Organophosphate Flame Retardants in Car Dust from Thailand and Implications for Human Exposure

Premrudee Kanchanapiya, Benjawan Nilyok, Supachai Songngam, and Sun Olapiriyakul

***Abstract*—Organophosphorus flame retardants (PFRs) are a group of compounds frequently detected in indoor dust that pose high health risks to exposure subjects. The present study reports on the investigation of the levels and profiles of seven target PFRs in car dust samples from Thailand. The samples were collected from the discarded air conditioning (AC) filter of 14 private cars (called AC dust samples) and from the bag filter installed in vacuum cleaners of 10 car washing services (called settled dust samples) in 2019. The concentrations of Σ_7PFRs in AC dust samples were approximately 3,800-91,000 ng/g, whereas those from settled dust samples were about 11,000 to 15,000 ng/g. Tris (2-butoxyethyl) phosphate (TBEP) was found to be the most prominent of PFRs detected in both types of car dust with the highest concentration of 39,000 ng/g for AC filter dust and 10,000 ng/g for settled dust. The main PFR contributors in both dust types were TBEP (80%, 75%), followed by Tris (2-ethylhexyl) phosphate (TEHP) (9%, 5%) and tris(1-chloro-2-propyl)phosphate (TCPP) (7%, 4%), respectively. Tris(2-chloroethyl)phosphate (TCEP) and tri cresyl phosphate (TCP) were not detected in both types of car dust and tris(1,3-dichloro-2-propyl) phosphate (TDCPP) was found only in settled dust samples. According to the results of exposure assessment to PFRs in car dust, the human exposures via ingestion for adults and toddlers ranged from 1.69×10^{-2} to 2.67 and 10.6 to 2,360 ng/kg/day. The human exposures via inhalation for adults and toddlers ranged from 3.27×10^{-4} to $5.17\text{x}10^{-2}$ and 2.58×10^{-1} to 40.9 ng/kg/day. The highest exposure among PFRs corresponded to TBEP for both adults and toddlers and the risk through ingestion was higher than inhalation intake. Toddlers were more exposed to PFR contaminants in comparison to adults. When comparing the estimated average daily intake (ADI) values with the reference doses (RfDs) for PFRs, it was found that exposure to PFRs in car cabins via inhalation and dust ingestion is unlikely to have adverse human health effects.**

***Index Terms*—Organophosphorus flame retardants (PFRs), indoor dust, car dust, human exposure.**

I. Introduction

Organophosphorus flame retardants (PFRs) are a group of organic compounds used as flame retardants, plasticizers, or antifoaming agents in a wide range of products especially in furniture, textiles, building materials, electronic and car interior parts to enhance fire safety and comply with safety regulations. They were used as a substitute for brominated flame retardants (BFRs), which were banned and phase-outs due to their persistence, bioaccumulative and toxic properties [1], [2]. Since the ban, PFRs have increased from ~0.3 to 1.0 million tons [3]-[5]. In most applications of PFRs, they are not reactive FR, which means they are not covalently bound to the polymeric structure [6]. Thus, these PFRs in the products are easily released into the environment during use or after disposal. The toxicity of some PFRs show the evidence of adverse development, neurotoxicity and carcinogenicity. Tris (1-chloro-2-propyl) phosphate (TCPP), tris (2-chloroethyl) phosphate (TCEP), and tris-(1,3-dichloro-2-propyl) phosphate (TDCPP) have been proven to be neurotoxic and carcinogenic [7], [8]. Tris-(1,3-dichloro-2-propyl)phosphate (TDCPP) was classified as a level two carcinogen in the risk assessment report of the European Union in 2008 [9]. Tris (2-ethylhexyl) phosphate (TEHP), triphenyl phosphate (TPP) and tricresyl phosphate (TCP) have the potential to cause neurotoxicity [3]. Recently, PFRs have been detected in indoor dust especially in homes, offices and cars due to their migration from the products. Their concentrations detected in indoor air are often higher than BFRs (PBDE) concentrations and the human exposure due to PFRs appears to be higher than that of PBDE [3], [9]–[11]. Due to low vapor pressures at room temperature, most organophosphates including some PFRs are classified as semi-volatile organic compounds (SVOCs), which can sorb strongly to dust particles. Therefore, indoor dust bound PFRs play an important role in human exposure to PFRs [12], [13]. Additionally, the main pathways of human exposure to organic contaminants in indoor environments are inhalation and ingestion of suspended dust particles [14]. Ingestion pathways are particularly relevant to children, who contact the dusty floors and put their hands and toys into their mouths. Settled dust that is sampled from floors and other objects by vacuum cleaner has been widely used as an indicator of indoor contamination since it can collect and accumulate SVOCs pollutants in the same as a passive sampler [15]. Moreover, the alternative method to collect dust particles in an indoor environment is the use of filters in air conditioning (AC) systems. The dust samples from the built-in filter, installed for relatively long periods of time, can be collected conveniently and low-cost. They also represent the integrated concentration of collected particles from large volumes of air over a long period of time [13], [16]. Car is another indoor environment that humans spend a lot of time with them for daily transportation. The PFRs concentration detected in a car is ten times higher than other indoor environment and the reason may be due to the higher

