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Exploring personal chemical exposures in China with wearable air pollutant monitors: A repeated-measure study in healthy older adults in Jinan, China

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ABSTRACT

The health impact of airborne contaminants has been challenging to assess due to current limitations in measurement technologies. The emergence of wearable passive samplers coupled with high resolution mass spectrometry (HR-MS) chemical analysis has enabled comprehensive characterization of personal exposures. We conducted a repeated-measure study among 84 older adults in Jinan, China, as part of the Biomarkers for Air Pollutants Exposure (China BAPE) study. Study objectives were: 1) to characterize the occurrence, magnitude, and distribution of personal exposure to airborne contaminants; 2) to evaluate the temporal variation of chemical exposures across the study population; and 3) to identify behavioral and environmental factors that influence the observed variance in chemical exposures. The FreshAir wristband was worn by participants for three consecutive days each month from September 2018 to January 2019 and collected with paired time-activity logs. Passive air samplers were also deployed in parallel at a local outdoor air monitoring station. Spearman's Rho trend test and trajectory cluster analysis were used to identify exposure trends and variation patterns, respectively. Out of the 70 airborne compounds of potential concern screened, 26 compounds from 10 chemical classes were found to be above detection thresholds across >70% of the study population. Personal exposures were predominantly characterized by nine polycyclic aromatic hydrocarbons (PAHs), four phthalates, three nitroaromatics, and two volatile organic compounds (VOCs). Phthalate personal exposures were positively correlated with outdoor temperatures while the inverse relationship was observed for certain PAHs ($p < 0.05$). Specifically, dimethyl phthalate ($r_s = 0.31$) decreased as temperatures declined, while nitrobenzene ($r_s = -0.35$) and naphthalene ($r_s = -0.40$) increased as temperatures decreased. Compared to levels measured at the outdoor air monitoring site, personal exposure of phthalates was elevated ($p < 0.05$) and hexachlorobutadiene was lower across participants ($p < 0.01$). Personal exposure of these chemicals was further found to be weakly associated with daily duration participants spent outdoors. Individuals formed distinct clusters based on trajectories of chemical exposures across the sampling period (September to January), potentially suggestive of distinct emission sources. In conclusion, we demonstrate the feasibility of characterizing the occurrence and magnitude of personal exposure to airborne chemical contaminants using passive wristband samplers. The temporal variability of these personal exposure profiles was highlighted and with distinct trends identified across different groups of individuals. Future studies will integrate this data with other omics datasets collected from this population of Chinese older adults to investigate associations between exposure profiles and health relevant biomarkers, to provide evidence in feasibility of disease prevention through environmental improvements.

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1. Introduction

Complex mixtures of airborne chemicals have been detected in indoor environments (Farmer et al., 2019), including a diverse range of volatile organic compounds (VOCs) and semi volatile organic compounds (SVOCs) released from various consumer products, indoor activities, and building materials and furnishings (Allen et al., 2007; Demirtepe et al., 2019; Lucattini et al., 2018). The extended time spent by individuals indoors (>90%) has prompted interest in better understanding personal chemical exposures and the influence of these exposures on health (US EPA, 2018d). A growing number of studies have reported that poor indoor air quality is associated with various adverse health outcomes such as reduced cognitive function (Allen et al., 2016), heart and respiratory symptoms (Prüss-Üstün et al., 2016; Sakellaris et al., 2020), stroke, chronic obstructive pulmonary disease (COPD) and lung cancer (Prüss-Üstün et al., 2016; WHO, 2020).

Personal exposures to airborne exposome are dynamic (Jiang et al., 2018), exhibiting temporal and spatial variation which are influenced by diurnal or seasonal fluctuations in emissions, features of the built environments (Huang et al., 2020), and human behaviors (Hystad et al., 2009). Multiple time-variant factors can affect chemical levels in a microenvironment, such as meteorological conditions (Amoatey et al., 2018; Huang et al., 2020), indoor ventilation (Huang et al., 2020; Ye et al., 2017) and consumer product use (Lucattini et al., 2018). As time spent in certain locations of the house and consumer products used depend on individual preferences and domestic roles, even within the same house individuals may have vastly differing profiles (Bergmann et al., 2017; Chung et al., 2018; Cowell et al., 2017). Capturing these individual-level differences requires personal exposure assessment.

With the advancement of the exposome approaches (Vermeulen et al., 2020; Wild, 2005), wearable air pollutant sensors have emerged as an attractive tool which can be used for evaluating personal exposures. Silicone wristband passive samplers have been used in a growing number of external exposome related studies to characterize personal exposure to environmental chemicals (Allen et al., 2007; Craig et al., 2019; Hammel et al., 2018; O'Connell et al., 2014). The FreshAir wristband has recently been introduced as a lower-cost, wearable tool for collecting airborne contaminants onto a custom fabricated polymeric membrane sorbent bar composed of polydimethylsiloxane (PDMS) (Lin et al., 2020). The FreshAir wristband uniquely encases the PDMS sorbent in a housing chamber, restricting uptake to airborne contaminants (Lin et al., 2020). This design feature further limits airflow around PDMS sorbent minimize variance on the rate of contaminant uptake from activities including biking and those involved rapid arm movements, as well as from changes in wind and other factor affecting air flows around the sampler (Harner et al., 2003; Seethapathy et al., 2008; Zhang et al., 2013). Off-line chemical analysis of contaminants absorbed by the PDMS sorbent provides a time-integrated measurement of exposure.

These wearable passive samplers have been used to evaluate exposure to various VOCs, polycyclic aromatic hydrocarbons (PAHs), phthalates, pesticides, polychlorinated biphenyls (PCBs), and polybrominated diphenyl ethers (PBDEs) (Doherty et al., 2021; Koelmel et al., 2021a; Koelmel et al., 2021b; Lucattini et al., 2018). Detected VOCs and SVOCs have been linked to adverse health with select compounds recognized to be carcinogens or mutagens (Dodson et al., 2012; Encarnação et al., 2019; Rudel et al., 2010; US CDC, 2009; Vardoulakis et al., 2020). Wearable environmental samplers present opportunities to characterize chemical exposures at an individual-level to identify personal behaviors and sources that affect exposure profiles.

Countries are facing challenges with respect to the aging population. Notably, the impacts of modifiable environmental risk factors of noncommunicable diseases (NCDs) are disproportionately distributed across age; children under five and older adults are the most vulnerable populations with increased risks of NCDs (Choi et al., 2017; Guan et al., 2016; Koelmel et al., 2021a). Older populations are highly susceptible to the toxicity of air pollutants (Prüss-Üstün et al., 2016; Rückerl et al., 2011). Data is limited in personal exposures to airborne toxicants in older adults in urban China. In this study, we used these wearable sensors to quantify longitudinal personal exposures of older adults in Jinan, China over a five-month period to a panel of to 76 airborne contaminants (Koelmel et al., 2021a). This chemical assessment was conducted as part of China Biomarkers for Air Pollutants Exposure (BAPE) Study which also included collection of detailed activity logs and home characteristics. This rich dataset enabled us to overcome the caveats of previous studies and explore the temporal dynamics of personal exposures and the impact of behavioral and environmental factors. The objectives of this study were: 1) to characterize the occurrence, magnitude, and distribution of personal exposure to airborne contaminants; 2) to evaluate the temporal variation of chemical exposures across the study population; and 3) to identify behavioral and environmental factors that influence the observed variance in chemical exposures. Previous studies suggest that a large proportion of NCD burden can be mitigated through environmental interventions among vulnerable populations (Koelmel et al., 2021a; Prüss-Üstün et al., 2016). Findings from this longitudinal study may improve our understanding of long-term exposures to airborne contaminants and identify efficient targeted prevention and intervention strategies.

2. Material and methods

2.1. Study population and study design

Eighty-four healthy non-smoking healthy older adults (50% male), aged 60 to 69 years, living in Dianliu Community, Jinan, Shandong Province, China were recruited to participate in the China BAPE Study. The study was approved by the Ethics Review Committee of National Institute of Environmental Health (NIEH), Chinese Center for Disease Control and Prevention (China CDC, No. 201816). The consent form was given to in written and signed by each study participant. Jinan is a large city in eastern China with a population of approximately seven million residents. The study design, recruitment and enrolment process, inclusion and exclusion criteria of the China BAPE study have been previously described (Fang et al., 2020; Zhou et al., 2020). Briefly, environmental exposures of participants were comprehensively assessed over a series of 3-day assessment periods. Five of these assessment periods were conducted across monthly over five consecutive months from September 2018 to January 2019. Each participant wore a passive air pollutant sampler over the 3-day period each month and completed a time-activity log each day.

