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Assessment and Quantification of Polybrominated Diphenyl Ethers (PBDEs) in Soils of E-Waste Dumpsites in Benin City, Nigeria

Osemudiamen Anao Edene[1*], Stanley O. Edene[3,4], Collins O. Eigbike[1], David J. Onaiwu[1], Daniel I. Olorunfemi[1], Iyere O. Onoagbe[2]

[1]Department of Environmental Management and Toxicology, University of Benin, BeninCity, Edo State.
[2]Department of Biochemistry, University of Benin, BeninCity, Edo State.
[3]Enville Institute of Management and Technology, Lagos, Lagos state.
[4]Department of Environmental Management and Toxicology, Federal University of Petroleum resources, Effurun, Delta State.

*Corresponding author; Email: osemudiamen.anao@uniben.edu, Tel: +234 706 185 7637

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ABSTRACT: Polybrominated diphenyl ethers (PBDEs) are a class of synthetic halogenated organic compounds used in a number of polymer-based commercial and home-use products. They are incoporated in the manufacture of electronics devices, furniture and textiles to increase resistance to flames and to meet set out fire safety standards. Generally, PBDEs are characterized by long range atmospheric transport, high bioaccumulation and toxicities. There is a dearth of information on the concentrations of PBDEs and the pattern of distribution at the various e-waste dump and dismantling sites in Nigeria. In this study, we assessed the concentration of PBDEs in areas where waste electrical and electronic equipment (WEEE) plastics have been dumped and dismantled over a period of time. Three (3) different locations in Benin City, Edo State, Nigeria, where these WEEE materials are dumped were studied. Soil samples were analysed using Gas Chromatography Mass Spectrometer (GC-MS). The results showed that most of the PBDEs assessed were below detectable limits (<0.01ng/g) but Octa brominated diphenyl ether (OctaBDE-79) was present at concentrations ranging from 1.91-1.94ng/g. Conclusively, PBDEs are present in WEEE dumpsites as burning releases some of the PBDEs present in these materials and also as a result of leaching from the WEEE plastics into the soil over time.

Keywords: Waste electrical and electronic equipment (WEEE), Polybrominated diphenyl ethers (PBDEs), Synthetic halogenated organic compounds, Dumpsites, Benin City

Introduction

Electronic waste also known as e-waste contains a wide range of substances, some of which are valuable economically whilst others are harzardous. Some of these harzardous substances have drawn so much attention environmentally and they include metals, products of incomplete combustion and/or reformation products (Osibanjo and Nnorom, 2007). Some of the organic compounds are persistent organic pollutants (POPs) such as Brominated Flame Retardants (BFRs) for example, Polybrominated Diphenyl Ethers (PBDEs), non-dioxin like Polychlorinated Biphenyls (PCBs), Polycyclic Aromatic Hydrocarbons (PAHs), Polychlorinated Dibenzo-p-dioxins, and Furans (PCDD/Fs) (Ohajinwa *et al*., 2019). BFRs are ubiquitous chemicals with large and global industrial use, and many of them are still produced in large volumes. Informal recycling sites for e-waste are located in most developing countries where environmental regulations are usually not adhered to strictly. Nigeria is a principal player in this and serves as a hub for importation of these waste electrical materials from

other more developed countries. BFRs are considered to be one of the most important flame retardants, making up about 25% halogenated organics produced annualy (Alaee *et al*., 2003). Polybrominated diphenyl ethers (PBDEs) are additive flame retardants that are present in many commercial and house products such as electronic devices (computers, televisions, electronic circuit boards, etc) plastics, furniture, carpets, toys, paints, textiles, foam and rubber (Alaee *et al*., 2003). PBDEs are environmentally persistent chemicals. Some, especially the lower brominated congeners (e.g. "penta-BDE"), are also highly bioaccumulative. Their manufacture and use as additives in plastics and other polymers, in which they are not tightly bound to the polymer matrix, has led to their widespread presence in the environment and this may pose some significant environmental health risk (UNEP, 2007, Brigden *et al*., 2008, Chen *et al*., 2018).

