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Considerations for emission monitoring and liner analysis of thermally manufactured sewer cured-in-place-pipes (CIPP)

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ABSTRACT

Cured-in-place-pipes (CIPP) are plastic liners chemically manufactured inside existing damaged sewer pipes. They are gaining popularity in North America, Africa, Asia, Europe, and Oceania. Volatile and semi-volatile organic compound (VOC/SVOC) emissions from storm sewer CIPP installations were investigated at a dedicated outdoor research site. Tedlar bag, sorbent tube, and photoionization detector (PID) air sampling was conducted for five steam-CIPP installations and was coupled with composite characterizations. New CIPPs contained up to 2.21 wt% volatile material and only 6–31% chemical mass extracted per CIPP was identified. Each 6.1 m [20 ft] liner contained an estimated 5–10 kg [11–22 lbs] of residual chemical. Extracted chemicals included hazardous air pollutants and suspected and known carcinogens that were not reported by others. These included monomers, monomer oxidation products, antioxidants, initiator degradation products, and a plasticizer. PID signals did not accurately represent styrene air concentration differing sometimes by 10s- to 1000s-fold. Multiple VOCs found in air samples likely affected PID responses. Styrene (> 86.4 ppm$_v$) and methylene chloride (> 1.56 ppm$_v$) air concentrations were likely greater onsite and phenol was also detected. Additional studies are needed to examine pollutant emissions so process monitoring can be improved, and environment impacts and associated human exposures can be minimized.

1. Introduction

In the U.S., more than 2 million kilometers [1.3 million miles] of sewer pipes require repair or replacement, and many were installed after World War II [1,2]. Sewer pipe failures can endanger public health and safety by enabling explosions, flooding, and roadway collapses. Instead of open-trench pipe replacement, the popular cured-in-place-pipe (CIPP) technology is being used for repairs. CIPPs are new plastic liners manufactured inside existing damaged host pipes or culverts. The manufacturing process requires a limited construction site footprint, can avoid roadway shutdowns, and sometimes the damaged pipe is only out-of-service for a few hours. The U.S. is predicted to represent roughly 40% of the $2.6 billion-dollar market by 2023 [2]. This technology is gaining popularity across the rest of the world [2].

Health concerns with CIPP manufacture have been expressed by the public [3], U.S. federal and state agencies [4–7], and organizations outside the U.S. [8–11]. During CIPP manufacture, a flexible uncured resin tube containing raw chemicals is inserted into the damaged culvert. Initiators, felt, plastic films and coatings, fillers, and reinforcements may also be in the tube. Once inserted, polymerization is expedited by hot water, steam, or ultraviolet (UV) light exposure. Next, the ends of the hardened plastic are mechanically removed and the new CIPP is placed into service. At present, the steam method is the most popular U.S. CIPP installation practice [2]. Styrene-based resins, such as polyester and vinyl ester, are the most popular due to their low cost, but non-styrene resins are also used [12,13].

More than 100 air contamination incidents have been associated with CIPP manufacturing sites (Table SM-1), and little information exists regarding the chemicals emitted and their fate. In 2018, CIPP related chemical exposure contributed to a worker fatality where blood

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styrene levels indicated a 220–270 ppm_v exposure [7–14]. In 2017, the California Department of Health issued public safety warnings [4,5]. The U.S. Agency for Toxic Substances and Disease Registry (ATSDR) [6] previously declared that a CIPP installation caused an indoor air pollution 'public health hazard'. Several emission control and monitoring recommendations issued in the Netherlands implied (1) styrene was the only compound of concern, (2) monitoring should include a photoionization detector (PID), and (3) a fan should be installed on manholes that can move thousands of m^3 air/hr during and for at least 24 h after CIPP installation [15]. In 2019, the U.S. National Institute for Occupational Safety and Health (NIOSH) found 140 ppm_v styrene at a CIPP manufacturing site exceeded the 15-min short-term exposure limit of 100 ppm_v [16]. In response, the federal agency recommended that workers ventilate manholes, bag excess liner immediately, and change gloves regularly when they contact resin. Also previously reported was that styrene concentration remained unchanged 1 km down a sanitary sewer [17], and chemicals traveled "kilometers from the worksite" aboveground [18].

Existing chemical identification and air concentration data are lacking. The few air monitoring studies have focused on worker exposure, only styrene, and assumed that PID signals accurately represented styrene air concentration (Table 1). Styrene is sometimes used for CIPP manufacture, is a hazardous air pollutant (HAP), and is reasonably anticipated to be a carcinogen [19,20]. Other VOCs and SVOCs including HAPs, endocrine disrupting compounds, and carcinogens have recently been found emitted into air [3]. To estimate VOC and SVOC contributions to urban and residential air pollution, additional chemical identification and quantification data are needed.

To better understand what chemicals may be emitted into the air, CIPP water contamination studies were reviewed. Li et al. [32] confirmed and quantified the loading of 19 chemicals in a styrene-based uncured resin tube used for a UV CIPP installation; only 4 chemicals were reported on the product safety data sheets (SDS). The manufactured UV CIPPs contained more than 30–70+ unidentified compounds including HAPs, carcinogens, and endocrine disrupting compounds. Ra et al. [33] reported that initiators previously used for CIPP manufacture could degrade into 32 different compounds, including VOCs. Also, 13 storm water, lake, river, or creek water contamination incidents were found. Several storm water studies focused on styrene release [34–39], but VOCs and SVOCs have also been released into waters [39–42]. Except for Li et al. [32] no studies were found that examined chemical residual loading inside new CIPPs, and CIPPs can emit chemicals into the air after they are installed [15,16]. To predict chemical air concentrations, the amount of chemical in the CIPP should be estimated.