Passive air sampling in October 2018 was limited to 26 participants from the total 84 that were enrolled in the study due to shipping-related issues. The distribution of individual and household characteristics was not found to differ between participants included in the October sampling period and all study participants, except for participant higher body mass index (BMI) and residential location: the decreased number of participants assessed in October, on average, had an approximately one-unit higher BMI and were more likely to live further away from major road arteries (>200 m) (Tables S1-S2).

2.2. Sample and data collection

2.2.1. Personal chemical exposure assessment

A novel wearable passive air sampler known as the FreshAir Wristband was developed to evaluate personal chemical exposures (Fig. S1). The sampler design, wristband preparation, and sample analysis has been previously described (Lin et al., 2020). Briefly, the wristband contains polydimethylsiloxane (PDMS) sorbent bars housed in a polytetrafluoroethylene (PTFE) chamber. As sorption properties of PDMS are proportional to octanol air partition coefficient (log K_{OA}), this sampler collects the mass of a gas-phase chemical from air through molecular diffusion driven by the fugacity gradient across air and the sampler (Koelmel et al., 2021a; Lin et al., 2020; Padukudru et al., 2020). The

diffusion process continues through the linear and curvilinear sampling phases until the chemical attains dynamic equilibrium between air and the sampler (Koelmel et al., 2021a; Lin et al., 2020; Padukudru et al., 2020). We previously published results from an uptake study examining a range of chemicals exposures (Lin et al., 2020): in an indoor environment, comparable to the homes of BAPE study participants, linear uptake was demonstrated over a one-week period for compounds with log K_{oa} ranging from 4.15 to 11.72. Based on these findings, we selected a three-day exposure assessment period in BAPE study. This sampling period with the FreshAir wristband provided a sufficient duration to capture detectable levels of chemical exposure without saturating the sorbent material.

PDMS sorbent bars were cleaned at the Yale School of Public Health and shipped together with other wristband materials to the China BAPE study team in Jinan. Immediately prior to deployment, a member of the study team placed four cleaned PDMS sorbent bars into the PTFE chamber and delivered a FreshAir wristband to each participant at their home. Participants wore the FreshAir wristband for three consecutive days monthly. Participants were requested to remove the wristband while bathing or swimming. For participants that preferred not to wear the wristband while sleeping were directed to place the sampler on an elevated surface near their sleeping area. At the end of the exposure assessment period, a member of the study team collected the sorbent bars from participants' home and removed the PDMS sorbent bars from the wristbands. Cold chain transport was used to ship samples back to the Yale School of Public Health and stored at −80 °C until analysis.

To accommodate study team logistics, participants were divided into three approximate 25-person sub-groups and the sampling period of each group was staggered through each month. The start date and end date of each sampling period were the same in each sub-group. Details of enrolment dates has been reported (Fang et al., 2020; Zhou et al., 2020). A total of 298 wristbands were collected from participants and seven passive samplers deployed at an outdoor stationary site were collected for chemical analysis. Wristband samples were retrieved from all participants.

2.2.2. Outdoor airborne chemical and temperature monitoring

Airborne chemical levels were monitored at a fixed outdoor air monitoring station operated by the China National Environmental Monitoring Center and located 1.5 km from the participant's home at Dianliu Community. Similar to personal exposure assessment (Section 2.2.1), a FreshAir sampler was also used for stationary exposure monitoring and was positioned 1.5 m above ground (proximity to a person's breathing zone). A total of seven passive samplers were deployed for three consecutive days which aligned with participant exposure assessment: two sampling periods were conducted in September 2018, two additional samples were collected in October 2018, and three outdoor samples were deployed in November 2018. Ambient temperature was monitored (hourly averages) across the study period (September 10th, 2018 - January 19th, 2019) at the Jinan Meteorological Station.

2.2.3. Personal behavioral and environmental characteristics

At the start of the study, a member of the study team visited each participant's home and completed a questionnaire about the participant (*i.e.*, age, gender, education level, income) and characteristics of their home (*i.e.*, floor level, building area, distance from the home to the nearest main road, ventilation). Participants also completed daily activity logs during each of the five 3-day exposure assessment periods. The content of questionnaires on personal and household characteristics has been previously described (Koelmel et al., 2021a; Zhou et al., 2020). Briefly, participants visited the study office at the local community hospital on each day of the assessment period. Daily time activity logs were completed together with detailed the amount of time spent at home/outdoors/in transit and the length of time windows at home were opened. All logs and surveys were conducted on tablets by trained investigators.

2.3. Data acquisition

Chemical analysis of PDMS sorbent bar has been previously described (Koelmel et al., 2021a; Lin et al., 2020). Briefly, PDMS sorbent bars were loaded with a panel of internal standards, extracted using a thermal desorption method, and then separated and analyzed using GC-HRMS. Samples extracted by thermal desorption were directly transferred to the GC column (TG-5SILMS, 30 m × 0.25 mm × 0.25 μm; ThermoFisher, Waltham, MA, USA) from the thermal desorption unit. The carrier gas flow (helium) was set to 1.4 mL/min and the GC oven was held at 70 °C for one minute and then ramped at 7 °C/min to 300 °C. The final temperature was held for 4.0 min for a total run-time of 37.86 min. During the analysis, full-scan electron ionization (EI) mass spectra (m/z 53.4–800) was recorded at an acquisition rate of 4 Hz (30,000 resolution) on a Q-Exactive Orbitrap mass spectrometer (ThermoFisher, Waltham, MA, USA). QCs and blanks (laboratory and transport) were run every 5 samples. Samples were introduced in randomized batches, which were limited to a maximum of 80 acquisitions (samples, controls, and standards). A cleaning protocol was conducted between each batch.

2.4. Data processing

Raw mass spectral data were analyzed by TraceFinder 4.1 (Thermo). A seven-point calibration curve (0 to 1000 pg/uL) was set up for each compound and subsequent quantification, then applied for quantification for a total of 70 compounds. All coefficients of determination (R^2) for calibration curves were>0.95. Peak integration was checked manually before data export. Blank feature filtering (BFF) was performed using instrument blanks to eliminate compounds with high background contaminations after chromatographic peak-picking (Patterson et al., 2017). The equation below shows how BFF was conducted:

$$x_{95\%} > 10\left(\bar{b} + 3\sigma_b\right) \quad (1)$$

where $x_{95\%}$ is the sample 95th percentile, ($\bar{b}$) is the field blank average, and $\sigma_b$is the standard deviation of the field blank.

To account for batch effects, features were normalized using batch-wise median normalization (BMN). This technique has previously been described in detail (Koelmel et al., 2021a). Chemicals with non-detects over 30% were excluded (Helsel, 1990; Lubin et al., 2004). Other non-detects were replaced by half the lowest detected level (Xia and Wishart, 2011). All chemicals were log2 transformed (x + 1) before further analysis.

2.5. Statistical analysis

To explore correlations across targeted chemical compounds and chemical classes, the Spearman's rank correlation was conducted since most of the chemical exposures did not follow a normal distribution after transformation. Correlation globes were modified from chord diagram plotting using *chorddiag* R package to show inter-class correlations (Holtz, 2018).