Environmental concerns about brominated flame retardants (BFRs) such as the polybrominated diphenyl ethers (PBDEs) has increased over the last two decades (Covaci *et al*., 2003). Polybrominated diphenylethers can be leached into the environment as a result of disposal when polymeric substances or products with sizeable quantities of PBDEs are exposed to environmental conditions that support their leaching (Olutona *et al*., 2017). The receptors of man-made organic pollutants are commonly soils and sediments at the bottoms of rivers and water reservoirs. Studies conducted at Chinese (Tang *et al*., 2010; Wang *et al*., 2012; Labunsk *et al*., 2013; Chakraborty *et al*., 2018), Indian (Huang *et al*., 2011, Li *et al*., 2016), Nigerian (Adetunde *et al*., 2014, Isimekhai *et al*., 2017) and Ghanaian (Daso *et al*., 2016; Tue *et al*., 2016; Akortia *et al*., 2017) e-waste sites found large concentrations of heavy metals and organic contaminants such as PBDEs, PCBs and polycyclic aromatic hydrocarbons (PAHs) in the air, in dust, soil, vegetation and in the blood of workers and residents on and in the vicinity of the studied sites (Moeckel *et al*., 2020).

PBDEs are released into the environment by abrasion of the polymer during its life cycle and release is accelerated by heating, also PBDEs can be transported with dust (Frederiksen *et al*., 2009; Kalachova *et al*, 2012). From this point of view, existing products containing PBDEs are a reservoir of these POPs. Uncontrolled burning of e-waste plastics also contributes to the emissions of PBDEs which remain in the ashes and more toxic chemicals such as polybrominated dibenzofurans and dibenzo-p-dioxins (PBDD/Fs) could also be generated (Estrellan and Iino, 2010; Sepúlveda *et al*., 2010).

Several studies have shown high concentrations of heavy metals, PBDEs or PAHs in soil and vegetation at Agbogbloshie (Daso *et al*., 2016; Akortia *et al*., 2017; Fosu-Mensah *et al*., 2017, Oteng-Ababio *et al*., 2020). Moeckel and his co-investigators reported that PBDEs concentrations in Agbogbloshie area of Ghana ranged between 6.3 and 7,700 ng/g. Agbogbloshie samples showed significantly higher ($p = 0.010$) total PBDE concentrations than Kingtom samples where 1.2–100 ng/g were found. The concentrations at both sites were much higher than those reported for background areas in Kenya and Tanzania (Moeckel *et al*., 2020).

Typically, PBDE concentrations in soils are of the order of several ng/g (Thorez *et al*., 2010). In soils near Bratislava where the concentrations of ΣPBDEs ranged from 0.087 to 0.627ng/g (Thorez *et al*., 2010).

In rural areas and woodlands in UK and Norway, the concentrations of ΣPBDEs ranged from 0.065 to 12ng/g. On a local scale the increase in the PBDE levels was detected in flooded areas and in agricultural regions (up to several hundred ng/g), where the sewage sludge is used (Eljarrat *et al*., 2004) or where the land is irrigated with treated effluents (Zhang *et al*., 2008).

There is a dearth of information on PBDE concentrations in Edo State, Nigeria hence this study assessed soils of e-waste dump sites identified in Benin City, Edo State.

Materials and Methods

Study area: The samples were collected from three sampling locations representing Ikpoba Okha and Oredo Local Government areas of Benin City, Edo State, Nigeria. The first sampling location designated **S1** as shown in Figure I, is located at Mission road on an adjoining street called Iwehen street, where local e-waste collectors gather and dismantle these e-waste materials and extract the plastics for sale to recyclers.

The second sampling location designated **S2** as shown in Figure 1, is also located at Mission road on an adjoining street called Idahosa Lane where these e-waste materials were dumped for over a decade but was excavated for other uses about three(3) months prior to sample collection in May, 2017.

The third sampling location designated **S3** as shown in Figure 1 is located at Sapele road on an adjoining street called NEPA Street, where local e-waste collectors gather and dismantle these e-waste materials and extract the plastics for sale to recyclers. The control samples were collected about 300 metres away from each sampling location.

