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Simultaneous targeted and non-targeted analysis of plastic-related contaminants in e-waste impacted soil in Agbogbloshie, Ghana

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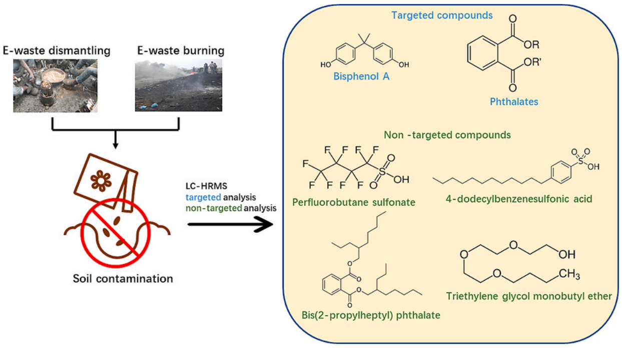
HIGHLIGHTS

- DEHP and BPA were the two dominant targeted contaminants in e-waste soils.
- Non-targeted analysis was applied to e-waste soils for the first time.
- Plastic-related chemicals, some previously unreported, were identified.

GRAPHICAL ABSTRACT



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ABSTRACT

An LC-MS based analytical method was developed and validated for the simultaneous targeted analysis and suspect screening of plastic-related contaminants in e-waste impacted soils. Satisfactory recoveries (97 ± 13 %) were achieved using ultrasound-assisted extraction for 14/15 of the targeted analytes (7 bisphenols and 8 plasticizers) in a range of agricultural and non-agricultural soils. The method was applied to 53 soil samples collected in May 2015 in the region of Agbogbloshie (Ghana) at e-waste facilities (incl. Dump, trade, and burn sites), neighboring non-agricultural (incl. upstream, downstream, and community) and agricultural fields, and at two control agricultural sites away from e-waste recycling facilities. Bisphenol A (BPA) and bis(2-ethylhexyl) phthalate (DEHP) were the two dominant contaminants in e-waste soil (with concentrations up to 48.7 and 184 $\mu g\ g^{-1}$, respectively), especially at the trade site, where e-waste was sorted and dismantled. The non-targeted workflow was successfully applied to identify additional plastic-related contaminants previously unreported in e-waste impacted soils, including bis(2-propylheptyl) phthalate, diisononyl phthalate, trioctyl trimellitate, 4-dodecylbenzenesulfonic acid, perfluorooctanesulfonic acid, perfluorobutanesulfonic acid, diphenyl phosphate, and triethylene glycol monobutyl ether. The agricultural soils surrounding the e-waste sites were also contaminated by plastic-related chemicals (especially DEHP), highlighting the impact of e-waste activities on the surrounding agricultural system.

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1. Introduction

Rapid technological developments since the 1990s have led to an unprecedented increase in the global production of electronic products (Forti et al., 2020; Murthy and Ramakrishna, 2022). High levels of disposable income, along with increasing urbanization and industrialization, have resulted in growing amounts of electronic waste (e-waste or also known as waste electric and electronic equipment) (Forti et al., 2020; Tanskanen, 2013), which refers to electric and electronic equipment that has been discarded by its owner as waste without the intent of reuse (Forti et al., 2020).

Electric and electronic equipment can be classified into six general categories according to waste management characteristics (Forti et al., 2020): temperature exchange equipment, screens and monitors, lamps, large equipment, small equipment, and small IT and telecommunication equipment. The composition and structure of e-waste products can vary across and within these different categories (Tanskanen, 2013), but they mainly contain metals and plastics. Metals can be largely recovered, while plastics are often separated from the metal parts and incinerated or disposed of because they are considered impurities in recycling (Tanskanen, 2013). Plastics are estimated to constitute around 30 % of e-waste by weight (Schlummer et al., 2007).

Toxic substances are released during the combustion or disposal of plastics, leading to contamination of the local environment as well as potential adverse effects on human populations (Daum et al., 2017; Moeckel et al., 2020). Polybrominated diphenyl ethers, polychlorinated biphenyls, and polychlorinated naphthalenes are considered as legacy chemical contaminants (used for electric and electronic products production) and have been reported in e-waste impacted environmental matrices and biological samples from people working at or living near e-waste sites (Cai et al., 2020; Han et al., 2010; Moeckel et al., 2020; Niu et al., 2021). In addition to the legacy chemical contaminants mentioned above, some contaminants of emerging concern have been reported in e-waste-impacted environmental matrices such as water (Huang et al., 2014b), soil (Huang et al., 2014a; Ma et al., 2013), air (Gravel et al., 2019), and dust (Deng et al., 2021). These chemicals include organophosphate esters (OPEs) possibly used as replacements for legacy flame retardants (Blum et al., 2019), bisphenols used in the production of polycarbonate and epoxy resins (Zhang et al., 2019a), and phthalates used as plasticizers for thermoplastics (Deng et al., 2021). These chemicals are classified as plastic-related chemicals (PRCs), which are used for the production of plastics (e.g., monomers and polymers residual, plasticizers, and antioxidants, etc.) and their degradation products (Tian et al., 2019; Tisler and Christensen, 2022).

Considering that a majority of these PRCs have not been investigated in e-waste impacted soil, there is a need to expand the chemical coverage of current monitoring programs based on targeted analytical methods, to include potential unknown and unexpected chemical contaminants, and discover as early as possible any potential toxicants. Using high resolution mass spectrometry (HRMS), such as quadrupole time-of-flight (QToF), for non-targeted data acquisition is capable of capturing more comprehensive contaminant profiles of environmental matrices (Mullin et al., 2020) owing to its high mass accuracy for molecular formula generation (Knolhoff et al., 2016).

