Summary Report

Characterization of Volatile Organic Compound Emissions from Consumer Level Material 3D Printers and Their Relationship with Particle Emissions

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SUMMARY REPORT

Characterization of Volatile Organic Compound Emissions from Consumer Level Material Extrusion 3D Printers and Their Relationship with Particle Emissions

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INTRODUCTION

A fused filament fabrication (FFF) 3D printer works by heating a coil of thermoplastic filament and extruding it onto a moving platform, which builds an object layer by layer. There are numerous filaments available for FFF 3D printers. These filaments are a blend of thermoplastics (e.g., polylactic acid (PLA), acrylonitrile butadiene styrene (ABS), nylon) with coloring dye, metal, wood, and other additives. Extrusion temperature ranges between 180 °C and 500 °C depending on material types, resulting in emissions of both gases and particles that may deteriorate indoor air quality.[1–3] Gaseous emissions are complex and contain a mixture of volatile organic compounds (VOCs) that may include odorants, irritants, and carcinogens.[4–6] Both acute and chronic exposure may occur to users and occupants in the surrounding space, especially when operating in small-scaled or not well-ventilated indoor environment.

This study investigates VOC emissions from multiple 3D printers operating various filament materials, based on both controlled environmental chamber experiments, and exposure modeling. VOC emissions were characterized based on different printing conditions and compared with other existing 3D printer studies. Furthermore, the relationship between VOC and particle emissions was also studied.

METHODS

Chamber emission test

Each 3D printer was tested in a 1 m^3 well-mixed stainless-steel emission test chamber according to standards.[7] Five brands of commercially available desktop 3D printers were tested, with 20 different 1.75mm diameter filaments, including ABS, PLA, nylon, polyvinyl alcohol (PVA), and high impact polystyrene (HIPS) materials from eight different distributors.

VOC and low-molecular-weight aldehyde samples were collected from the chamber onto solid sorbent cartridges separately. At least three sets of VOC and aldehyde samples were collected for each print run: one 30-minute collection during pre-operating phase (i.e., printer and filament loaded in the chamber but not printing), at least one 1-hour collection during the last hour of printing phase, and one 1-hour collection after two hours into post-printing phase (i.e., print has finished). VOC samples were analyzed using gas chromatography-mass spectrometry. [8–10] Each individual VOC (IVOC) was specifically identified using mass spectral databases and quantitated using multipoint calibration standards if available. Further, total VOC (TVOC) concentrations were calculated by adding all IVOC responses obtained by the mass spectrometer and converting the total mass to a toluene equivalent. Aldehyde samples were analyzed by high-performance liquid chromatography.[11–13] Limit of detection is 2 μg m^{-3} for all chemicals identified. In addition, particle concentrations in the chamber were measured continuously through the three phases.[14]

The emission rate of chemical i, $ER_{g,i}$ (μg h^{-1}), was calculated from the measured concentration using Equation 1:

$$ER_{g,i} = \frac{Q\left(C_{it} - C_{io} exp\left(-\frac{Q}{V}(t - t_0)\right)\right)}{1 - exp\left(-\frac{Q}{V}(t - t_0)\right)} \tag{1}$$

where Q is air flow rate into the chamber, C_{it} is VOC$_i$ concentration measured at time t, C_{i0} is background concentration at time t_0, V is chamber volume.

<u>Exposure model</u>

Potential exposure concentrations of users and building occupants were calculated based on a steady-state mass balance model assuming emissions were well-mixed within the modeled volume. The estimated exposure concentration for chemical i, $C_{i,m}$ (μg m^{-3}), for a particular room model m, was calculated using Equation 2:

$$C_{i,m} = ER_{g,i}\left(\frac{A}{V_m}\right)\left(\frac{1}{N_m}\right) \tag{2}$$

where A is the number of printers in the modeled room, which was assumed to be 1 in this study. V_m and N_m are the volume and air exchange rate of the modeled room, specific to two scenarios in this study: residential room and personal exposure. Briefly, these scenarios represented and simulated the emission concentrations from 3D printing in a single-family residence room or within about 0.5 m near the printer.

RESULTS and DISCUSSION

<u>VOC emissions</u>

The average TVOC ERs of 3D printers were lower than those of laser printers, dry process copiers, and personal computers (Table 1), while some of the high emitting filaments like ABS and nylon had similar TVOC ERs as reported from laser printers and personal computers. TVOC ERs varied significantly depending on filament material; TVOC ERs of ABS were comparable to that of HIPS, which both were higher than those of PLA and PVA in general. The two nylon filaments differed significantly, and the high emitting nylon was the highest emitting filament in this study.