Table 1: Designation, location and GPS coordinates of the sampling locations in this study

Designation	Location	GPS Coordinates
SI	Upper Iwehen Street	N6.342 º E5.625º, Elevation 318ft
C1	Control 1	N6.342 º E5.627º, Elevation 318ft
S2	Idahosa Lane	N6.341 º E5.625º , Elevation 306ft
C2	Control 2	N6.341º E5.628º, Elevation 306ft
S3	NEPA Street	N06º.17´ 41.88´´, E05º 37´ 42.26´´ Elevation 205ft
C3	Control 3	N06º.17´ 68.35´´, E05º 42´ 69.44´´ Elevation 190ft

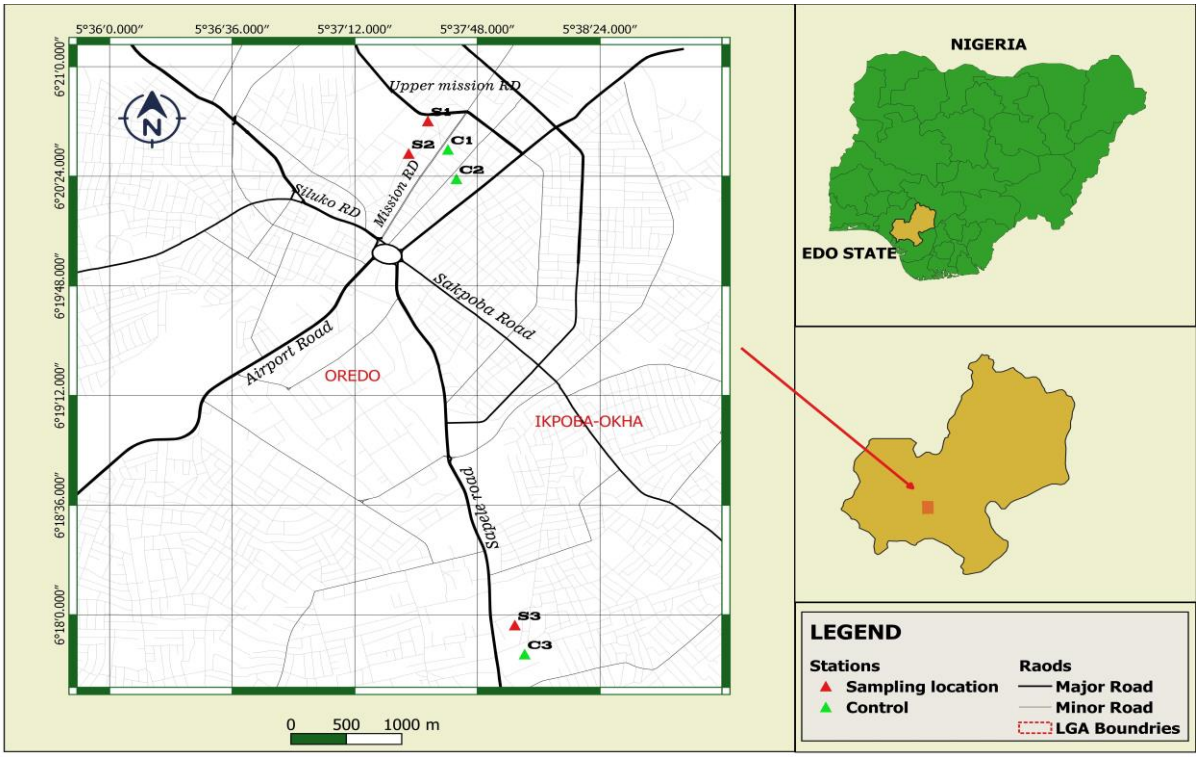

Figure 1: Map of study area showing sample collection points at S1, S2 and S3

Key: SI-Iwehen street, S2-Idahosa Lane, S3-NEPA Street; C1, C2, C3-Control

Collection of samples: On the field for the soil sampling, each selected site was divided into grids of about 5-10m wide depending on the size of the site. Samples were systematically collected from three to five points within each site. Samples were collected with the aid of a sterile hand held soil auger at a depth of 0-15 cm (top soil) and 15-30 cm (sub soil), respectively. A composite mixture was obtained from both top and sub soils and a soil trowel was used to transfer soil from the auger into aluminium foil to avoid cross contamination, the soil auger and trowel were decontaminated (cleaned first with a brush and wiped thoroughly with wipes before sample collection at each sampling site. They were kept in aluminium foil, placed in polyethylene zip bags and transported to the laboratory.

