Survey (USGS) elevation derivatives for national applications (EDNAs) map information. NOAA 2010 Coastal Change Analysis Program (C-CAP) Land Cover Atlas data were used to determine the percent of land use distribution (e.g., developed, forested, and wetlands). Watershed location, delineation, and land use is illustrated in Figure 1A, B. The sampling location is also shown (star; 32°49′38″N, 80°01′44″W). Additionally, ArcGIS software tools were used to delineate the watershed upstream of the sampling location and to categorize land use (Figure 1B). The upstream watershed area of ~430 ha delineated by ArcGIS digital elevation modeling extends beyond the EDNA watershed boundaries, but upstream land use distribution followed the pattern of the entire watershed. The watershed is dominated by low- and medium-density development but also has some intact wetlands. Bull Creek watershed has a reported impervious cover of 38% (Sanger et al., 2015).

### FIELD SAMPLING AND LABORATORY ANALYSES

Sampling dates and schedule are shown in Table 1. Sampling was conducted on five dates, with samples spaced over the course of the day between 9 a.m. and 5 p.m. and at different stages of the tidal cycle. Bull Creek exhibits a semi-diurnal tidal pattern, with two high tides and two low tides.

<table>
<thead>
<tr>
<th>Date</th>
<th>Tidal Cycle Sampled</th>
<th>Sample Type and N</th>
<th>TSS/PAH</th>
<th>N, Ebb/Flood</th>
</tr>
</thead>
<tbody>
<tr>
<td>9/22/15</td>
<td>Flood</td>
<td>TSS</td>
<td>8/0</td>
<td></td>
</tr>
<tr>
<td>10/9/15</td>
<td>Ebb &amp; Flood</td>
<td>TSS</td>
<td>3/1</td>
<td></td>
</tr>
<tr>
<td>1/25/16</td>
<td>Ebb</td>
<td>TSS</td>
<td>4/0</td>
<td></td>
</tr>
<tr>
<td>3/24/16</td>
<td>Ebb &amp; Flood</td>
<td>TSS</td>
<td>3/1</td>
<td></td>
</tr>
<tr>
<td>4/11/16</td>
<td>Ebb &amp; Flood</td>
<td>TSS</td>
<td>4/3</td>
<td></td>
</tr>
</tbody>
</table>


Figure 1. A. Location of the study site in Bull Creek adjacent to the Ashley River near Charleston, SC, with the USGS EDNA watershed shown in black outline. The sampling site within Bull Creek is shown with a star (32°49'38"N, 80°01'44"W). B. Watershed delineation and land use classification according to the USGS EDNA map system (black; area 778 ha), additionally with the watershed area delineated upstream of the sampling location by ArcGIS (red; area 430 ha).
over a lunar day (24 hours 50 minutes). The maximum depth of the channel transect over the tidal range was between 2.2 m and 4.0 m during the sampling dates. Antecedent precipitation data were acquired from the National Climate Data Center (ncdc.noaa.gov) at a local gage site (#US1SCCR0087).

Stream discharge, turbidity, and general water quality parameters were measured in the field. Discharge measurements were obtained every 30–60 minutes using a Teledyne RD Instruments acoustic Doppler current profiler (ADCP) (Ellis et al., this issue). The timing of these measurements was normalized to high tide slack (when discharge is negligible as the creek current switches direction between flood and ebb), which typically occurred about 40 minutes after high tide (the maximum stage of the creek). The ADCP was attached to the back of a kayak and pulled across the width of Bull Creek, collecting water velocity and depth data that are used to calculate average discharge. Two optical backscatter sensors were used to measure the turbidity (cloudiness) of the water: a Thermo-Scientific Orion Aquafast handheld portable turbidity meter and a YSI Multiparameter Water Quality Sonde (6600 V2). These two instruments measure turbidity at different angles of light scatter. The former detects light at a 180° angle with color compensation, while the later detects light at a 90° angle without color compensation. The handheld turbidity meter was used on sampling dates 9/22/15 and 10/9/15, and the sonde was used on 1/25/16, while both instruments were used to collect turbidity data on 3/24/16 and 4/11/16. A comparison of results for the turbidity meter and sonde on these two dates showed good agreement between the instruments, with the sonde generally indicating slightly higher turbidity but within 1 S.D. of the turbidity meter average (e.g., 3/24 11 am 8.09 ± 0.66 NTU vs. 8.7 NTU, and 4/11 4 pm 19.24 ± 0.89 NTU vs. 19.6 NTU). On the dates that both instruments were used, data from the handheld turbidity meter were reported. Temperature, pH, salinity, and conductivity were measured with the YSI.

