

# Colloidal Side-Chain Fluorinated Polymer Nanoparticles Are a Significant Source of Polyfluoroalkyl Substance Contamination in Textile Wastewater

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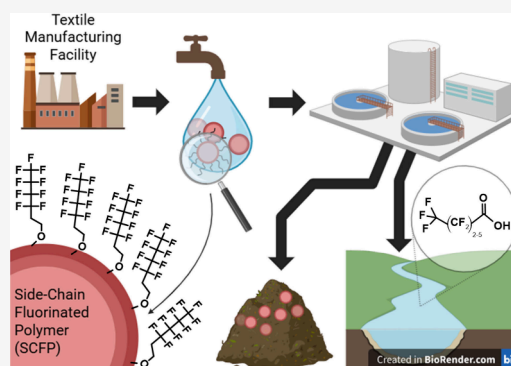
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**ABSTRACT:** Side-chain fluorinated polymers (SCFP) are a class of per- and polyfluoroalkyl substances (PFAS) that are extensively used in functional textiles as water and stain repellents. The potential for environmental contamination through the release of SCFP into aqueous waste streams during textile manufacturing is poorly understood. In this study, SCFP in textile wastewater were characterized using targeted analysis, total oxidizable precursor (TOP) and total hydrolyzable precursor (THP) assays, ultrafiltration, and asymmetric-flow field-flow fractionation (AF4). An investigation into point sources of PFAS precursors in Burlington, NC's wastewater treatment plant (EBWWTP) using the TOP assay revealed a significant PFAS burden stemming from textile manufacturing operations within the city's sewershed (max: 12,000,000 ng/L after oxidation), far outweighing domestic contributions. TOP and THP profiling of textile manufacturing wastewater showed that the dominant precursors contained 6:2 fluorotelomer functionality, and ultrafiltration and AF4 showed them to be colloidal in size (hydrodynamic diameter:  $\sim 100$  nm), resulting in the determination of SCFP in textile wastewater. PFAS mass loading analysis showed that most SCFP exiting the EBWWTP were associated with sludge rather than effluent, where  $\sum$ PFAS concentrations up to 150,000 ng/g were measured using the direct TOP assay; thus, the land application of SCFP-contaminated biosolids may represent a significant route of environmental contamination.

**KEYWORDS:** polyfluoroalkyl substances (PFAS), side-chain fluoropolymers (SCFP), total oxidizable precursor (TOP), total hydrolyzable precursor (THP)



## INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS) have been widely used in textiles since the 1950s to impart water and oil repellancy to performance and functional textiles, including outdoor equipment and firefighting gear.<sup>1–7</sup> Common textile applications of PFAS include expanded polytetrafluoroethylene (e-PTFE) barriers and durable water repellent (DWR) impregnations which incorporate side-chain fluoropolymers (SCFP) into fabrics.<sup>8</sup> The structure of SCFP consists of fluorinated alkyl chains, or PFAS moieties, present as side-chains along a nonfluorinated polymer backbone.<sup>2,3</sup> SCFP in modern DWR formulations predominately utilize perfluoroalkonyl fluoride (PACF), perfluoroalkanesulfonyl fluoride (PASF), or fluorotelomer side-chains ester-linked to acrylate, methacrylate, and urethane polymer backbones,<sup>2</sup> which often exist as copolymers with nonfluorinated polymers.<sup>2,4,9</sup>

Fluorotelomer-based SCFP in functional textiles have been shown to degrade to intermediate PFAS such as fluorotelomer alcohols (FTOHs) and terminal perfluoroalkyl acids (PFAAs) over their lifespan due to a wide range of external influences (e.g. sunlight, abrasion, high temperatures) and laundering.<sup>7</sup> Degradation primarily occurs through scission of the polymer

backbone, releasing oligomeric fragments, and hydrolysis of ester-linked fluorotelomer side-chains to release  $n:2$  FTOHs.<sup>2</sup> Once released,  $n:2$  FTOHs can undergo a series of biologically mediated transformations to yield end points of  $n:3$  fluorotelomer carboxylic acids (FTCA), hydrogen-substituted carboxylic acids (H-PFCAs), and PFAAs of varying alkyl chain lengths,<sup>10–12</sup> which are highly persistent in the environment and in some cases bioaccumulative and toxic.<sup>13–18</sup> There are limited data regarding SCFP degradation rates in the environment, but half-lives on the scale of decades have been reported for 8:2 fluorotelomer acrylate polymers in aerobic soil microcosms.<sup>19</sup> The initial hydrolytic conversion to FTOHs from SCFP in the environment appears to be rate limiting, where a half-life of 55–89 years was estimated for abiotic hydrolysis of an 8:2