A total of thirty (30) representative samples [five (5) from Iwehen street (S1), five (5) from Idahosa Lane (S2)] were collected at five (5) sampling points (SP) in triplicates on the identified e-waste dumpsites. At NEPA Street (off Sapele road), a total of nine (9) representative samples were collected from three (3) sampling points on the e-waste dump site in triplicates.

They were then transported to the laboratory where the samples were homogenized, ground with a mortar and pestle, and sieved through a 2 mm mesh sieve to remove bigger particles. Next, they were transferred into individual 10 ml amber bottles, labelled and stored at -4ºC until chemical analysis began.

Methodology

Extraction and clean up: The dried samples were sieved (with 1mm mesh) to eliminate extraneous materials before extraction. The USEPA, method 3570 (USEPA, 2002) was adopted for the extraction of soil samples. 20g of air dried and pre-sieved soil samples were transferred into a quick-fit extraction flask with a firmly fitted stopper. 5 g of homogenized sample was thoroughly mixed with 0.6 g DE with a mortar and pestle. Each sample was thereafter spiked with 2 ng 13C- labeled PCB-208 and 10 ng PCB-209 standards, and allowed a static equilibration of 5 minutes in two cycles. 5 g of homogenized sample was thoroughly mixed with 0.6 g DE with a mortar and pestle. Each sample was thereafter spiked with 2 ng 13C- labeled PCB-208 and 10 ng PCB-209 standards, and allowed a static equilibration of 5 min in two cycles. The sample was then extracted using an ASE 350 accelerated solvent extraction system (Dionex, USA) with n-hexane/dichloromethane (v/v, 1/1) at 90°C, 1500 psi. After extraction, acid washed copper sheets were added to the extracts to remove sulfur present in the samples. The extracts were evaporated to about 10mL under a gentle stream of N_2, and transferred to a conical centrifuge tube. 1 ml of concentrated sulfuric acid (98 %) was added to the concentrated extracts to carbonize part of the impurities present. The supernatants were transferred to a preconditioned glass cleanup column, which was packed with 0.5 cm neutral aluminum oxide, 3.0 cm neutral silica gel, 3.0 cm acid silica gel, and 1.0 cm anhydrous sodium sulfate from the bottom to the top. The columns were then eluted with 20 ml hexane, and the eluent was evaporated to about 0.3 ml and transferred to a 1.5 ml sample vial. After the internal standard (10 ng BDE-77) had been added to the vial, the volume of the solution was made up to 0.5 ml.

GC-MS analysis of samples: The samples (1µl) were injected in splitless mode. Helium was used as carrier gas at a flow rate of 1.2 ml/min, and the temperature program was set as follows: 90°C for 2 min, increased to 320 °C at 15 °C/min and held for 7 min. The temperature of GC inlet, transfer line, ionization source and quadrupole were set at 290 °C, 300 °C, and 150 °C. The compounds were monitored for brominated BDEs at, m/z 79, 81, 487 and 489 for BDE-47, 99, 153 and 209, m/z. A temperature programme was used in conjunction with the gas chromatograph to facilitate separation of PBDE congeners. Detection was achieved by a Mass Spectrometer.

Results

The concentrations of PBDEs obtained from the analyzed samples at S1 (Iwehen Street) is presented in Table 2. It was observed that the concentrations of PBDEs (decaBDE-209, pentaBDE-99, tetraBDE-47 and hexaBDE-153) ranged from below detectable limits to 1.94 ng/g in all of the sampling points. Pecisely, samples from Sampling Point 2 (SP2), were < 0.01ng/g which did not differ from the control, for decaBDE-209, pentaBDE-99, tetraBDE-47 and hexaBDE-153, while octaBDE-79 was detected at Sampling Point 1 (SP1), Sampling Point 3 (SP3), Sampling Point 4 (SP4) and Sampling Point 5 (SP5) at 1.91 ng/g, 1.92ng/g, 1.94ng/g and 1.91ng/g concentrations respectively. This was a higher concentration when compared with the control.