This study was conducted to better understand VOC and SVOC emissions from CIPP storm sewer installations and chemical residual in new CIPPs. Specific objectives were to: (1) capture and detect chemicals in air using real-time and grab sample supported sampling, (2) chemically identify compounds that can be extracted from new CIPPs, and (3) characterize new CIPP physical and thermal characteristics. While this study was conducted, two parallel studies were conducted for the same installations. Teimouri et al. [3] captured and analyzed condensed materials from two fixed chemical emission points, but did not measure concentrations in air, chemical loading in new CIPPs, or the CIPP final properties. Currier [42] evaluated short-term chemical release into water by the new CIPPs, but did not consider the array of chemicals in the resin, residual left in the new CIPPs, and differences between degree of cure, or manufacturing conditions.

2. Materials

In August 2016, five CIPPs were installed in corrugated steel pipes (CSP) [pipes [1,3–5]] and a reinforced concrete pipe (RCP) [pipe 2] in California, USA. CSPs were buried in parallel at the California State University at Sacramento outdoor research site on campus. Air samples were collected at fixed locations and as the authors moved around the worksite. Stainless-steel manifolds were used to capture materials emitted into the air at two fixed locations referred to as the exhaust emission point and fugitive emission points (Fig. SM1 and SM2). At these locations, air was sampled using PIDs and Tedlar bags. Sorbent tubes were only used for fugitive emission point sampling because of the high amount of liquid discharged at the exhaust emission point. Area chemical monitoring inside the worksite was also conducted at breathing zone level using sorbent tubes to understand the type, intensity, and spatiotemporal variability of emissions.

Two air sampling teams each carried a PID, and the team that monitored the exhaust emission point wore a sorbent tube along their lapel. All sorbent tubes (worn and located at fixed monitoring locations) were replaced every 30 min. A detailed description of field and laboratory methods can be found in the Supplemental Material section (SM): analytical standards, PID devices and calibration, procedures for sample preparation, Tedlar bag and sorbent tube procedures, methylene chloride and hexane solvent extractions, and gas chromatography/mass spectrometry (GC/MS) methods. Thermal and chemical properties were determined using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).

2.1. Analytical standards, installation conditions, and materials

A list of the analytical standards used can be found in the SM. CIPP installation conditions were pre-determined and overseen by Currier [42]. All CIPPs were manufactured using the steam curing process by the same contractor. The steam temperature and monitoring location was not reported, but steam temperature readings were listed on the contractor's post-installation paperwork and indicated recordings every 5 min. Contractors also recorded the temperature between the host pipe and uncured resin tube every 5 min, at the very end of the host pipe. This temperature was referred to as the interfacial temperature.

The contractor's submittal indicated that CIPPs were to be manufactured using either a styrene-based resin or non-styrene-based resin. Each CIPP was designed with polyethylene terephthalate (PET) polyester staple fiber felt, outer layer of 17 mil (0.4318 mm) thermoplastic polyurethane coating on the felt's interior [43], as well as a polymer sealing tape on top of the outer layer. Pre-liners were inserted into pipes 1, 3, and 4 before the uncured resin tubes were inserted. Component composition, where available, can be found in the SM.

Some installation conditions did not match the contractor's submittal and video monitoring helped identify these differences (SM). A 34.5 kPa [5 psi] pressure was recorded by contractors for resin tube expansion, while 40.7 kPa [5.9 psi] was recommended in the submittal. Steam exposure time for all CIPPs described in the submittal was listed as 1 h, but exposures occurred for at least 1.5 h (Table 2). Volumetric airflow rates into and exiting the uncured resin tubes were not reported or monitored. The authors attempted to use a vane anemometer, but force from the exhaust emission point sheared off a stainless-steel blade.

Contractors cut solid specimens from the end of each CIPP. Samples were stored in sealed bags at 4 °C until analysis. Teimouri et al. [3] described chemical characterization results for the uncured resin tubes for each CIPP.

2.2. Air monitoring

Air monitoring utilized a combination of sorbent tubes (Markes International, Inc., Sacramento, CA) packed with quartz wool, Tenax TA and Carbograph 5 TD and a low flow pump (ACTI-VOC) (Markes International, Inc.), Tedlar bags (1 L, Restek, Inc., Bellefonte, PA), and a PID connected to the stainless-steel manifold (Fig. SM2). A prior study by Teimouri et al. [3] described the chemical composition of condensed materials which were removed from the manifold before air sampling at the manifold fugitive emission point. Their absence likely affected the present study's follow-up air sampling and PID, Tedlar bag, and sorbent

Table 1
Comparison of Chemical Air Monitoring Studies Found for CIPPs.