Water samples were collected at elbow depth (~0.3 m) from a dock or kayak in coordination with discharge measurements to determine the PAH concentrations and to characterize the dissolved and suspended matter. A flow chart depicting the sample analysis is shown in Figure 2. Whole water samples collected in 1 L amber glass bottles were processed to quantify the total suspended solids, organic matter content of the solids, DOC concentration, and aromaticity of the DOC (SUVA254). Total suspended solids were determined as the dry mass of the particulates captured on a GF/F glass fiber filter (0.7 μm pore size) after drying in an oven at 105°C to constant weight. The organic matter content of the solids was determined by loss on ignition after combusting the filter at 450°C for 4 hours (ASTM, 2014). The filtrate was acidified to pH 2–3 with 1 N HCl, purged in a sonication bath, and analyzed for DOC using a Shimadzu elemental analyzer (TOC-VPN) against calibration standards prepared with potassium hydrogen phthalate (C₇H₅KO₄).

The specific UV absorbance at 254 nm wavelength (SUVA254) was determined on an unacidified filtrate using a UV-Vis spectrophotometer (Thermo Scientific Evolution 220). SUVA254 was determined as the absorbance of the sample normalized to its DOC content (Weishaar et al., 2003).

Whole water samples for PAH analysis were collected in 2 L glass bottles with Teflon-lined caps and kept refrigerated until sample processing. The samples for PAHs were collected on four of five sampling dates (Table 1). Liquid-liquid extraction was performed in the 2 L bottles by adding 15 mL of hexane and gently shaking on a horizontal shaker table for 24 hours (USEPA, 1996). The samples were allowed to settle for several hours to allow the layers to separate, as emulsions were common in the relatively high-DOC water matrix. Hexane was recovered by directly pipetting the top hexane layer off the bottle or with the aid of a separatory funnel. A second liquid-liquid extraction with hexane was performed by hand-shaking for 2 minutes. The recovered hexane layers were pooled for each sample, dried with sodium sulfate, and condensed to <350 μL. Blank DI water extractions were also performed to ensure no laboratory background or cross-contamination. PAHs were analyzed by an Agilent Technologies gas chromatograph with mass spectrometer detection in selective ion monitoring mode (GC-MS; model 7890A GC with directly coupled model 5975C MS). Separation was performed following injection and introduction of the sample in pulsed splitless mode onto an Agilent DB-XLB column (0.18 μm, 20 m x 0.180 mm I.D.), with He carrier gas (0.6 mL/min) and stepped oven temperature ramps from 55°C to 310°C during the 46 minute analytical run. Twelve PAHs were quantified against their 13C-labelled internal standards by an isotope dilution method (Boden and Reiner, 2004), including: three-member ring compounds, acenaphthylene (ANY), acenaphthene (ACE), fluorene (FLN), phenanthrene (PHE), and anthracene (ANT);
Polycyclic Aromatic Hydrocarbons and Suspended Materials in a Semi-urbanized Tidal Creek

Polycyclic Aromatic Hydrocarbons and Suspended Materials in a Semi-urbanized Tidal Creek

four-member ring compounds, fluoranthene (FTH), pyrene (PYR), benzo(a)anthracene (BaA), and chrysene (CHR); and five-member ring compounds, benzo(b)fluoranthene (BbF), benzo(k)fluoranthene (BkF) and benzo(a)pyrene (BaP). The detection limit for each PAH was 1 ng L⁻¹.

CALCULATION OF PARTICLE AND PAH FLUXES

A full ebb tide was captured with periodic sampling on 10/9/15, 1/25/16, and 3/24/16. Discharge was measured at least hourly over the course of the ebb tide (PAHs two to three times and TSS three to four times). To calculate flux, TSS and PAH mass concentrations were averaged (C_avg, mass/L) and multiplied by the total water volume discharged past the sampling point in Bull Creek (V_total, L) divided by the duration (in hours) of the ebb cycle:

\[ \text{Flux} = \frac{C_{\text{avg}} V_{\text{total}}}{t} \]

where \( V_{\text{total}} \) was determined by integrating the discharge values measured over time. This approach is not flow-weighted and assumes that the surface water sample is representative of the stream cross section.