The results for the concentrations of PBDEs in the samples obtained from the second location at S2 (Idahosa Lane) is presented in Table 3. The concentrations of all the components of PBDEs (decaBDE-209, octaBDE-79, pentaBDE-99, tetraBDE-47 and hexaBDE-153) measured were below (< 0.01ng/g) detectable limits this was similar with the concentrations of PBDEs at the control site.

The result obtained from the analyzed samples at S3 (NEPA Street) as shown in Table 4 gives details of the concentrations of the PBDE congeners (decaBDE-209, octaBDE, pentaBDE-99, tetraBDE-47 and hexaBDE-153). The measured components were below (< 0.01 ng/g) detectable limits.

Table 2: PBDE mean concentrations (ng/g dry weight) on soil samples from an e-waste dumpsite at Iwehen Street (S1), Benin-City, Edo State Nigeria

PBDE	Control	SP1	SP2	SP3	SP4	SP5
DecaBDE-209	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
OctaBDE-79	0.00±0.00	1.91±0.03	0.00±0.00	1.92±0.02	1.94±0.03	1.91±0.00
Penta BDE-99	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Tetra BDE-47	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Hexa BDE-153	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
∑PBDEs (ng/g)	**< 0.01**	**1.91**	**< 0.01**	**1.92**	**1.94**	**1.91**

Values are expressed as Mean± SEM, n=3

Table 3: PBDE mean concentrations (ng/g dry weight) on soil samples from an e-waste dumpsite at Idahosa Lane (S2), Benin-City, Edo State Nigeria

PBDE	Control	SP1	SP2	SP3	SP4	SP5
DecaBDE-209	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
OctaBDE-79	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Penta BDE-99	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Tetra BDE-47	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Hexa BDE-153	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
ΣPBDEs (ng/g)	**< 0.01**	**< 0.01**	**< 0.01**	**< 0.01**	**< 0.01**	**< 0.01**

Values are expressed as Mean± SEM, n=3

Table 4: PBDE mean concentrations (ng/g dry weight) on soil samples from an e-waste dumpsite at NEPA Street(S3), Benin-City, Edo State Nigeria

PBDE	Control	SP1	SP2	SP3
DecaBDE-209	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
OctaBDE-79	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Penta BDE-99	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Tetra BDE-47	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Hexa BDE-153	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
ΣPBDEs (ng/g)	**< 0.01**	**< 0.01**	**< 0.01**	**< 0.01**

Values are expressed as Mean± SEM, n=3

Discussion

Over the last two decades there have been indications of increased environmental PBDE concentrations, although their levels are still generally lower than those of PCBs, due to different usage volumes (De Wit, 2002).

PBDEs do not occur naturally; their presence was not registered in deep rock strata (Van *et al*., 2008). Manufacturing processes, decomposition of products containing PBDEs, and dismantling and recycling of scrapped electronic devices are the main sources of PBDE emissions throughout the entire lifecycle of these products. PBDE concentrations in areas distant from local sources of pollutants are of the order of several pg/m^3 The highest concentrations were registered in the neighbourhood of the electronics recycling plants. The receptors of man-made organic pollutants are commonly soils and sediments on the bottoms of rivers and water reservoirs (Winid, 2015).

Typically, PBDE concentrations in soils are of the order of several ng/g. In soils near Bratislava the concentrations of ΣPBDEs ranged from 0.087 to 0.627 ng/g (Thorez *et al*., 2010). In rural areas and woodlands in UK and Norway, the concentrations of ΣPBDEs ranged from 0.065 to 12 ng/g. On a local scale the increase in the PBDE levels was detected in flooded areas and in agricultural regions (up to several hundred ng/n), where the sewage sludge is used or where the land is irrigated with treated effluents. In Nigeria, Ohajinwa, *et al*., reported that PBDE congeners were detected in all sampling sites studied across Lagos, Ibadan and Aba, indicating that PBDEs were widespread pollutants in this research area. The total concentration (∑13PBDE) ranged from 1.702 to 149,770.560 ng/g. The most abundant PBDE congener in all the sites and samples was BDE-209, with concentrations ranging from 0.850 to 147,091.400 ng/g at e-waste dismantling sites in Lagos, Ibadan and Aba (Ohajinwa, *et al*., 2019). This finding was inconsistent with that from our studies as we recorded concentrations mostly below detectable limits.