Study	Project Details			Monitoring and Analysis Methods		Observations	Ref
	Country	CIPPs	Method	Resin			
2019	USA	4	UV	Styrene	Field-test: PID; FTIR; evacuated canisters Bench-test: sorbent tubes, evacuated canisters	Styrene exceeded NIOSH exposure limit of 100 ppm for 15 min at 140 ppm_v; Only examined styrene in air; Ventilation blowers seemed to reduce air concentrations; Styrene was emitted into air from the manufactured liner in the lab (5100 ppm_v); Worker safety recommendations provided.	[16]
2018	USA	3	Steam	Styrene	PID; Charcoal sorbent tubes with GC/FID	Odors prominent within 10 ft surrounding the sources and up to 200 ft downwind. PID Upwind 0.25–269 ppm_v vs. PID Downwind 0–4 ppm_v; max: 1050 ppm_v Sorbent Tube: $<$ 0.011 to 6.32 ppm_v. Used only SDS to determine what chemicals to test for.	[21]
2018	NZL	1	Steam	Styrene	PID	Only PID, instrument calibration and environmental conditions not reported; Max. 12 ppm_v reported.	[22,23]
2018	USA	2	Hot Water	nr	PID; Passive badges with GC/FID	PID signals: Inside ($<$ 20 ppm) and outside ($>$ 20 ppm) manholes; During site cleanup ($>$ 20 ppm); Detected max. 167 ppm_v and $>$ 100 ppm_v for more than 15 min; Badges: 58 ppm_v styrene for 1 installation	[24]
2017	USA	2	Steam	nr	PID; Passive badges with GC/FID	PID signal: Ma × 16.5 ppm and max. 104 ppm Badges: $\leq$ 0.4 ppm_v styrene	[25]
2017	USA	7	Steam	Styrene, non-styrene	PID with humidity prefilter; Condensed material capture with GC/MS	A multi-phase mixture of solids, liquids and gases was emitted into air; multiple VOCs and SVOCs, not just styrene detected; styrene emitted during a non-styrene CIPP manufacture; PID: Max. (6321 ppm_v) styrene CIPP; non-styrene CIPP (9.6 ppm_v); mouse lung cell toxicity detected for some condensed materials; Did not report air concentrations; Site safety recommendations provided	[3]
2016	USA	3	Steam	Styrene	Tedlar bag with GC/MS; Velocimeter for flowrate	During steam curing (250–1070 ppm_v styrene) and cool down (3.6–76.7 ppm_v styrene); Flowrate exiting the exhaust pipe $>$ 240 m^3/hr	[26]
2007	USA	1	nr	nr	Passive monitors with GC/FID	Only 1 sample reported, styrene only reported, and construction practices not reported; Max. styrene level of 9.955 ppm_v next to end of pipe	[27]
2006	NED	1	Hot Water	nr	PID; Anemometer for flowrate	Flowrate was estimated to be 6000 m^3/hr; PID only applied with a max. signal of 87 ppm; chemicals detected not confirmed	[28]
2006	NED	3	Hot Water	nr	PID; Passive samplers	Method description and results unclear; Ventilation recommended.	[29]
2005	NED	1	Hot Water	Styrene	PID; Portable GC/PID; Portable IR	Method description and results unclear; 1 km downstream in the sewer no decrease in styrene concentration was found.	[17]
2004	DEU	24	Steam, Hot Water, UV	nr	nr	Method description and results unclear; Air testing results not reported; Recommended max. 400 mg/kg of styrene in pilot experiments should remain in new CIPP, and 500 mg/kg for pipes $<$ 61 cm [24 in. diameter and 1000 mg/kg for pipes at and larger than 61 cm [24 in. diameter; Site safety recommendations provided.	[a]
nr	DEU	2	UV	nr	Charcoal sorbent tubes with GC (detector not reported)	Sorbent: Max. 0.008 ppm_v styrene; Method description and results unclear	[a]
nr	DEU	1	Steam	nr	Draeger tubes	Method description and results unclear; 10 ppm_v detected 20 m away; 0 ppm_v Draeger tube detection limit; Of 32 samples, five $>$ 10 ppm_v; Max. 20 ppm_v styrene concentration reported	[a]
nr	UK	4	Steam, Hot Water	nr	PID; Sorbent tubes with GC	Method description and results unclear; Media and detector not reported; 6 ppm_v about 1 m away from the manhole; Max. reported 165 ppm_v styrene in manhole.	[a]
2004	CAN	2	Steam	Styrene	PID	PID only applied with a max. signal of 110 ppm_v	[18]
2001	CAN	nr	Hot Water	nr	Charcoal sorbent tubes with GC/MS	Installation among other details not reported; Sorbent: Max. 3.2 ppm_v styrene	[31]

nr = information not reported.

[a] Mentioned by Najafi et al. [30], but reports cited were not made available when requested from Najafi et al. and IKT and they were not found for review.

Table 2
Installation Conditions.

Pipe	Host pipe (L/D-m/cm)	Pre-liners Used	Resin Type	Steam Exposure Duration, min	Cool down Method, Duration in min
1	CSP (6/45.7)	Yes, 1	L713	92	Ambient Air, 35**
2	CSP (6/48.2)	No, 0	EcoTek	111	None
3	CSP (6/45.7)	Yes, 2	L713	107	Hot Air, 60**
4	RCP (6/45.7)	Yes, 1	L713	100	None
5	CSP (6/45.7)	No, 0	L713	104	None

According to the contractor's submittal all L713-LTA (styrene-based resin) and EcoTek (non-styrene based resin).

** An asterix corresponds to the contractor's reported cool down time.

tube results. For the exhaust emission point, high velocity emissions prevented air sampling device connections. Therefore, at the exhaust emission point, Tedlar bag air samples were captured next to, not directly above, the exhaust outlet at the same time fugitive emissions were captured. Sampling with sorbent tubes and Tedlar bags began once the steam was injected into the uncured resin tube: at the start of the CIPP curing (every 5 min) and every 10 and 20 min as steam injection continued. Methods for sampling pump calibration, flows, equipment cleaning, determining analyte adsorption onto the walls of new Tedlar bags, and analytical methods for air sample analysis are described in the SM.

2.3. CIPP characterization

CIPPs were visually, physically, thermally, and chemically examined. Sample thickness and bulk density were measured and compared against the contractor submittal and literature values. Wall thickness was measured using a Mitutoyo absolute digital caliper. The bulk density mean and standard deviation value for each CIPP was calculated by using the sample mass of three replicate cubes (6 mm × 7 mm × 8 mm).

CIPP thermal stability, volatile content, and residue content were determined using TGA (Q-500, TA Instruments, Inc., New Castle, DE). Detailed analytical methods are described in the SM. A Q-2000 DSC (TA Instruments Inc., New Castle, DE) was used to determine if uncured resin remained in the CIPPs. Sample weight was approximately 10 mg in aluminum pans. Scans were performed at 10 °C/min from −25 °C to 200 °C.