To determine variable importance in explaining the greatest variance of compounds, Random Forest (RF) machine learning approach (Breiman, 2001), as implemented in the *randomForest* R package (Liaw and Wiener, 2002), was used to estimate the contributions of behavioral/environmental variables collected from questionnaires on the binary PC scores obtained via PCA on 26 log-transformed compounds scaled to unit variance. A total of 232 questionnaire responses were collected from 66 participants that completed the study questionnaire at least once across five months study period. It is useful to note that results based on all 232 questionnaire responses were deemed to be robust as we did not observe a substantial difference in chemical exposure profiles between 232 records from participants surveyed and 66 records from participants without available questionnaires in any months (Table S3,

Fig. S2). We developed a RF algorithm based on a training set randomly sampled from 70% of the 232 records (N = 162) and used the remaining 30% of records as a testing set (Alderden et al., 2018). In the testing set, we tested the agreement between measurements and predictions by calculating the average area under the curve (AUC) using the receiver operating characteristic (ROC) approach. We used 1000 trees to build the RF (bootstrap samples N = 162). All available predictors (M = 85) were entered at the first step, then at the second step we retained the top 10 predictors (M = 10) that were most likely associated with PC scores according to variable importance measure using 'importance' function in the *randomForest* package (Liaw and Wiener, 2002). Data collection of predictors are described in Sections 2.2.2 and 2.2.3. out-of-bag (OOB) estimate of error rate was 22.49% at the first step, 20.71% at the second step. Using the selected 10 predictors from the second step, AUC was 0.660 in the testing set. We then plotted variable importance on the PC scores of the 10 variables using *ggplot* package. All calculation and visualization for PCA were conducted using *rexposome* R package (Hernandez-Ferrer et al., 2019). Missing data imputation for behavioral/environmental variables was handled by *mice* R package (Buuren and Groothuis-Oudshoorn, 2010). To identify the seasonal trends of chemical exposures by season and personal activity, the Spearman's Rho trend test was used in chemicals versus ambient outdoor temperature and time spent outdoors.

To compare exposure levels across months and between personal measurements and outdoor stationary measurements, the pair-wise multiple comparison method, Dunn's test following a Kruskal-Wallis test was performed. The relationship between chemical exposure levels and temperature was displayed using locally weighted regression smoothing plots (implemented using *ggplot2* 'loess' method in R).

To explore individual variation in exposure across seasons, a k-mean based trajectory cluster analysis was conducted for each time-varying chemical on individuals with at least four complete data points using *kml* and *kml3d* R packages (Beckers et al., 2020; Genolini et al., 2015). In choosing initial configurations, the 'kmeans' method was deployed to achieve optimal center repartition and minimum complexity. The number of clusters was determined by five non-parametric criteria within the *kml* and *kml3d* R packages.

Given the exploratory nature of these analyses, no statistical adjustments were conducted for multiple comparisons. We performed sensitivity analyses to evaluate if the decreased sample size for the October assessment period biased results. As participant included in this sampling month, on average had a higher BMI or longer distance from major roads ($p > 0.05$), we stratified analyses by BMI (>=75% percentile, <75% percentile) and by distance from major roads (<50 m, >=50 m).

3. 3.Results

3.1. Characteristics of study participants

For the subset of participants (66/84) who filled out individual and household surveys, details are provided in Table 1. Of the 66 participants, 50% were females, with an average age of 65 years. >20% of participants completed a bachelor or graduate level degree. Participants resided in homes constructed between 1977 and 2016. Most participants lived in a building with less than six floors (76.9%) and over 50 m away from a major road (69.7%).

A total of 682 of the 24 -hour time-activity logs were collected and are summarized by month in Table 2. Participants spent a median of 21 h at home. In warmer weather participants kept the window open nearly all day (median 24 h), while window opening time decreased sharply starting in November (median 10 h), with windows open only 1.5 h on average in January.

Table 1
Individual and household characteristics of study participants (N = 84).

Characteristic	*n* (%) or Mean ± SD
Sex	
Male	33 (39.3)
Female	33 (39.3)
Unspecified‡	18 (21.4)
Education level	
Below middle school	7 (10.6)
Secondary school	45 (68.1)
Bachelor and above	14 (21.2)
*Floor level**	
1st-5th floor	50 (76.9)
6th-33rd floor	15 (23.1)
*Year of Home Construction**	
1977–1999	33 (70.2)
2000–2016	14 (29.8)
Distance from major road arteries (m)	
<50	20 (30.3)
50–200	31 (46.9)
>200	15 (22.7)
Age (years)	65.1 ± 2.8
Income (10 k yuan/year)	4.5 ± 3.7
BMI (kg/m^2)	24.9 ± 2.7
Indoor room area (m^2)	79.4 ± 36.6

Abbreviations: SD, standard deviation. BMI, body mass index.
‡ Sex listed as unspecified if questionnaire was not returned.
* 1 missing in floor level, 19 missing in year of home construction.

3.2. Distribution of detected chemical exposures across participants

Wristband samplers were analyzed for 70 compounds, spanning 15 chemical classes (Table S4). A total of 39 of these compounds were excluded after BFF. These compounds included herbicides, fungicides, PCBs, polychlorinated naphthalene derivatives, brominated flame retardants (BFRs), dioxins, and furans. Among the other 31 compounds, five compounds (organophosphate ester (OPE) flame retardants, smoking-related chemicals, pesticides) were excluded due to detection in<70% of samples. A total of 26 chemicals were included in further exposure analyses (Fig. S3) and included 9 PAHs, 4 phthalates, 3 nitroaromatics, isophorone, and 2 volatile organic compounds (VOCs) (Fig. 1). Generally, phthalates had the highest detected mass loading with medians ranging from 2,749 pg/g (Q1 to Q3: 1,544 to 5,176) for diethyl phthalate to 28,042 pg/g (Q1 to Q3: 16,103 to 50,723) for dimethyl phthalate, whereas polybrominated diphenyl ethers (PBDE) was found to have the lowest detected mass loading: median of 11 pg/g (Q1 to Q3: <1 to 13) for BDE-154. The median exposure levels of PAHs were highest for naphthalene (median 6,198 pg/g, Q1 to Q3: 3,264 to 13,055), and lowest for pyrene (114 pg/g, Q1 to Q3: 69 to 162). Exposure levels of nitroaromatics and isophorone ranged from 1,166 pg/g (Q1 to Q3: 759 to 1,772) for 4-nitroaniline to 7,157 pg/g (Q1 to Q3: 3,796 to 13,534) for isophorone. The median exposure levels of VOCs ranged from 458 pg/g (Q1 to Q3: <1 to 1,630) for 1,4-dichlorobenzene to 2,351 pg/g (Q1 to Q3: 1,067 to 8,207) for 1,3-dichlorobenzene.

Two chemicals showed significant gender difference in both overall and month-specific personal exposures. Males had 15%-70% higher personal exposures to 2-methylnaphthalene than females ($p = 0.006$), with the largest difference in November. Males had over 60% higher 1,4-dichlorobenzene ($p = 0.015$) than females in October, but the difference became narrower in colder seasons.

3.3. Intra-class and inter-class correlations among chemical exposures

Correlations were identified across compounds within (intra-correlations) and between (inter-correlations) all 10 chemical classes; variation in the strength of correlations were found across seasons (Fig. 2). Overall, of the 325 unique pairwise correlations, 240 were found to be significant ($p < 0.05$). There were high intra-class correlations observed across PAHs and phthalates irrespective of month (season), with

Table 2
Overview of study participants' activity patterns over the 5-month exposure assessment period.

	Median (Q1,Q3), Hours per day					
Activity	*September*	*October*	*November*	*December*	*January*	*Overall*
Indoors	21.0 (18.5,22.0)	21.0 (19.0,22.0)	20.3 (19.0,22.0)	21.5 (19.5,22.5)	22.0 (21.0,23.0)	21.0 (19.5,22.5)
In-transit	1.0 (0.5,2.0)	1.0 (0.5, 2.0)	1.0 (0.5,2.0)	1.0 (0.5,1.5)	1.0 (1.0,1.5)	1.0 (0.5,1.7)
Outdoors	2.0 (0.6,4.0)	2.0 (1.0, 3.0)	2.0 (1.0,4.0)	1.5 (0.5,3.0)	1.0 (0,1.5)	1.5 (0.5,3.0)
Open windows	24.0 (17.0,24.0)	24.0 (15.0, 24.0)	10.0 (6.0,14.0)	4.0 (0.6,10.0)	1.5 (0.5,5.0)	10.0(2.0,24.0)
Cooking	0.2 (0,0.7)	0.4 (0,1.3)	0 (0,0.9)	0.2 (0,0.6)	0 (0.2,0.8)	0.2 (0,0.8)
Range Hood use	0.8 (0.2,1.2)	1.0 (0.2,1.3)	1.2 (0.7,2.2)	1.0 (0.3,1.5)	0.9 (0.5,1.4)	1.0 (0.4,1.5)

Abbreviations: Q1, the first quartile value. Q3, the third quartile value.