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K. Premrudee is with the National Metal and Materials Technology Center, Thialand (e-mail: premrudk@mtec.or.th).

N. Benjawan and O.Sun are with Sirindhon International Institute of Technology, Thaialand (e-mail: benjawan.nilyok@gmail.com, and suno@siit.tu.ac.th).

S. Supachai is with the National Metal and Materials Technology Center, Thialand (e-mail: supachais@mtec.or.th).

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levels of PFRs applied in various polymers used in car interiors [17], [18]. Additionally, when the car is exposed to rigorous sun radiation the temperature in the car cabin highly increases and it could cause a higher release of PFRs from interior materials into the gas/particle-phase [19], [20]. To assess the impact of PFRs exposure on human health based on PFRs concentration in car dust sampled from vehicles in Thailand the present study carries out to (1) examine the concentration levels and profiles of PFRs (TCPP, TCEP, TDCPP, TEHP, TPP, TCP and TBEP (Tris(2-butoxyethyl) phosphate)) in the dust samples collected from AC filter of 14 private cars and the settled car dust from bag filter installed in the vacuum cleaner used in 10 car wash services, in Thailand and (2) estimate the human exposure to PFRs via dust inhalation and ingestion.

II. Materials and Methods

A. Chemicals and Reagents

TPP-d_{15} was used as internal standards (IS) for seven PFRs analysis. All PFRs and IS were purchased from Cambridge isotope laboratories, Sigma-Aldrich, and Albemarle Europe Sprl. Florisil, alumina and diatomaceous earth used for clean- up were purchased from Thermo Fisher Scientific Inc. All solvents (n-hexane and acetone) were chromatography grade and purchased from EOS Scientific (Thailand). Helium gas (Ultra high purity 99.999%) was purchased from LINDE (Thailand).

B. Dust Sampling

AC filter dust samples and settled dust samples were collected from the discarded filter in car service centers and from the vacuum cleaner used in car wash services, respectively, located in Klong Luang, Pathumthani province from March to June 2019. In generally, AC filter dust installed in private car are made from polypropylene melt-blown fabric and designed to filter particles smaller than 2.5 μm, generated by the fuel combustion of vehicle. In general, car owners mostly change their air filters after more than a year of use. The brands of sampled cars were Asia (n=13) and U.S.A (n=1) and the year of manufacture ranged from 2007-2019. During sampling, the discarded AC filters were sealed in PE zip-lock bags and transported back to the laboratory within the same day of sampling. Then, the dust samples were removed from the AC filter by a soft toothbrush and they were kept in PE zip lock bags at -20 ℃ until further processing. Settled dust was sampled from the bag filter by putting it into a large PP bag, and then it was manually shaken and knocked to remove the dust from the bag filter. Then it was transported back to the laboratory. Normally, the bag filters installed in vacuum cleaners of car wash services are made from cotton and polypropylene (PP) and they can filter the particles in a wide range from 0.05 to 500 μm [21]. In the laboratory, the settled dust samples were sieved to remove any large debris and to obtain the particle size in the range of 125-500 μm. They were kept in PE zip-lock bags at -20 ℃.