CIPP extracts were also analyzed using ^{1}H NMR and GC/MS. For ^{1}H NMR, CIPP was extracted with deuterated chloroform ($CDCl_3$) at room temperature. For GC/MS analysis, CIPP samples were also placed in methylene chloride and hexane at room temperature. Details for extraction, ^{1}H NMR and GC/MS analysis methods are described in the SM.

3. Results and discussion

3.1. Chemical detection at fixed monitoring points and personal exposures

Multiple VOCs were emitted into air during all CIPP installations. At both fixed emission points, styrene and methylene chloride concentrations were determined (Table 3). Both compounds are "reasonably anticipated to be human carcinogens" [19–44], and have been associated with CIPP water contamination incidents and studies [33–40]. The greatest air concentrations were not always found at the start of steam injection (Fig. 1). Styrene was detected for all CIPP installations, whereas methylene chloride was only found at styrene-based resin CIPP pipes 3, 4, and 5 (Table 3). The sample with the maximum styrene level (> 86.4 ppm_v) was greater than 7 days old when analyzed. A styrene adsorption experiment onto the walls of new Tedlar bags showed 31 ppm_v styrene was reduced by 72.8 ± 5.1% during 7 days storage at room temperature (SM). Therefore, Tedlar bag results likely underestimated the total number and magnitude of chemicals emitted. The minimum styrene concentration (> 2.35 ppm_v) was found during the non-styrene resin CIPP installation. Styrene levels were less than what OSHA estimated [220–270 ppm_v] [7], NIOSH found [140 ppm_v] [16], and a maximum reported by others [1,070 ppm_v] [26]. Multiple compounds detected on sorbent tubes have been associated with prior CIPP water contamination incidents. Compounds found on the sorbent tubes included: acetaldehyde, acetophenone, benzaldehyde, benzene, benzoic acid, (1-methylethenyl)-benzene, nonanal, 1-pentanol, phenol, styrene, and 1-tetradecanol (Table SM1).

Table 3
The maximum styrene and methylene chloride concentration detected in Tedlar bag samples for each CIPP installation.

Pipe	Methylene Chloride, ppm_v		Styrene, ppm_v	
	Fugitive Emission Point	Exhaust Emission Point	Fugitive Emission Point	Exhaust Emission Point
1	–	nc	86.4^{8d} (> 317)	nc
2	–	–	2.35^{2d} (> 4)	< MRL^{7d}
3	–	1.48^{3d}	3.31^{2d} (> 5)	86.5^{3d} (> 236)
4	–	1.46^{3d}	23.3^{2d} (> 36)	3.88^{3d} (> 11)
5	1.41^{2d}	1.56^{2d}	45.2^{2d} (> 69)	36.3^{2d} (> 56)

The minimum reporting limit (MRL) for styrene was 0.88 ppm_v and for methylene chloride was 1.29 ppm_v; Superscripted numbers indicate the age (days old) of the Tedlar bag sample when analyzed; (-) Not detected; nc = No Tedlar bag samples were collected at the site I exhaust emission point. Styrene results in parentheses are estimated levels based on the styrene adsorption experiment onto the walls of new Tedlar bags.

Phenol and *N,N*-dimethylacetamide were confirmed in Tedlar bag samples from all sites, and were not found in control samples (Tedlar bags with UHP nitrogen). While these compounds have been previously attributed to Tedlar bag artifacts [39,40], Teimouri et al. [3] found phenol in the condensed materials (captured in glass, not Tedlar bags) from the same CIPP installations. Currier [42] detected phenol in water from the same CIPPs and did not use Tedlar bags. Therefore, phenol was emitted and is also a HAP that has been associated with CIPP water contamination [33]. *N,N*-Dimethylacetamide may have been a Tedlar bag artifact.

PID signals did not accurately represent styrene concentration (Fig. 1). PIDs were equipped with humidity filters to remove water vapor and protect the PID lamp. The PID signal and corresponding Tedlar bag styrene concentration sometimes differed by 10- to 1000s-fold, possibly due to (1) analyte adsorption onto Tedlar bag walls before GC/MS analysis, (2) differences in sample characteristics: time-integrated Tedlar bag result vs. single PID spot measurement, and (3) other unknown factors. PIDs may have responded to other organic compounds collected on sorbent tubes: acetaldehyde, acetone, benzene, methylene chloride, and phenol [45]. Interferents can prompt PID signals to underestimate and overestimate organic vapor concentrations and can be affected by the calibration environment and humidity

