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Is e-waste a source of phthalate and novel non-phthalate plasticizers? A comparison study on indoor dust

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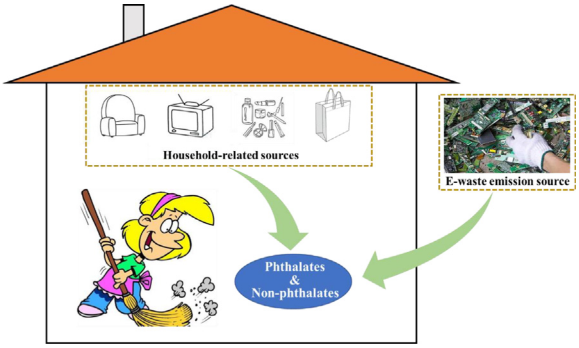
HIGHLIGHTS

- 33 novel non-phthalate plasticizers were determined for the first time in indoor dust in e-waste recycling area.
- DINP/DEHP could be a promising indicator to evaluate the impact of e-waste source emission.
- E-waste source emissions have little effect on plasticizers except DINP in indoor dust in e-waste recycling area.
- The EDIs for phthalates and non-phthalates through dust ingestion presented a low health risk in *e*-waste recycling area.

GRAPHICAL ABSTRACT



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ABSTRACT

Nine traditional phthalate plasticizers and 33 novel non-phthalate plasticizers were determined in indoor dust from a typical e-waste recycling area. The median concentrations ranged from <LOQ to 22,700 ng/g for phthalates and from <LOQ to 1250 ng/g for non-phthalates. Bis-(2-ethylhexyl) phthalate (DEHP) and di-isononyl phthalate (DINP) were the two major phthalates in dust, while glycerol monooleate (GMO) and methyl oleate (MO) were the predominant non-phthalates. Different pollutant patterns among different sites implied multiple sources of the plasticizers. Using the ratio of DINP/DEHP as an indicator, we evaluated the impact of e-waste source emission on the sampling sites, resulting in an impact rank of Matou > Dakeng > Baihetang > Shiding > Jieyang, which was consistent with the local e-waste dismantling activities and supported by polybrominated diphenyl ethers (PBDEs) levels. The correlations between chemical levels and the indicators indicated that most phthalates and non-phthalate plasticizers in the dust, might not be primarily influenced by e-waste emission sources. Additionally, the estimated median human exposures of phthalates and non-phthalates via dust ingestion were 30.6 and 1.82 ng/kg/day for adults, and 299 and 17.8 ng/kg/day for toddlers respectively, indicating negligible health risks.

1. Introduction

Plasticizers are additives which are widely used in polymer industries to improve the flexibility and processability of polymers (Hahladakis et al., 2018). Phthalates, a class of manufactured plasticizer chemicals with

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widespread use in industrial and consumer products, is one of the widely used plasticizers with the annual global production estimated at around 6–8 million tonnes per year (Net et al., 2015). Phthalates are typically semi-volatile organic compounds and not covalently bound to the host polymers, allowing them to enter the environment during production, processing, use and waste disposal, which results in environmental contamination and human exposure (Malarvannan et al., 2019; Subedi et al., 2017). Numerous studies have demonstrated that the exposure to phthalates can cause adverse health effects, including irreversible changes in the reproductive system, immune system dysfunction and potential carcinogenicity (Kay et al., 2014; Koike et al., 2010; Huang et al., 2019). Because of health concerns, restrictions in many regions and countries including the EU, US and China have come into force concerning some phthalates such as benzylbutyl phthalate (BBP), bis-(2-ethylhexyl) phthalate (DEHP), di-n-butyl phthalate (DNBP), di-isobutyl phthalate (DIBP) and di-isononyl phthalate (DINP) (Kamrin, 2009; Negev et al., 2018).

Such restrictions/bans for phthalate plasticizers have resulted in their replacement by new chemicals, which are being largely produced and used worldwide, to comply with the safety rules (Hahladakis et al., 2018). Many of these novel plasticizers are non-phthalates, including benzoates, sebacates, azelates, adipates, citrates and oleates etc. Similar to the traditional phthalate plasticizers, most non-phthalate plasticizers can leach out from the materials (Hahladakis et al., 2018), rendering potential accumulation in the environment. Although for those ones are generally recognized as safe (GRAS) alternatives to phthalates, the toxicological and concentration information in the environment is missing. In a limited number of reports, certain novel non-phthalate plasticizers like dipropylene glycol dibenzoate (DPGDB), diethylene glycol dibenzoate (DEGDB) and 2,2,4-trimethyl-1,3-pentanediol-diisobutyrate (TMPDDiB) exhibited adverse effects (Björkblom et al., 2018; Roy et al., 2013). According to a previous developed multicriteria model, some novel non-phthalate plasticizers including DPGDB, DEGDB and TMPDDiB were also suggested to be prioritized in future screening studies (Björkblom et al., 2018). Therefore, the occurrence of these non-phthalates plasticizers needs to be investigated.

Contamination by e-waste has been a critical global environmental health issue due to its increasing amounts and health risks. E-waste comprises a variety of discarded electrical/electronic appliance, of which 30 % are plastics in composition (Deng et al., 2021). During the e-waste recycling operations such as manual/mechanical dismantling, burning and dumping, these plastic-containing products could emit plasticizers to the environment in e-waste area, posing potential threat on wildlife and human. To date, studies on occurrence of phthalates and/or other plasticizers in e-waste polluted area have been performed in different environmental matrix including soil, dust, and plant (Deng et al., 2021; Liu et al., 2019a; Ma et al., 2013). However, previous studies primarily investigated the pollution and exposure status of phthalates, with little focus on non-phthalate plasticizers which are novel ones. Qingyuan, located in south China, has been one of the most intensive e-waste dismantling areas for more than two decades. In recent years, to meet the environmental requirements, large numbers of informal family-run e-waste dismantling workshops have been restricted and transferred to industrial parks (Tang et al., 2022). Despite positive development, the e-waste recycling industrial parks are mostly closed to adjacent villages (<3 km), which may still act as source of e-waste emissions.