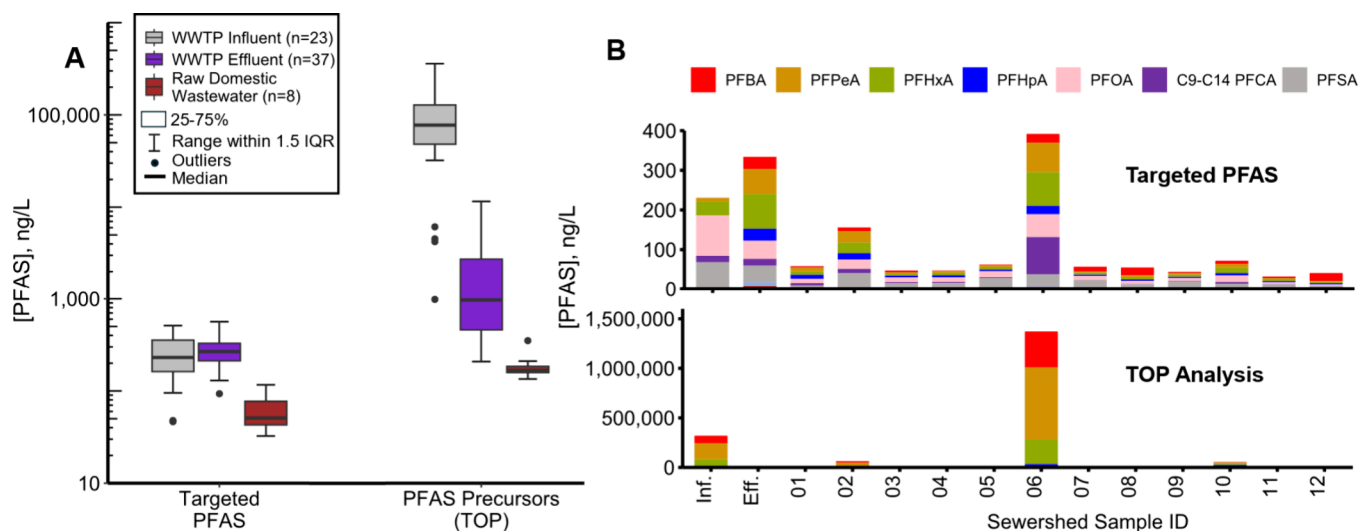
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**Figure 1.** (a) Average PFAS concentrations measured by targeted analysis and TOP assay in EBWWTP influent, effluent, and raw domestic wastewater samples collected between 2020 and 2024 (IQR = interquartile range). (b) PFAS concentrations of a targeted analysis (top) and TOP assay (bottom) of WWTP influent (abbreviated Inf.), effluent (abbreviated Eff.), and sewer samples 1–12, sampled on 6/8/2021. Figure S7 shows a time-series of EBWWTP influent and effluent samples after targeted and TOP analysis.

fluorotelomer acrylate SCFP.<sup>20</sup> In contrast, degradation half-lives spanning days to months have been reported for 6:2 and 8:2 FTOH in soil.<sup>10,21</sup>

The polymeric nature of SCFP often makes them inaccessible to commonly used PFAS measurement techniques such as liquid-chromatography mass-spectrometry (LC-MS), which relies on detection of soluble molecular species. Consequently the fate, occurrence, and exposure of these materials during manufacturing, release, and disposal are relatively understudied. Currently, there are no established methods to determine SCFP in environmental samples. The estimated decades-scale environmental half-life of SCFP warrants the development of sensitive methods to determine their presence in environmental samples.