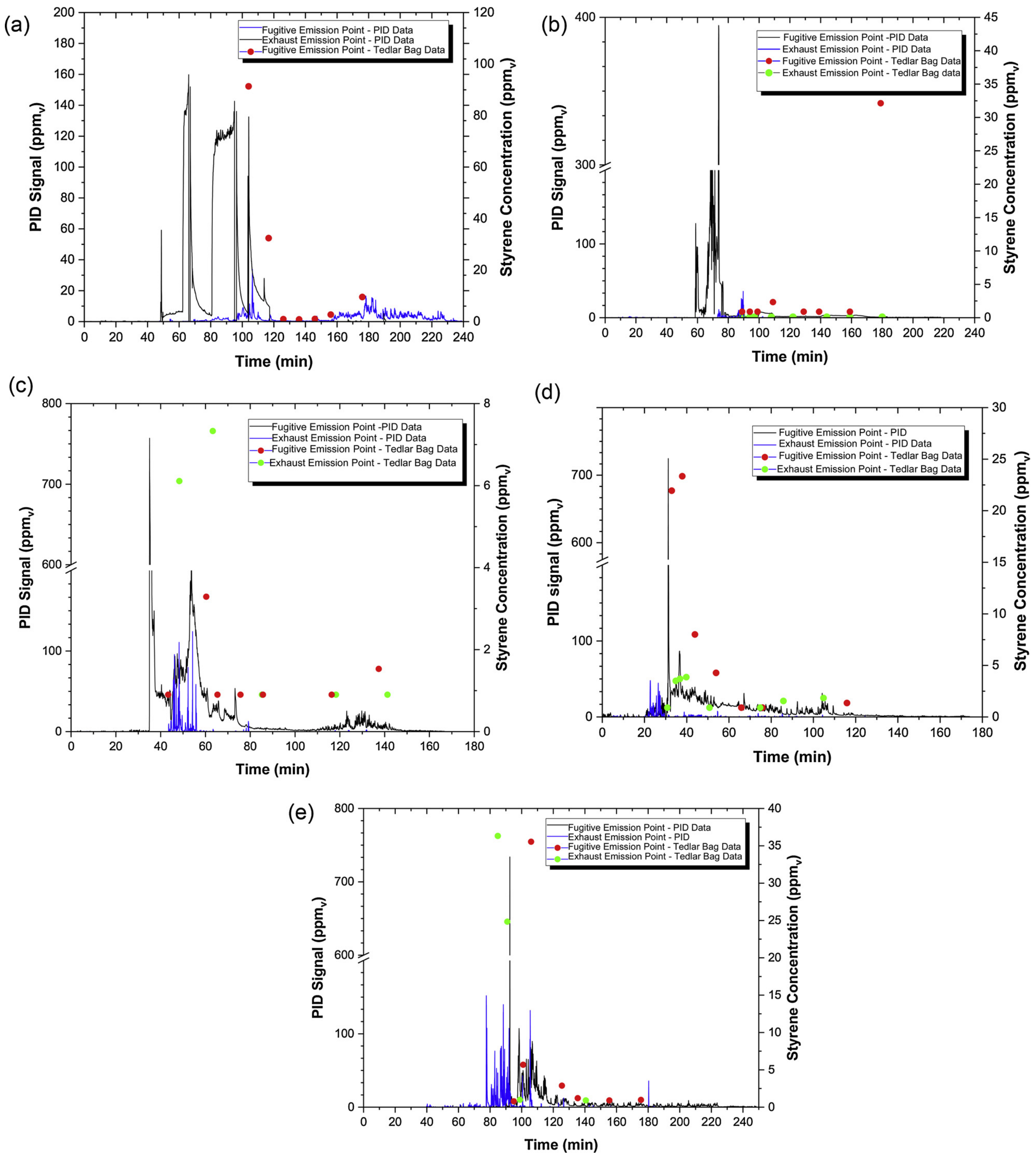

Fig. 1. PID results vs. Tedlar bag data at fugitive and exhaust emission points (a) Site I, (b) Site II, (c) Site III, (d) Site IV, and (e) Site V. Out of range data were excluded. New Tedlar bag data are shown with fugitive and exhaust emission point PID results retrieved from Teimouri et al. [3]. Number of samples the Tedlar bag styrene concentration > > PID reading at the fugitive emission point [Site I: 7 of 7, Site II: 2 of 8, Site III: 0 of 7, Site IV: 0 of 7, and Site V: 0 of 6], and at the exhaust emission point [Site I: no Tedlar bag samples, Site II: 7 of 8, Site III: 6 of 6, Site IV: 8 of 8, Site V: 1 of 4].

[46–48]. Discrete air samples at fixed emission points were not collected when the greatest PID signal was detected. PID signals from personal monitoring (this study) were on average lower than fixed emission point PID signals [3]. These were sometimes greatest for teams when they approached the emission points, but not always (Fig. SM3, Table SM2). Video monitoring indicated that chemical fate was affected by wind direction (Fig. SM4, Videos SM1, SM2).

3.2. CIPP visual, physical, and thermal properties

Visual, physical, and thermal property differences were found across the CIPPs, but no studies were found that determined the influence of CIPP installation conditions on associated emissions or final CIPP chemical properties. The inner and outer CIPP surfaces differed visually (Fig. SM5). As expected, pre-liners were seen in pipes 1, 3, and 4. Though, only pipe 5 had a thermoplastic layer on the CIPP's inner and outer layer, whereas other CIPPs had the thermoplastic layer only on their inner surface. Blisters that contained liquid were observed on pipe 5′s outer wall, and small color splotches were observed on its inner wall. Blisters for steam-CIPPs have been reported by others [49,50], but the blister formation mechanism was not found in the CIPP literature. A prior study found blisters formed at high temperature for vinyl ester resin laminates due to hydrolysis and de-bonding [51]. The blistered pipe had the greatest interfacial temperature (139 °C) compared to other styrene-based CIPPs (˜121 °C) (Fig. SM6). Liquid bubbling inside one blister contained benzoic acid (5.7 mg/L), benzaldehyde (< 0.06 mg/L), styrene (< 0.1 mg/L), and phenol (< 0.22 mg/L) (Fig. SM4, Video SM3). CIPP bulk density was 1.069–1.204 g/cm^3, similar to values reported by others [1,52]. Particulates entered the air and deposited on the ground when the end of each new CIPP was cut (Video SM4).

CIPPs contained volatile material and the amount of noncombustible materials in CIPPs differed. The wide endothermic peaks around 110 °C shown by DSC thermograms indicated VOCs were present in pipes 4 and 5 (Fig. SM7). Sharp endothermic peaks around 120 °C for pipes 1 and 3 likely represented the thermoplastic pre-liner's melting point, but lesser peaks were also detected for pipes 2, 4 and 5. The broad endotherm around 170 °C represented the disordering of high segment crystallite of the thermoplastic polyurethane coating [53,54]. Because of differences in host pipe type (concrete vs. steel), components (pre-liner vs. no pre-liner), steam exposure duration (100 min vs 92–107 min), post-cure temperature range (71–77 °C vs. 66–99 °C), styrene loading in the uncured resin tube (66.2 mg/g vs. 45.2–72.9 mg/g), and possibly other factors, the authors were unable isolate the reason for observed differences.