In this study, different components of polybrominated diphenyl ethers were analyzed for in soil collected from three different locations. It is not uncommon for low recoveries of PBDEs in soil samples, as some other studies have also reported concentrations ranging from undetectable to very low (La Guardia *et al*., 2006, Brown *et al*., 2006, Bodin *et al*., 2007). The concentration of octaBDE-79 (Table 2) seen in this study is in agreement with concentrations reported by Ohajinwa *et al*., that total PBDE concentrations (∑13PBDE) ranged from 1.702 to 149,770.560 ng/g in Lagos, Ibadan and Aba.

Furthermore, results obtained from our other sampling locations in this study (Table 3 and 4), shows that all components analyzed were below detectable limits (<0.01 ng/g). This result did not agree with some studies

where high concentrations of PBDEs were reported (Adetunde *et al*., 2014; Daso *et al*., 2016; Tue *et al*., 2016; Akortia *et al*., 2017 and Isimekhai *et al*., 2017). These values negates that reported by Ohajinwa *et al*. (2019); whose report showed values with concentrations ranging from 0.850 to 147,091.400 ng/g with BDE-209 being the most abundant PBDE congener in all the sites, although it was consistent with the findings of Oros *et al*., who reported falls in the range of concentrations of PBDEs in water from different parts of the globe which mostly ranged between (<0.1 and 500ng/mL) (Oros *et al*., 2005). Tang *et al*. (2010); Wang *et al*. (2012); Labunsk *et al*. (2013); Chakraborty *et al*. (2018); Li *et al*. (2016); Adetunde *et al*. (2014); Isimekhai *et al*. (2017); Daso *et al*. (2016); Akortia *et al*. (2017) and Thorez *et. al*. (2010) reported high concentrations of PBDEs in their study sites. In trying to understand the reason for these deviations, we tied the result seen in our study location at S2 to the fact that the activities of gathering and dismantling these WEE equipment was stopped few months before our sampling took place and the land had been excavated. Also, the low detection especially at sampling location 3, could be attributed to the detection efficiency of the equipment used and the extraction eficiency, as some other studies have shown that some of the detection equipment may not be so sensitive, which may be responsible for poor detection hence higher resolution equipment could be employed for proper detection of most cogeners of the PBDEs (Xiao *et al*, 2007, Pietroń and Małagocki, 2017).
Thermal degradation and mass discrimination of higher molecular weight compounds are main drawbacks of this technique (Björklund *et al*., 2004). Therefore, injector temperature and splitless time need to be optimised for maximum sensitivity. Abou-Elwafa reported that thermal degradation and isomeric interconversion are the main challenges facing analytical chemists with the GC/MS analysis of BFRs. Therefore, several parameters of the GC/MS system need to be carefully optimised according to the properties of target analytes. This includes injection technique, stationary phase, column dimensions, and mass spectrometric parameters (Xiao *et al*, 2007, Abou-Elwafa, 2014).

Conclusion

This result therefore shows that PBDEs are present in soils where waste electrical and electronic equipment are dumped as burning releases some of the PBDEs present in these materials and also as a result of leaching from the WEEE plastics into the soil over time. Although most of the measured PBDEs in this study were below detectable limits, this we strongly attribute to the detection sensitivity of the equipment used. Hence, there is a need for more studies on e-waste dumpsites in Benin City with more advanced methods of analysis of PBDEs in order to confirm concentrations of these congeners in her soil relative to that from other studies in Nigeria. Also, measures should be put in place to monitor the disposal methods as well as management of these e-waste materials so as to eliminate potential environmental and health effects.

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