The state of North Carolina, particularly the Piedmont Triad, has been a center for the textile industry since the 1820s,<sup>22,23</sup> including manufacturers, chemical suppliers, and other industrial facilities that may utilize SCFP in fabric finishing processes. Discharges from these facilities have the potential to introduce SCFP-laden wastewater into municipal systems, with the eventual release of PFAS into downstream surface waters. In 2022, Petr  et al. reported particularly high levels of C<sub>4</sub>–C<sub>8</sub> perfluorocarboxylic acids (PFCAs) in river water several miles downstream of the Burlington, NC, wastewater treatment plant (WWTP), located along the Haw River basin.<sup>24</sup> Total PFAS concentrations measured in water samples collected downstream of Burlington, NC, ranged from 50 to 1000 ng/L,<sup>24</sup> suggesting a significant PFAS burden associated with wastewater discharges from this area. Elevated concentrations of PFAS measured in WWTP effluent compared to influent from the same plant<sup>24</sup> suggested the conversion of unknown PFAS precursors in wastewater to PFCAs, with the precursors likely derived from one or more of the many textile manufacturing operations within the municipality. PFAS precursors are compounds that are reactive and can transform into terminal PFAS such as PFCAs and perfluorosulfonic acids (PFSAs) in the environment. While some PFAS precursors are measurable, there are thousands that are not due to a lack of neat analytical standards, unknown chemical structure, or incompatibility with commonly used analytical methods.<sup>3,25</sup> This may result in an

underestimation of the total PFAS burden in systems where precursors are present.

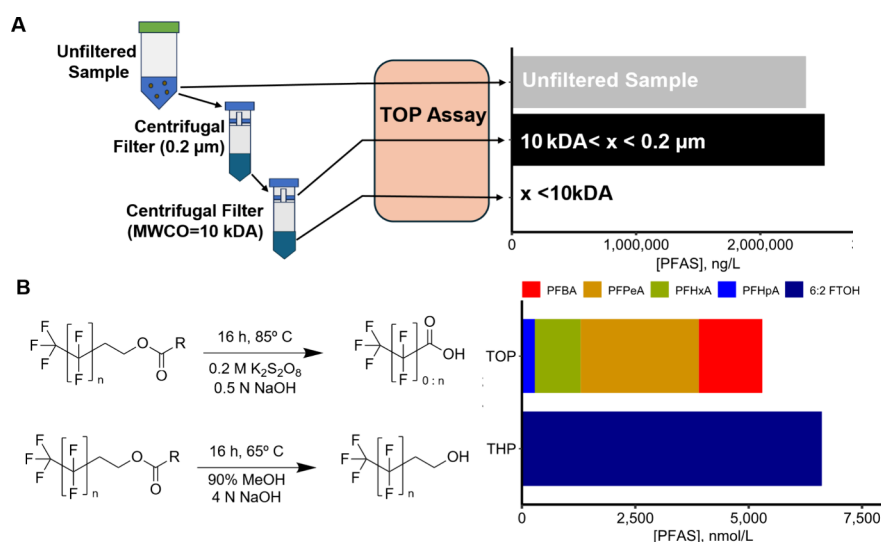
Here, we present findings of a comprehensive source-tracing study to investigate elevated concentrations of PFAS measured in wastewater and drinking water sources downstream of textile industries in central North Carolina. We hypothesized that SCFP-related precursors released into textile wastewater contribute significant PFAS burdens to the WWTP. Experiments were designed to trace, identify, and quantify the relevant PFAS precursors. PFAS precursors were characterized according to size using ultrafiltration (UF) and asymmetric-flow field-flow fractionation (AF4) coupled to the total oxidizable precursor (TOP) assay, marking a novel use of these techniques for PFAS measurement and a functional method to measure SCFP in environmental samples.

## MATERIALS AND METHODS

**Materials.** Authentic standards of all measured PFAS, isotopically labeled standards for use as internal standards (IS), other materials, and solvents are described in Supporting Information Section S1A and Table S1.