Table 1. Averages and ranges of TVOC (toluene equivalent) ERs for different filament materials, compared to those of laser printers, dry process copiers, and personal computers.[15,16]

TVOC ER ($\mu g\ h^{-1}$)	ABS	PLA	Nylon	HIPS	PVA	Laser Printer[15,16]	Dry Process Copier[15]	Personal Computer[15]
Average	835	193	1662	888	147	26400	36400	12200
Range	506-1455	149-269	276-3048			1200-130000	4600-108000	50-24200

In general, ABS and nylon filaments were more likely to have higher TVOC ERs than HIPS and PLA filaments (Figure 1). Overall, large differences of VOC ERs were found (Figure 1), which may have occurred as a result of the differences among filament materials, and the differences in testing and measurement methods used. In addition, print duration, chamber construction/material, testing environment, may also be associated with the variances in ERs.

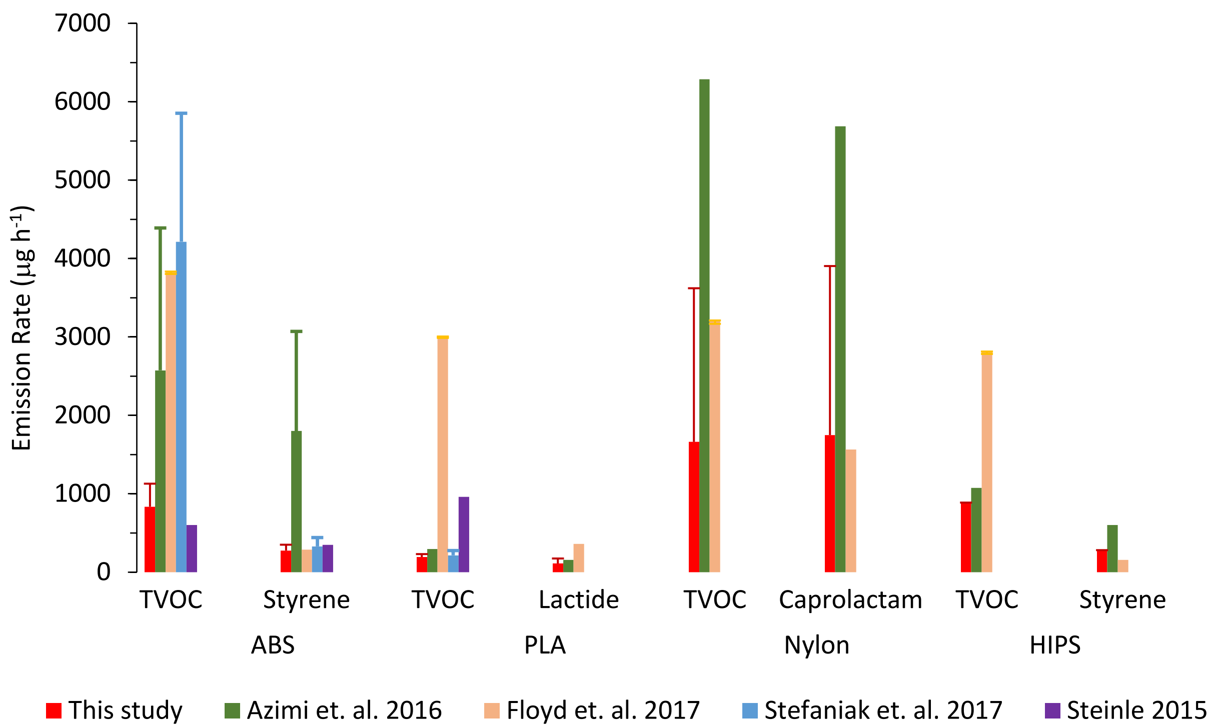


Figure 1. TVOC (toluene equivalent) and individual VOC (styrene, lactide, and caprolactam) ERs from this study compared to other literature by filament material. Error bars represent standard deviations. 3D printer

malfunctioned results in Stefaniak et al.[5] are not included. Only one test with HIPS was performed in the study, thus standard deviation is not available for HIPS.