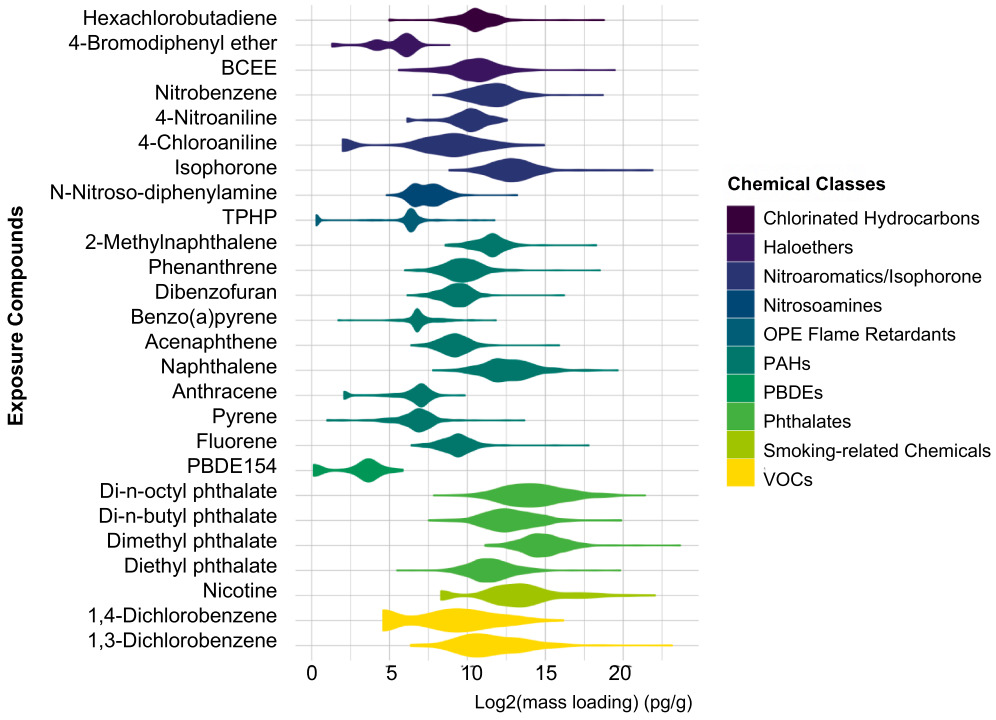

Fig. 1. The distribution of targeted exposure compounds among the study population of older adults in Jinan, China. Abbreviations: OPE, organophosphate ester. PAHs, polycyclic aromatic hydrocarbons. PBDEs, polybrominated diphenyl ethers. VOCs, volatile organic compounds. BCEE, bis(2-chloro-1-methylethyl) ether. TPHP, triphenyl phosphate.

naphthalene and di-n-octyl phthalate least correlated with other chemicals within the chemical class. Most PAHs demonstrated moderately stronger correlations with other PAHs in warmer seasons than those in colder seasons. For example, the average correlation coefficient between naphthalene and other PAHs in September was 0.51, but were 0.57, 0.56 0.41, 0.43 in the following months, respectively. Nitroaromatics, isophorone, VOCs, and haloethers did not show strong intra-correlations regardless of month. Specifically, the two detected VOCs (1,3-dichlorobenzene and 1,4-dichlorobenzene) constantly exhibited different trends despite their structural similarity. PAHs were found to be strongly correlated with phthalates across months (Fig. S4A). PAHs and phthalates were also both significantly correlated with nitrosoamines, nitroaromatics, isophorone, haloethers, and OPE flame retardants for the study period (Fig. S5; Fig. S6).

3.4. Seasonal trends of personal chemical exposures and comparison with outdoor monitoring

Correlations between principal component (PC) scores and chemicals, and behavioral/environmental characteristics were determined to understand which variables explained the greatest variance of compounds (Fig. 3). The first two principal components (PC) explained approximately 50% of variance across exposure compounds (Fig. 3A). The standardized loadings represent correlations between PCs and chemicals (Fig. 3B). No significant correlations were found between PC1 scores and behavioral/environmental characteristics (data not shown). By contrast, PC2 scores were significantly correlated with certain behavioral/environmental variables ($p < 0.05$). To interpret and quantify the relative importance of these characteristics to PC2 scores, Random Forest model was used for selecting and ranking the top ten important variables in Fig. 3C. The top three variables that were found to vary with PC2 scores were related to season: ambient outdoor temperature, assessed month, and window opening time. Exposure levels were poorly predicted by the other seven variables (Fig. 3C). These seven variables included anthropometrics (*i.e.*, BMI, weight, height), cooking behaviors (*i.e.*, range hood using time), housing characteristics (*i.e.*, home construction year, indoor room area), and air conditioner (AC) use last month. Chemical exposures that were well-defined by PC2 included bis(2-chloro-1-methylethyl) ether (BCEE), HCBD, nitrobenzene, phthalates, naphthalene and naphthalene-derivatives (Fig. 3D).

Chemical exposures varied with ambient outdoor temperature, exhibiting different trends according to chemical class. Generally, phthalates tended to decrease at lower temperatures while the inverse relationship was observed for certain PAHs such as naphthalene and