In the present study, a method based on ultrasound-assisted liquid extraction and LC-QToF-MS analysis was developed and applied for the

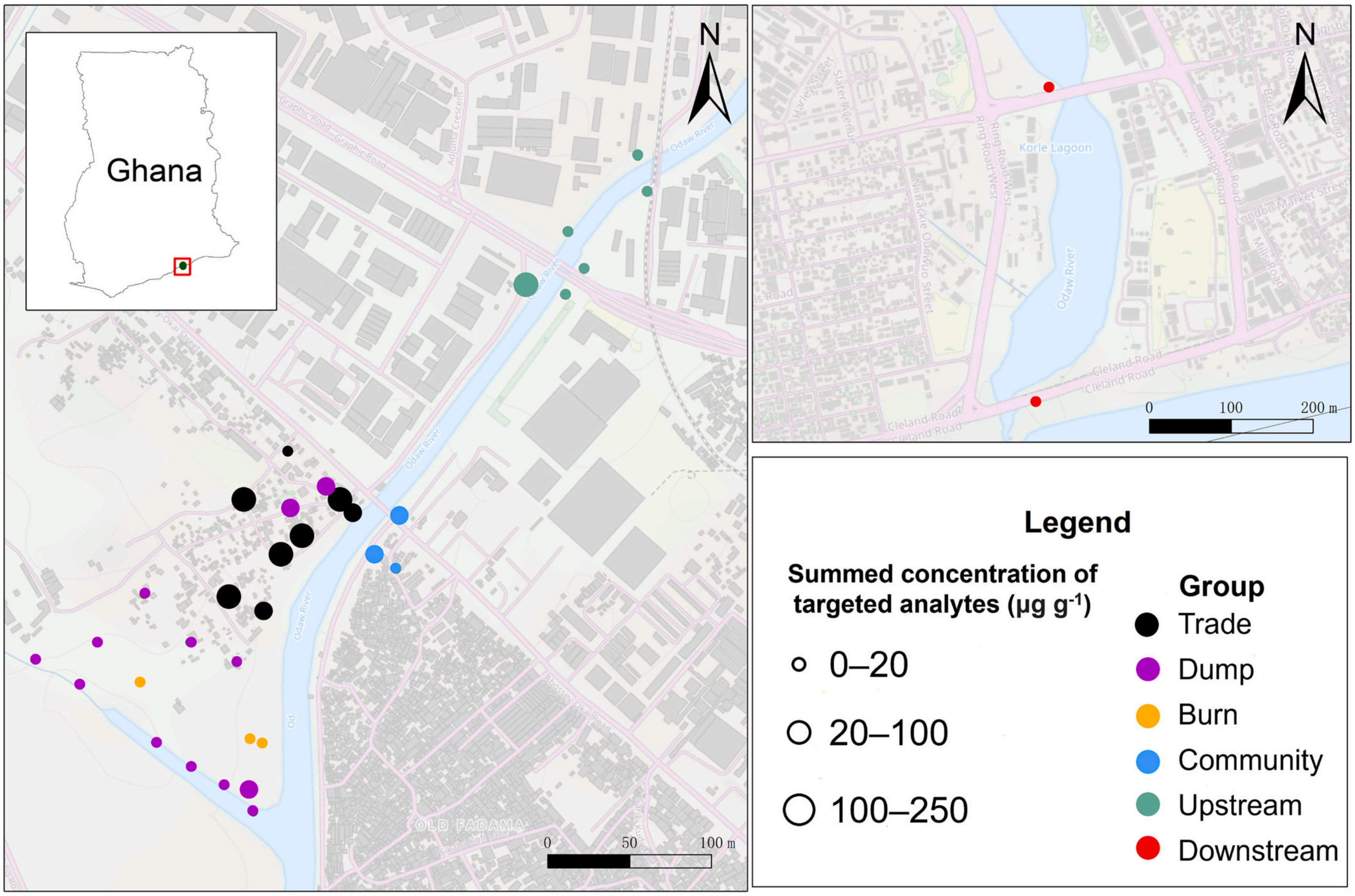

Fig. 1. Sampling locations of non-agricultural e-waste impacted soils in Agbogbloshie, Accra, Ghana, as well as the spatial distribution of the summed concentrations of targeted analytes in these soils. The size of dots reflects the range for the sum concentration of all targeted analytes. Samples from different groups are demarcated using dots in different colors as detailed in the legend. The map on the left (A) displays the locations of e-waste, community, and upstream sites, while the smaller map on the right (B) shows the two downstream sampling sites. The sampling map was created using ArcGIS Pro 3.1.

simultaneous targeted analysis of 7 bisphenols and 8 plasticizers, and non-targeted screening of other PRCs in e-waste impacted soil collected in Agbogbloshie, Ghana. Among the 15 targeted analytes, 6 bisphenol analogues (bisphenol S (BPS), bisphenol F (BPF), bisphenol AF (BPAF), bisphenol B (BPB), bisphenol E (BPE), and bisphenol AP (BPAP)) and 1 plasticizer (diheptyl phthalate (DHpP)) have not been studied before in e-waste impacted soils. To the best of authors' knowledge, the current study is the first to perform non-targeted analysis of organic chemical contaminants in e-waste related soils (including the agricultural soils impacted by e-waste activities). This is also the first study on the levels of bisphenols and phthalates in the e-waste and e-waste impacted agricultural soils in Agbogbloshie, Ghana.

2. Methods and materials

2.1. Chemicals and analytical standards

The analytical standards for 7 targeted bisphenols, 8 targeted plasticizers, 14 mass-labelled surrogates, and other compounds were purchased from various vendors (Section 1.1 of Supplementary Materials for details and purity). Ammonium acetate (LC–MS grade) and HPLC grade solvents (methanol, 2-propanol and water) were purchased from Fisher Scientific (Whitby, Canada). Stock solutions of the individual target compounds were prepared in methanol at 1000 μg mL^{-1}. Two working standard mixtures (1 μg mL^{-1} each, in methanol) were prepared the day before use for the seven targeted bisphenols (bisphenol A (BPA), BPS, BPF, BPAF, BPB, BPE, BPAP) and the eight targeted plasticizers (bis(2-ethylhexyl) phthalate (DEHP), DHpP, dipentyl phthalate (DPP), diisobutyl phthalate (DiBP), dibutyl phthalate (DBP), butyl benzyl phthalate (BBzP), diethyl phthalate (DEP), bis(2-ethylhexyl) adipate (DEHA)). The isotope-labelled internal standard mixture solutions (BPAF-^{13}C$_{12}$, BPS-^{13}C$_{12}$, BPA—^{13}C$_{12}$, BPF-^{13}C$_{12}$, DEP-d$_{14}$, DiBP-d$_4$, BBzP-d$_4$, dicyclohexyl phthalate-d$_4$ (DcHP-d$_4$), DEHP-d$_{38}$, and di-n-octyl phahalte-d$_4$ (DnOP-d$_4$) was also prepared at 1 μg mL^{-1} in methanol the day before use. All the standard solutions were prepared in amber glass vials and stored in −20 °C freezer. The mortar and pestle were washed with soap and rinsed with 2-propanol three times and dried in a fume hood between samples, in order to avoid cross-contamination. Amber glass vials for standards and soil samples were baked at 320 °C for 4 h prior to use.