The chemical profile of emitted IVOCs varies significantly by filament material. Specifically, 218 IVOCs have been identified from 3D printer emissions, with 177 compounds detected from ABS (most detected), 70 from HIPS, 57 from PLA, 49 from PVA, and 47 from nylon. ERs and detection frequencies for the top 15 IVOCs from each filament material are listed in Table 2. Top emitting IVOCs were associated with filament material monomers, e.g., styrene from ABS and HIPS, lactide from PLA, and caprolactam from nylon. Out of hundreds of chemicals identified, only five (for PLA) to eight chemicals (for ABS) were consistently detected for each filament material. The inconsistency in the rest of the chemical identifications may imply the existence of many chemical/formulation combinations (e.g. additives) available in the market.

Table 2. Average ERs of the top 15 IVOCs detected for different filament materials with detection frequency (freq.). n lists the number of tests performed. Italicized chemicals are species not on referenced indoor air quality (IAQ) health risk tables. Asterisk means listed only for its odor threshold.

ABS (n=12)			PLA (n=9)			Nylon (n=2)			HIPS (n=1)		PVA (n=1)	
	freq.	ER (μg h^{-1})		freq.	ER (μg h^{-1})		freq.	ER (μg h^{-1})		ER (μg h^{-1})		ER (μg h^{-1})
Styrene	100%	275.7	*Lactide*	100%	110.8	Caprolactam	100%	1749	Styrene	281.4	Acetic acid	183.8
Benzaldehyde	100%	71.5	Acetaldehyde	100%	18.8	Acetaldehyde	100%	11.3	Benzaldehyde	157.6	Acetaldehyde	87.0
Benzene, ethyl	100%	69.3	1-Butanol	100%	17.8	*Lactide*	100%	8.2	Benzene, ethyl	113.9	2-Butenal	56.0
Acetaldehyde	100%	53.6	Formaldehyde	100%	7.0	Benzaldehyde	100%	8.0	*Cyclotrisiloxane, hexamethyl*	80.6	Formaldehyde	21.6
Formaldehyde	100%	24.7	Decanal	100%	4.1	Decanal	100%	6.6	*Benzene,1,1'-(1,2-cyclobutanediyl) bias-, cis*	47.7	Pentanal	18.5
1-Butanol	100%	19.8	Benzaldehyde	89%	4.1	Nonanal	100%	6.2	Cyclotetrasiloxane, octamethyl	41.5	Benzaldehyde	18.5
Cumene	100%	18.1	Nonanal	89%	2.9	Formaldehyde	100%	6.1	Acetaldehyde	35.0	1,2-Ethanediol	15.7
Acetophenone	92%	63.1	Caprolactam	56%	7.4	1-Hexanol, 2-ethyl	100%	4.0	Formaldehyde	30.2	2-Butenal	14.6
p-, m- Xylene	92%	6.8	Styrene	56%	1.6	Acetophenone	100%	4.0	Pentanal	26.1	Cyclohexane	13.8
Vinyl cyclohexene	83%	20.4	Methyl methacrylate	22%	19.8	Toluene	100%	3.2	TXIB (2,2,4-Trimethyl-1,3-pentanediol diisobutyrate)	24.6	Caprolactam	11.9
Decanal	83%	6.5	2,4-bis(1,1-dimethylethyl)-Phenol	22%	8.3	Octanal	100%	3.1	Acetophenone	20.8	*Cyclotrisiloxane, hexamethyl*	11.5
Toluene	83%	5.9	1-Dodecanol*	22%	2.6	Pentanal	100%	2.9	*2-Phenylpropenal*	18.8	TXIB	10.9
Cyclotrisiloxane, hexamethyl	75%	19.7	Butyl acrylate	22%	1.1	Styrene	100%	2.6	Nonanal	15.2	Diethylene glycol	9.3
Benzene, propyl	75%	10.2	Dodecane	22%	0.8	Formamide, N,N-dimethyl	100%	2.5	Cumene	14.6	Isooctyl acrylate	9.3
Phensuximide	75%	7.9	Ethanol, 2-butoxy	11%	2.1	*Octane, 1,1'-oxybis*	100%	2.5	1H-Trindene, 2,3,4,5,6,7,8,9-octahydro-1,1,4,4,9,9-hexamethyl-	10.5	2,4-Hexadienal*	6.9

Effects of 3D printer and filament parameters

Filament color, printer brand, and filament brand were the top three parameters affecting VOC ERs (Table 3). TVOC and monomers released were influenced by printer brand, filament color, and nozzle temperature in the same pattern. Printer brand had the largest effect on TVOC ERs for both ABS and PLA. ERs of certain VOCs, including formaldehyde, other aldehydes, and acetophenone, varied significantly by filament brands for both PLA and ABS. Some PLAs emitted methyl methacrylate depending on the manufacturer. Emissions decreased with the decrease of nozzle temperature. The case with HEPA filter installed released higher styrene, hydrocarbons, xylenes, and benzaldehyde, which may be due to the filter itself.