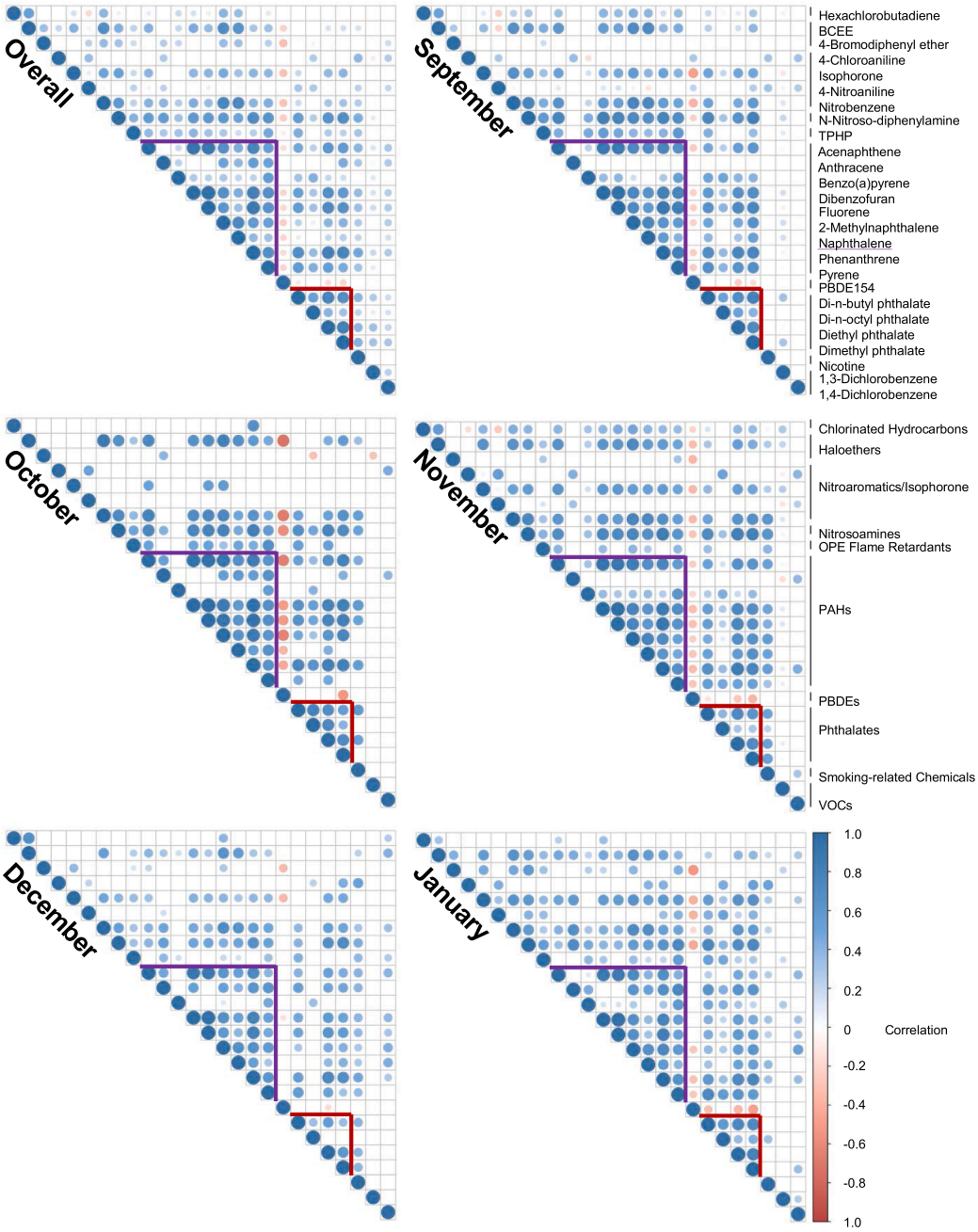

Fig. 2. The chemical exposure correlation heatmaps across months. Statistically significant correlations ($p < 0.05$) are included in heatmaps. The names of chemicals are shown in the overall heatmap while the names of chemical classes were shown in the heatmap of January. The intra-class correlations of PAHs are highlighted in the triangle area with purple line outline, the intra-class correlations of phthalates were highlighted in the triangle area with red outlines. Abbreviations: OPE, organophosphate ester. PAHs, polycyclic aromatic hydrocarbons. PBDEs, polybrominated diphenyl ethers. VOCs, volatile organic compounds. BCEE, bis(2-chloro-1-methylethyl) ether. TPHP, triphenyl phosphate.

benzo(a)pyrene (Fig. 4A). Four chemicals were found to have moderate correlations with ambient outdoor temperature ($r_s > 0.30$, Fig. 4B). 1,3-dichlorobenzene ($r_s = 0.40$, $p < 0.001$) and dimethyl phthalate ($r_s = 0.31$, $p < 0.001$) decreased at lower temperatures. In contrast, nitrobenzene ($r_s = -0.35$, $p < 0.001$) and naphthalene ($r_s = -0.40$, $p < 0.001$) increased at lower temperatures. Among all 26 personal chemical exposures, 13 were statistically significantly correlated with temperatures ($p < 0.05$). Exclusion of samples collected during the October sampling period did not change chemical-temperature correlations patterns observed for the complete dataset (Table S5). However, removing October samples were, however, observed to weaken the correlation between selected exposure chemicals, including phthalates, VOCs, and several PAHs (fluorene, acenaphthene, phenanthrene). These weakened correlations were limited to our stratified sensitivity analysis: participants with a higher BMI (>=75% percentile; 27.2) or that lived further away from a major road (>=50 m) appeared to have weaker positive correlations in select chemicals (phthalates, VOCs, and some PAHs) than those with lower BMI (<27.2) or shorter DMR (<50 m) (Tables S6-7). Thus, the observed weakened correlations due to reduced number of personal samples in October did not change our conclusions.

Passive air samplers (FreshAir Clips) that were deployed at the stationary outdoor monitoring site and the FreshAir wristbands worn by study participants were found to detect similar chemical exposures (Fig. S3). Fig. 5 shows the fluctuation of chemical exposure levels with

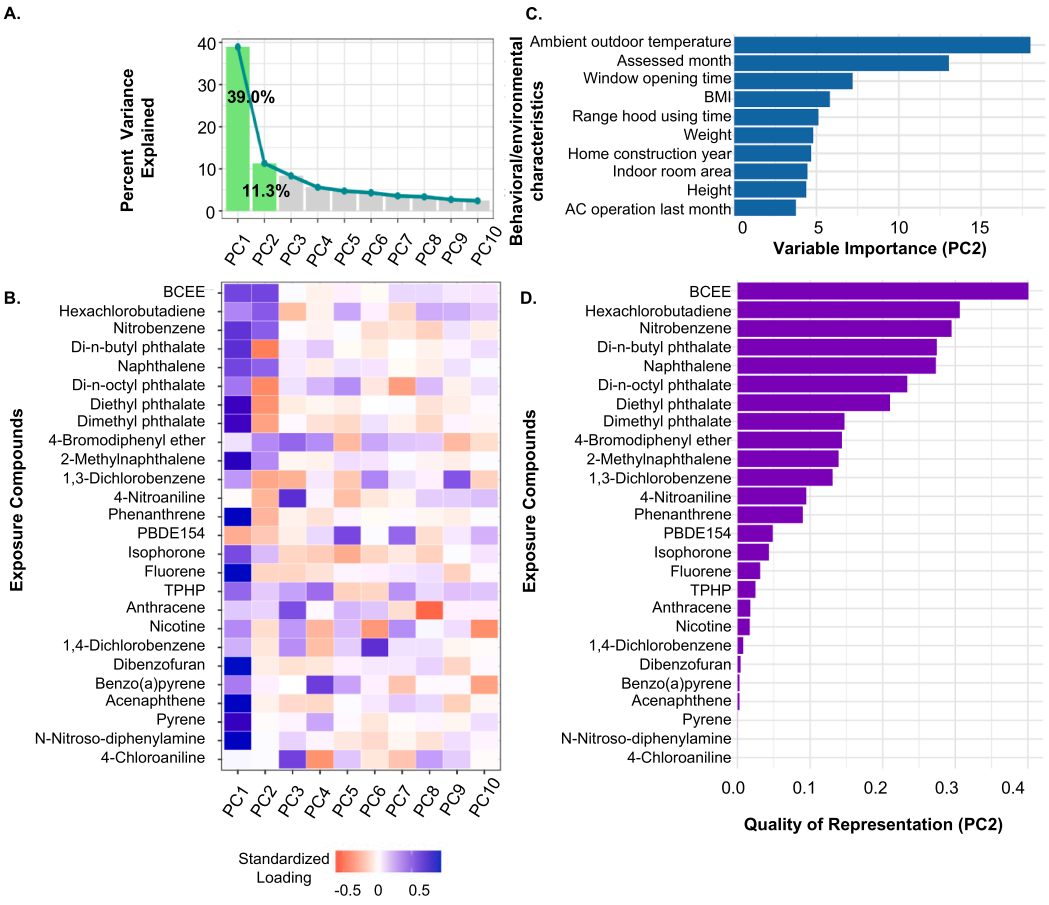

Fig. 3. Principal component analysis (PCA) of participants' personal chemical exposures and Random Forest (RF) model of behavioral/environmental characteristics on the PC2 scores. AC, air conditioner. **A**) Principal components 1 (PC1) and 2 (PC2) explained around 50% of variance across exposure compounds in total. **B**) PC1 had the highest loadings among PAHs, PC2 had the highest loadings among nitrobenzene, naphthalene, hexachlorobutadiene (HCBD), BCEE, 1,3-dichlorobeneze, and phthalates. **C**) Three variables indicating seasonality (ambient temperature, assessed month and window opening time) were the top three variables covarying with the PC2 scores. **D**) Among all chemicals, BCEE, HCBD, nitrobenzene, naphthalene and phthalates were most well-defined by PC2. Abbreviations: BCEE, bis(2-chloro-1-methylethyl) ether. TPHP, triphenyl phosphate. PBDEs, polybrominated diphenyl ethers. AC, air conditioner.

time spent outdoors, and distributions of chemical levels in outdoor site samples. Among the 31 chemicals assessed using wristband samplers, five of these compounds, including dieldrin, 2,4-dinitrotoluene, TCPP, methoxychlor, and delta-9-tetrahydrocannabinol, were below detection limits, reflective of indoor emission sources. Outdoor levels of phthalates were also found to be lower compared to personal exposure concentrations ($p < 0.01$), consistent with well-documented indoor residential sources of these compounds or proximity to products used (e.g., cosmetics). In contrast, two compounds (HCBD, BCEE) were found to have significantly elevated levels for outdoor samplers compared to personal exposure levels ($p < 0.01$), which supports prevalent emission from industrial sources in the study region.

3.5. Exposure-specific trajectory patterns

Fifty-nine of the 66 study participants participated in at least four exposure assessment periods. Two of these participants were excluded as it was not possible to assign these two participants into clusters formed with the remaining study population using five non-parametric criteria within the *kml* and *kml3d* packages. A total of 57 participants with measurements spanning 4 or 5 months were included in trajectory cluster analysis for each quantified chemical exposure. The overall seasonal trend of time-varying personal exposures consisted of several distinguishable trajectory patterns (Fig. 6).