Indoor dust can act as a sink and a repository for many indoor contaminants including plasticizers to reflect indoor exposure and public health (Christia et al., 2019; Huang et al., 2020). However, different from urban buildings where indoor dust is usually collected from, dwellings in e-waste area are generally rural houses with much ventilation. Consequently, it is very likely that pollutants in indoor dust in e-waste area originate from different sources, that is, e-waste source emission and household-related products. Nonetheless, the reality may be more complicated: some traditional plasticizers like DEHP are certainly incorporated in both e-waste and household products, but the levels may vary considerably; novel plasticizers may not be added to old e-products or only be used in certain types of products; in addition, the recycling process for e-waste may accelerate the leaching of some compounds (Robinson, 2009). In summary, e-waste and household-related products acting as primary/or secondary sources could variously contribute to plasticizers in indoor dust. To better understand the role e-waste played in the contamination of phthalate and non-phthalate plasticizers, investigation on the impacts of e-waste source emission on these chemicals is needed.

To fill the existing knowledge gap, the present study investigated phthalates and novel non-phthalate plasticizers in indoor dust from an e-waste recycling area in South China. The primary objectives of this study were to a) investigate the occurrence and concentrations of phthalates and non-phthalate plasticizers in indoor dust collected from an e-waste recycling area; b) explore the sources as well as the impact of e-waste source emissions of phthalates and non-phthalate plasticizers in indoor dust; c) estimate the ingestion exposure for human health risk assessment.

2. Materials and methods

2.1. Chemicals and reagents

Details of the target analytes, including 9 traditional phthalates and 33 novel non-phthalate plasticizers, were provided in the Supplementary materials (SI), and the description of novel non-phthalate plasticizers is listed in Table S1. Standards of phthalates and isotope-labeled standards (DMP-d4, DEP-d4, DNBP-d4, DIBP-d4, DEHP-d4, DPP-d4, and DOP-d4) were purchased from AccuStandard (New Haven, CT). Coumaphos-d10 (Toronto Research Chemicals) was used as internal standard for phthalates analysis. Standards of novel non-phthalate plasticizers were obtained from AccuStandard (New Haven, CT). DMP-d4, DEP-d4, di-n-propyl phthalate-d4, tributyl *O*-acetyl citrate-d3, DEHP-d4 and DOP-d4 were used as surrogate standards for non-phthalates. DNBP-d4 was used as internal standard for non-phthalates analysis. The organic solvents used to extract the analytes and to prepare the standard solutions were methanol and acetonitrile, both of which were LC-grade with purity ≥99.9 % from Fischer Scientific (Hanover Park, IL). LC-grade ultrapure water was also supplied by Fischer Scientific.

2.2. Sampling

Indoor dust samples ($n = 46$) were collected from five villages located in Guangdong Province, South China during July to September 2020 (Fig. 1). Four of the studied villages (including Baihetang (site BH), Shiding (site SD), Matou (site MT), and Dakeng (site DK)) are located in Qingyuan City, where e-waste dismantling and recycling activities have been conducted for decades. Jieyang (site JY) is about 360 km away from Qingyuan City with less e-waste dismantling industry existed nearby, and was selected as a control site. Sampling was conducted according to a previous method (Zheng et al., 2015). Indoor dust samples were obtained from the furniture surface, windowsills, and floors of the residence houses using woolen brushes that were pre-cleaned by ethyl alcohol. Samples from a particular house were combined into one pooled sample, and were wrapped in aluminum foil and sealed in zip bags. After being transported to the laboratory, all dust samples were passed through a 100-mesh sieve to remove debris and were stored at −20 °C until further analysis. Field blanks ($n = 5$) were prepared using pre-cleaned sodium sulfate and treated identically to those used for field samples.

2.3. Chemical analysis

Phthalates were analyzed according to a previous method (Liu et al., 2019b) with minor modifications. Briefly, approximately 30 mg of sieved dust was spiked with surrogate standards (DMP-d4, DEP-d4, DNBP-d4, DIBP-d4, DEHP-d4, DPP-d4 and DOP-d4) before extraction using a ultrasonicator with 3 mL of a mixture of methanol and water (6:4, v/v), 3 mL of a mixture of acetonitrile containing 0.2 % formic acid and water (8:2, v/v), 3 mL of a mixture of acetonitrile and isopropanol (1:1, v/v), and 3 mL of a mixture of hexane and isopropanol (1:1, v/v). the combined