Table 3. Maximum ER differences (diff.) from each printer or filament parameters and the corresponding relative mean absolute difference (RMAD).

	ABS				PLA			
	TVOC diff.		Styrene diff.		TVOC diff.		Lactide diff.	
	$\mu g\ h^{-1}$	RMAD	$\mu g\ h^{-1}$	RMAD	$\mu g\ h^{-1}$	RMAD	$\mu g\ h^{-1}$	RMAD
Filament color	522	63%	94.4	34%	21.5	11%	41.3	37%
Filament brand	439	53%	125	45%	25.3	13%	38.8	35%
Printer brand	594	71%	86.4	41%	56.8	29%	8.7	8%
Nozzle temperature	323	39%	80.6	29%				
Internal HEPA filter	110	19%	28.8	22%				

Relationship between VOC and particle emissions

For the overall dataset, there was a positive monotonic relationship between TVOC and styrene ERs versus particle ERs (Spearman rho, r_s, of 0.5 or higher), but not for lactide ER (r_s < -0.4). 3D printer emissions are heavily dependent on filament material. For ABS filaments, an increase in styrene ER was associated with an increase in particle mass ER ($r^2 = 0.74$, p-value = 0.0003) and particle number ER ($r^2 = 0.40$, p-value = 0.03). Benzothiazole ER had better correlation with particle ERs than other IVOCs. For PLA, lactide ER versus total particle mass/number ERs were both positively monotonic, but no strong linear relationships were observed. ABS filaments generated 4.3 times higher TVOC ER than PLA, but this ratio was much lower than ABS to PLA ratio of particle number ERs (138 times) and particle mass ERs (58.1 times). The two nylon filaments showed an inverse relationship between caprolactam emissions and particle emissions. Printer brands influenced both particle and VOC emissions, however, that was not the case for filament color or filament brand. Increase in nozzle temperature has a much greater effect on particle emissions than VOC emissions. Though the particle count decreased for larger particles (i.e. greater than 300 nm in diameters) with the internal HEPA filter, other emissions (especially VOC emissions) increased with the use of the filter.

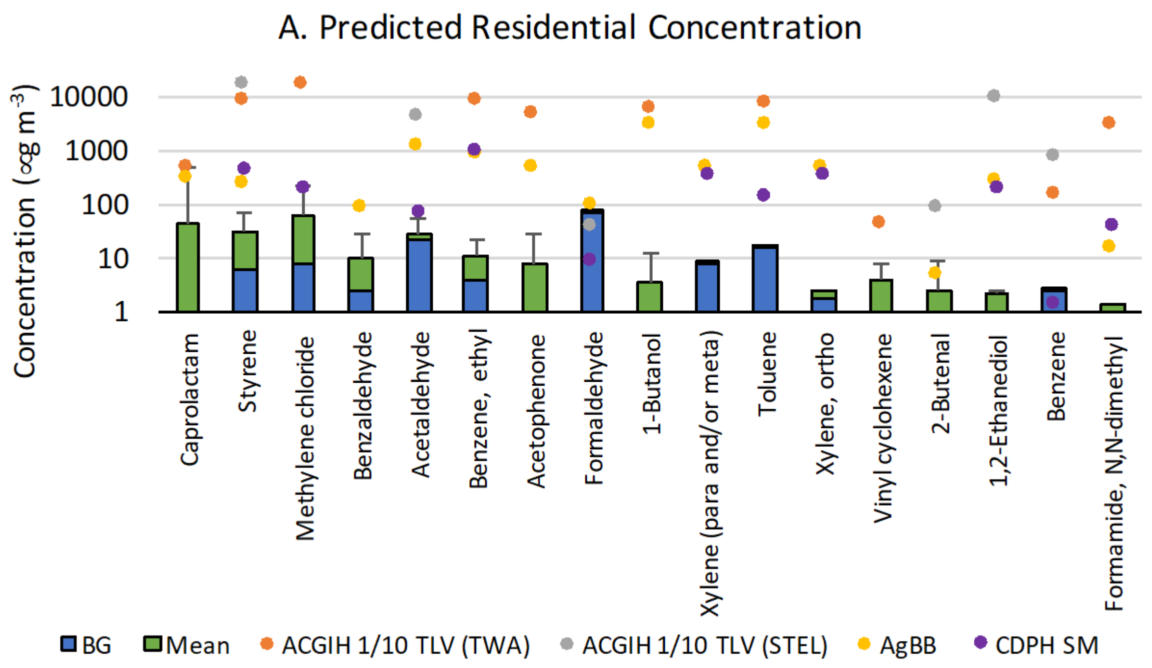
Predicted exposure levels and health implications