When heated to 120 °C, TGA results showed CIPPs decreased by 0.37–1.16 wt%, and by 1.02–2.21 wt% at 160 °C (Table 4). This significant loss was likely due to water and VOC emission. Since the boiling points of styrene (monomer for pipes 1, 3, 4 and 5) and TPGDA (monomer for pipe 2) were 145 °C and 160 °C, respectively, the authors assumed that the residual TPGDA, styrene, and their oligomerized products would be emitted from CIPPs at 160 °C [55,56]. CIPPs contained 12–17 wt% noncombustible material, and the multiple decomposition temperatures observed were likely associated with the resin, pre-liner, and felt plastic coating (Fig. SM8, Table 4). Residue at 900 °C likely represented silica from talc in both resins (20–30 wt%) [43], which is common [40,57–59]. Residue differences could be associated with silica losses during steam exposure [3], liner mechanical abrasion during installation, unequal distribution during resin tube impregnation, or undisclosed compounds that did not decompose at 900 °C.

Steam exposure duration and temperature likely influenced the CIPP thermal and compositional characteristics. The extended steam exposure periods may have allowed excess resin to polymerize, removed monomer and initiators that could have been used for a more complete crosslinked network, removed fillers, or had other unknown consequences. Because CIPP samples were collected at the end of each CIPP, farthest away from the steam injection point, the samples' thermal exposure profile may have differed from nearer to the steam injection point. CIPP samples also were not in contact with the host pipe. Chemical volatilization through the outer layer may have contributed to chemical loss. Chemical volatilization may or may not differ around the CIPP's circumference (i.e., top vs. bottom, etc.).

3.3. Chemical residual in the CIPPs

^{1}H NMR spectroscopy indicated that several compounds extracted from CIPPs were also found in the uncured resin tubes (Fig. SM9). Styrene was extracted from pipes 1, 3, 4 and 5 with ^{1}H NMR peaks around 5.25, 5.76, 6.72, 7.33 and 7.41 ppm (Table SM3). These CIPPs were manufactured with an isophthalic polyester styrene resin. Styrene was not extracted from CIPP pipe 2; this CIPP was manufactured with a non-styrene vinyl ester resin. Instead, TPGDA was found in CIPP pipe 2 and was suspected to be an active monomer. Styrene oxidation products such as benzaldehyde and 2-phenyl acetaldehyde were also detected by ^{1}H NMR. 4-*tert*-Butylcyclohexanol, a known degradation product of initiator (Perkadox®), was extracted from all CIPPs. Also, acetophenone was only found in pipe 2, the non-styrene resin CIPP, and is a known degradation product of Trigonox®. Phenol and benzaldehyde were found in all styrene-based CIPPs. Acetone, bis(*tert*-butylcyclohexyl) peroxydicarbonate, 1-tetradecanol, and *tert*-butyl peroxy-2-ethylhexanoate (initiators) were also detected in CIPPs. Similar to a styrene-based CIPP installed in an Indiana sanitary sewer, analyses in the present study also revealed the presence of styrene dimer and trimer compounds in styrene-based CIPPs (Fig. SM10, Table SM4), but not styrene oxide [60]. Because no barrier existed between the resin impregnated felt and steam, steam may have catalyzed styrene oligomerization and degradation of other compounds in the uncured resin tube [43].

The new CIPPs contained a significant amount of volatile material, roughly 1.02–2.21 wt% (Table 4). If this result is representative of the entire CIPP, a 6.1 m [20 ft] liner could potentially contain 5–10 kg [11–22 lbs] of residual chemical. Others have reported residual ranging from 0.4 to 1.6 wt% [60] in 1 steam-CIPP and 1.0–9.3 wt% in 4 UV-CIPPs [32]. While no significant relationship was found between total volatile weight loss and CIPP styrene loading (Fig. SM11), some weight loss was certainly due to styrene emission. Chemical emission would depend on a variety of factors and may be greatest early in the CIPP's service life. A multitude of chemicals were present in the CIPPs to include, but are not limited to, initiator degradation products (i.e., acetophenone, 4-*tert*-butylcyclohexanone, 1-tetradecanol, benzoic acid), monomers (i.e., styrene, TPDGA), an oxidation product (i.e., benzaldehyde), plasticizer (i.e., dibutyl phthalate), and a compound previously associated with CIPP water contamination incidents (i.e.,

Table 4
Residual found in newly manufactured CIPPs and their thermal characteristics.

Pipe	TGA Analysis: Weight Loss		Solvent Extraction and GC/MS Analysis		TGA Analysis: Transitions & Residue		
	At 120 °C, %	At 160 °C, %	Total mass unidentified, Methylene chloride	Total mass unidentified, Hexane	1st step Decomposition Temp., °C	2nd step Decomposition Temp., °C	Residue Content at 900 °C, %
1	0.37 ± 0.06	1.32 ± 0.02	79%	94%	376.75 ± 1.42	549.30 ± 6.16	15.86 ± 1.92
2	1.16 ± 0.05	2.21 ± 0.15	82%	93%	397.10 ± 0.91	584.38 ± 2.37	17.05 ± 0.17
3	0.54 ± 0.07	1.02 ± 0.36	69%	92%	381.02 ± 1.18	550.73 ± 2.68	16.03 ± 1.76
4	0.60 ± 0.01	1.60 ± 0.31	76%	94%	379.44 ± 1.18	546.61 ± 2.72	16.14 ± 0.96
5	0.56 ± 0.04	1.64 ± 0.43	75%	94%	377.70 ± 2.37	551.20 ± 1.43	12.77 ± 0.47

The weight loss was determined by first measuring the mass of initial sample and then weight after heating. The "residue" pertains to materials that remained from the initial sample after heating to 900 °C. The total mass of unidentified chemicals extracted in methylene chloride and hexane solid-liquid extractions was determined by comparing the weight loss at 160 °C to the amount of chemical quantified in extracts. The weight of each 6.096 m CIPP using measured bulk density was estimated to be: 80.5 kg (pipe 1), 89.6 kg (pipe 2), 87.9 kg (pipe 3), 92.0 kg (pipe 4), 100.6 kg (pipe 5).