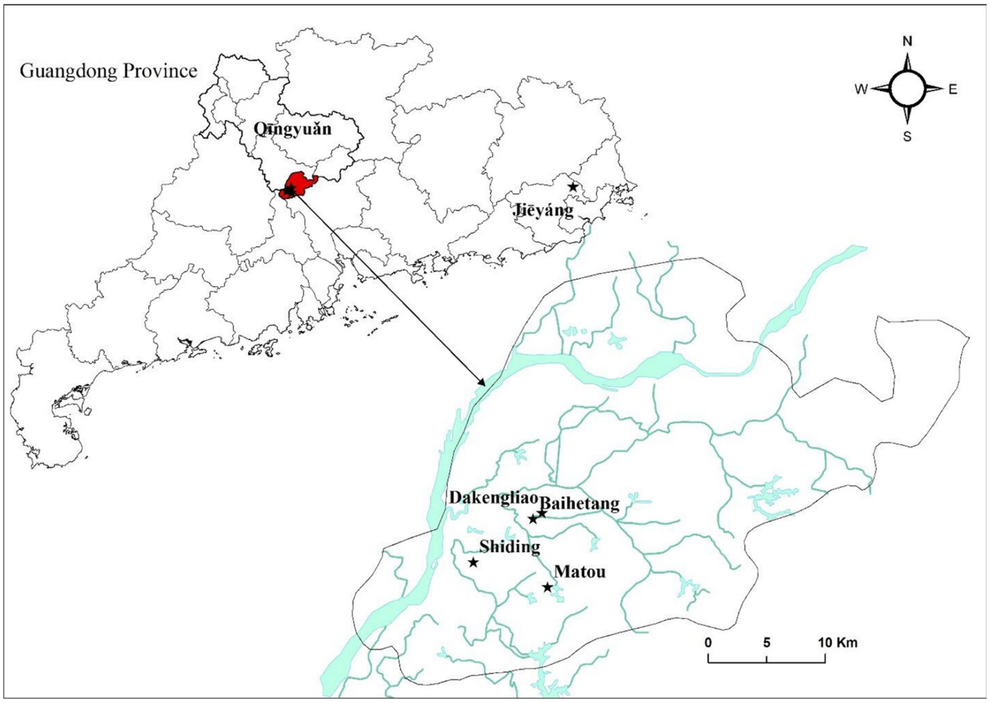

Fig. 1. The graphic of sampling areas located in Guangdong Province, South China.

supernatant was concentrated to 2 mL, reconstituted to 6 mL with water, and then purified with an HLB cartridge. Target analytes were eluted out with 5 mL of acetonitrile and then 5 mL of methanol. The elution was condensed to about 100 μL and filtered through a 0.22 μm centrifugal filter (VWR International). A known amount (5 ng) of internal standard (coumaphos-d10) was added prior to instrumental analysis.

Non-phthalates were extracted using the following procedure. An aliquot of approximately 30 mg dust was transferred to a 10 mL glass tube. After spiking with 5 ng of surrogate standards (DMP-d4, DEP-d4, di-n-propyl phthalate-d4, tributyl *O*-acetyl citrate-d3, DEHP-d4 and DOP-d4), each sample was extracted twice with 3 mL acetonitrile, using an ultrasonicator. The tubes were centrifuged and the supernatants were combined into a new glass tube. The extracts were evaporated to near dryness under a gentle nitrogen flow, then reconstituted in 0.5 mL methanol. The final extracts were filtered through a centrifugal filter (VWR International, 0.22 μm), concentrated to 100 μL, and spiked with 5 ng internal standards (DNBP-d4) prior to instrumental analysis.

The determination of phthalates was conducted on a ultraperformance liquid chromatograph coupled with an AB Sciex 3200 or 5500 Q Trap triple quadrupole mass spectrometer (MS/MS, Toronto, Canada) and an Agilent gas chromatograph coupled with an Agilent 5977 A single quadrupole mass analyzer. Detailed information on phthalate instrumental analysis can be found in the previous report (Liu et al., 2019b). The novel non-phthalate plasticizers were determined using an ultraperformance liquid chromatograph coupled with an AB Sciex 5500 Q Trap triple quadrupole mass spectrometer. The chromatographic separation was performed on a Luna 2.5 μm C18(2) 100 Å column (100 mm × 2 mm, 3 μm particle size; Phenomenex, Torrance, CA, U.S.). The mobile phases consisted of purified water with 5 mmol/L ammonium acetate (A) and methanol (B), with a flow rate at 0.2 mL/min. Positive electrospray ionization (ESI) source with multiple reaction monitoring (MRM) mode was used for analysis of the analytes. The gradient conditions used for MS analysis in the positive ion mode were: 0–2 min, 10 % B; 2–11.5 min, followed by an increase to 100 % B, then held for 10.5 min; 22–28 min, followed by a decrease to 10 % B, then held for 2 min. Both mobile phase gradients were linearly ramped. Information on ion transitions monitored for the non-phthalate plasticizers were provided in the SI (Table S2).

2.4. Quality assurance and quality control (QA/QC)

A procedural blank was processed along with each batch of 9 samples. DEHP, DNBP, DINP, GMO, MO, TBC, DPGDB, TMPDDiB and IPP were detected in the procedural blanks ($n = 5$), with average concentrations ranging from 3.96 to 23.0 ng which were all below 5 % of levels of compounds in authentic samples, and were subtracted from the finally reported concentration data. Field blanks were found to be close to the procedural blanks, suggesting that sampling was not contaminated. The matrix spiked recoveries (mean ± standard deviation) for phthalates and novel non-phthalate plasticizers were between 81.3 ± 18.5 % to 123.6 ± 19.1 % and 60.2 ± 6.8 % to 126.6 ± 20.5 % (with exception of DBM/DBF, BARO and DESU for 40.0 ± 7.8 %, 41.4 ± 3.7 % and 51.3 ± 14.6 %) (Table S3), respectively. Recoveries of surrogate standards during the analysis of authentic samples ranged from 65.7 ± 13.3 % to 85.2 ± 12.4 % for phthalates and from 61.5 ± 15.3 % to 96.3 ± 13.4 % for non-phthalates. The limit of quantification (LOQ) was defined as the average blank plus 10 times of the standard deviation for the analytes detected in the procedural blanks, and a signal/noise ratio of 10 was used for analytes undetected in the procedural blanks. The determined LOQs ranged from 1.0 to 200 ng/g for phthalates analysis and from 2.0 to 71 ng/g for non-phthalates analysis.