Predicted TVOC concentrations can vary by one order of magnitude between personal space (breathing zone) and residential room scenarios. Personal TVOC exposure near a 3D printer with any type of filaments studied is expected to exceed 900 $\mu g\ m^{-3}$, higher than the TVOC criteria recommended by the Leadership in Energy and Environmental Design (500 $\mu g\ m^{-3}$).[17] The predicted TVOC exposure concentrations for PLA were an order of magnitude smaller than those for ABS and nylon for both model scenarios. Just two nylon printers working at the same time may result in TVOC exposure concentrations exceeding 500 $\mu g\ m^{-3}$ in a typical residential room.

All IVOCs identified were compared with national and international indoor air regulatory and health risk tables. Out of the top 15 IVOCs detected (Table 2), 13 chemicals for ABS, 13 for PLA, 13 for nylon, 11 for HIPS, and 14 for PVA are known to be toxic, irritant, and/or cause odor. Furthermore, acetaldehyde, ethylbenzene, formaldehyde, methylene chloride, styrene, and toluene are commonly detected for emissions from ABS, PLA, and nylon filaments. Specifically, formaldehyde is listed as human carcinogen, styrene and methylene chloride as probable human carcinogens, acetaldehyde and ethylbenzene as possibly human carcinogens under the International Agency for Research on Cancer (IARC)[18] and California Proposition 65 (Prop 65),[19] and toluene is listed under Prop 65 for its reproductive and developmental toxicity. The six chemicals are also listed under the American Conference of Governmental Industrial Hygienists' Threshold Limit Values (ACGIH TLV®),[20] Ausschuss fur gesundheitlichen Bewertung von Bauprodukten's Lowest Concentration of Interest (LCI),[21] and the California Department of Public Health Standard Method v1.2–2017 (CDPH SM).[22] 51 chemicals (29%) for ABS, 36 chemicals (63%) for PLA, and 30 chemicals (64%) for nylon out of the total number of IVOCs identified are listed at least once in the five risk tables mentioned previously. Chemicals such as benzenes, toluene, xylenes, hydrocarbons, and aldehydes, common thermal degradation byproducts of filament monomers, are also commonly identified. For ABS, PLA, and nylon respectively, 30 chemicals (59%), 19 chemicals (53%), 14 chemicals (47%) out of all the IVOCs associated with health effects are newly introduced to indoor air quality (IAQ) environment solely from 3D printing.[23,24] Caprolactam, acetophenone, 1-butanol, vinyl cyclohexene, and 2-butenal are some chemicals that are introduced by 3D printing (Figure

2). Caprolactam has an ocular and respiratory toxicity and has a low 8-hour chronic reference exposure level (CREL) of 7 µg m^{-3} (1.4 ppb).[25] Acetic acid (from PVA) is an irritant and is listed under ACGIH TLV® and LCI.[21] PLA is the only filament that its primary detected monomer (lactide) is not listed under major regulatory/health risk lists for IAQ. However, the chemical with the second highest ER from some PLA filaments, methyl methacrylate, is an irritant.[21]

For the top emitting VOCs that are cancerous, hazardous, or irritant to humans, their predicted exposure levels are compared to CDPH SM, 1/10 ACGIH TLV®, and Gabbi ICL in Figure 2. Caprolactam, methylene chloride, formaldehyde, and benzene, all under IARC, not only exceed some criteria listed above for personal exposure but also for maximum residential exposure levels. Caprolactam exposure from 3D printer operating nylon filament can exceed the criteria in AgBB, CREL, and ACGIH (1/10 TLV® TWA). The predicted personal exposure of formaldehyde can exceed limits set by CDPH SM and ACGIH (1/10 of TLV® STEL). Benzene emission may be as high as five times greater concentration than the CDPH SM's criteria for personal exposure. The average styrene concentration for personal exposure is above the allowable concentration by AgBB and CDPH SM; it is likely that most ABS filaments will exceed the two criteria for personal exposure. Benzaldehyde, acetaldehyde, and vinyl cyclohexene exceed one of the three risk table values with maximum and/or average predicted personal exposure concentrations.