2.2. Study sites

Fifty-three soil samples were collected in this study in May 2015 (Supplementary Fig. S1A and Supplementary Table S1), including twenty-four from e-waste sites, eleven from e-waste surrounding sites, and eighteen from agricultural sites. The e-waste sites are situated on the banks of the Korle Lagoon in Agbogbloshie (Ghana). Trade site (13), dump site (8), and burn site (3) soil samples were collected directly at the e-waste sites. Samples that were collected from upstream (6), downstream (2), and community (3) sites will be collectively referred to as e-waste surrounding sites. The upstream and downstream samples were collected from the roadside near the upstream and downstream of the Korle Lagoon, respectively. The sampling locations are shown in Fig. 1. Twelve agricultural soil samples used for cultivating vegetables were collected from some gardens near an e-waste dump site in Agbogbloshie (Ghana). Six reference agricultural samples were collected from a garden over 100 km away from the e-waste sites in Asesewa (Ghana). In addition, six additional agricultural soils were collected for method development at the Horticultural Center (3) and Macdonald Student-Run Ecological Gardens (3) of Macdonald Campus of McGill University (Sainte-Anne-de-Bellevue, Canada). These soil samples were considered to be relatively "clean" control samples, as there are no known sources of contamination in these soils used for conventional and organic farming, respectively.

2.3. Sample collection and preparation

Each surface soil sample (approximately 10 g) was collected from the top half inch (approximately 1.5 cm) soil while avoiding collecting rocks, twigs, and roots. Each soil sample from Ghana was preserved in an individual Whirl-Pak® (polyethylene) bag and shipped to McGill University (Canada). The samples were then transferred into 20 mL amber glass vials and freeze-dried (Flexi-Dry MP) at −90 °C for 24 h. The freeze-dried samples were further sieved (1 mm^2 stainless steel) to remove large rocks and roots, homogenized using mortar and pestle, and finally stored into amber glass vials in the dark at −20 °C until analysis.

Extracts were also prepared from the Whirl-Pak® bags made of polypropylene ($n = 3$) with 10 mL methanol (1 h, 23 °C) and analyzed to test for potential chemical leaching from the polyethylene bags used for sampling.

2.4. Sample extraction

The extraction procedures were adapted from Liu et al. (2021). Briefly, 1 g (±0.05 g) of soil was weighed and transferred into a 50 mL polypropylene centrifuge tube. Acetonitrile (4 mL) was added, and vortexed for 30 s., followed by ultrasound sonication for 30 min using a Branson 3510 sonication bath (40 kHz). Extracts were centrifuged (3000 ×*g*, 10 min.), and 1.5 mL of the supernatant was transferred into a 10 mL glass vial and spiked with 30 μL of the internal standard mixture (1 μg mL^{-1}). This aliquot was concentrated under nitrogen gas to ~100 μL and reconstituted to ~1 mL using methanol. The final extract was filtered through a 0.22-μm PTFE filter (Fisher Scientific, Whitby, Canada) prior to LC-QToF-MS analysis. Internal standards were purposely added after the extraction (as another aliquot of the extracts was used for toxicity assessment). Method validation data are presented in Supplementary Tables S2–S5.

Based on the preliminary analysis of the samples (data not shown), further dilution was required for the agricultural e-waste, trade, dump, burn, and e-waste surrounding sites extracts in order to fit the concentrations of the targeted analytes into the linear range of the matrix-matched calibrations and reduce the strong matrix effects. Extracts were diluted using an internal standard mixture solution as proposed by

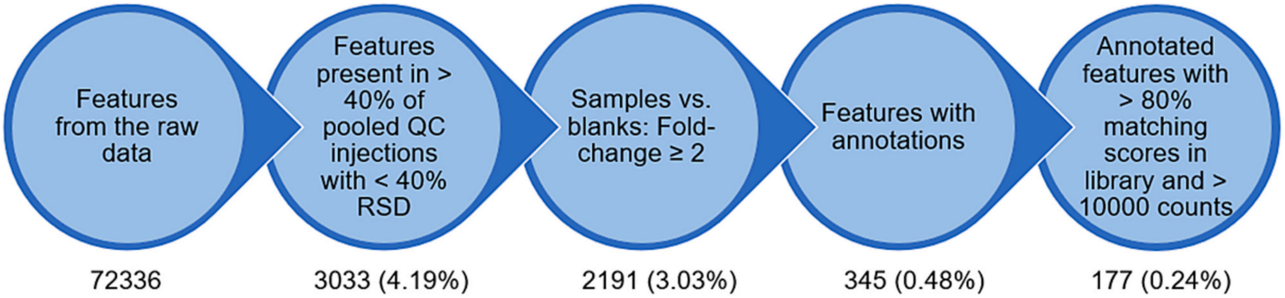

Fig. 2. Prioritizing features for identification in the non-targeted workflow and the number of features in each step (the percentage of the total original features from the raw data is also indicated).

Wang and Zhou (2016), a strategy maintaining the concentrations of the internal standards identical during the dilution. For the agricultural e-waste soil extracts, a 2-fold dilution was applied using methanol containing 30 ng mL^{-1} of the isotope-labelled internal standard mixture solution. Trade, dump, burn, and e-waste surrounding sites extracts were diluted 10 times using the same internal standard mixture solution. The quantification of DEHP in e-waste sites (trade, dump, and burn) and e-waste surrounding sites specifically required a 200-fold dilution.

2.5. Instrumental analysis

Extracts were analyzed using an Agilent 1290 Infinity II LC system equipped with Agilent 6545 QToF-MS (Agilent Technologies, Santa Clara, USA) in both positive and negative electrospray ionization (ESI+ and ESI-). The detailed settings and parameters of the LC separation and MS data acquisition are presented in Section 1.4 of Supplementary Materials.

2.6. Targeted analysis of the 7 bisphenols and 8 plasticizers

The quantification of the target analytes was performed using Quantitative Analysis (B.10.00) from Agilent MassHunter Workstation Software. The most abundant isotope of either $[M + H]^+$ in ESI+ or $[M-H]^-$ in ESI- was selected as the quantifier ion for each target analyte (Supplementary Table S2). A mass extraction window of ±20 ppm and retention time (RT) of ±0.15 min were applied for the extraction of extracted ion chromatograms (EICs) with symmetric and sharp peak shape (Liu et al., 2021). The concentration of each analyte was calculated using relative response factor, and the formula is presented in Section 1.3 of Supplementary Materials.

