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Measurement of Phthalates in Settled Dust in University Dormitories and Its Implications for Exposure Assessment

Jiahui Wang [1], Fangzhou Yuan [2], Haitian Ye [2] and Zhongming Bu [2,*]

[1] School of Urban Construction, Hangzhou Polytechnic, Hangzhou 311402, China
[2] Department of Energy and Environmental System Engineering, Zhejiang University of Science and Technology, Hangzhou 310023, China
* Correspondence: zmbu@zust.edu.cn

Abstract: Six phthalates: dimethyl phthalate (DMP), diethyl phthalate (DEP), di(n-butyl) phthalate (DnBP), butyl benzyl phthalate (BBzP), di(2-ethylhexyl) phthalate (DEHP), and di(n-octyl) phthalate (DOP) in settled dust on different indoor surfaces were measured in 30 university dormitories. A Monte Carlo simulation was used to estimate college students' exposure via inhalation, non-dietary ingestion, and dermal absorption based on measured concentrations. The detection frequencies for targeted phthalates were more than 80% except for DEP (roughly 70%). DEHP was the most prevalent compound in the dust samples, followed by DnBP, DOP, and BBzP. Statistical analysis suggested that phthalate levels were higher in bedside dust than that collected from table surfaces, indicating a nonuniform distribution of dust-phase phthalates in the sleep environment. The simulation showed that the median DMP daily intake was 0.81 μg/kg/day, which was the greatest of the targeted phthalates. For the total exposures to all phthalates, the mean contribution of exposures during the daytime and sleeping time was 54% and 46%, respectively.

Keywords: phthalates; settled dust; dormitory; sleep environment; exposure assessment

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1. Introduction

Phthalates have been recognized as a group of emerging indoor air pollutants in the past few decades. Phthalates are typically used as plasticizers or solvents during the manufacturing process of a wide range of daily products, such as soft polyvinyl chloride (PVC) and polymers, wallpapers, stickers, children's toys, and food packages [1,2]. These chemicals can be released from artificial materials since they are not chemically bound to the material matrices, leading to the deterioration of indoor air quality [3]. Phthalates are semi-volatile organic compounds, a group of chemicals with high boiling points and low vapor pressure. Besides the vapor phase in the air, phthalates can also be easily absorbed by suspended particles, settled dust, indoor surfaces, and even the human skin surface [3]. Therefore, humans can be exposed to phthalates via inhalation, oral ingestion, and dermal pathways [4]. Scientific evidence has indicated that exposure to phthalates may lead to a variety of adverse health effects, such as endocrine disorders [5,6], reproductive system dysfunction [7,8], children's asthma [9,10] and neurodevelopment problems [11,12], diabetes [13], and obesity [14].

Phthalates accumulated in settled dust are a result of contact transfer between the source and dust, dust-air partitioning, and particle deposition [15,16]. The abundance of dust-phase phthalates could be treated as an indicator of phthalate pollution and is widely used for estimating exposures in a given indoor environment. In previous studies, settled dust from one certain location in a room was usually collected [17–21]. They assumed that phthalate concentration was uniformly distributed in the target space, without taking into account the influence of airflow or occupant activities. This simplicity could be acceptable for most cases, but not for a sleep environment. During the sleeping time, concentrations

of air pollutants could be higher in the vicinity of the human body (i.e., the sleeping microenvironment) than in the bulk room air [22–24]. Liang and Xu [25] found that the contents of several phthalates or their alternatives in crib mattress covers could reach 10% or even greater. Furthermore, the emission strengths of those chemicals could be further increased at night due to the heat transfer from occupants to bedding materials [25–27]. On the other hand, the air exchange (normally driven by the thermal plume) in a sleep environment might not be enough for the dispersion of pollutants [24]. For instance, typical air exchange rates in bedrooms were found to be less than 1 h^{-1} at night [28,29]. Therefore, to improve the accuracy of exposure estimates in the sleep environment, sampling from multiple locations is required.