Participants were subdivided into one of two groups based on their naphthalene exposure levels; one group included a smaller number of participants ($n = 9$) and consistently exhibited elevated exposure concentrations. For BCEE, phthalates, and nitrobenzene, participants were found to have similar exposure levels in winter regardless of different trajectories in autumn. The major cluster of participants for dimethyl phthalate (25/57) exhibited a decreasing trend over the study period. The other two dimethyl phthalate clusters decreased two-fold in autumn before reaching the same average level in winter. The trajectory of di-n-butyl phthalate exposure levels also declined slowly across the study period for most participants (39/57); exposure levels of the remaining 18 participants dropped three-fold in October compared to other study months. Compared with other chemical exposures, decreased variation was observed for HCBD across seasons and between individuals.

4. Discussion

We present the first longitudinal assessment study quantifying personal exposure of older adults to a comprehensive mixture of airborne contaminants. Using a novel wearable exposure sensor, we found distinct season trends in personal exposures for our study population in Jinan, China. Phthalates ($n = 4$) and PAHs ($n = 9$) dominated exposures with a trend towards decreased phthalate levels as outdoor temperatures declined and the inverse relationship observed for certain PAHs such as naphthalene. We additionally considered the toxicity of detected chemical exposures using the prototype EPA Hazard Comparison Dashboard (EPA-HCD) (Vegosen and Martin, 2020). EPA-HCD ranks health hazards of chemicals, based on experimental and predicted

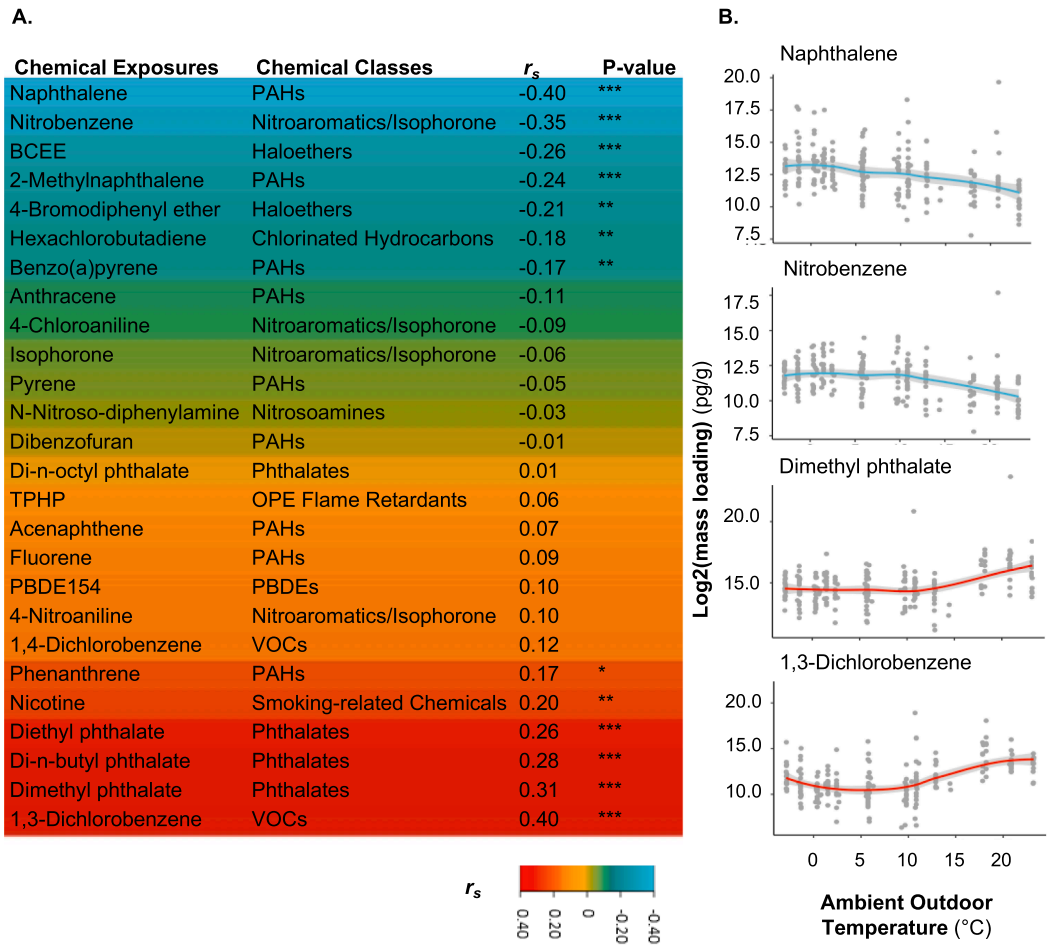

Fig. 4. Chemical exposures varied with ambient outdoor temperature. A) Spearman's rho correlation (r_s) between individual exposure levels of chemicals and the average outdoor temperature (*$p < 0.05$, **$p < 0.01$, and ***$p < 0.001$). B) Smoothed trend plots of four chemicals with the strongest positive and negative correlations between exposure levels and ambient outdoor temperature ($|r_s|>0.30$). Abbreviations: OPE, organophosphate ester. PAHs, polycyclic aromatic hydrocarbons. PBDEs, polybrominated diphenyl ethers. VOCs, volatile organic compounds. BCEE, bis(2-chloro-1-methylethyl) ether. TPHP, triphenyl phosphate.

toxicities available through the Alternatives Assessment Dashboard (Table S8).

4.1. Phthalates

Amongst the 70 measured airborne contaminants assessed, the high exposure concentrations were found for phthalates. Four phthalates were detected using FreshAir wristbands worn by study participants, diethyl phthalate, dimethyl phthalate, di-n-butyl phthalate, and di-n-octyl phthalate, all commonly used plasticizers. The high exposure levels detected for the BAPE study participants is in line with the growing number of studies that have also reported airborne phthalate concentration detected with international study populations (Agency for Toxic Substances and Disease Registry ATSDR, 2001; Bergmann et al., 2017; Blanchard et al., 2014; Bu et al., 2016; Chen et al., 2018; Dixon et al., 2019; Fromme et al., 2004; Huang et al., 2020; Lunderberg et al., 2019; Mitro et al., 2016; Rudel et al., 2010; Wang et al., 2014; Zhang et al., 2014). Despite this emerging literature describing the potential for inhalation exposure, most toxicological and epidemiological studies have focused phthalates exposures from consumer or food products (Grosse et al., 2011; Kim et al., 2020; Romero-Franco et al., 2011; Wang et al., 2017). The elevated levels detected for our population of older adults reinforce the need to characterize airborne phthalate exposures and further suggest prioritization of these airborne compounds for future studies as they can potentially increase the risk of metabolic disorders and even more severe diseases in aging populations (Dales et al., 2018; Kim et al., 2013). All of the four phthalates detected have high endocrine disruption toxicity, while three of them also have high acute inhalation toxicity, according to EPA-HCD.

Detected phthalates included three lower-molecular-weight compounds: dimethyl phthalate, diethyl phthalate, di-n-butyl phthalate. On average, personal exposure to these airborne chemicals was elevated compared to the stationary outdoor air samplers. While we acknowledge our outdoor sampling was limited to a single site, which may not be representative of different sources, other studies have similarly reported decreased airborne phthalate levels outdoors compared to indoor environment in China (Chen et al., 2018; Ouyang et al., 2019) and other countries (Anh et al., 2020; Başaran et al., 2020). A range of indoor residential sources of these compounds have been well documented, including various consumer products, such as shampoos and soaps as well as other personal-care and cosmetic products (Huang et al., 2020; Koch et al., 2013; Mitro et al., 2016; National Research Council, 2008; Pei et al., 2013; Sakhi et al., 2019). Personal exposure levels to the low-molecular-weight phthalates were positively correlated with outdoor temperatures. Similar seasonal changes have been reported for other China-based studies, including in Tianjin (Zhang et al., 2014) and Beijing (Chen et al., 2018; Huang et al., 2020). Elevated exposure levels may be attributed to increased air emissions of phthalates from indoor sources at higher temperatures, as shown in controlled laboratory testing (Wei et al., 2018; Zhou et al., 2021). The increased exposure during warmer periods may also be reflective of seasonal behavioral patterns such as personal care product use (*i.e.*, sunscreen).