**Sample Collection.** Sampling was conducted over the period of November 2019 to August 2024 from designated points within the sanitary sewer system of Burlington, NC (Figure S1). Samples included composite and grabs of raw wastewater from textile industries, trunk-line sampling of combined raw wastewater, WWTP influent and treated effluent, domestic wastewater influent devoid of industrial sources, primary sludge, and biosolids. Additional sample collection details are provided in SI Section S1B.

**PFAS Precursor Assays.** A modified version of the TOP assay was applied to all samples in this work to screen for the presence of PFAS precursors.<sup>26,27</sup> Solid-phase samples (e.g., sludge and biosolids) were screened for PFAS precursors using a modified direct-TOP (dTOP) assay. In some cases, the total hydrolyzable precursor (THP) assay was performed alongside TOP to further inform the presence and identity of PFAS precursors in aqueous samples.<sup>28</sup> Detailed methods are provided in SI Sections S1E–S1F.



**Figure 2.** (a) TOP analysis of textile wastewater after size fractionation by ultrafiltration revealed PFAS precursors were retained on a 10 kDa MWCO filter but passed through a 0.2 μm filter. (b) Reaction schemes for the TOP assay and THP. Application of these assays on wastewater from Textile Manufacturer A yielded a 1:1 molar conversion of the 6:2 FT-based precursors to their end points, validating the identity of the side-chains.

**PFAS Nanoparticle Size Fractionation.** Particulate PFAS precursors in textile wastewater were fractionated by size using ultrafiltration and asymmetric-flow field-flow fractionation (AF4). Additional information regarding ultrafiltration and AF4 methods and parameters is described in SI Sections S1G and S1H, respectively.

**Data Acquisition and Processing.** PFAS analysis was performed using an ultrahigh-performance liquid chromatograph (UHPLC, Vanquish Flex, ThermoFisher Scientific, San Jose, CA), coupled with a triple quadrupole mass spectrometer (TSQ Altis, ThermoFisher Scientific, San Jose, CA). Data was acquired using multiple reaction monitoring (MRM), and analytes were quantified over a calibration range of 1–1000 ng/L. Full method details, including nontargeted analysis, are provided in SI Sections S1J–S1K. QA/QC measures are described in SI Section S2, with results shown in SI Section S4.

## RESULTS

**Point Source Investigation and Initial PFAS Precursor Characterization.** Targeted analysis of effluent from the East Burlington WWTP (EBWWTP) during 2019–2020 yielded ΣPFAS concentrations up to 33,000 ng/L, significantly higher than concurrent measurements of influent (2,500 ng/L, Figure S6). Zimpro wet oxidation had been utilized in the EBWWTP prior to May 2020 to reduce the production of biosolids from the plant. Measured PFAS levels in Burlington’s effluent declined dramatically after May 2020, suggesting that the influent contained unknown PFAS precursors and that the Zimpro process may have converted them to detectable PFAS, which were then released in WWTP effluent. These results are consistent with previous studies showing that PFAS precursors can degrade to PFAAs through advanced oxidation processes (AOPs) used in wastewater treatment.<sup>29–31</sup> Additionally, targeted ΣPFAS concentrations of ~850 ng/L were measured in the town of Pittsboro’s drinking water, downstream of the EBWWTP, by our laboratory in May 2019 (Figure S7). The PFAS detected in Pittsboro’s drinking water were dominated by C<sub>4</sub>–C<sub>7</sub> PFCAs, consistent with the PFAS composition detected in EBWWTP effluent, further suggesting that discharge from

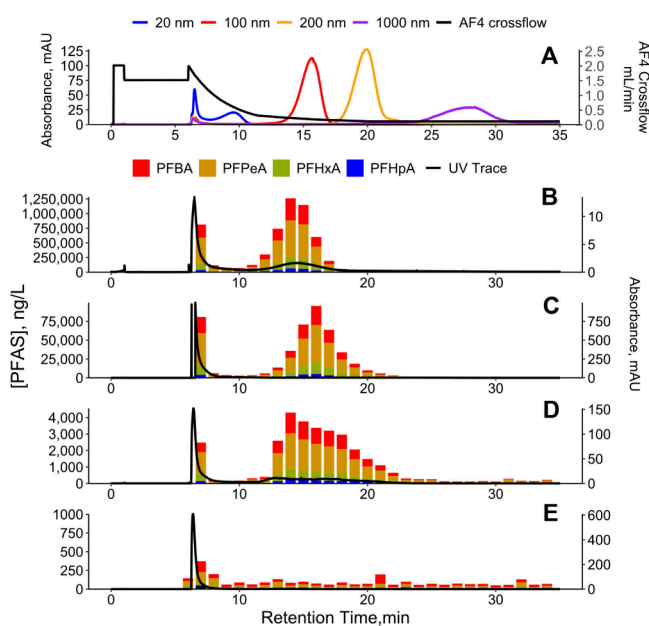
EBWWTP could be a significant source of PFAS in the Haw River.