A university dormitory is one of the most important indoor environments for university students (provided that students stay in their dormitories for at least 8–10 h per day). They live in private spaces, which are full of daily products, furniture, and decoration materials, for at least four years. A dormitory can be treated as a sleep environment at night. The specific indoor environment of university dormitories may result in significant exposures to phthalates and associated health risks [30]. During the SARS-CoV-2 epidemic, students had to stay in dormitories (roughly 12–14 h/day) and take online courses to reduce their infection risk. The daily intakes may increase with respect to the elevated time spent in dormitories. Therefore, using university dormitories as examples, the objectives of this study are as follows: (1) measure six phthalate concentrations in settled dust from two different locations in dormitories (bedside and table dust), and (2) estimate exposures to phthalates in university dormitories using the measured data. The detailed information can provide a better understanding of the fate of phthalates as well as associated exposures in such sleep environments.

2. Materials and Methods

2.1. Study Design

The field test was carried out as reported previously by Yao et al. [31]. Briefly, our investigation was carried out from November 2020 to December 2020 at Zhejiang University of Science and Technology, Hangzhou, China. A total of 30 male undergraduate dormitories on the campus were selected. Each dormitory contains a public room and a bathroom, accommodating four students. There are four beds, four bedside cupboards, and one table in the public space (roughly 5 × 4 m). A university dormitory can be treated as a sleep environment, where the air pollutant concentrations may not be uniformly distributed due to the source-proximity effect (as a result of strong source emissions and the occupants' thermal plume during sleeping time) and insufficient air exchange [24]. In each dormitory, settled dust on the table (roughly 2 m away from the bed) and the top of one bedside cupboard (right next to the bed) were both collected. Therefore, phthalates in collected dust could represent personal exposures in the dormitories during daily activities and sleep, respectively.

2.2. Chemicals

Six phthalates, i.e., dimethyl phthalate (DMP), diethyl phthalate (DEP), di(n-butyl) phthalate (DnBP), butyl benzyl phthalate (BBzP), di-2-ethylhexyl phthalate (DEHP), and di(n-octyl) phthalate (DOP), were selected as the target compounds since they were commonly-used plasticizers and frequently detected in Chinese indoor environments [32]. Analytical standard mixtures of these chemicals (2000 mg/L of each phthalate in hexane) were purchased from Organic Standard Solutions International Co., LLC (North Charleston, SC, USA). Dichloromethane (DCM, HPLC grade, TEDIA Co., Inc., Fairfield, OH, USA) was used as the solvent for sample extraction.

2.3. Dust Sampling and Chemical Analysis

Settled dust was collected with a household vacuum cleaner equipped with a glass fiber membrane, as detailed in our previous studies [17,31]. The membrane was inserted

into a self-made stainless-steel collector. The internal surfaces of the collector were carefully wiped with gauze pads (wetted with DCM) to remove any phthalates remaining in the collector before each sampling. Impurities like hair, lint, or paper scraps were excluded from the collected samples using pre-cleaned tweezers. The glass fiber membrane was baked at 350 °C in a muffle furnace for at least 3 h to remove any phthalates remaining in the sampling media. The membranes were weighed on an electric balance (precision of ±0.1 mg) before and after sampling. Collected samples were wrapped in pre-cleaned aluminum foil, transferred to the laboratory, and stored at −20 °C until chemical analysis.

Dust samples were extracted with DCM in a Soxhlet extractor at 70 °C for 8 h. The extracts were concentrated to about 20 mL using a rotary evaporator, filtered through a 0.45 μm polytetrafluoroethylene (PTFE) microporous membrane, and then transferred into a Kuderna–Danish (K–D) tube. Thereafter, the clean extracts were further concentrated to 1 mL under a purified nitrogen stream. Finally, 200 μL of the concentrated extracts were transferred from the K–D tubes into 2 mL Agilent sample vials equipped with 250 μL microvolume inserts. Final samples were stored at 4 °C in a laboratory refrigerator until chemical analysis.