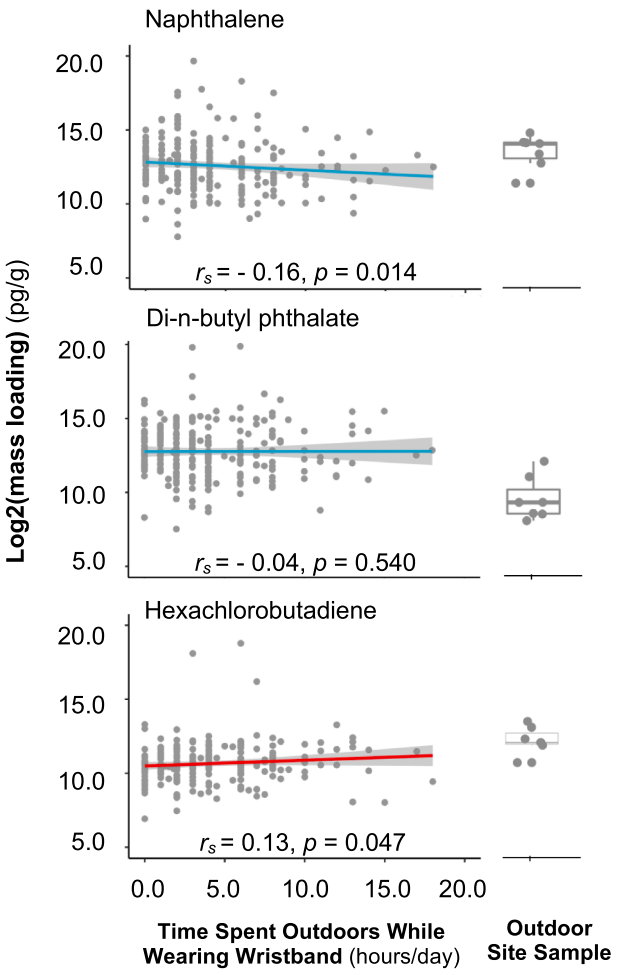

Fig. 5. Fluctuation of chemical exposure levels with time spent outdoors. Wristband personal exposure levels were found to decrease with time spent outdoors for naphthalene and di-n-butyl phthalate. In contrast, increased personal exposure levels of hexachlorobutadiene were found with increasing time spent outdoors. Personal exposure levels are compared to samples collected at the stationary outdoor air monitoring site.

No seasonal trend was observed for the higher-molecular-weight phthalate (di-n-octyl phthalate) detected in this study. High-molecular-weight phthalates with high boiling points and low volatility tend to exist primarily in the particle phase and partially in gas phase (Wei et al., 2018; Zhou et al., 2021). Two China-based studies have evaluated indoor airborne levels of this compound. Our results agree with the assessment of homes in Beijing (Chen et al., 2018), however, increased di-n-octyl phthalate levels were reported in the summer in Tianjin (Zhang et al., 2014). Unlike the lower-molecular-weight phthalates, which are released from consumer products that may have varied use across seasons, higher-molecular-weight phthalates are commonly found in food packaging materials, vinyl floor coverings, and building materials (National Research Council, 2008). The distinct sources of the various phthalates measured in this study were further supported through our correlation analysis. Personal exposure levels of triphenyl phosphate, a flame retardant and plasticizer in a wide variety of materials, were correlated with a single phthalate, di-n-octyl phthalate. This relationship was maintained across the entire study period (September to January), suggestive of common emission sources for the two compounds.

4.2. PAHs

We evaluated the exposure of BAPE Study participants to 8 of the 16 EPA priority PAHs. Elevated concentrations were detected for naphthalene, 2-methylnaphthalene, and benzo(a)pyrene. Multiple PAHs are acknowledged to have a 'very high' level of carcinogenicity (naphthalene, benzo(a)pyrene, anthracene) or genotoxicity mutagenicity (benzo (a)pyrene, anthracene, pyrene, fluorene). Among them, naphthalene and benzo(a)pyrene are listed as a Group 2 carcinogen and a Group 1 carcinogen by the International Agency for Research on Cancer (IARC), respectively. Exposure to naphthalene and benzo(a)pyrene has been associated with incremental lifetime cancer risk (Han et al., 2020; Liu et al., 2020; Zhang et al., 2016). In addition, inhalation of benzo(a) pyrene has been associated with age-related macular degeneration, a major cause of central vision loss in the elderly (Wang et al., 2009). Seven PAHs detected are labelled with high risk of endocrine disruption effect, which may lead to metabolic disorders. Detected PAHs also have adverse respiratory effects (WHO, 2010). Typically, pyrene has a 'very high' level of acute inhalation toxicity.

The eight lower molecular weight PAHs detected using the wristband samplers were found to be correlated across each of the sampling months. The strength of the identified correlations increasing during warmer exposure assessment periods, suggested different primary emission sources depending on season such as tobacco, diesel fuels, grilled food, petroleum products, coal tar, coal burning and agricultural smoke (WHO, 2010). Participants' benzo(a)pyrene exposure was only weakly correlated with the lower molecular weight PAHs, potentially reflective of distinct sources or partitioning behavior (Wei et al., 2018; WHO, 2010). Only pyrene was moderately or strongly correlated with benzo(a)pyrene across seasons.

Higher exposure levels were found for most PAHs, notably naphthalene and related derivatives, during cool periods. A similar trend was reported for a study in Chile, which evaluated the personal exposure of adults using silicone wristbands (Manzano et al., 2019). Various sources may be attributable to the elevated levels of PAHs detected in the current study, including cooking and heating systems (Liu et al., 2020; Sinha et al., 2005; Vardoulakis et al., 2020; WHO, 2010). Smoking may also be a source, but unlikely, as nicotine levels measured using the FreshAir wristband were low for all participants. Despite a ban on the production and sale of naphthalene-containing mothballs by the Chinese Government in 1993 (WHO, 2010), they are still available in the supermarket and network marketing in China. Household surveys undertaken in the BAPE study revealed the common use of mothballs, which can also be a source of naphthalene (Koelmel et al., 2021a; Moss et al., 2017; Sudakin et al., 2011; Zhu et al., 2009).

4.3. Other airborne contaminant exposures

Personal exposure to airborne pesticides was low and none of the 11 PCBs, dioxins, furans evaluated was detected for BAPE study participants. A recent study of individuals from 14 communities in Africa, North America, and South America similarly reported PCB exposures were not commonly detected (Dixon et al., 2019). This is consistent with the global phase-out of these chemicals (Hung et al., 2016). Furthermore, of the 14 organochlorine pesticides assessed, only two insecticides were detected with quantifiable levels detected in<30% wristbands (methoxychlor and dieldrin, Fig. S3). The low detected frequency of pesticide exposure is reflective of the urban study location. This finding is consistent with previous studies showing that these two pesticides were detected in the environmental samples (e.g., air and water) of Jinan, and the distribution pattern of the level of atmospheric organochlorine pesticides were directly or indirectly influenced by meteorological conditions (Jiao et al., 2018).