Targeted analysis of EBWWTP influent and effluent samples over a period of several months between late 2020 and 2024 ( $n = 21$  influent,  $n = 36$  effluent; Figure 1A) yielded mean ΣPFAS concentrations of 260 (±140) and 270 (±90) ng/L, respectively. Effluent PFAS concentrations measured during this time period were insufficient to explain the magnitude of PFAS contamination observed in Pittsboro drinking water. Following TOP analysis, mean ΣPFAS concentrations increased to 99,525 (±92,416) ng/L in influent samples and 2715 (±2784) ng/L in effluent samples (Figure 1A), representing a 383- and 7.5-fold increase in measurable PFCAs, much higher than similar measurements reported for other WWTPs across the United States ( $n = 38$ ), where TOP analysis led to only 2- to 3-fold increases in measurable PFCAs.<sup>32</sup> Application of the TOP assay to WWTP influent from domestic sources within Burlington ( $n = 8$ , Figure 1A) yielded only a 3-fold increase in measurable PFCAs, indicating that industrial discharges were likely the primary source of PFAS precursors in this sewershed. Our results illustrate that reliance on targeted analysis alone would lead to significant underestimation of total PFAS loadings on wastewater in this case.

Further investigation was conducted in 12 strategically chosen sewersheds upstream of the EBWWTP to isolate the sources of PFAS precursors using targeted and TOP analysis. A point-source of PFAS precursor emission, hereafter named “Textile Manufacturer A”, was identified after the TOP assay yielded a ΣPFAS concentration of 1,370,000 ng/L in sewershed 6 (Figure 1B). The TOP profile of PFAS precursors in Textile Manufacturer A wastewater and EBWWTP influent (Figure 2B) was dominated by C<sub>4</sub>–C<sub>6</sub> PFCAs, consistent with a 6:2 fluorotelomer (FT) based precursor oxidation profile,<sup>26,33</sup> suggesting the primary PFAS precursors in EBWWTP influent were 6:2 FT-based precursors. Nontargeted analysis and suspect screening of EBWWTP and Textile Manufacturer A wastewater samples using high-resolution LC-MS failed to identify abundant molecular PFAS precursors that could explain the large concentration discrepancy between targeted and TOP analysis. We hypothesized that the precursors were not

amenable to electrospray ionization and mass spectrometry analysis due to their high molecular weight. Ultrafiltration of Textile Manufacturer A wastewater (Figure 2A) revealed that the PFAS precursors were colloidal in nature, as they were quantitatively retained by a 10 kDa molecular weight cutoff (MWCO) membrane but passed through a 0.2  $\mu\text{m}$  filter. Based on this result, we hypothesized that the unknown precursors were likely SCFP with fluorotelomer functionality ester-bonded to an acrylate or urethane polymer backbone. Application of the THP assay to Textile Manufacturer A wastewater (Figure 2B) yielded quantitative generation of 6:2 FTOH, clearly verifying the presence of carboxy ester-functionalized fluorotelomer side-chains in the precursors. Additionally, comparing THP results to TOP (Figure 2B) showed that 6:2 FT-based, carboxy ester-functionalized SCFP was the dominant PFAS in wastewaters. These findings demonstrate that the combination of TOP and THP can be advantageous for characterizing total PFAS precursor concentration and composition in environmental media, particularly in cases (e.g., for SCFP) where targeted and nontargeted analysis fails to yield informative results.