The performances of the extraction and instrumental methods (Supplementary Tables S2-S5) were assessed using procedural blanks ($n = 10$), solvent blanks ($n = 10$), solvent and matrix-matched calibrations, and recovery samples (spiking). The background signals for the targeted analytes in blanks are presented in Supplementary Table S9, and the sample-to-blank ratios of targeted analytes are shown in Supplementary Fig. S2). Method detection limits (MDLs), matrix effects, recoveries, precisions, and mass accuracy for the target compounds are detailed in Sections 1.3 and 2 of the Supplementary Material.

2.7. Suspect screening and non-targeted analysis

Full-scan (MS1) data including samples, pooled QC injections, and procedural blanks were imported to Agilent Profinder (B.10.00) for retention time alignment and feature extraction under the "*Batch Molecular Feature Extraction*" mode. Agilent Profinder parameters for retention time alignment and molecular feature extraction are listed in Supplementary Table S6. Results were exported as Profinder Archive files (.pfa files) and later imported to Agilent Mass Profiler Professional (MPP, version B14.0) software for feature filtration and peak identification. Identifying all the extracted features in a non-targeted analysis is presently not feasible due to the limitation of chemical databases and spectral libraries. Thus, features with relatively high abundance and stability were prioritized for further identification and confirmation during the non-targeted workflow in this study (Fig. 2). Data were firstly log2 transformed and scaled with Pareto algorithm. A QC-based data filtration step was applied in this study (adapted from Gravert et al. (2021)) to select features present in >40 % of the pooled QC injections with <40 % RSD). Then, features with a fold-change value >2 in samples compared with their respective procedural blanks (e.g., with the same dilution factor) were retained, and tentatively annotated using the *Agilent Extractables & Leachables PCDL* (Personal Compound Database and Library) (1006 compounds). Tentatively annotated features with matching scores >80 % were subsequently manually inspected for their peak shape and peak height, and features with good peak shape were included in a final list of suspects to investigate. Targeted MS/MS data were obtained for the pooled QC and compared with experimental MS/MS from the literature or spectral libraries (https://mona.fiehnlab.ucdavis.edu/), or with predicted *in-silico* MS/MS spectra (CFM-ID; https://cfmid.wishartlab.com/) when experimental MS/MS information were not available. Finally, targeted MS/MS data were also analyzed with SIRIUS (v 5.5.7, https://bio.informatik.uni-jena.de/software/sirius/) for molecular formula prediction, fragmentation tree construction, and structure prediction based on MS/MS fragmentation patterns at different collision energies (Dührkop et al., 2019). Parameters are listed in Supplementary Table S7. As a best practice in the field, the NTA Study Reporting Tool (SRT) was used in the preparation of this manuscript (Peter et al., 2021) and is provided in a separated supplementary document "220,912-SRT_V2".

In additional to the features identified through library search, the identity of the top 10 largest peaks without annotations in ESI+ and ESI- after the "ID Browser" steps were also investigated using targeted MS/MS, with subsequent interpretation MS/MS from data from the literature, libraries, in silico prediction, using SIRIUS and eventually through comparison with pure standards.

The final list of suspect identities (level 1 and 2 identification confidence) with *m/z* (Schymanski et al., 2014), retention time, annotations and MS/MS matching information is summarized in Supplementary Table S8. Detailed information for the filtration and identification steps is shown in Section 4 of the Supplementary Material.

Table 1
Detection frequencies and concentrations of the 8 detected target analytes in e-waste impacted and reference agricultural soils.

		DEP	DBP	DiBP	BBzP	DHpP	DEHP	DEHA	BPA
Reference agricultural ($n = 6$)	DF	17 %	17 %	33 %	0 %	0 %	17 %	0 %	0 %
	Min	N/D	N/D	N/D	N/D	N/D	N/D	N/D	N/D
	Max	1320	19.4	133	N/D	N/D	72.2	N/D	N/D
	Mean	220	3.23	33.2	N/A	N/A	12.0	N/A	N/A
Agricultural e-waste ($n = 12$)	DF	0 %	67 %	67 %	100 %	100 %	100 %	8 %	83 %
	Min	N/D	N/D	N/D	2.35	9.45	800	N/D	N/D
	Max	N/D	77.8	31.5	9.35	54.2	6670	18.1	24.8
	Mean	N/A	27.6	16.5	4.89	22.2	2930	1.93	12.1
E-waste surrounding sites ($n = 11$)	DF	64 %	45 %	36 %	91 %	82 %	82 %	0 %	73 %
	Min	N/D	N/D	N/D	N/D	N/D	N/D	N/D	N/D
	Max	3020	595	1080	4880	3210	62,500	N/D	62,300
	Mean	898	176	219	467	573	16,800	N/A	6300
E-waste ($n = 24$)	DF	50 %	50 %	50 %	79 %	100 %	71 %	25 %	83 %
	Min	N/D	N/D	N/D	N/D	N/D	N/D	N/D	N/D
	Max	5560	2670	5040	557	10,440	184,000	263	48,700
	Mean	1220	500	645	104	2550	36,600	29.8	5940

The concentrations (min, max, and mean) are presented in ng g^{-1} dw. DF: detection frequency; N/D: not detected; N/A: not available.