2.5. Daily exposure assessment

The estimated daily intake (EDI, ng/kg/d) of phthalates and novel non-phthalate plasticizers via dust ingestion were calculated using the following equations:

$$\mathrm{EDI}_{\mathrm{ingestion}} = \Sigma(C_{\mathrm{dust}} \times \mathrm{IR} \times \mathrm{EF} \times \mathrm{ED})/(\mathrm{BW} \times \mathrm{AT}) \tag{1}$$

where C_{dust} is the concentration of the target analyte in dust (ng/g), IR is the ingestion rate of the indoor dust (mg/day), EF is the exposure frequency (days/year), ED is the exposure duration (year), BW is the body weight (kg), AT is the average time (days). Parameters used in the equations were given in the SI (Table S4).

Table 1

Concentrations (median (min–max), ng/g) of phthalate and non-phthalate plasticizers in indoor dust from 5 villages in e-waste recycling area, South China.

Compounds	DF for total samples (%)	Qingyuan				Jieyang (control)	Total Qingyuan samples	Total samples
		Site SD (*n* = 10)	Site BH (*n* = 12)	Site DK (*n* = 8)	Site MT (n = 8)	Site JY (n = 8)		
Phthalate plasticizers								
DMP	100	26.3 (9.09–53.1)	19.4 (5.42–144)	28.9 (8.10–99.0)	79.6 (17.7–220)	61.2 (7.10–198)	30.4 (5.42–220)	35.9 (5.42–220)
DEP	93	98.5 (39.3–227)	228 (<LOQ-590)	157 (<LOQ-850)	94.3 (28.6–597)	379 (<LOQ-1270)	102 (<LOQ-850)	112 (<LOQ-1270)
DNBP	96	1760 (630–2840)	1860 (<LOQ-3380)	1460 (446–2280)	1930 (250–3610)	863 (<LOQ-1510)	1730 (<LOQ-3610)	1690 (<LOQ-3610)
DIBP	93	691 (<LOQ-1510)	1110 (<LOQ-5250)	979 (101–5370)	1950 (24.4–7550)	621 (167–2650)	1000 (<LOQ-7550)	1040 (<LOQ-7550)
BBP	100	4.58 (1.42–11.9)	4.16 (1.66–42.0)	8.45 (2.50–23.9)	23.7 (7.53–73.2)	12.8 (4.40–22.6)	7.00 (1.42–73.2)	8.67 (1.42–73.2)
DEHP	100	20,600 (2560–47,900)	25,700 (2220–67,500)	17,600 (1850–47,500)	32,900 (4560–70,000)	20,200 (1630–67,600)	22,700 (1850–70,000)	21,900 (1630–70,000)
DOP	32	<LOQ[a] (<LOQ-7.78)	<LOQ (<LOQ-29.2)	22.8 (<LOQ-122)	14.1 (<LOQ-56.3)	<LOQ (<LOQ-44.9)	<LOQ (<LOQ-122)	<LOQ (<LOQ-122)
DPP	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DINP	100	6340 (522–12,900)	4640 (255–35,200)	7620 (1160–54,800)	13,500 (7590–32,400)	2980 (1880–13,300)	7200 (522–54,800)	7600 (522–54,800)
ΣPhthalates	100	30,200 (3900–64,300)	41,000 (2540–96,000)	28,600 (8550–79,000)	63,200 (18900–102,000)	40,000 (17000–83,000)	36,300 (2540–102,000)	36,300 (2540–102,000)
Non-phthalate plasticizers								
DMA	78	181 (82.8–385)	215 (114–335)	265 (88.1–415)	265 (215–509)	<LOQ (<LOQ-34.8)	219 (82.8–509)	181 (<LOQ-509)
DEA	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DHAZ	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DIAZ/DEHAZ	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DMAZ	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DEGDB	55	<LOQ (<LOQ-8.31)	3.58 (<LOQ-14.1)	5.13 (<LOQ -23.4)	14.2 (<LOQ -22.1)	6.35 (<LOQ −62.7)	3.52 (<LOQ −23.4)	3.54 (<LOQ −62.7)
DPGDB	59	4.20 (<LOQ-7.97)	7.85 (<LOQ-54.2)	4.55 (<LOQ–16.9)	4.90 (<LOQ–12.9)	<LOQ (<LOQ-214)	4.93 (<LOQ-54.2)	4.90 (<LOQ-214)
TMPDMiB	86	127 (50.5–293)	70.9 (<LOQ-710)	131 (58.0–233)	126 (<LOQ-372)	38.1 (<LOQ −163)	115 (<LOQ-710)	100 (<LOQ-710)
TMPDDiB	68	79.4 (<LOQ-234)	39.8 (<LOQ-567)	85.4 (32.6–184)	58.0 (<LOQ-192)	<LOQ (<LOQ-5.60)	54.7 (<LOQ-567)	34.8 (<LOQ-567)
ATEC	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
BTHC	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
TBC	64	31.1 (<LOQ-204)	121 (<LOQ-467)	37.3 (<LOQ-374)	48.6 (<LOQ-304)	78.7 (<LOQ-5480)	32.3 (<LOQ-467)	32.3 (<LOQ-5480)
TEC	64	5.26 (2.78–14.6)	3.06 (<LOQ-32.3)	2.52 (<LOQ-13.8)	5.43 (<LOQ-15.4)	<LOQ	3.75 (<LOQ-32.3)	2.65 (<LOQ-32.3)
DBM/DBF	2	<LOQ	<LOQ (<LOQ-20.8)	<LOQ	<LOQ	<LOQ	<LOQ (<LOQ-20.8)	<LOQ (<LOQ-20.8)
DEHM	70	6.05 (<LOQ-23.3)	3.33 (<LOQ-32.2)	6.53 (<LOQ-13.1)	4.80 (<LOQ-26.1)	2.81 (<LOQ-5.93)	3.90 (<LOQ-32.2)	3.45 (<LOQ-32.2)
IPMS	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
IPP	76	77.1 (<LOQ-221)	128 (48.9–234)	112 (<LOQ-132)	63.4 (<LOQ-140)	<LOQ (<LOQ-74.5)	74.8 (<LOQ-234)	70.1 (<LOQ-234)
BO	15	<LOQ (<LOQ-66.8)	<LOQ	<LOQ	<LOQ (<LOQ-79.1)	<LOQ (<LOQ-175)	<LOQ (<LOQ-79.1)	<LOQ (<LOQ-175)
BARO	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
BRO	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
GMO	82	1600 (250–15,100)	2290 (747–9200)	555 (<LOQ-3800)	858 (286–2040)	<LOQ (<LOQ-1140)	1250 (<LOQ-15,100)	1120 (<LOQ-15,100)
MO	56	584 (214–1020)	375 (<LOQ-1660)	35.4 (<LOQ-833)	344 (<LOQ-1050)	<LOQ	288 (<LOQ-1660)	169 (<LOQ-1660)
MARO	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
PO	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
THFO	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DBS	28	<LOQ (<LOQ-16.2)	<LOQ (<LOQ-30.9)	<LOQ (<LOQ-13.4)	<LOQ (<LOQ-66.1)	<LOQ	<LOQ (<LOQ-66.1)	<LOQ (<LOQ-66.1)
DEHS	15	<LOQ (<LOQ-14.7)	<LOQ	<LOQ (<LOQ-8.39)	6.80 (<LOQ-19.5)	<LOQ	<LOQ (<LOQ-19.5)	<LOQ (<LOQ-19.5)
DMS	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DESU	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
TiNTM	50	42.5 (<LOQ-186)	<LOQ (<LOQ-28.0)	<LOQ (<LOQ-110)	232 (133–522)	11.5 (<LOQ-2010)	26.5 (<LOQ-522)	26.3 (<LOQ-2010)
THTM	0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
ΣNon-phthalates	98	3380 (1300–15,900)	3700 (950–9830)	1650 (780–4610)	2160 (950–4760)	700 (<LOQ-6950)	2720 (780–15,900)	2330 (<LOQ-15,900)