OPE, and to a lesser extent, PBDE flame retardants were detected in the air. This is in line with China's phasing-out of PBDE brominated flame retardants in 2004 (Dodson et al., 2012; Ma et al., 2017; Yu et al.,

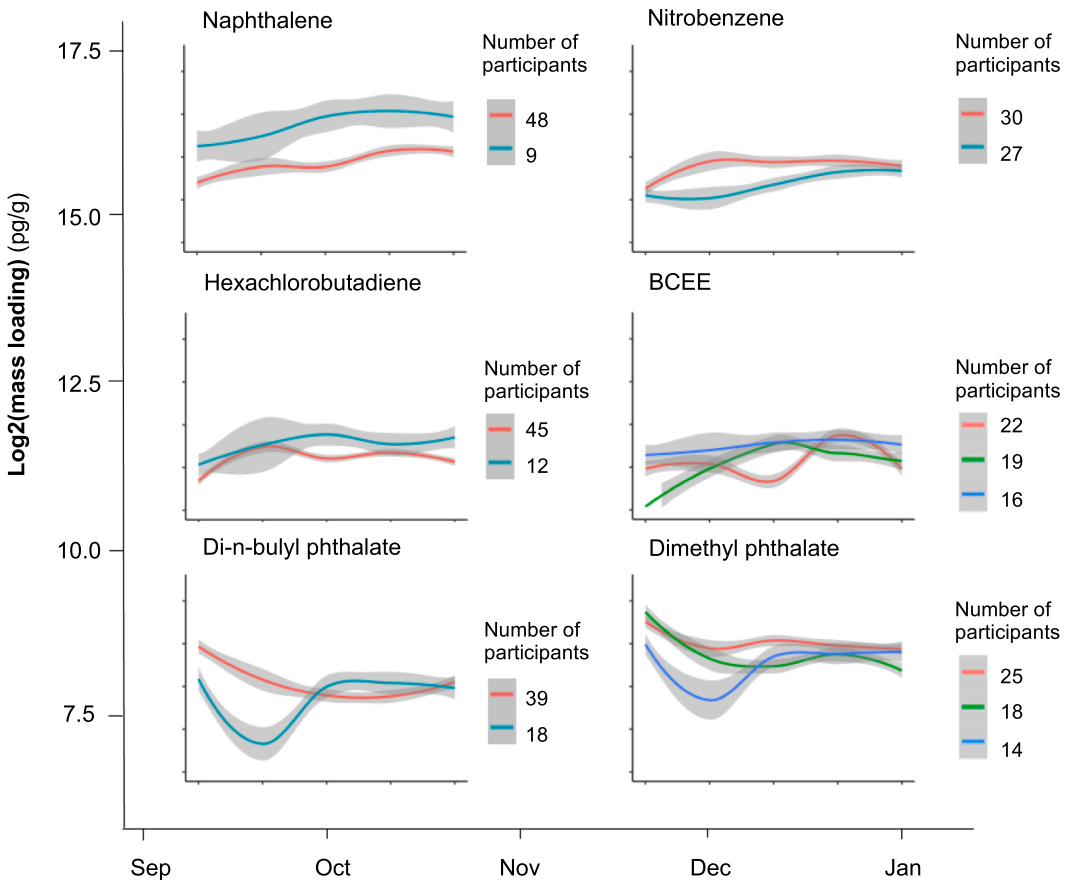

Fig. 6. Exposure-specific trajectory patterns among potential time-varying personal chemical exposures. Trajectory cluster analysis was applied in each chemical exposure respectively. Smoothing trends were displayed by loess method (local regression fitting). Grey shaded areas represented 95% confidence intervals. Abbreviations: BCEE, bis(2-chloro-1-methylethyl) ether.

2016) due to their bioaccumulation and toxicity, especially in endocrine disruption (Dodson et al., 2012), and replacement with OPE flame retardants (Rauert et al., 2018; Wang et al., 2020). The impact of this phase out has also been reported by other studies of urban communities near major water systems, which have observed a sharp decrease in PBDEs detected in environmental matrices (sediments in the aquatic environments) and human biological samples (breast milk) (Yin et al., 2020; Yu et al., 2016).

Apart from aforementioned compounds, some chemicals may pose great risk to older adults with high and very high toxicities. Five chemicals have very high acute mammalian toxicity, including nicotine, HCBD, BCEE, 4-nitroaniline, and pyrene. Nitroaromatics, isophorone, N-nitroso-diphenylamine, nicotine and BCEE are recognized to have very high carcinogenicity and very high genotoxicity mutagenicity. Nitrobenzene, 1,3-dichlorobenzene, and nicotine have high endocrine disruption toxicity. Although some of these chemicals did not show much abundance in the study population, they might be able to cause harm with minimum exposure levels.

Wristband samplers were assessed for 70 compounds, spanning 15 chemical classes. While exposure to a broad panel of compounds was quantified, we acknowledge our targeted list only represent a small percentage of airborne contaminants. To enhance chemical resolution, we additional performed a suspect screening analysis of the FreshAir wristbands collected from BAPE study participants. Exposure to known and unexpected environmental contaminants was obtained through this complementary analysis, albeit yielding semi-quantitative results (Doherty et al., 2021; Koelmel et al., 2021a).

4.4. Strengths and limitations of the FreshAir wristband for monitoring exposures

Airborne contaminants have traditionally been measured with active air samplers that draw air through a filter using a pump to enhance the sampling rate. The invasive nature of these samplers (*i.e.*, noise, cords/tubes, need for electrical power/batteries) significantly limit deployment. Their weight and size present challenges when evaluating the personal exposure, especially for vulnerable populations (*i.e.*, individuals with pre-existing health conditions, seniors). The high cost of active samplers presents further potential restrictions on the number of available units and the geographic region where they are deployed. Evaluation of personal chemical profiles over time is critical to understanding the dynamic nature of exposures and the impact on associated disease risk (Jiang et al., 2018). Technologies traditionally used to evaluate personal exposure present challenges in conducting repeated deployments with larger populations. The FreshAir wristband used in this study is a lower cost, passive air sampler that addresses these major drawbacks. This wearable device enabled assessment of a time-integrated exposure to a comprehensive panel of chemicals at the individual-level. The light-weight design promoted participant compliance, with wristband collected from 90% of participants for at least four monthly sampling time points (n = 298). These characteristics are promising for long-term monitoring of airborne contaminants across the life course in future applications (Doherty et al., 2021).

We acknowledge several limitations of this wristband exposure assessment approach and opportunities for future development, including uncertainties regarding the uptake rate of gas and particulate contaminants. Chemical uptake by the passive air samplers is influenced

by environmental conditions, including temperature. Elevated ambient temperatures may enhance uptake of airborne contaminants through increased diffusivity, sampler-air partition coefficient, and the sorption capacity of a passive sampler (Armitage et al., 2013). While this may have impacted samplers positioned at the stationary outdoor air monitoring site, the effects of extreme outdoor temperatures on personal sampling, was expected minimal as participants spent most of their time in indoor spaces. The median time outdoors ranged from 1 to 2 h per day over the study period. Passive samplers, including the FreshAir wristbands, exhibit efficient diffusive uptake for gas phase compounds as well as particles sized < 0.1 μm; particles sized > 0.1 μm can also be absorbed by these devices through gravitation and inertial processes (Nash and Leith, 2010; Wagner and Leith, 2001). We are currently evaluating particle uptake rates for different particle size fractions. These results are being prepared as part of another manuscript. Finally, the fact that our study population was focused on healthy older adults residing in an urban region in China may limit the generalizability of our findings to the population at large scale.

5. Conclusions

Wearable passive air samplers were used to evaluate the longitudinal personal exposure of older Chinese adults to 26 chemicals and were found to be dominated by phthalates and PAHs. Overall, during cooler seasonal periods phthalate exposure decreased while exposure to several PAHs increased. Distinct trends over the five-month assessment period (September to January) were further identified for individual chemicals across subgroups of participants. The comprehensive characterization of personal exposure to airborne containments may be used to guide targeted interventions that account for individuals' specific activities and behavior patterns. Future integration of this detailed personal exposure data collected through the China BAPE Study with health-related measures presents further opportunities to investigate associated biological responses. Efficient targeted environmental interventions then can be conducted to minimize the adverse health impact and disease burden.

CRediT authorship contribution statement

Pengfei Guo: Conceptualization, Data curation, Investigation, Methodology, Formal analysis, Validation, Visualization, Writing - original draft. **Elizabeth Z. Lin:** Conceptualization, Investigation, Data curation, Methodology, Writing - original draft. **Jeremy P. Koelmel:** Conceptualization, Data curation, Formal analysis, Investigation, Writing - original draft. **Enmin Ding:** Methodology, Investigation, Data curation. **Ying Gao:** Investigation, Data curation. **Fuchang Deng:** Investigation, Data curation. **Haoran Dong:** Investigation, Data curation. **Yuanyuan Liu:** Investigation, Data curation. **Yu'e Cha:** Investigation, Data curation. **Jianlong Fang:** Investigation, Data curation, Project administration. **Xiaoming Shi:** Conceptualization, Data curation, Funding acquisition, Investigation, Methodology, Project administration, Resources, Writing - review & editing. **Song Tang:** Conceptualization, Data curation, Funding acquisition, Investigation, Methodology, Project administration, Resources, Writing - review & editing. **Krystal J. Godri Pollitt:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Visualization, Writing - original draft. **:** .

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 material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envint.2021.106709.

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