C. Analytical Procedure

A Dionex ASE 300 system, equipped with 22 mL stainless-steel cells, was used to extract PFRs from dust samples. A cellulose filter was placed at the bottom of the extraction cell, and then 2 g of alumina, 2 g of florisil to remove nonpolar lipids and colored compounds were loaded into the cell. The remaining free space was filled with 5 g of diatomaceous earth mixed with 0.2 g of dust sample. Hexane: Acetone (1:1, v/v) was used to extract PFRs from the dust samples with the pressure of 1500 psi and the temperature of 150 ℃ [22], [23]. A static time of 8 minutes, purge time of 120 seconds, and 100% flush volume was used with three static cycles. The extracts were evaporated to 5 mL, using a rotary evaporator. Finally, it was filtered by a 0.22 μm nylon syringe filter and transferred to a 2 ml injection vial for further GC analysis [24].

PFRs were analyzed by Shimadzu single quadrupole GCMS-QP2010 SE gas chromatograph (GC)-mass spectrometer (MS) operated in electron impact (EI) mode. The GC system was equipped with a DB-5ms capillary column (15 m, 0.25 mm, 0.1 μm film thickness). The injection temperature was set at 40 ℃, held for 1 min, ramped at 2 ℃/min to 60 ℃, ramped at 30 ℃/min to 210 ℃, ramped at 5 ℃/min to 280 or 340 ℃, held for 2 min. The final injector temperature was set at 280 ℃ for PFRs except for TBEP (340 ℃). The injection was performed with splitless mode. Helium was used as a carrier gas with a flow of 1 ml/min until the end analyze time (~36 min). The temperatures of ion and interface source were maintained at 200 ℃ and 280 ℃ for PFRs except for TBEP at 200 ℃ and 330 ℃, respectively. The MS was run in selective ion monitoring (SIM) mode [24].

D. Quality Control

TPP-d_{15} was used as an IS in a concentration of 150 ppb for all sample analysis. Recovery rates were examined by method blank sample spiked with a known concentration of target PFRs standard solution. The range of recovery percentage was between 81 and 97 %. The limit of detection (LOD) and limit of quantification (LOQ) were calculated based on the standard deviation (SD) of the response and the slope. At least two method blanks were extracted and analyzed in parallel with each sample batch (5 samples per batch) to check the laboratory contamination. Blank concentrations were periodically checked and controlled to be lower than LOD. In addition, the repeatability of GC-MS analysis was checked by running one sample per batch in duplicate and the repeatability was controlled to be lower than 20%.

E. Exposure Assessment and Potential Health Risk

The estimation of human exposure was based on the Agency for Toxic Substances and Disease Registry (ATSDR) approach [25]. The average daily intake (ADI) for PFRs via dust ingestion and inhalation was calculated by the following equations:

$$ADI_{Ingestion}(ng/kg/day) = C_{dust} \times IG \times AB \times EF/BW \quad (1)$$

$$ADI_{Inhalation}(ng/kg/day) = C_{dust} \cdot CP \cdot IH \cdot AB \cdot EF/BW \quad (2)$$

where C_{dust} is the concentration of the compound in indoor dust (ng/g); IG is the ingestion rate of dust (0.05 g/day for adults and 0.06 g/day for toddlers [26]); AB is the absorption rate (100%); EF is the exposure frequency or the time spent in a car (assumed 1 hr/day for adults and toddlers [26], [27]); BW is body weight (assumed 63 kg for adults and 12 kg for toddlers [10], [28]); CP is estimated concentration of particles with a diameter equal to or less than 10 μm in the atmosphere (assumed $48.6x10^3$ ng/m^3 [29]); IH is the inhalation rates (0.83 and 1.25 m^3/hr for adults and toddlers [30]). The potential health risk was calculated for target PFRs, for which reference doses (RfDs (ug/kg/day)) were available.