[a] LOQ: limit of quantitation.

2.6. Statistical analysis

Statistical analysis was performed with SPSS 26.0 (SPSS, Inc., Chicago, IL, USA). The normality was checked by Shapiro-Wilk test, and the data was close to normal distribution after log transformation. Concentrations below the LOQ were substituted with a value equal to LOQ divided by the square root of 2 for the calculation of the mean value. Spearman correlation analysis was used to investigate the possible association between chemical concentrations in dust samples. One-way analysis of variance (ANOVA) was conducted to determine the difference in chemical concentrations and patterns among different sites. The criterion for significance was set at p <0.05 throughout the study.

3. Results and discussion

3.1. Concentrations and profile of phthalate plasticizers

Descriptive data on detection frequency (DF) and concentrations of phthalates and non-phthalate plasticizers in the indoor dust are summarized in Table 1. Among the 9 target phthalates, DMP, BBP, DEHP, and DINP were 100 % detected, whereas DEP, DNBP, and DIBP were frequently detected in >90 % dust samples. The DF of DOP was only 32 %, while DPP was not detected in any of the sample. The total concentrations of the nine phthalates ($\Sigma_9$phthalates) ranged from 2540 to 102,000 ng/g with a median concentration of 36,300 ng/g, which was lower than those of indoor dust from the U.S., 2007, 2008 and 2010 (396 μg/g) (Guo and Kannan, 2011), China, 2010 (295 μg/g) (Guo and Kannan, 2011), the U.S., 2017 (341 μg/g) (Subedi et al., 2017) and China, 2019 (54.1 μg/g) (Zhu et al., 2019). Additionally, the $\Sigma_9$phthalates level in the present study was much lower than that of total phthalate concentrations (median 300 μg/g) in rural home dust collected from the same e-waste polluted area (Deng et al., 2021). A possible explanation for the low phthalate levels in the present study is the difference in sampling time. It should be noted that the dust samples used in Deng et al. (2021) were collected in 2016, whereas the dust samples in the present study were collected in 2020. With many restrictions on the use of phthalates in consumer products and further treatment of crude e-waste recycling activities (Net et al., 2015; Zhu et al., 2019), it is reasonable that much lower phthalate levels was observed in the present study.

DEHP dominated over other phthalates in the current indoor dust, reaching a median concentration of 22,700 ng/g for Qingyuan samples and 21,900 ng/g for total samples, which approximately constituted 65 % of the total phthalates. Similar results were also observed in other indoor environments (Guo and Kannan, 2011; Liu et al., 2019b; Zhu et al., 2019), indicating that DEHP was broadly used in household products. Interestingly, DINP was detected as the second abundant analyte (median 7600 ng/g for all samples, contributing 25 % of the total phthalates) in the present dust collected from e-waste recycling area. Although DEHP are gradually been replaced by DINP and that of high levels of DINP in dust from western European countries (Jo et al., 2018; Bornehag et al., 2015; Nagorka and Koschorreck, 2020; Blanchard et al., 2014), DEHP typically contributed 70–90 % to the total phthalates in China home dust, whereas DINP had a much lower proportion (two orders of magnitude lower than DEHP) (Liu et al., 2019a, 2019b; Zhang et al., 2020; Sun et al., 2017). In agreement with our findings, a recent study revealed the predominance of DINP in indoor dust collected from e-waste dismantling workshops (Deng et al., 2021), implying that e-waste could be an important source of DINP.