**Size Separation and Analysis of Colloidal PFAS Precursors from a Wastewater Matrix.** Following the results observed in ultrafiltration size-separation experiments, we used AF4 fractionation coupled to the TOP assay of collected fractions to characterize size distributions of SCFP in textile industry-impacted wastewaters. Comparing the size distribution and TOP profile of fractionated wastewaters with polystyrene size standard separation profiles (Figure 3A) demonstrated a



**Figure 3.** AF4-TOP fractograms detailing (a) cross-flow parameters and size standard separation profiles and (b) the size distributions of colloidal PFAS precursors of a commercially obtained 6:2 fluorotelomer SCFP and wastewaters from Burlington, NC, including (c) textile wastewater, (d) EBWWTP influent, and (e) effluent.

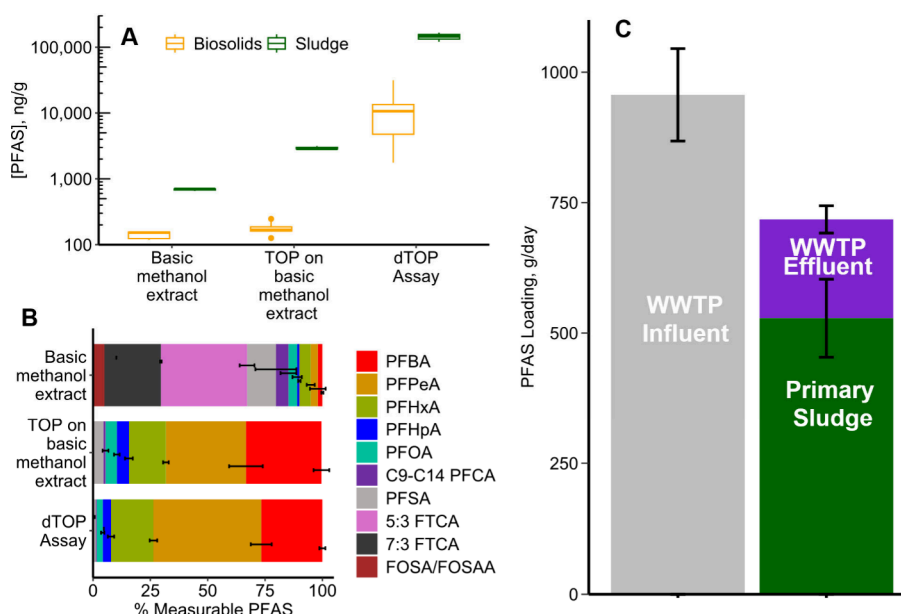
mean size of  $\sim 100$  nm for SCFP in wastewater from Textile Manufacturer A (Figure 3C), which aligned well with size distribution measured for a sample of commercial 6:2 fluorotelomer-based SCFP textile treatment formulation (Figure 3B). In WWTP influent, the majority of PFAS precursor mass (60%) eluted close to the 100 nm size standard (Figure 3D), further evidencing that Textile Manufacturer A was the

source of SCFP in EBWWTP. However,  $\sim 30\%$  of the SCFP particle size distribution in WWTP influent was eluted in a region consistent with a size of  $\sim 200$  nm. This suggests that SCFP may have agglomerated with suspended solids or organic matter in the wastewater distribution system prior to entering the EBWWTP. The AF4-TOP elution profile for WWTP effluent (Figure 3E) was similar to those of negative control samples containing no PFAS precursors (Figure S5), suggesting that most SCFP was removed during conventional wastewater treatment via agglomeration and partitioning to sludge. Additional discussion of the AF4 results can be found in Section S3D. Further discussion of the applicability of UF, AF4, and TOP analysis to other types of SCFP (e.g., those with PACF and PASF side-chain moieties) is presented in SI Section S3F.