RESULTS

ENVIRONMENTAL CONDITIONS

Water temperature, pH, and conductivity, along with antecedent rainfall data for each of the sampling dates, are shown in Table 2. Of particular note is the dramatic reduction in conductivity in October, which reflects the high volume of freshwater runoff delivered to Bull Creek from the precipitation event related to Hurricane Joaquin (Oct 1–5). Water salinity is typically brackish at the site but was classified as freshwater in October 2015.

SUSPENDED MATTER: DISSOLVED AND PARTICULATE

The concentration and quality of particulate matter and DOC in water samples are shown in Figure 3. Samples collected on October 9, 2015, are plotted separately from the other sampling dates. Total concentration of suspended solids was generally lower in October but with similar organic matter content (Figure 3A,B). In contrast, although the DOC content was at a level consistent with other sampling dates, the SUVA₂₅₄ measurement was elevated (Figure 3C,D). The higher SUVA₂₅₄ values in October are indicative of a more aromatic, terrestrial source of DOC.

Although the organic matter content of suspended particulates was consistent across sampling dates (Figure 3B), the correlation between turbidity and TSS deviated in October (Figure 4). Turbidity is measured by optical light scattering, and the characteristics of the particles in solution that affect light scattering include the general type and, in particular, the particle size, geometry, density, and color (Gippel, 1995; Rügner et al., 2013). The offset correlation indicates that some fraction of the suspended material measured as TSS in the water samples following the October 2015 storm event scatters light differently and therefore has a different quality.

Table 2. Precipitation and water condition parameters for sampling dates (S.D. of average measures in parentheses)

<table>
<thead>
<tr>
<th>Date</th>
<th>Antecedent precipitation</th>
<th>T (°C)</th>
<th>pH</th>
<th>Conductivity (mS/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9/22/15</td>
<td>10 d 1.8</td>
<td>26.0</td>
<td>7.0 (0.8)</td>
<td>26.7 (3.8)</td>
</tr>
<tr>
<td></td>
<td>5 d 0.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10/9/15</td>
<td>10 d 413.3</td>
<td>21.8</td>
<td>6.1 (0.9)</td>
<td>0.9 (0.5)</td>
</tr>
<tr>
<td></td>
<td>5 d 42.4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1/25/16</td>
<td>10 d 91.7</td>
<td>9.5</td>
<td>7.2 (0.8)</td>
<td>8.4 (5.1)</td>
</tr>
<tr>
<td></td>
<td>5 d 39.7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3/24/16</td>
<td>10 d 4.8</td>
<td>19.5</td>
<td>5.8 (0.7)</td>
<td>14.7 (1.2)</td>
</tr>
<tr>
<td></td>
<td>5 d 2.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4/11/16</td>
<td>10 d 26.1</td>
<td>17.8</td>
<td>5.7 (1.4)</td>
<td>16.4 (3.1)</td>
</tr>
<tr>
<td></td>
<td>5 d 1.0</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 3. Box and whisker plots show data points, quartiles, and outliers for (A) total suspended solids (TSS), (B) suspended solids organic matter (OM) content, (C) dissolved organic carbon (DOC), and (D) specific absorbance of DOC (SUVA₂₅₄) in units of L mg⁻¹m⁻¹. Samples taken on 10/9/15 are plotted separately from the other dates.
POLYCYCLIC AROMATIC HYDROCARBONS

The total concentration of 12 PAHs (PAH-12) in water was higher in October 2015 compared to the other sampling events (Figure 5). The concentrations of PAHs expressed on a particle mass basis indicated an even larger difference (data not shown), since the TSS concentration was lower in October. The distribution pattern of PAHs showed a larger contribution of lower molecular weight three-ring PAHs (ANY, ACE, FLN, PHE, ANT) in October 2015 (45% of the total PAH-12 concentration) compared to January, March, and April 2016 (0%, 0%, and 4% of the total PAH-12, respectively). The concentrations of PAHs in all samples analyzed were below the ecological risk assessment screening values for surface waters for the southeastern United States (US EPA, 2015).

Diagnostic ratios of PAH compounds were used to further distinguish sources as either petroleum or combustion derived, following Yunker et al. (2002) (Figure 6). The ratios of FTH/FTH+PYR and BaA/BaA+CHR were applied due to the robustness of these ratios and the consistent detection of these compounds in whole water samples in the present study. Further, since these PAHs are hydrophobic and are primarily associated with particles (octanol-water partition coefficient, log $K_{ow} > 5$), it is more appropriate to apply the diagnostic ratios that are established for sediments and suspended particles. The diagnostic ratios cross-plot indicates a combination of biomass, coal, and petroleum combustion, with a stronger mixed-sources signal indicated by the lower BaA/BaA+CHR ratio for the October 2015 samples (Figure 6).