3.2. Concentrations and profiles of non-phthalate plasticizers

Concentrations of the target non-phthalate plasticizers in indoor dust from e-waste recycling area, South China were presented in Table 1. A total number of 12 compounds including DEGDB, DPGDB, TMPDMiB, TMPDDiB and GMO etc., were frequently detected (DF > 50 % (50–98 %)), with median concentrations from 3.52 to 1250 ng/g. However, 15 compounds (i.e., DEA, DMAZ, DHAZ, DIAZ/DEHAZ, ATEC, BTHC, IPMS, BARO, BRO, MARO, PO, THFO, DMS, DESU and THTM) were not detected in dust. To date, little is known about the occurrence of these novel plasticizers in the environment. DPGDB, DMS and DMAZ were identified at frequencies ranging from 12 % to 56 % in the indoor dust from Flemish region, Belgium, but no concentrations data are available (Christia et al., 2021). BTHC was reported below LOQ in the household dust from Guangzhou, China (Tang et al., 2020), which was consistent with the present result. According to our previous study (Tao et al., 2022), most novel non-phthalate plasticizers were detected at higher DF and levels (>1–3 orders of magnitude) in indoor dust (17 novel non-phthalates with DF in 100 %, ranging from 1.85 to 198,000 ng/g) collected from the urban dwellings (Guangzhou, South China) than from the present rural houses, which could be attributed to urban-rural discrepancies such as relative higher density of consumer products in room of the urban buildings.

Among the novel non-phthalate plasticizers, GMO and MO exhibited the highest proportions in our dust except for site JY, collectively accounting for 26–96 % of Σ_{18}non-phthalates. This was consistent with the predominance of GMO and MO (64 % and 20 % respectively) detected in the urban indoor dust in our previous study, reflecting their broad application. However, significantly (one-way ANOVA, p <0.05) higher proportions of DMA and TMPDMiB (4.2–5.8 % of total non-phthalates in the present dust, 0.8–0.9 % in the home dust) and lower proportions of DPGDB, DBM/DBF, DMAZ, PO, IPMS and THTM (<LOQ–0.2 % of total non-phthalates in the present dust, 1.3–7.4 % in the home dust) were found in the present dust than the urban home dust, indicating the different profile patterns of non-phthalate plasticizers between the two types of indoor environments.

Novel non-phthalate plasticizers generally contributed 3–10 % (calculated using mean values) to the total plasticizers (sum of phthalates and non-phthalate plasticizers) in dust in this study (Fig. 2 and Table S5), with relatively high proportion in several samples (20–50 %), which suggested that the major plasticizers present in e-waste recycling area are phthalates. Nonetheless, it is likely that the contribution of non-phthalate plasticizers will increase over time because of the safety rules (Björkblom et al., 2018). Due to the lack of toxicity information, long-term monitoring on these novel plasticizers in the environment is warranted.

3.3. Site variations

Significantly (one-way ANOVA, p <0.05) higher Σ_9 phthalates concentrations were found in site MT than site SD, BH, and JY, which are mainly attributed to the higher concentration of DINP in site MT. It is reasonable as site MT is the closest village to the local e-waste recycling parks, and

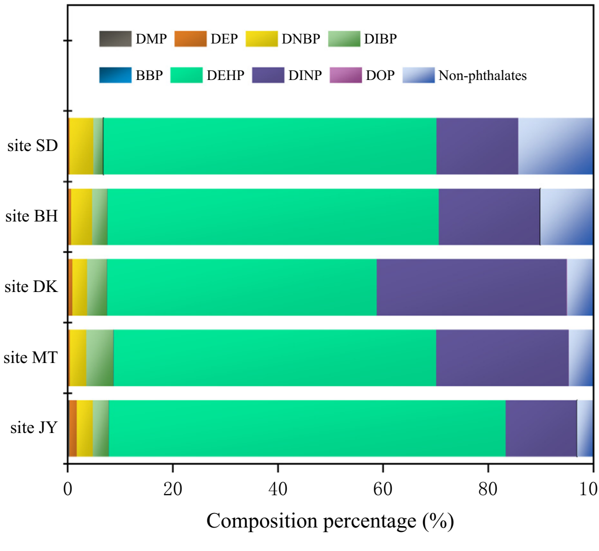

Fig. 2. Composition patterns of target plasticizers (individual phthalate and Σnon-phthalates).

that the impact of e-waste recycling activities could be more intensive than other sites. In addition, the control site JY showed much lower (one-way ANOVA, $p < 0.05$) DINP concentrations than other sites in Qingyuan, while comparable DEHP levels were observed among all the sampling sites. These results indicated that DINP may be highly associated with e-waste dismantling activities, which is consistent with Deng et al. (2021).

Regarding the variations on novel non-phthalate plasticizers, significantly (one-way ANOVA, $p < 0.05$) lower Σ_{18} non-phthalate plasticizers concentrations were observed in site JY than site MT, BH, and SD. Specifically, higher levels of GMO, MO, DMA, TMPDMiB and TEC were detected in indoor dust collected from the four sites in Qingyuan than those from control site JY. Although these findings might owe to the impact of e-waste dismantling activities on the emission of certain novel non-phthalate plasticizers, other factors, e.g., different types of indoor household materials and different living habits, may be more important because of much higher concentrations of novel plasticizers determined in urban dust. Therefore, the observed variations among sampling sites could imply the influence of e-waste source emissions for some of the target plasticizers, while other analytes in indoor dust may be related to household products.