Table 5
Mass loading of confirmed compounds for each CIPP sample.

Extraction Solvent	Compound Detected	Pipe [Resin Type, Pre-liner Use] mg compound/kg CIPP sample[d]				
		Pipe 1 [1 pre-liner]	Pipe 2 [0 pre-liner]	Pipe 3 [2 pre-liners]	Pipe 4 [1 pre-liner]	Pipe 5 [0 pre-liner]
Methylene Chloride	Styrene[a,b,c]	86 ± 22	124 ± 165	322 ± 21	562 ± 44	235 ± 62
	1-Tetradecanol	2,140 ± 193	394 ± 77	2,200 ± 91	2,360 ± 69	2,650 ± 133
	Benzaldehyde	55 ± 14	72 ± 60	92 ± 18	242 ± 13	364 ± 20
	4-TBCH	–	786 ± 440	–	–	–
	Acetophenone[c]	–	1,090 ± 10	–	–	–
	Phenol[c]	37 ± 23	–	37 ± 1	39 ± 1	33 ± 3
	Benzoic Acid	491 ± 113	1,800 ± 284	470 ± 80	590 ± 16	828 ± 66
	Sum	*2,809*	*4,266*	*3,121*	*3,793*	*4,110*
Hexane	Styrene[a,b,c]	75 ± 6	32 ± 15	93 ± 5	52 ± 23	62 ± 11
	1-Tetradecanol	749 ± 48	433 ± 285	719 ± 71	817 ± 101	872 ± 183
	Benzaldehyde	14 ± 2	14 ± 1	25 ± 2	28 ± 7	94 ± 11
	4-TBCH	–	220 ± 661	–	–	–
	Acetophenone[c]	–	305 ± 7	–	–	–
	TPGDA	–	565 ± 15	–	–	–
	Sum	*838*	*1,569*	*837*	*897*	*1,028*

Mass loading is reported as the mass of chemical detected per mass of CIPP sample that was analyzed. Mean and standard deviation shown; (-): compound not detected.

[a] Carcinogenic compound [69].

[b] Endocrine disruptors [70].

[c] HAP [20].

[d] Najafi et al. [30] reported a prior study had identified different allowable styrene loadings in new CIPPs [400, 500, and 1000 mg/kg], but the source document could not be obtained or reviewed by the authors.

phenol) (Table 5). Though, different compounds were extracted from the same CIPP by different solvents and the quantity of compounds detected varied across CIPPs. Also notable was that most of the chemical mass extracted from CIPPs was not identified (69–94%) (Table 4). Additional work should focus on characterizing residual chemical in CIPPs as well as determining chemical emission rates, fate, and toxicity.

Notable differences were detected for contaminant loading across CIPPs (Table 5). Styrene was extracted from pipe 2, the non-styrene resin based CIPP. Teimouri et al. [3] did not find styrene in the pipe 2 uncured resin tube. These investigators however subsequently found styrene in the condensed materials emitted into the air from that same pipe when it was installed. Currier [42] found styrene leaching into water from pipe 2 after installation. Therefore, the contractors likely contaminated pipe 2 with styrene during installation. 1-Tetradecanol, a potential thermal initiator degradation product [61], was present with the greatest abundance for styrene-based CIPPs, and limited toxicological data were found [62]. The relative loading of other compounds in styrene-based CIPPs was benzoic acid > styrene > benzaldehyde > phenol. For the non-styrene resin CIPP (pipe 2), benzoic acid was the next most abundant chemical followed by acetophenone > 4-*tert*-butylcyclohexanone > 1-tetradecanol > styrene > benzaldehyde. TPGDA (suspected monomer) was in greatest abundance for CIPP pipe 2 and was found in the uncured resin tube [3]. No studies were found that examined chemical loading or differences in radial directions for a single CIPP. A recent UV CIPP study did find the styrene loading (mg/kg CIPP) of upstream and downstream samples for a single CIPP differed by a factor of 68 [32]. No contractor procedures or requirements were found for equipment cleaning, nor was this practice observed onsite or recommended in training courses and other guidance [50,63–68].

3.4. Future research needs and recommendations

To predict short- and long-term chemical emissions from CIPP installations, additional research is needed, and results of the present study can inform future investigations. Factors that control chemical emission from and residual in CIPPs should be investigated to include CIPP components (i.e., resin, initiators, fillers, pre-liners, reinforcement), the curing process (i.e., airflow rate, post-cure temperature, exposure duration, reflective heat of the host pipe, degree of resin distribution), and cool down process (i.e., air flowrate, temperature). Chemical residual in the CIPP closer to the steam injection point should be compared against chemical loadings further down the CIPP. The presence and extent larger polymers or more polar organics existed in the resin tube or were formed and remained in the CIPP remains unclear because only ^{1}H NMR and GC/MS were used for quantification (Table SM5). Other methods such as liquid chromatography mass spectrometry, proton transfer reactor time-of-flight (TOF)-MS, and GC/GC TOF-MS may be helpful in identifying other materials emitted. Chemical loading characterization for new CIPPs would also help identify chemicals that remain and their magnitudes. Additional air monitoring studies are also recommended because publicly available data are lacking. Analysis of photochemical transformation reactions in air should also be considered.