All samples were analyzed using a GC-MS system (Agilent Technologies, GC-7890N and MS-5975C, Santa Clara, CA, USA) equipped with an auto-liquid injector (Agilent G4513A). A chromatographic column with the dimensions 30 m × 0.25 mm × 0.25 μm (Agilent HP-5MS, helium gas at 1.0 mL/min) was used for chromatography separation. Mass spectrometry was operated in both scan and SIM (selected ion monitoring) modes. The targeted compounds were quantified by the selected molecular ions: m/z = 163 for DMP and m/z = 149 for the other phthalates. The GC oven temperature was maintained at 80 °C for 2 min, increased to 220 °C at 10 °C/min and maintained for 3 min, and further increased to 300 °C at 20 °C/min and maintained for 3 min, i.e., for a total of 26 min. The temperatures of the injection port and ion source were 280 °C and 250 °C, respectively. For solvent extracts, 1 μL extracts were injected into the GC injection port.

2.4. Quality Assurance and Quality Control (QA and QC)

An eight-point calibration curve was obtained by 1 μL injections of a standard mixture with concentrations of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, 10, and 20 μg/mL (of each phthalate). A linear function between the peak area and the injected amount of each phthalate was assumed valid when R^2 of the linear function was greater than 0.99. For each sampling event, a field blank was prepared by directly wrapping up a pre-cleaned glass fiber membrane in aluminum foil. All blanks were analyzed by the same method as the samples. Phthalate concentrations in the field blanks were all below the detection limit of quantification (LOQ). The recovery rates were determined by adding 10 μg of a standard mixture to blank glass fiber membranes and were found to be 78–112%. The precision of the measurement method was assessed by replication spikes (n = 9) of a 10 μg/mL standard mixture solution. The relative standard deviation (RSD) ranged from 3.7% to 8.6%.

2.5. Statistical Analysis

Statistical analyses were performed with SPSS software (v.18.0 for Windows), with significance defined as p = 0.05. Concentrations below detection limits were replaced by half of the detection limit. A Shapiro–Wilk test was conducted to examine whether the measured data were normally or log-normally distributed. Statistical differences among dust-phase phthalate levels in different locations were evaluated using Wilcoxon tests for paired samples. The correlations between concentrations of different compounds were evaluated using Spearman's rank correlation analysis.

2.6. Exposure Assessment

Exposures to phthalates in university dormitories were separated into two parts, i.e., exposures during the daytime and sleeping time. Exposures in the daytime were calculated as the summed daily intakes of phthalates via inhalation, dust ingestion, and dermal

absorption, which were detailed elsewhere (see Supporting Information (SI) Table S1) [17,32]. The inhalation and dermal pathway accounted for phthalate exposures during sleeping time. The exposure level of the gas-phase phthalate (C_g, μg/m^3) was estimated from the measured abundance in settled dust (X_{dust}, g/g) based on a dust-air partitioning model [33]:

$$C_g = \frac{X_{dust}}{K_d}, \quad (1)$$

where K_d is the dust-air partitioning coefficient of a given phthalate, m^3/μg.

The particle-phase concentration (C_{sp}, μg/m^3) was estimated from C_g based on a particle-air partitioning model [33]:

$$C_{sp} = C_g C_p K_p, \quad (2)$$

where C_p is the mass concentration of suspended particles indoors, μg/m^3; K_p is the particle-air partitioning coefficient, m^3/μg. Note: particle-phase concentrations only account for DnBP, BBzP, DEHP, and DOP since the other two phthalates (i.e., DMP and DEP) exist almost entirely as vapor phases in the air [3].