3.4. Source implication and impact of e-waste source emission

To explore the sources of phthalate and non-phthalate plasticizers in indoor dust, the correlations among analytes were assessed using Spearman analysis. As shown in Table 2, significantly positive correlations (r = 0.30–0.81, $p < 0.05$) were found among most phthalates, suggesting similar sources of these chemicals in industrial appliance in products and/or e-waste emissions. However, poor correlations were found among most non-phthalate plasticizers, possibly due to their diverse sources in household-related products. With respect to the relationships between phthalates and non-phthalates, most pairs were not significantly associated with each other, indicating that traditional plasticizers and most novel plasticizers may be released from different sources. Notably, certain pairs like DMP vs DEGDB, DEP vs DPGDB and DEHP vs GMO (r = 0.58, 0.51 and 0.34) showed significant correlations, which might be attributed to their similar application and good compatibility of each pair in products, e.g., polymeric membrane, PVC plasticizers and cosmetic products, etc. (Björkblom et al., 2018; Totu et al., 1998). Stronger correlations than ours among phthalates were previously reported in dust collected from e-waste recycling workshops (Deng et al., 2021). On the other hand, less correlations were observed among traditional phthalates in indoor dust from non-e-waste area, especially in urban home dust (Deng et al., 2021; Liu et al., 2019b). Therefore, phthalates in indoor dust collected from village houses, which are at some distance from the workshops, could be influenced by both e-waste source and other sources like household-related products (e.g., food, furniture, and personal care products). Taken together, multiple sources of the plasticizers were considered to exist in our dust samples.

Of the multiple sources, we focused on the e-waste emission in the studied area. We noted that the ratio of DINP/DEHP might be a promising indicator for assessing the impact of e-waste source emission. A previous study reported that urban indoor emissions of DEHP were estimated to be 2 orders of magnitude higher than DINP (Cousins et al., 2014), and accordingly DEHP in urban indoor dust typically constituted >70–90 % of the total phthalates before the replacement of DEHP by DINP and other plasticizers (Ait Bamai et al., 2014; Bornehag et al., 2005; Lan et al., 2012; Liu et al., 2019b; Zhang et al., 2013). Whereas Deng et al. (2021) first revealed that DINP was the predominant phthalate plasticizer in dust from e-waste recycling workshop, accounting for 66 % of the total traditional phthalates. Moreover, in Deng's report, the highest DINP/DEHP (median value) ratio was detected in dust in e-waste recycling workshop (2.21), followed by dust from e-waste rural area (0.30), and then urban home dust (0.19). Although both DEHP and DINP were plasticizers with high production volume and broad applications (Jo et al., 2018; Bornehag et al., 2015; Nagorka and Koschorreck, 2020), these findings might be caused by the more effective surface coatings materials in preventing the release of DINP relative to

Table 2

Spearman's correlation coefficients of plasticizers in indoor dust from e-waste recycling area.

	DMP	DEP	DNBP	DIBP	BBP	DEHP	DINP	DMA	DEGDB	DPGDB	TMPDMiB	TMPDDiB	TBC	TEC	DEHM	IPP	GMO	MO	TiNTM
DMP	1.00																		
DEP	0.56**	1.00																	
DNBP	0.40**	0.44**	1.00																
DIBP	0.32*	0.46**	0.54**	1.00															
BBP	0.81**	0.43**	0.35*	0.32*	1.00														
DEHP	0.03	0.16	0.23	0.56**	0.13	1.00													
DINP	0.27	0.24	0.25	0.41**	0.30*	0.40**	1.00												
DMA	−0.09	−0.08	0.23	0.29	−0.01	0.22	0.05	1.00											
DEGDB	0.58**	0.51**	0.36*	0.48**	0.56**	−0.03	0.37*	0.11	1.00										
DPGDB	0.46**	0.51**	0.37*	0.28	0.47**	−0.06	0.29	−0.06	0.51**	1.00									
TMPDMiB	0.03	0.04	0.38*	0.34*	0.04	0.06	0.31*	0.28	0.19	0.16	1.00								
TMPDDiB	0.09	0.04	0.18	0.11	0.04	−0.05	−0.04	0.20	0.06	0.15	0.42**	1.00							
TBC	0.48*	0.37*	0.28	0.24	0.35*	0.07	0.40*	−0.21	0.23	0.60**	0.24	0.10	1.00						
TEC	0.21	0.003	0.34*	0.21	0.19	0.01	−0.04	0.18	0.13	0.19	0.27	0.38*	0.06	1.00					
DEHM	0.39*	0.26	0.23	0.12	0.29	−0.03	0.01	0.08	0.29	0.22	0.22	0.60**	0.14	0.53**	1.00				
IPP	−0.01	0.06	0.34*	0.13	−0.07	−0.15	−0.02	0.08	0.08	0.16	0.28	0.25	0.04	0.25	0.24	1.00			
GMO	−0.06	0.22	0.45**	0.22	−0.05	0.34*	0.15	0.12	−0.07	0.44*	0.25	0.26	0.27	0.02	0.29	0.42*	1.00		
MO	−0.07	−0.08	0.23	0.13	−0.28	0.16	−0.03	0.19	−0.17	0.06	0.39*	0.31	0.08	0.30	0.12	0.24	0.33	1.00	
TiNTM	0.28	−0.12	0.06	0.15	0.36*	0.15	0.44**	0.21	0.28	0.03	0.07	0.09	−0.03	0.38*	0.17	−0.15	0.01	0.05	1.00

* $p < 0.05$.

** $p < 0.01$.

DEHP (Jeon et al., 2016), and therefore the coatings could be ruined during the recycling activities, allowing much of the DINP to leach out.

According to the ratios of DINP/DEHP in the present study (35.1 %, 30.0 %, 22.5 %, 21.5 % and 10.8 % for site MT, DK, BH, SD and JY, median value), the e-waste source emission could make the greatest impact on site MT and DK, followed by BH, SD and then JY. This was consistent with the actual background. In fact, village MT was the closest to the local e-waste dismantling industrial parks, while village DK had crude, family-run e-waste recycling workshops in operation during the sampling period. Village BH and SD were relatively far from the e-waste dismantling parks. Although e-waste recycling activities used to be very common for BH and SD, the e-waste dismantling workshops had been abandoned and no recycling activities were observed in the two villages. Village JY, as a control site, was considered to be less influenced by e-waste source emissions. To further verify the accuracy of the indicator used in this study, we examined the levels of polybrominated diphenyl ethers (PBDEs) for each site, considering the highly association between PBDE levels and e-waste source emission in the sampling area (Zhao et al., 2009; Zheng et al., 2015). As a result, the villages with higher DINP/DEHP ratios also exhibited higher ΣPBDE (BDE 47, 100, 99, 85, 154, 153, 183, and 209) concentrations (3161, 2596, 571, 381, and 97 ng/g of ΣPBDEs in site MT, DK, BH, SD and JY, respectively, median value) (Fig. S1), which supported the inference that e-waste recycling activities have a significant effect on DINP release.