The hazard quotient (HQ) was generally used for investigating the potential health risk characterization. The formula of hazard quotient is:

$$HQ = ADI/RfD \quad (3)$$

III. Results and Discussion

A. Concentrations and Distribution of PFRs

The concentrations of PFRs were summarized in Table I. TCEP and TCP were not detected in all car dust samples. The concentration of Σ_7PFRs in AC filter dust samples ranged from 3,818 to 90,557 ng/g, with mean concentrations of 42,683 ng/g while the concentration in settled dust samples ranged from 11,375 to 15,383 ng/g, with mean concentrations of 13,159 ng/g. TBEP was the highest concentration detected in both types of car dust at ~39,000 ng/g for AC filter dust and ~10,000 ng/g for settled dust. Possibly, this could be attributed to the commonly use of TBEP as a plasticizer in plastics and also as a chemical stabilizer in artificial rubber and polisher in floors. TDCPP was not detected in AC filter dust while it was found in all settled dust samples with a mean concentration of ~1,500 ng/g. When comparing the concentrations of PFRs from this research with other studies focused on car dust, it was found that the maximum levels of the total PFRs in this study (90,557 ng/g) were lower than those reported in car dust from Saudi Arabia, Kuwait, German, and Greek (109,000; 185,000; 870,000; 89,962 ng/g) [10], [17], [31], [32] while they were higher than those reported in car dust from Egypt and Pakistan (3,367 and 5,600 ng/g) [10], [32]. According to the previous researches TCPP and TDCPP were the main PFR contributors in car dust [10], [33] but not in dust samples from this research. From Fig. 1 and Fig. 2the most abundant PFRs in both AC filter car dust (n=14) and in settled car dust (n=10) were TBEP (80%, 75%), followed by TEHP (9%, 5%) and TCPP (7%, 4%), respectively. TBEP is added mostly in PVC coverings and floor polishes and waxes and it was found in higher concentration levels in various indoor environments [10]. From another study of PFRs in residential dust and road dust samples in Thailand, TBEP was the most prominent PFRs [34] which was similar to this study. TEHP and TCPP are widely used in polyurethane foam which is a component of the upholstery in the car.