Lack of chemical air testing results as well as fate and transport models inhibit evidence-based health risk assessments. Available evidence does not support prior claims that health risks are “medium and low” and the “problem applies only until the resin is cured” [71,72]. A 2017 CIPP worker fatality investigation revealed chemical emission exposure during a CIPP installation was a contributing factor [7,14]. Other U.S. federal and state agencies as well as organizations outside the U.S. also have raised public health and safety concerns and found chemicals traveled into nearby buildings prompting the need for medical assistance. Contractors and infrastructure owners should consider implementing engineering and administrative controls like negative pressures, emission capture systems, ventilation systems, online monitoring, work area access restrictions, hazard awareness training, and PPE. Setback distances of at least 5 m from CIPP installation sites, continuous air monitoring, and the capture of all emissions during and after 24 h of installation have been recommended in Europe (Table 1), but practices were not found in the U.S. [64,66,67]. In the present study, chemicals were emitted into areas where contractors resided, emissions migrated offsite, and contractors were not wearing PPE to protect against inhalation exposure. The newest CIPP industry construction guidance in the U.S [67] directed contractors to setup their exhaust pipe at a minimum of 2.4 m [8 ft] above the ground. Though, it is unclear if this action increases or decreases the chemical exposure risks to workers and nearby public.

A unique discovery of this study was that the contractor's installation practices (i.e., temperatures, exposure time) did not match the procedures provided to a client before the installation. No prior studies were found that scrutinized CIPP contractor practices in such detail. It may be that existing CIPP air monitoring studies (Table 1) are not representative of the CIPPs intended/designed to be installed. Moreover, the contractor's extended steam injection in the present study may have stripped out residual chemicals so that Currier [42] underestimated CIPP leaching into water. Contamination of a non-styrene CIPP with styrene was also discovered and therefore, contractor equipment cross-contamination expands the list of chemicals potentially emitted. While some testing has been conducted in other composite manufacturing applications [73–78], CIPP bench- and field-scale studies are needed to determine the factors that control and minimize emissions through design and construction practice upgrades.

Several measurements should be considered during future investigations. These include chemical identification and quantification of the uncured resin tube and newly installed CIPPs. The variety and magnitude of volatiles emitted *during* the manufacturing process where raw chemicals are potentially more subject to emission should be further investigated. These materials should also be considered in environmental impact assessments for urban and rural environments. Documentation of contractor material handling practices and manufacture conditions to include work activity, time, and temperature, ambient environment monitoring, and video recording should also be considered. The application of multiple air sampling approaches, analytical methods and material characterization techniques should be considered to identify different contaminants and material properties. The link between design and construction practices to chemical emission can be better understood by these approaches. Evidence from the present study indicated that while PIDs can inform testing decisions, these devices should not be solely used to assess CIPP related chemical emission and exposures. PIDs can both significantly underestimate and overestimate the air concentration of some contaminants of environmental and health concern.

4. Conclusions

This study was conducted to better understand what chemicals can be released from steam-CIPP storm water culvert installations into air, their concentration, and loading in new CIPPs. Many chemicals not detected by prior investigators were found in the present study. A few chemicals found by parallel investigators at the same worksite were also detected in the present work (Table SM4). Results of the present study include several unique discoveries that pertain to the interpretation of prior studies and future investigations. A detailed comparison of contractor CIPP manufacturing practices (i.e., exposure time, temperature) to air and composite chemical testing data is unique to this study and should be repeated in future investigations.

VOCs were emitted into the air and PID signals did not accurately reflect styrene air concentration. Styrene, methylene chloride, and phenol were found in Tedlar bag samples. Tedlar bag styrene adsorption experiments indicated that chemical levels in air were likely much higher onsite than confirmed in the laboratory (i.e., styrene > 86.4 ppm$_v$). Multiple VOCs that can affect PID signals were also found in sorbent tube air samples. Grab samples were not collected when the PID signals were the greatest. More expansive chemical identification and quantitation is needed for field- and bench-scale studies. PID signals should be further examined under the complex emission atmospheres associated with CIPP installations [79]. PIDs should not be solely relied upon to monitor instantaneous or prolonged (minutes to hours) chemical exposure for the complex atmospheres generated by the process.

Much of the chemical mass extracted from CIPPs was not chemically identified (69–94%). Chemical loading differed across CIPPs, even those created by the same contractor, with the same resin and host pipe characteristics. Compounds extracted included initiator degradation products (i.e., acetophenone, 4-*tert*-butylcyclohexanone, 1-tetradecanol, benzoic acid), monomers and oligomers (i.e., styrene, dimers, trimers, TPDGA), an oxidation product (i.e., benzaldehyde), and a compound associated with CIPP water contamination incidents (i.e., phenol). Some compounds were also found in air samples and were likely created during CIPP manufacture and/or were not found on SDSs.

Recommendations to upgrade existing outdoor plastic pipe construction practices, capture and confirm emissions were captured, and provide more oversight were provided. Future studies have also been proposed. To interpret the representativeness of future studies, parameters outlined in the present study as well as those proposed should be considered for future investigations. The outdoor CIPP manufacturing process requires engineering and administrative controls as well as safety upgrades to protect the health of workers and the nearby public from harm.

Conflict of interest disclosure

AJW, JAH, BEB, ENC, and SMTS are named in a patent application (PCT/US18/28173) filed April 18, 2018 by the Purdue Research Foundation. The patent application pertains to the technologies for capturing, identifying, analyzing, and addressing emissions that are potentially hazardous to the environment and humans. The invention was developed with support from US National Science Foundation CBET-1624183 grant.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.jhazmat.2019.02.097.

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