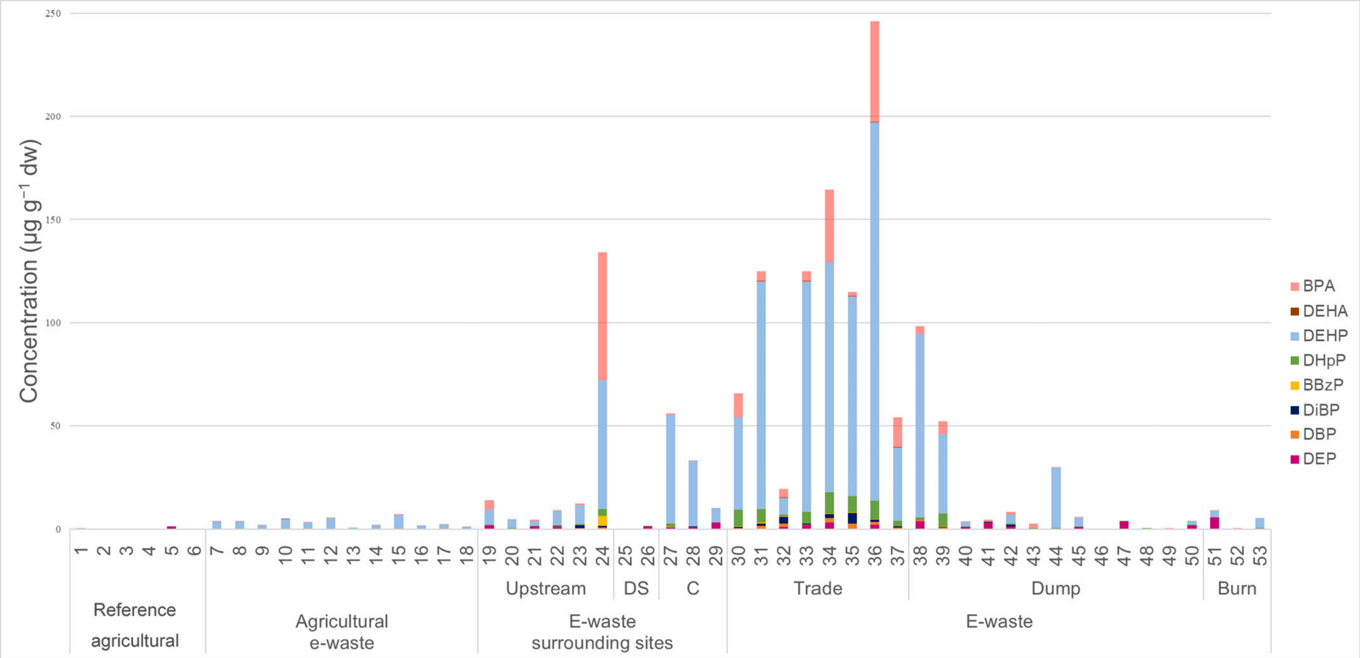

Fig. 3. Concentrations of target analytes in soils (μg g^{-1} dw) from Ghana. DS: downstream; C: community.

2.8. *Statistical analysis*

Student's *t*-tests (two-tailed, two-sample unequal variance) were performed using Microsoft Excel to assess differences in the mass error (ppm) for the 15 target analytes in solvent and matrix-matched calibrations. Statistical analysis was conducted using MetaboAnalyst 5.0 (Pang et al., 2022). Prior to data import into MetaboAnalyst 5.0, the "Not Detected" (N/D) values for the concentrations of targeted analytes in soils were replaced with a value equal to half of the MDL. Data from downstream sites ($n = 2$) were excluded from statistical analysis, as a minimum of three replicates is required for statistical analysis. Subsequently, the concentration data were log-transformed and Pareto-scaled to approximate a normal distribution. Principal Component Analysis (PCA) was performed on the concentrations of targeted analytes in each individual e-waste impacted soil sample, including those from agricultural e-waste soils. One-way Analysis of Variance (ANOVA) was employed to assess differences in analyte concentrations across various sites. Post-hoc analysis using Fisher's LSD test (Least Significance Difference) was conducted to identify significant differences between individual sites. A *p*-value <0.05 ($p < 0.05$) was deemed statistically significant for this study.

3. Results and discussion

3.1. *Targeted analysis of the 7 bisphenols and 8 plasticizers*

According to the method performances (Supplementary Tables S2-S5), the background signals of targeted analytes in solvent blank ($n = 10$) and procedural blanks (n = 10) (Supplementary Table S9) and the sample-to-blank ratios of targeted analytes (Supplementary Fig. S2), the analytical method was appropriate for the analysis of target analytes in this study. Details of method validation results are presented in Section 2 of the Supplementary Material. BPA and 7 plasticizers (DEP, DBP, DiBP, BBzP, DHpP, DEHP and DEHA) were detected in soils at various levels. Their detection frequencies are presented in Table 1. Except for DEP and DEHA, the detection frequencies for the remaining detected target analytes were higher in agricultural soils (median: 92 %) than in e-waste (median: 75 %) and e-waste surrounding soils (median: 78 %). *E*-waste and e-waste surrounding sites samples had similar detection frequencies (median: 61 % vs. 69 %) for the 8 detected target analytes.

Reference agricultural soils (#1–6) contained low levels of DBP, DiBP, and DEHP in 1/6, 2/6, and 1/6 samples, respectively. DEP was detected in only one sample (#5, see Supplementary Table S9) at 1320 ng g^{-1} dw. Agricultural soils impacted by e-waste activities contained additional contaminants (BBzP, DHpP, and DEHA, and BPA) compared to the control sites at levels below 100 ng g^{-1}; however, DEHP had concentrations ranging from 800 to 6670 ng g^{-1}. Overall, the concentrations of contaminants in e-waste and e-waste surrounding sites soils were higher than that in agricultural e-waste soils, especially for the levels of DEHP and BPA (Table 1 and Fig. 3). The mean concentrations of DEHP were more than ten times higher (N/D-184,000 ng g^{-1}) in e-waste and five times higher (N/D-62,500 ng g^{-1}) in e-waste surrounding sites, while BPA had the mean concentration (approximately 6000 ng g^{-1} for both sites) hundreds of times higher

Table 2 summarizes the concentration ranges for BPA and plasticizers determined in this study compared to those reported for other countries in the literature. As compared to levels in studies in India and China, two main countries involved in e-waste recycling, the present results revealed comparable levels for most of the target analytes, except for DEP, DEHP, and BPA, whose concentrations were up to 57, 132, and 34 times higher than in other studies, respectively. It is important to note that differences exist among studies in terms of sampling depth (top half inch in this study vs. top 20 cm in other studies). In the absence of further information on contaminant profiles in soil, it is not possible to compare data among studies. The concentrations of the target analytes in individual samples are summarized in Supplementary Table S9, and the sum concentrations for the targeted analytes are presented in Fig. 1. DBP ranged from the low ng g^{-1} range to >2 μg g^{-1}, while DiBP and BBzP ranged from N/D to >5 μg g^{-1}. The concentration range of DEHA was from N/D to 263 ng g^{-1}. Additional discussion on the individual analytes in e-waste and e-waste impacted soils is presented in Section 3 of Supplementary Material.

Among the various e-waste sites (i.e., trade, dump, and burn), higher concentrations and detection frequencies of targeted contaminants were observed at trade sites (Fig. 1), probably because of the processes of dismantling activities (Huang et al., 2014a), during which dust and other suspended particles containing contaminants can be produced and gradually contaminate the surrounding areas (e.g., agricultural e-waste sites and e-waste surrounding sites in this study) by short- and long-range atmospheric transport (Huang et al., 2014a). The authors also

Table 2
Concentration ranges of BPA and plasticizers (DEP, DBP, DEHP, BBzP, and DEHA) in e-waste impacted soils in the literature.