PM_{10} was assumed to represent indoor suspended particles, and C_p can be calculated as per the method of Liu et al. [34] as follows:

$$C_p = C_{p,out}\left(\mathrm{mf}_{2.5}\frac{ACH}{ACH + v_{d,2.5}}P_{2.5} + \mathrm{mf}_{2.5-10}\frac{ACH}{ACH + v_{d,2.5-10}}P_{2.5-10}\right), \quad (3)$$

where the subscripts "2.5" and "2.5–10" indicate $PM_{2.5}$ and $PM_{2.5\text{-}10}$, respectively; $C_{p,out}$ is the mass concentration of outdoor suspended particles, μg/m^3; mf is the mass fraction for outdoor particles, unitless (68% and 32% for $PM_{2.5}$ and $PM_{2.5–10}$, respectively); *ACH* is the air exchange rate, h^{-1}; v_d is the particle deposition rate constant, h^{-1} (0.09 h^{-1} and 4 h^{-1} for $PM_{2.5}$ and $PM_{2.5–10}$, respectively); and *P* is the penetration coefficient of outdoor particles, unitless (0.8 and 0.3 for $PM_{2.5}$ and $PM_{2.5–10}$, respectively).

2.7. Uncertainty and Sensitivity Analysis

In the present study, an uncertainty and sensitivity analysis was performed with the Oracle Crystal Ball software (fusion edition, 64-bit for Windows, v. 11.1.2.2). The number of trials was set to be 10,000 (i.e., enough to reach a stable mean or standard deviation for the exposure estimates). Phthalate concentrations were deduced to be log-normally distributed according to the Shapiro–Wilk tests. For other input parameters, coefficients of variations (CVs), defined as the ratio of standard deviation to the mean, were assigned to describe the uncertainty and variability. For instance, CV values between 0.1 and 0.3 reflect typical measurement variability and uncertainty. A CV between 0.3 and 3 indicates a parameter obtained with a somewhat reliable estimation method [35]. Physical properties of each phthalate were assigned log-normal distributions with a CV of 1.0 (see Table S2). Exposure factors were assumed to be log-normally distributed except for body weight, which was assigned a normal distribution (listed in Table S3).

3. Results

3.1. Phthalate Concentrations in Settled Dust

Statistics of the dust-phase concentrations of targeted phthalates in two different locations are listed in Table 1. Phthalate levels in bedside dust were previously reported by Yao et al. [31]. Among all dust samples, the detection frequencies for targeted compounds were more than 80%, except for DEP (roughly 70%). DEHP levels were significantly higher, followed by DnBP, DOP, and BBzP. Dust-phase concentrations of DMP and DEP were the lowest. This trend was similar among settled dust on two different surfaces. Based on the results of Wilcoxon tests, dust-phase levels for bedside dust were significantly higher ($p < 0.05$) than those for table dust (except for DMP and DEP), suggesting that phthalate

levels in the sleeping microenvironment could be higher than other spaces in the room. The co-occurrence between phthalate levels in table dust and bedside dust is shown in Table 2. The concentrations of DnBP, BBzP, DEHP, and DOP between those two locations were significantly correlated. These results suggest that phthalates in table dust and bedside dust might come from the same sources in the dormitories.

Table 1. Dust-phase phthalate concentrations in university dormitories (μg/g).

	DMP	DEP	DnBP	BBzP	DEHP	DOP
Bedside						
Mean	8.5	5.2	264	54.7	958	95.0
25th%	4.2	0.3	48.0	7.3	329	36.2
50th%	6.1	4.3	195	42.6	660	54.0
75th%	7.5	7.2	309	59.6	1597	114
frequency	96	70	100	100	100	100
Table						
Mean	4.5	4.1	43.8	27.3	210	28.4
25th%	1.0	0.3	4.6	9.9	57.3	10.0
50th%	2.7	2.8	16.0	21.2	130	16.8
75th%	4.9	5.7	35.9	45.2	220	40.6
frequency	80	72	92	96	100	100

Table 2. Correlation coefficients between phthalates in table dust and bedside dust by Spearman's rank correlation analysis.