To further investigate the impact of e-waste source emission on target compounds in the present indoor dust, the correlations between the levels of phthalate and non-phthalate plasticizers (DF > 50 %) and the indicators were also estimated in the e-waste recycling area in this study. As shown in Fig. S2, only DINP were significantly associated with DINP/DEHP ratio ($r = 0.41$, $p < 0.05$), suggesting DINP might primarily originate from e-waste emission source. The results also indicated that most phthalate and non-phthalate plasticizers in the dust in e-waste recycling area had significant influences by other sources apart from e-waste emission. This can well explain the fact that the levels of plasticizers in house dust (e-waste dismantling workshop is not included as it is a spot source) in e-waste polluted area is not higher than those from other areas (Deng et al., 2021; Giovanoulis et al., 2018).

3.5. Human exposure assessment

Dust ingestion and dermal absorption are the main pathways for human exposure to indoor environmental pollutants (Zhu et al., 2019). Previous studies indicated that dermal absorption was very limited human exposure pathway due to the small dermal absorption fraction (0.000031–0.000778) for phthalates, and that dust ingestion is the predominant exposure pathway (Giovanoulis et al., 2018). In this study, human intakes of phthalates and non-phthalate plasticizers through dust ingestion were estimated under median exposure and high-end exposure scenarios, which were calculated based on the median and 95th percentile concentrations in indoor dust, respectively.

The EDIs of phthalates for toddlers and adults were 299 and 30.6 ng/kg/day, respectively, which were 10 to 20 folds higher than those of non-phthalate plasticizers (17.8 and 1.82 ng/kg/day for toddlers and adults, respectively) under the median exposure scenario. The calculated EDIs of toddlers were about one order of magnitude higher than those of adults. Much higher risk for toddlers than adults indicating that more attention should be given to toddlers due to their frequent hand-to-mouth contact and mouthing toys that contact with floors (He et al., 2018; Jones-Otazo et al., 2005). At the high-end scenario, the EDIs of phthalates for toddlers was as high as 725 ng/kg/day, which was three to four orders of magnitude lower than the U.S. EPA recommends maximum oral daily intakes for phthalates (DEP, DNBP, BBP, and DEHP at 0.8, 0.1, 0.2, and 0.02 mg/kg/day, respectively) (USEPA, 2011). However, the EDI of non-phthalate plasticizers is approximately 10 % of the EDI of traditional phthalates, indicating a co-exposure in indoor environment. More studies are needed to evaluate the toxicities of the novel non-phthalate plasticizers, in order to better understand the potential health risks of these chemicals to human.

4. Conclusions and implications

Our study reports for the first time the presence of a suite of novel non-phthalate plasticizers in indoor dust in e-waste recycling area. Our analysis for traditional phthalate plasticizers further demonstrates that DINP in the dust could be highly influenced by e-waste dismantling activities. Thus, we tentatively used DINP/DEHP, appeared to be a promising indicator, to estimate the impact of e-waste source emission on different sites. Despite the need for future improvement, this could provide a quick approach to assess e-waste impact by comparing the indicator with neighboring urban dust, and could be applied to define the impact range of e-waste emission.

It is expected that novel plasticizers in our study were not influenced by e-waste emission, because/suggesting these chemicals may not be added in older electronic devices. Interestingly, on the other hand, e-waste source emission also has little effect on traditional plasticizers except DINP in our dust. Despite the high concentrations of traditional plasticizers, such as DEHP, in dust of e-waste dismantling workshop, it seems that these compounds could not be efficiently transferred to adjacent communities. This reveals that, in order to reduce exposure to plasticizers, humans should preferentially target and reduce sources of plasticizers in household-related products. Moreover, as DINP has being replacing DEHP in recent years (Jo et al., 2018; Bornehag et al., 2015; Nagorka and Koschorreck, 2020; Blanchard et al., 2014), the impact of e-waste recycling activities on plasticizers pollution is expected to increase. In particular, concerns are growing about DINP as a safe plasticizer, and thus it is necessary to have a clear understanding of the role e-waste source emission played in plasticizers contamination.

Admittedly, there are several limitations in this study. First, more supporting is needed to better verify the accuracy of the indicator used in this study. Second, different conditions of home micro-environment for instance temperature, humidity, and types of household materials, may interfere with the impact assessment of e-waste source emission on the studied chemicals. Third, the sample size for each site is not very sufficient. More in-depth and quantitative research are warranted to better trace the source of phthalate and non-phthalate plasticizers in indoor environment in e-waste polluted area.

CRediT authorship contribution statement

Zong-Rui Li: Investigation, Validation, Formal analysis, Funding acquisition, Writing-original draft.

Chang He: Investigation, Validation, Formal analysis, Writing-review & editing.

Jing Yang: Investigation, Formal analysis.

Tian-Rui Gao: Formal analysis-review & editing.

Yi-Chao Huang: Investigation, Validation, Formal analysis-review & editing.

Lin Tao: Investigation, Validation, Formal analysis, Supervision, Writing-review & editing.

Data availability

Data will be made available on request.

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.

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Appendix A. Supplementary data

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