**SCFP from Textile Manufacturing Wastewater Becomes Incorporated into Sludge during Wastewater Treatment.** Our observation of SCFP agglomeration within wastewater prompted an evaluation of PFAS precursor extractability from sewage sludge and biosolids from EBWWTP. Solvent extraction with methanolic  $\text{NH}_4\text{OH}$  revealed intermediate fluorotelomer degradation products such as 5:3 and 7:3 FTCA present in biosolids and sludge (Figure 4B);<sup>10,11</sup> however, comparison of solvent-extractable PFAS levels with those generated by dTOP of WWTP sludge and biosolids (Figure 4A) revealed that sludge-associated SCFP were not solvent-extractable but yielded high PFAS concentrations after direct oxidation. A detailed discussion of these results is presented in SI Section S3E. Analysis of PFAS precursor mass loadings within EBWWTP showed that the majority of SCFP were removed to sludge during wastewater treatment (Figure 4C). SCFP loadings in EBWWTP sludge were approximately 3-fold higher than in effluent, whereas previous mass loading studies of other WWTP showed that effluent loadings were approximately double those in sludge.<sup>32</sup>

Agglomeration of colloidal SCFP during wastewater treatment and incorporation into sewage sludge have important implications for the fate of these PFAS precursors. Biosolids contaminated with PFAS have been shown to contaminate nearby surface and groundwater systems when land-applied.<sup>34–36</sup> Degradation of SCFP in soils to form molecular PFAS could contribute to atmospheric release of FTOHs<sup>20</sup> and uptake of PFAS by plants and biota in soils.<sup>37–39</sup> In the current scenario, application of EBWWTP biosolids near Cane Creek Reservoir, the drinking water supply for Chapel Hill, NC, has historically resulted in targeted  $\Sigma$ PFAS concentrations as high as 2,000 ng/L downstream of the application fields.<sup>40</sup> Crucially, the use of Zimpro wet oxidation at EBWWTP, applied prior to May 2020, actively converted sludge-associated SCFP into PFAS in the effluent (Figure S4), contaminating drinking water supplies for downstream communities, including Pittsboro (Figure S5). This finding indicates the use of Zimpro was a direct driver of environmental PFAS contamination in the Haw River, highlighting that AOPs may be a major pathway for precursor transformation and PFAS release.

This study is the first report documenting the release to the environment of nanoparticulate, colloidal PFAS precursors derived from textile manufacturing. Our results reveal a significant analytical gap created by the failure to detect SCFP by targeted analysis alone, and in this case, PFAS loadings to the EBWWTP would have been underestimated by 2–3 orders of magnitude by relying solely on targeted analysis. We have demonstrated that colloidal SCFP from textile manufacturing becomes incorporated into sludge during wastewater treatment.



**Figure 4.** (a) Average PFAS concentrations and (b) their molar compositions measured through a basic-methanol extraction, application of the TOP assay on a basic-methanol extract, and a direct TOP assay on WWTP sludge and biosolids revealed PFAS precursors to be agglomerated to sludge and biosolids. (c) A mass balance of WWTP influent, effluent, and primary sludge shows that SCFP entering the WWTP is removed to sludge. PFAS extraction recoveries for the basic methanol extraction are listed in Table S10.

As such, early identification of SCFP and other particulate PFAS precursors in industrial wastewater prior to discharge to WWTPs could prevent the release of contaminated biosolids and effluent from WWTPs. More work is needed to assess the fate, transport, and transformation of colloidal SCFP following its release to soil and surface waters. This is particularly the case for current-use, 6:2 fluorotelomer-based SCFP, as previous research has focused on 8:2 fluorotelomer-based SCFP.<sup>19,41,42</sup> In the particular case of textile-derived SCFP contamination of Burlington, NC, wastewater, the city used its pretreatment authority under the Clean Water Act to require industrial dischargers within the sewershed to reduce or eliminate the SCFP discharges we discovered before they entered the EBWWTP. As a result, phase-out of PFAS by Textile Manufacturer A led to a sharp decline in PFAS precursors in raw wastewater at the EBWWTP (Figure S8), with a corresponding decrease of PFAS release to the Haw River.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.estlett.5c01014>.

Additional sampling details, materials, experimental details, targeted and nontargeted instrument methods, UHPLC and AF4 separation parameters, quality control measurements and mass balances with data tables, discussion of solvent extraction, AF4 results, and applicability of methods to other SCFP, Pittsboro water quality data, and EBWWTP time-series data (PDF)

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## Notes

The authors declare no competing financial interest.

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