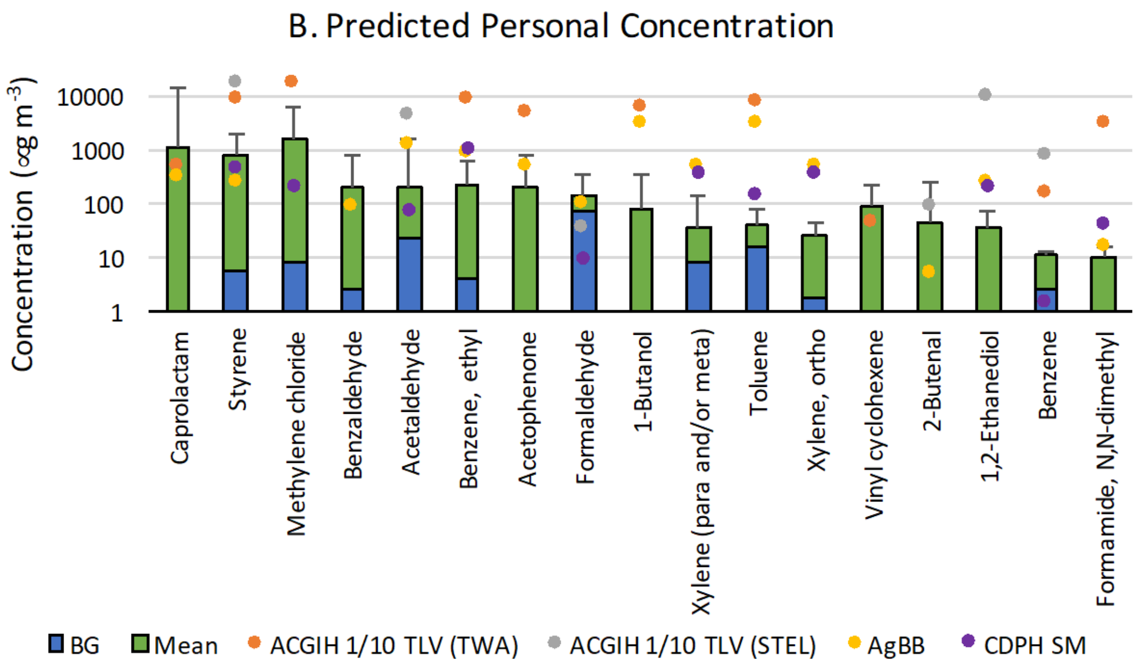


Figure 2. Model predicted concentrations for residential (A) and personal (B) scenarios, comparing to corresponding IAQ standards. Line extends to the predicted concentration using maximum ER measured. Residential background (BG) concentrations were from Logue et al.[23]

It is not unusual for one to be in an environment with exposure concentrations exceeding the criteria from 3D printers if users are not aware of the emission information and the mitigation strategies. However, emission reduction methods that a typical user can apply are limited and ineffective at reducing exposure levels especially for chemicals with low allowable concentrations. A standardized method to test and assess 3D printer emissions can lead to accurately measuring ERs, assessing their risks, and enabling manufacturers to produce lower emitting 3D printers and filament formulations. Differences exist between emissions of VOCs and UFPs for various filaments, therefore both must be considered when selecting a filament that minimizes possible adverse health effects. However, reduction of nozzle temperature or selecting a filament that operates with lowest nozzle temperature leads to least exposure to both VOC and UFPs.

CONCLUSIONS

TVOC and IVOC emissions from 3D printing vary largely among filament materials, while top emitting IVOCs were always associated with filament material monomers. The filament additives potentially contributed to the variance of IVOCs inconsistently detected among filaments. Specifically, styrene was released from ABS, caprolactam from nylon, lactide and methyl methacrylate from PLA. Printer operating conditions like nozzle temperature, filament type, filament and printer brand, and filament color all affected VOC emissions. The exposure modeling results showed the personal and room exposure concentrations of known or suspected to be carcinogens or irritants may exceed levels known to cause health effects especially to sensitive communities.

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