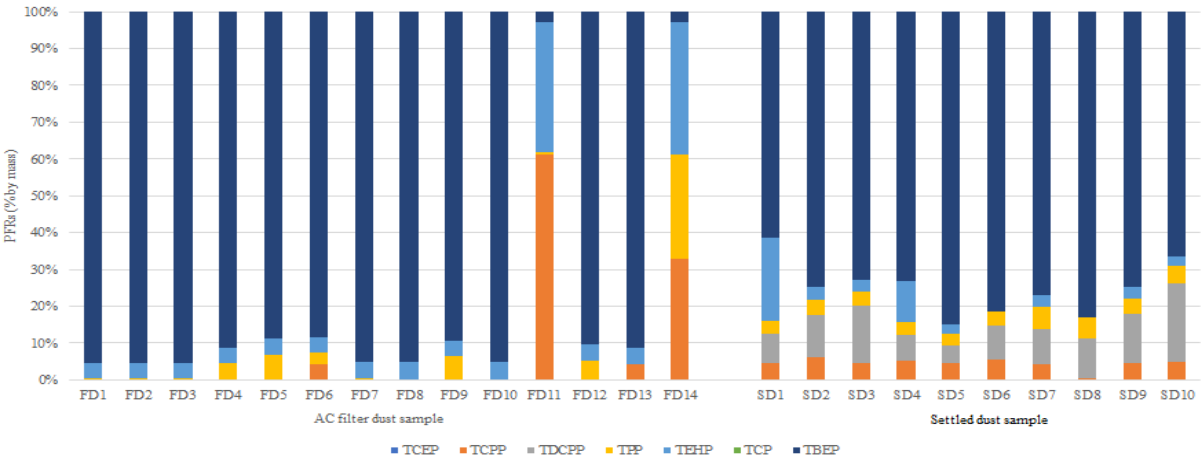

Fig. 1. Target PFR profile per car dust sample.

TABLE I: Mean, SD, and Range Concentrations (ng/g) of PFRs in Car Dust Samples

Comp.	LOQ	Mean[1]	SD[1]	Range	DF(%)[2]
AC filter dust					
TCEP	28		<LOQ		0
TCPP	105	519	817	<LOQ-2,484	29
TDCPP	65		<LOQ		0
TPP	27	1,127	1,756	<LOQ-5,309	43
TEHP	13	2,086	1,244	1,178-4,088	100
TCP	14		<LOQ		0
TBEP	225	38,952	29,060	<LOQ-82,080	86
Σ_7PFRs		42,683	30,682	3,818-90,557	
Settled dust					
TCEP	28		<LOQ		0
TCPP	105	578	183	<LOQ-723	90
TDCPP	65	1,481	703	670-3,299	100
TPP	27	560	153	410-866	100
TEHP	13	718	936	<LOQ-3,303	80
TCP	14		<LOQ		0
TBEP	225	9,822	942	8,689-11,544	100
Σ_7PFRs		13,159	1,235	11,375-15,383	

[1]Mean and SD: if the value is less than LOQ, LOQ/2 was used in calculation;
[2]DF (%): Detection frequency

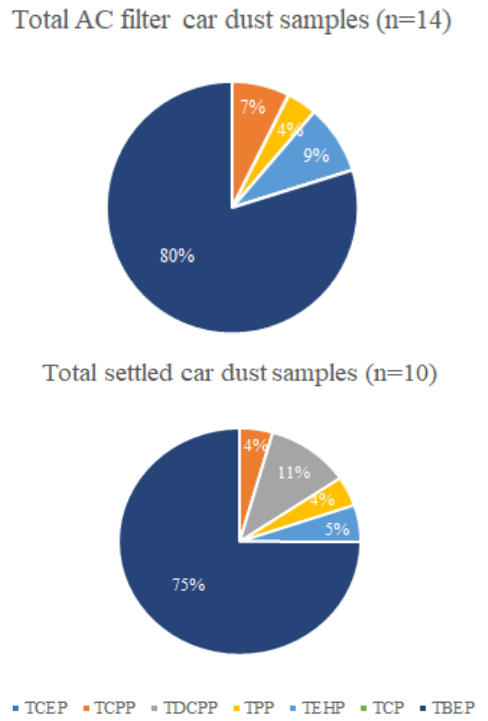

Fig. 2. Distribution of target PFRs in dust samples.

Moreover, the distribution profiles of both car dust groups were similar except for TDCPP (11%) which was detected in only settled dust. In other studies, the dominant Σ_7 PFRs in samples from Europe (n=13) and Asia (n=9) were TCPP (46%, 37%) and TDCPP (40%, 41%) while those from America was TCEP (51%) and TDCPP (14%) [10]. This data showed that there was a significant difference in the pattern of PFR distribution concerning in our samples compared to European, Asian, and America. In Europe, TCPP and TDCPP added in the upholstery foam of seats were considered as the important replacement PFRs after phase-out of PBDE [3], [17]. TCPP was more preferable for various applications than TDCPP because it was lower cost and has a similar structure and properties as TDCPP which was often used in higher safety standard applications [17]. Moreover, a small portion of TPP was detected in both AC filter and settled car dust samples about 4%, unlike floor dust from the e-waste dismantling facility that TPP was the major PFR contributors in Thailand [34].