Analyte	Concentration range (ng g^{-1} dw)	Detection technique	Country	Reference
DEP	N/D–5560	LC-MS	Ghana	Present study
	N/D–88	GC–MS	India	(Chakraborty et al., 2019)
	2.56–46.36	GC–MS	China	(Zhang et al., 2019b)
	214–879	GC–MS	China	(Liu et al., 2019)
DBP	N/D–2670	LC-MS	Ghana	Present study
	8–105	GC–MS	India	(Chakraborty et al., 2019)
	499.38–13,396.92	GC–MS	China	(Zhang et al., 2019b)
	1160–1510	GC–MS	China	(Liu et al., 2019)
DiBP	N/D–5040	LC-MS	Ghana	Present study
	156.22–4419.50	GC–MS	China	(Zhang et al., 2019b)
BBzP	N/D–4880	LC-MS	Ghana	Present study
	N/D–413	GC–MS	India	(Chakraborty et al., 2019)
	2.78–162.67	GC–MS	China	(Zhang et al., 2019b)
	111–432	GC–MS	China	(Liu et al., 2019)
DEHP	N/D–184,000	LC-MS	Ghana	Present study
	16–2804	GC–MS	India	(Chakraborty et al., 2019)
	95.38–8405.03	GC–MS	China	(Zhang et al., 2019b)
	8630–15,300	GC–MS	China	(Liu et al., 2019)
DEHA	N/D–263	LC-MS	Ghana	Present study
	N/D–310	GC–MS	India	(Chakraborty et al., 2019)
BPA	N/D–62,300	LC-MS	Ghana	Present study
	N/D–459	GC–MS	India	(Chakraborty et al., 2019)
	11.63–83.51	IFFM-D Chemiluminescence	China	(Qi et al., 2019)
	8.71–182.04	GC–MS	China	(Huang et al., 2014a)

observed that the farther the samples collected from the dismantling sites, the lower the concentrations of contaminants in the agricultural soils. This may explain why, in the present study, the surrounding soils also contained considerable levels of targeted analytes. In summary, the concentrations of target analytes in this study decreased in the following order: e-waste > e-waste surrounding sites > agricultural e-waste.

The contribution of each individual analyte to contaminant profile is illustrated in Fig. 4. DEHP emerged as the predominant contaminant in most of the sites. Remarkably, it accounted for >95 % of the targeted contaminants in agricultural e-waste soils. DEHP was also dominant in the contaminant profiles in community, trade, dump, and burn sites. In upstream sites, approximately 40 % of the total contaminants was attributable to BPA, whereas in trade and dump sites, it accounted for only about 10 %.

3.2. *Suspect screening and non-targeted analysis of other related contaminants*

3.2.1. *Key PRCs identified through suspect screening*

Based on the suspect screening using the Agilent Extractables & Leachables PCDL, 15 and 5 features were annotated in ESI+ and ESI-, respectively, as key plastic constituents and additives for further identification. From these 20 selected features, the identification confidence of 12 features was further increased (Table 3) by either chemical standards (level 1) or experimental MS/MS (level 2). The remaining 8 features were false positives, and their identities were limited to molecular formulas (Supplementary Table S10). All the targeted analytes (BPA and 7 plasticizers) detected in soil samples in this study were also correctly identified by the suspect screening workflow.

Further discussion on compounds previously unreported in e-waste soils is described below. For the features that have been reported in e-waste soil elsewhere, additional information on the identification workflows is described in Section 5 of Supplementary Material.

Features at *m/z* 279.1605 ([M + H]$^+$, 15.22 min), *m/z* 447.3482 ([M + H]$^+$, 19.38 min), and *m/z* 419.3175 ([M + H]$^+$, 18.61, 19.07 min) were tentatively identified by the PCDL as phthalates. The first feature was confirmed using a pure analytical standard as mono(2-ethylhexyl) phthalate (MEHP), a metabolite and degradation product of the ubiquitous plasticizer DEHP. DEHP can be hydrolyzed and photolyzed in the environment, but the degradation rate is very slow (Li et al., 2018). The occurrence of MEHP is likely to be associated with the relatively high levels of DEHP detected in this study in the e-waste and e-waste surrounding sites.

For other features discussed below, several tentative compounds which were structurally related were proposed by the suspect screening workflow. For example, feature at *m/z* 447.3482 ([M + H]$^+$, 19.38 min) was annotated as possibly diisodecyl phthalate (DiDP), didecyl phthalate (DDP), bis(2-propylheptyl) phthalate (DPHP), or di(isononyl) cyclohexane-1,2-dicarboxylate (DINCH) ([M + Na]$^+$). SIRIUS also proposed DiDP; however, when comparing with the RT of DiDP-d$_4$ and DDP-d$_4$ (isotopically labelled compounds are expected to have same or very close RT as their native versions), 0.3- and 0.6-min RT difference were found for DiDP-d$_4$ and DDP-d$_4$, respectively. The identity of this feature was further confirmed by chemical standards as DPHP (retention time difference <0.1 min and main MS/MS fragments at 10, 20, and 40 V match). Similarly, features at *m/z* 419.3175 ([M + H]$^+$, 18.61, 19.07 min) had more than one tentative identities. Since its RT did not match with decyl octyl phthalate (19.43 min), it could be dinonyl phthalate (DNP), diisononyl phthalate (DiNP), or even their isomers as proposed by SIRIUS. Feature at *m/z* 419.3175 (19.07 min) was confirmed using chemical standard as DiNP, and feature at *m/z* 419.3175 (18.61 min) could be an isomer of DiNP that has lower log*P* value. These long-chain phthalates have rarely been reported in agricultural soils (Lu et al., 2022; Ren et al., 2015) and never been reported in e-waste impacted soil, indicating that non-targeted analysis was capable of revealing the occurrence of new contaminants.

The feature at *m/z* 547.4015 ([M + H]$^+$, 20.56 min) was annotated by the PCDL as trioctyl trimellitate or tri-2-ethylhexyl trimellitate. These are two isomers replacing phthalate plasticizers, due to regulation on the use of phthalates (Lee et al., 2019). The identity was further confirmed by chemical standard as trioctyl trimellitate. This compound has been recently reported in environmental matrices such as sediment (Kim et al., 2020), sludge (Lee et al., 2019), and dust and air (Fromme et al., 2016); however, no data was available for soil or e-waste soil. This research would be the first research reporting this non-phthalate plasticizer in soil.