	DMP	DEP	DnBP	BBzP	DEHP	DOP
DMP	−0.111					
DEP		0.315				
DnBP			0.724 **			
BBzP				0.636 **		
DEHP					0.704 **	
DOP						0.460 *

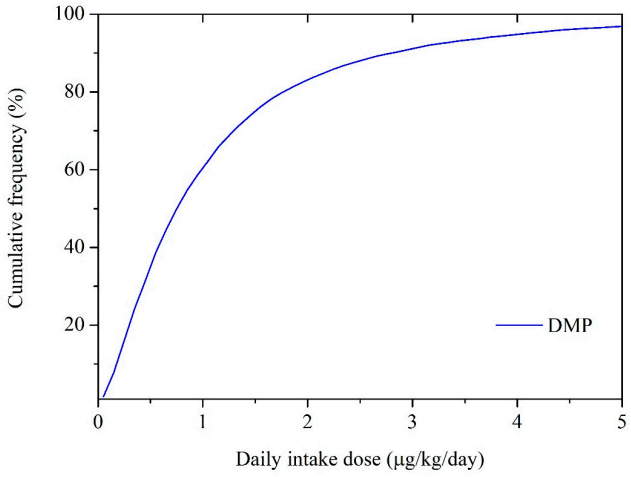
* $0.01 < p < 0.05$, ** $0.001 < p < 0.01$.

3.2. Exposure to Phthalates in Dormitories

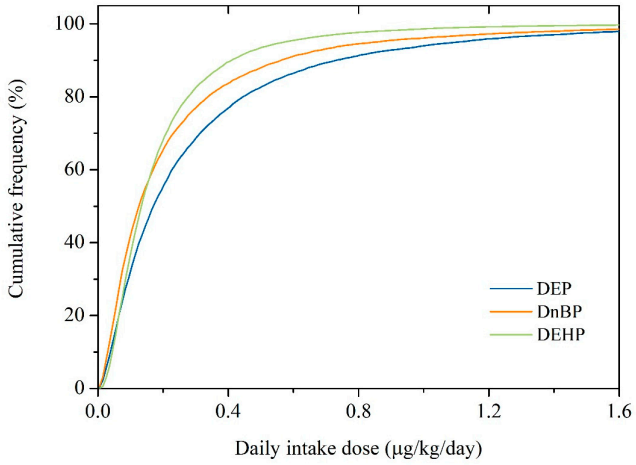
The cumulative frequency distribution of exposure to each phthalate is shown in Figure 1. Modeled results for BBzP and DOP are not included since their daily intake doses were a magnitude lower (with mean values of 0.02 and 0.03 μg/kg/day, respectively) than the other phthalates. For university students, the daily intake of DMP was higher (with a median of 0.81 μg/kg/day) than the other three targeted phthalates (medians ranging from 0.13 to 0.18 μg/kg/day). The mean value of the summed daily intake of the six phthalates was 2.31 μg/kg/day.

The contributions of each pathway to total exposure were also calculated. As shown in Figure 2, exposures via inhalation and dermal absorption mainly contributed to the total exposures for DMP, DEP, and DnBP (with ranges of 45–76% and 24–50%, respectively). Non-dietary exposure via dust ingestion was a predominant pathway for BBzP, DEHP, and DOP (roughly 70–80%).

The contributions of exposures in the different time periods (daytime vs. night) were further estimated. The results indicated that exposures during the day played a more important role for DEHP, BBzP, and DOP (mean contributions ranging from 80 to 88%) since oral intake was the most predominant pathway. For other lower-molecular-weight phthalates, the corresponding contributions were roughly 37–63%. For the summed daily intakes of all targeted phthalates, exposure during the daytime contributed roughly 54%.



(**a**)



(**b**)

Figure 1. Cumulative frequency distribution of exposure to phthalates in dormitories. (**a**) DMP; (**b**) Other phthalates.