FLUXES OF PAHS AND CARBONACEOUS MATERIALS

The total cumulative discharge volume of the ~6 hour ebb tides captured on 10/9/15, 1/26/16, and 3/24/16 were 291,800 m$^3$, 287,200 m$^3$, and 137,100 m$^3$, respectively. The average flood and ebb discharge at this site is reported in Ellis et al. (this issue). The high discharge measured in January is attributed to the full moon on 1/25/16 and recent precipitation (5d antecedent precipitation was similar for January and October sampling dates, Table 1). The average hourly fluxes of PAHs, DOC, TSS and particulate organic matter (POM, calculated from TSS and OM%) estimated for these dates are shown in Table 3. The fluxes reported are for the ebb tide only at a midpoint within the Bull Creek watershed and therefore do not represent the entire net flux of suspended materials in this watershed.

The average hourly flux of TSS was highest in January, while for PAHs, it was highest in October. This is the direct

Figure 4. Total suspended solids (TSS) versus turbidity for water samples collected on 10/9/15, in contrast to the other sampling dates.

Figure 5. Concentration of 12 PAHs in whole water samples from Bull Creek, SC. Error bars show ±1 S.D. of the mean for the sum of PAHs.

Figure 6. PAH diagnostic ratios can indicate sources of contamination, indicated in italics along the figure margins. Mixed sources include petroleum and combustion. Samples on 10/9/15 are encircled.
result of higher concentrations of the materials measured on these respective dates when similarly high discharge was experienced. The range in calculated flux over these three sampling dates was greater for PAHs than TSS. The flux of PAHs measured in October was 1.5 times greater than in January, and six times greater than in March. The lowest flux was observed in March when there was slower mean water velocity and less antecedent precipitation.

**DISCUSSION**

Several different measures of water quality collected in the present study indicated a significant alteration of the Bull Creek system in response to the historic flood event in October 2015. In addition to a decrease in salinity from brackish to freshwater, the nature of the suspended particle and DOC load changed, and a different profile of PAHs was mobilized. The precipitation and flooding that followed in October 2015 delivered particulate and dissolved organic material that was likely of terrestrial origin that may not typically be mobilized during smaller rain events (e.g., January 2016) and that was carrying a relatively high load of PAHs.

The PAH distribution pattern in October 2015 shifted toward PAHs with lower molecular weight (Figure 5) and also indicated mixed sources (Figure 6). These lower molecular weight compounds are relatively more mobile and bioaccessible due to higher water solubility and are capable of exerting an acute narcosis toxicity risk to aquatic organisms (Di Toro and McGrath, 2000). While the mixed-sources signal in October may have included both petroleum and DOC load changed, and a different profile of PAHs was mobilized. The precipitation and flooding that followed in October 2015 delivered particulate and dissolved organic material that was likely of terrestrial origin that may not typically be mobilized during smaller rain events (e.g., January 2016) and that was carrying a relatively high load of PAHs.

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et al., 2017). However, this level of understanding (e.g., outlier determination, or multi-parameter correlations) would require a longer data record and more extensive parameterization than are presently available for Bull Creek. Additional sampling and characterization may also uncover seasonal patterns that need to be accounted for, such as changes in terrestrial carbon and PAH sources and primary in-stream productivity and plankton assemblages (Osburn et al., 2015; Reed et al., 2015). While TSS versus turbidity correlations have been established for tidal creek salt marsh systems in other studies (Suk et al., 1998), it is possible that the impact of urbanization and stormwater complicates this approach, especially in smaller watersheds. This presents an opportunity for further study.

In conclusion, changes in both carbonaceous matter and PAH profile during storm events present a challenge for water quality monitoring in tidal creeks since the dynamics are difficult to capture with routine sampling approaches. These changes are important to understand due to the potential concomitant alteration of contaminant bioavailability and toxicity. Hydrology data collected in these systems, coupled with water quality monitoring results, will provide better data to guide management and regulatory decisions. Discharge conditions for ebb and flood tides in tidal creeks have been shown to be asymmetric (e.g., Ellis et al., this issue), and therefore both the duration and relative discharge of ebb and flood tides need to be accounted for to determine the net flux of materials. An additional aspect to consider is the accuracy of loading models, such as those for total maximum daily load (TMDL) predictions. Future work should aim to explore the parameterization needed to establish rating curves for tracking changes in water quality and contaminant transport in tidal creeks.

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