Triethylene glycol monobutyl ether (*m/z* 224.1865 [M + NH$_4$]$^+$, 12.97 min) was also confirmed using a pure analytical standard. As one of the glycol ethers, it is not naturally occurring in the environment (Shafiee et al., 2014). Information on its usage is scant in the literature; however, it can be found in a technical data sheet that it can be used for coatings, dye carrier, and even for production of plasticizers

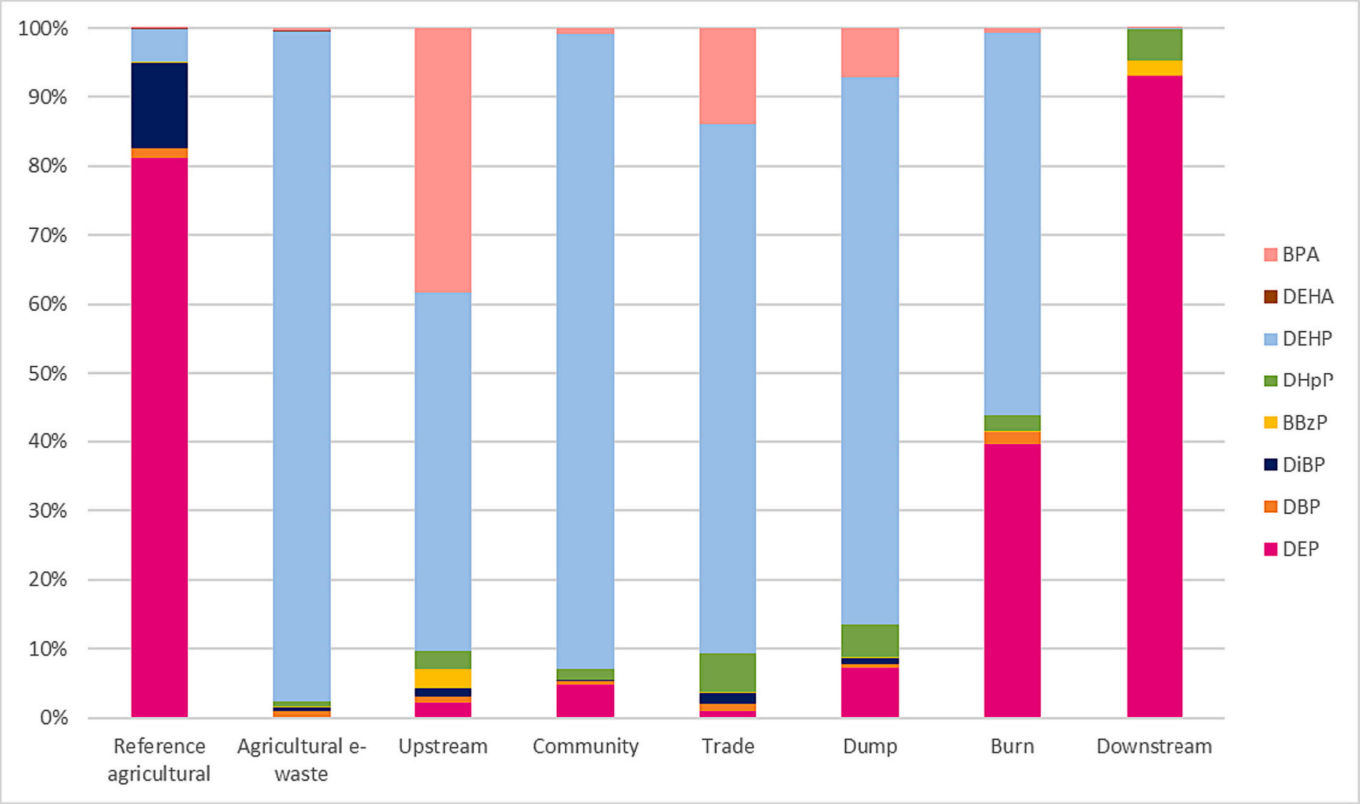

Fig. 4. Percent mass contribution of each targeted analyte to the total concentrations in different sites.

Table 3

Highlighted suspect PRCs at level 1 and 2 identification level in ESI+ and ESI- in e-waste impacted soils. The identities of eight (ESI+) and three (ESI-) features were successfully confirmed using chemical standards and at level 1 identification level. One (ESI+) feature was at level 2 identification level.

Feature (*m/z*)	Qualifier ion	Formula	RT (min)	Soil types[a]	Identification level (Schymanski et al., 2014)	Final identity
435.3602	$[M+H]^+$	$C_{24}H_{51}O_4P$	18.78	1, 2	1	Tris(2-Ethylhexyl) Phosphate
327.0796	$[M+H]^+$	$C_{18}H_{15}O_4P$	16.77	1, 2	1	Triphenyl phosphate
447.3482	$[M+H]^+$	$C_{28}H_{46}O_4$	19.38	1, 2, 3	1	Bis(2-propylheptyl) phthalate
419.3175	$[M+H]^+$	$C_{26}H_{42}O_4$	19.07	1, 2, 3	1	Diisononyl phthalate
279.1605	$[M+H]^+$	$C_{16}H_{22}O_4$	15.22	1, 2	1	Mono(2-ethylhexyl) phthalate (MEHP)
547.4015	$[M+H]^+$	$C_{33}H_{54}O_6$	20.56	1, 2, 3	1	Trioctyl trimellitate
224.1865	$[M+NH_4]^+$	$C_{10}H_{22}O_4$	12.97	1, 2	1	Triethylene glycol monobutyl ether
369.1264	$[M+H]^+$	$C_{21}H_{21}O_4P$	17.66	1, 2, 3	1	Tricresyl phosphate
146.0604	$[M+H]^+$	C_9H_7NO	12.30	1, 2, 3, 4	2	Indole-3-carboxaldehyde or its isomers
498.9299	$[M\text{-}H]^-$	$C_8HF_{17}O_3S$	14.68	1, 2	1	Perfluorooctanesulfonic acid (PFOS)
309.1870	$[M\text{-}H]^-$	$C_{21}H_{26}O_2$	17.87	1, 2, 3	1	Cannabinol
298.9424	$[M\text{-}H]^-$	$C_4HF_9O_3S$	12.14	1, 2, 3	1	Perfluorobutanesulfonic acid (PFBS)

[a] Numbers are used to represent soil types: 1 – e-waste sites, 2 – e-waste surrounding sites, 3 – agricultural e-waste sites, and 4 – reference agricultural sites.