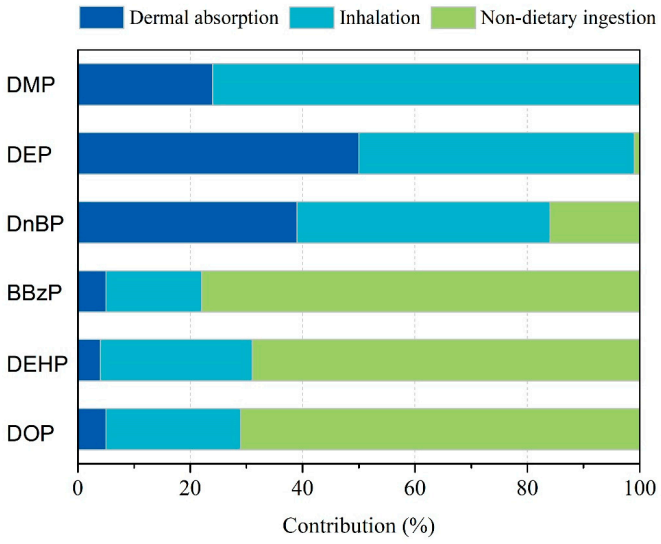


Figure 2. Mean contributions of exposure pathways for each phthalate.

3.3. Sensitivity Analysis

The sensitivity of input parameters to modeled daily intakes in university dormitories was analyzed for targeted phthalates (except for BBzP and DOP). As shown in Figure 3, the dust-air partitioning coefficient, dust-phase concentration, and transdermal coefficient (K_{p_g}) were found to be the major parameters. For DMP and DEP, the variations of the dust-air partitioning coefficient and dust-phase concentration of table dust contributed roughly 80–90% of the total variance. For DnBP, the dust-air partitioning coefficient and dust-phase concentrations (including both bedside and table dust) were predominant contributors. For DEHP, the phthalate level in the table dust was the most predominant input parameter (with a contribution of 85%) since dust ingestion was the most important exposure pathway during daily activities.

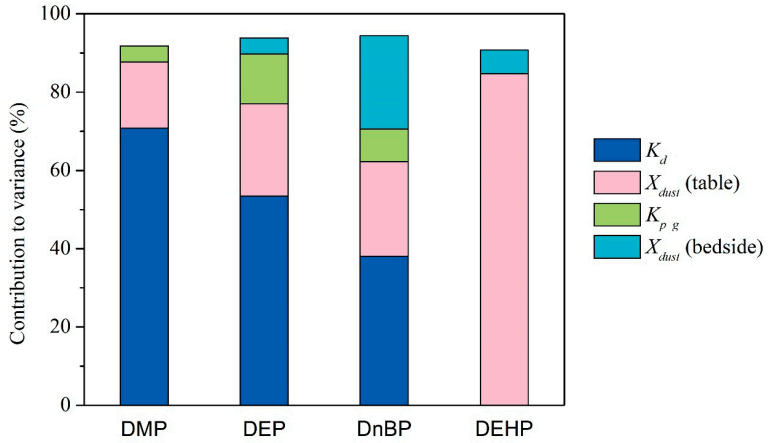


Figure 3. Sensitivity of major input parameters to total exposures.

4. Discussion

There was a significant difference between phthalate levels in settled dust in different locations, suggesting that phthalates were not uniformly distributed in dormitories. Phthalates accumulated in settled dust on bedside cupboards could be an indicator of phthalate pollution in the sleeping microenvironment. Previous studies pointed out that the concentrations of air pollutants (e.g., organic compounds, particles) in the sleeping microenvironment could be higher than those in the bulk room air, which might be a result of the source-proximity effect [24]. Some chemicals (both volatile and semi-volatile) might be added during the manufacturing process of certain materials in the sleeping microenvironment (e.g., mattresses, pillows, and covers) [22,23]. Liang and Xu [25] found that the contents of several phthalates or their alternatives in crib mattress covers could reach 10% or even greater. On the other hand, the air exchange (normally driven by the thermal plume) between the sleeping microenvironment and the bulk room air was limited, which might not be enough for the dispersion of pollutants [24]. Furthermore, the emission strengths of those chemicals could be further increased at night due to the heat transfer from occupants to those bedding materials [36]. This might lead to higher phthalate levels in the bedside dust. Therefore, sampling from different locations could improve the accuracy of exposure assessment in such sleep environments (e.g., bedrooms or dormitories).