B. Human Exposure via Dust Ingestion and Inhalation

The values of $ADI_{ingestion}$ and $ADI_{inhalation}$ for toddlers were higher than those for adults as shown in Table II and Table III. To estimate the normal-case and worst-case ADI values the average and the maximum PFR concentration were used, respectively. The order of PFRs concerning the ingestion of AC filter dust and settled dust, according to worst-case $ADI_{ingestion}$ values for toddler was TBEP > TPP > TEHP > TCPP and TBEP > TEHP > TDCPP > TPP > TCPP, respectively.

TABLE II: INGESTION EXPOSURE ASSESSMENT TO PFRS IN CAR DUST IN TODDLERS AND ADULTS

Target	RfD	ADI (ng/kg/day) via ingestion							
		AC filter dust				Settled dust			
		Normal-case		Worst-case		Normal-case		Worst-case	
		adult	toddler	adult	toddler	adult	toddler	adult	toddler
TCEP	22,000		NA				NA		
TCPP	80,000	1.69E-02	1.06E+01	8.08E-02	5.09E+01	1.88E-02	1.18E+01	2.35E-02	1.48E+01
TDCPP	15,000		NA			4.82E-02	3.03E+01	1.07E-01	6.76E+01
TPP	70,000	3.66E-02	2.31E+01	1.73E-01	1.09E+02	1.82E-02	1.15E+01	2.82E-02	1.77E+01
TEHP	35,000	6.78E-02	4.27E+01	1.33E-01	8.37E+01	2.33E-02	1.47E+01	1.07E-01	6.77E+01
TCP	13,000		NA				NA		
TBEP	15,000	1.27E+00	7.98E+02	2.67E+00	1.68E+03	3.19E-01	2.01E+02	3.75E-01	2.36E+02

TABLE III: INHALATION EXPOSURE ASSESSMENT TO PFRS IN CAR DUST IN TODDLERS AND ADULTS

Target	RfD	ADI (ng/kg/day) via inhalation							
		AC filter dust				Settled dust			
		Normal-case		Worst-case		Normal-case		Worst-case	
		adult	toddler	adult	toddler	adult	toddler	adult	toddler
TCEP	22,000		NA				NA		
TCPP	80,000	3.27E-04	2.58E-01	1.56E-03	1.24E+00	3.64E-04	2.88E-01	4.55E-04	3.60E-01
TDCPP	15,000		NA			9.32E-04	7.37E-01	2.08E-03	1.64E+00
TPP	70,000	7.10E-04	5.61E-01	3.34E-03	2.64E+00	3.53E-04	2.79E-01	5.45E-04	4.31E-01
TEHP	35,000	1.31E-03	1.04E+00	2.57E-03	2.04E+00	4.52E-04	3.57E-01	2.08E-03	1.64E+00
TCP	13,000		NA				NA		
TBEP	15,000	2.45E-02	1.94E+01	5.17E-02	4.09E+01	6.18E-03	4.89E+00	7.27E-03	5.75E+00

For the ingestion of settled car dust, the dominant PFRs were slightly different from that of AC filter dust, but still, the major compound (TBEP) was the same. The estimated $ADI_{ingestion}$ for an adult of TBEP in the AC filter dust and the settled dust were in the range of 1.27-2.67 and 0.319-0.375 ng/kg/day, respectively. The $ADI_{ingestion}$ for toddler of TBEP in the AC filter dust and the settled dust were in the range of 798-1,680 and 201-236 ng/kg/day, respectively. For human exposure via inhalation the order of PFRs concerning of AC filter dust and settled dust, according to worst-case $ADI_{inhalation}$ values for toddler was TBEP > TPP > TEHP > TCPP and TBEP > TEHP > TDCPP > TPP > TCPP,