(https://www.dow.com/content/dam/dcc/documents/en-us/productdatasheet/110/110-00972-01-butoxytriglycol-tds.pdf). It was also reported to be used for the biosynthesis of a bio-plasticizer in a lab (He et al., 2020), but to our knowledge there is no information available on its environmental occurrence.

Features at *m/z* 498.9299 ($[M\text{-}H]^-$, 14.68 min) and *m/z* 298.9424 ($[M\text{-}H]^-$, 12.14 min) were identified by chemical standards as polyfluoroalkyl substances (PFAS), namely perfluorooctanesulfonic acid (PFOS) and perfluorobutanesulfonic acid (PFBS), respectively. Due to the surface-active properties of PFAS, they have been widely used in textile, packaging, paints, surfactant agents, and fire-extinguishing formulations (Milinovic et al., 2015). The strong C—F bonds make them persistent and widely detected in environmental media such as water, sediments, and soils (Wang et al., 2018). Choi et al. (2017) reported that high PFOS level was found in agricultural soils adjacent to wastewater treatment plants, indicating that PFASs can be released to the environment directly or indirectly from the point sources. Similarly, in the present study, both PFOS and PFBS were detected in agricultural e-waste soils, suggesting that the occurrence of these two contaminants could result from atmospheric transport from the e-waste sites. PFOS and PFBS have also been reported in indoor dust from e-waste sites in China, and PFBS was one of the short-chain PFAS contributing significantly to the total PFAS concentrations in indoor dust (Zhang et al., 2020).

For the features not matching RT of chemical standards, they are limited to molecular formulas and are summarized in Supplementary Table S10. The identification steps are mentioned in Section 5 of Supplementary Material. The suspect screening workflow was able to prioritize features of interest in the current study and successfully confirm the identities of more than half of the highlighted features. The presence of features not matching RT of chemical standards revealed that MS/MS is necessary to improve the identification confidence of unknown features, and for some cases, even the MS/MS only partially matched the experimental MS/MS from literature or libraries, indicating that there is still a chance that the features are false positives, because of the presence of structurally similar compounds.

3.2.2. Identification of large peaks without annotations

The identification step in non-targeted workflows currently relies heavily on the capacity of databases and/or spectral libraries (Sobus et al., 2018), usually leaving a lot of features unannotated. In this study, only 345 features out of 2191 features were tentatively annotated using the "ID Browser" function in MPP using the Agilent Extractables & Leachables PCDL. In order to better understand features without annotations, the top 10 largest features in both ionization modes were inspected, and the unannotated features were selected to be further investigated. Six and nine unannotated features in the top 10 lists in each mode were highlighted (Supplementary Table S11), and their MS/MS fragmentation patterns at different collision energies were obtained by reanalyzing the pooled QC sample.

As examples, features at m/z 325.1848 ($[\text{M-H}]^-$, 16.94 min) were proposed for possible structures with a SIRIUS matching score >90 %. Using analytical standards (RT <0.1 min difference and main MS/MS fragments match), this feature was confirmed to be 4-dodecylbenzenesulfonic acid, which was not in the initial library, highlighting that there is a need to have broader libraries of synthetic organic chemicals for suspect screening. 4-dodecylbenzenesulfonic acid is a commonly used surfactant and catalyst for esterification (Toth et al., 2018); however, there is, so far, no published literature reporting its occurrence in the environment. 2-dodecylbenzenesulfonic acid, an isomer of 4-dodecylbenzenesulfonic acid, as well as 4-undecylbenzenesulfonic acid were reported in graywater samples in India (Athullya et al., 2022). The identification steps of some additional features are described in Section 6 of Supplementary Material.

Both the newly identified compounds and targeted compounds were searched against the US EPA CompTox Chemicals Dashboard (https://comptox.epa.gov/dashboard/) for toxicity information, and many of the newly identified compounds appeared to be as hazardous as the targeted compounds, as detailed in Supplementary Table S12. A scan of this data indicates that 18 of 22 compounds are associated with a range of adverse outcomes following short- and long-term exposures, with many associated with impairment to the reproductive and developmental systems and also having carcinogenic properties.

4. Conclusion

In this study, LC-MS based analytical workflows were developed for simultaneous targeted and non-targeted analysis of plastic-related contaminants and successfully applied to previously uncharacterized e-waste impacted soils from Agbogbloshie, Ghana. To the best of authors' knowledge, this is the first study reporting concentrations of bisphenols and phthalates in e-waste impacted soil using LC-MS instrumentation, and the first to apply a non-targeted approach to investigate plastic-related contaminants in such soils.

BPA and DEHP were the two predominant contaminants in soil, especially from trade sites, indicating that e-waste dismantling activities generated huge amounts of these chemicals. Relatively high concentrations of BPA, DEP, DBP, DiBP, DHpP, and DEHP were also observed in the adjacent areas (including agricultural sites), suggesting that contaminants gradually contaminated the surrounding areas by short- and long-range atmospheric transport, potentially affecting the health of those working and living nearby. The non-targeted workflows successfully identified another 13 plastic-related contaminants from a range of chemical families such as OPEs, glycol ethers, PFAS, trimetillates and benzenesulfonic acid derivatives. Limited information on their occurrence in e-waste impacted soil or even, for some, in any other environmental samples was available in the literature, highlighting the need for future research on these poorly described contaminants. Many of the unidentified chemical signals (approximately 2000 features in this study) may belong to other unexpected contaminant families. This work illustrates the complexity of e-waste soil matrices, and the necessity to develop non-targeted approaches for the characterization of contaminated soils.

Through searching against the CompTox Chemicals Dashboard, most of the targeted and non-targeted chemicals are toxic in long-term and/or short-term exposures. Toxicological profiles are lacking for several newly detected contaminants. Overall, the present study provides both qualitative and quantitative information for risk assessments. Future studies will also include understanding the effects of these e-waste extracts on rainbow trout and human cell lines to facilitate the development of efficient toxicity testing strategies that can inform environmental monitoring, management, and remediation activities.

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CRediT authorship contribution statement

Jingyun Zheng: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing. **Krittika Mittal:** Conceptualization, Investigation, Writing – review & editing. **Julius N. Fobil:** Conceptualization, Funding acquisition, Project administration, Writing – review & editing. **Niladri Basu:** Conceptualization, Funding acquisition, Project administration, Writing – review & editing. **Stéphane Bayen:** Conceptualization, Funding acquisition, Investigation, Project administration, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2024.170219.

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