We compared our measurements with those from previous studies, which also focused on dust-phase phthalates in Chinese university dormitories. As listed in Table 3, DnBP and DEHP were the most predominant compounds among all studies, suggesting that the two phthalates were the most commonly used in our country. Phthalate concentrations in bedside dust were higher in our present study.

Table 3. Medians of dust-phase concentrations of DnBP and DEHP (μg/g) in Chinese university dormitories.

References	Cities	Sample Size	DnBP	DEHP
Present study (bedside)	Hangzhou	30	195	660
Present study (table)			16.0	130
Qu et al. (2021) [37]	Beijing	102	32.7	171
He et al. (2016) [20]	Nanjing	8	76.2	202
Xu and Li (2021) [38]	Nanjing	23	38.8	134.9
Li et al. (2016) [30]	Harbin	18	45.2	270
Li et al. (2016) [30]	Baoding	8	29.2	65.2
Li et al. (2016) [30]	Shenyang	8	37.1	657
Hua et al. (2022) [39]	Tianjin	36	25.0	68.0

The investigation was conducted during the SARS-CoV-2 epidemic. Based on the requirement for epidemic prevention and control by the local government, although students were allowed to come back to the university campus, many courses still used online teaching. Students had to stay in their dormitories and take classes during the daytime. On the other hand, students preferred to stay on campus rather than go downtown to spend their leisure time. In the present study, the exposure frequency during the daytime was assumed to be 14 h/day for the exposure estimates, which might be somewhat higher than usual. If the value decreased by 40% (representing a time use pattern as usual), the mean total daily intake of targeted phthalates changed from 2.31 to 1.86 μg/kg/day (19% less). The contribution of exposures during the daytime decreased from 54% to 46%. Therefore, phthalate exposures in university dormitories might increase during the epidemic due to the change in time use patterns.

There are some limitations to this study. Firstly, the sample size was relatively small, which might not completely represent the concentration distributions of dust-phase phthalates in university dormitories. Settled dust from only two different surfaces was collected, which may not be enough to obtain a real distribution of dust-phase phthalates in such an indoor environment. Secondly, airborne concentrations were not directly measured but estimated from the dust-phase concentrations. This estimation may bring uncertainties for both inhalation and dermal exposure estimates, especially for lower-molecular-weight compounds. Thirdly, dermal absorption for clothing-covered skin was assumed to be negligible in our exposure estimates. However, exposure via the dermal pathway could increase when occupants wear dirty clothes (phthalates have been absorbed by the clothing materials before exposure) [40–42]. Although occupants' bodies were covered by quilts or clothing materials, our previous study found that dermal absorption contributed to phthalate exposure while sleeping [31]. Exposure via dermal pathways might be underestimated in our present work. These factors are possible refinements for future study.

5. Conclusions

Phthalates accumulated in settled dust in university dormitories were measured. DMP, DnBP, BBzP, DEHP, and DOP were frequently detected (more than 80%), but not DEP. Among the six commonly-used phthalates, DEHP was the most predominant compound. Phthalate concentrations in the bedside dust were significantly higher than those in the dust collected on table surfaces. For undergraduate students, the daily intake of DMP was higher than that of other phthalates. During the SARS-CoV-2 epidemic, the mean total daily intake of target compounds was 2.31 μg/kg/day. Exposures during the daytime contributed to 54% of the total exposures.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/atmos14040612/s1. Section S1, Detailed information of chemical analysis; Table S1, Equations used in the calculation of daily intakes; Table S2, Mean values of physical properties for targeted phthalates; Table S3, Values of exposure factors for Monte-Carlo simulation.

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