respectively. These orders were similar to that of $ADI_{ingestion}$. The estimated $ADI_{inhalation}$ for an adult of TBEP in the AC filter dust and the settled dust were in the range of $2.45x10^{-2}$ to $5.17x10^{-2}$ and $6.18x10^{-3}$ to $7.27x10^{-3}$ ng/kg/day, respectively. The $ADI_{inhalation}$ for toddler of TBEP in the AC filter dust and the settled dust were in the range of 194-409 and 4.89-5.75 ng/kg/day, respectively. Apparently, the estimated ADI values of PFRs via both ingestion and inhalation routes were significantly lower than their RfD values derived by Ali *et al*. [31], [35] as shown in Table 2 and Table III. However, the highest exposure among PFRs corresponds to TBEP for adults and toddlers. The risk through ingestion was higher than inhalation intake. Toddlers were more exposed to PFR contaminants in comparison to adults. HQ was calculated from ADI and RfD value in Table II and Table III. The results showed that all HQ values were less than one which means that no adverse health effects are expected as a result of this exposure.

IV. Conclusions

This study investigated the concentration of PFRs in car dust samples from 14 discarded AC car filters and 10 bag filters of vacuum cleaners from car washing services in Thailand. The highest average concentration of PFRs in AC filter dust was TBEP (38,952 ng/g), followed by TEHP (2,086 ng/g) and TPP (1,127 ng/g). The highest average concentration of PFRs in dust collected from bag filters was also TBEP (9,822 ng/g), followed by TDCPP (1,481 ng/g) and TEHP (718 ng/g). The profile of seven PFRs in dust samples showed that TBEP was the main contributor in both dust types, followed by TEHP and TCPP. In addition, the exposure assessment of PFRs in dust via dust ingestion and inhalation implied that toddlers proved to be more exposed to PFR contaminants in comparison to adults. Especially, in the worse-case, the TBEP exposure by dust ingestion and inhalation pathway in toddlers were 1,680 ng/kg/day and 40.9 ng/kg/day, not exceed the RfD value (15,000 ng/kg/day). The intake of PFRs was higher via dust ingestion than via inhalation. In conclusion, the intakes of PFRs via both ingestion and inhalation were several orders of magnitude lower than RfD values. All HQ values calculated from ADI and RfD values were less than one which means that no adverse health effects are expected as a result of this exposure.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

K. Premrudee and N. Benjawan carried out the car dust studies, participated in the sample collection, sample preparation, and data analysis. S. Supachai participated in the quantitative analysis by GCMS technic. K. Premrudee helped to draft the manuscript and all authors read and approved the final version.

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Premrudee Kanchanapiya is a Ph.D. graduated from graduate School of Natural Science & Technology, Division of Global Environmental SCI. and Engineering, Kanazawa University, Japan in 2005. She has worked as a researcher at National Metal and Materials Technology Center, Thailand. Her main research interests are in waste management and utilization, emerging pollutant monitoring/treatment and life cycle assessment.

Benjawan Nilyok is a graduate student in Advanced and Sustainable Environmental Engineering Program (EnvE). She received a scholarship from TAIST-Tokyo Tech. She is a graduate student at Sirindhorn International Institute of Technology (SIIT), Thammasat University. Her research interests include emerging pollutant monitoring and health risk assessment.

Supachai Songngam graduated from master of science in petrochemical and polymer science, Chulalongkorn University. He has worked as laboratory officer at National Metal and Materials Technology Center, Thailand. His has expertise in GC-MS and LC-MS analysis techniques.

Sun Olapiriyakul is a assistant professor of Industrial Engineering Program, School of Manufacturing Systems and Mechanical Engineering (MSME) at Sirindhorn International Institute of Technology (SIIT), Thammasat University. His fields of specialization are sustainability, supply chain management, life-cycle engineering and applied operations research.