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Current situation and health risk assessment of neonicotinoids insecticides in urban indoor dust from Ha Noi, Viet Nam

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Abstract. In the several decades, although neonicotinoids insecticides (typically imidacloprid and thiamethoxam) are widely used and account for 24 % of the total global pesticide production, they still have negative impacts on natural enemies such as kinds of bees as well as adversely affect human health. However, there are only a few studies evaluating the levels of imidacloprid and thiamethoxam in airborne environments including indoor dust. Therefore, the aim of this study was to investigate the distribution, concentration and health risk to human of imidacloprid and thiamethoxam in indoor dust samples collected from 6 inner districts of Hanoi. Imidacloprid was found in indoor dust samples at all samples with an average concentration of 0.079 µg/g (ranging from 0.028 to 0.216 µg/g, the detection frequency of 100 %). Meanwhile, the mean concentration of thiamethoxam was revealed lower than imidacloprid at 0.013 µg/g (ranging from 0.01 to 0.027 µg/g, the detection frequency of 60 %). In high-end exposure, based on the measured concentrations, daily intake doses (IDs) of imidacloprid and thiamethoxam were calculated to be 9.96×10^{-1} and 1.10×10^{-2} ng/kg-bw/day for adults, and 1.78 and 1.98×10^{-1} ng/kg-bw/day for children, respectively. The estimated values of hazard quotient (HQ) of imidacloprid and thiamethoxam were 1.75×10^{-6} and 9.20×10^{-7} ng/kg-bw/day for adults, and 3.13×10^{-5} and 1.65×10^{-5} ng/kg-bw/day for children, respectively. All HQ and HI values of the insecticides were less than 1 for both of adults and children, indicating the potential adverse effects to human health are negligible.

Keywords: insecticide, imidacloprid, thiamethoxam, indoor dust, risk assessment.

Classification numbers: 3.4.5, 3.6.2.

1. INTRODUCTION

Viet Nam is an agricultural country with 40 % of total agricultural land [1]. Pesticides have been used to control pests, protect and ensure crop yields. The pesticides include insecticides, fungicides and herbicides. According to a report of the Ministry of Agriculture and Rural Development on the situation and solutions of pesticides management, approximately 120,000 tons of pesticides including 83.2 % of insecticides was used in 2017 including nearly 0.3 % was used for 188,000 hectares of agricultural crops, floriculture and other purposes in Hanoi [2 - 4]. Most of the pesticides are extremely toxic even outdated. While EU and the US have initially restricted or even banned the application of exceedingly toxic pesticides [5]. Typical example, imidacloprid and thiamethoxam belong to the neonicotinoid group, which are used as insecticides to slay harmful insects such as aphids, planthoppers, worms, beetle [6, 7]. They are used globally and so common in pesticides that they have resulted in a rapid expansion in production from 11 - 15 % in 2005 to 25 % of total production of pesticides worldwide in 2019 [8]. Imidacloprid and thiamethoxam are known to be environmentally persistent organic compounds, their significantly characteristics are low volatility, long retention time and almost non-hydrolysis under normal conditions [9, 10]. Therefore, they exist for a long time in the environment, especially the airborne environment due to their attributes [11]. As a result, these compounds could be appeared into fruits, plants, and scattered to airborne dust.

The government and scientists have concerned about the toxicity of imidacloprid and thiamethoxam to a natural enemies, human health and the environment [12]. Actually, these compounds have been detected in a variety of environments, including outdoor air, outdoor dust, indoor air and indoor dust, surface water, groundwater, seawater and drinking water, soil and sediment [8, 13 - 17]. They are also present in food, fruits and vegetables, and affecting insects (typically bees), aquatic animals [18 - 23]. Imidacloprid and thiamethoxam have frequently been detected in environments, indicating that humans could be exposed even to intake of insecticides from various possible routes [24].

Due to their chronic toxicity and long-term exposure to these compounds, the higher incidence of cancer and potential disruption of the endocrine as well as the central nervous system are reported in previous studies [25 - 27]. This is reported by the fact that imidacloprid and thiamethoxam have been banned in all EU members due to their negative impact on the generation and development of bee species [5]. There are few studies on the risks of ingesting insecticides in indoor dust in Viet Nam even though we have already known about that poisoning was mainly through the ingestion of settled indoor dust accounting for 97.3 %, skin and inhalation only accounted for 1.9 and 1.8 % [28]. The widespread use of pesticides has become an alarming problem for human health. Therefore, providing more data on the toxicity and chronic exposure of individual pesticide have become the essential request in order to diagnose intimidation to human health [29].

The introduction should follow the key words and should be as brief as possible; it should concern itself with a clear justification for the work undertaken and the underlying theory and hypothesis; a short review of literature in the field of study is obligatory although any exhaustive review properly belongs to the discussion section. Citation of references is realized via using their numbers in the reference list, *e.g.* [1] or [1-3]. First citations of references have to follow the sequence of natural numbers. In particular, one paragraph must not contain only one sentence.

2. MATERIAL AND METHODS

2.1. Chemicals and materials

Imidacloprid and thiamethoxam standard solutions were purchased from Restek Japan and Kanto Chemical (Tokyo, Japan). The deuterium labelled standards that were used as internal standards (IS) and surrogates were purchased from Kanto Chemical and Hayashi Pure Chemical (Osaka, Japan). The stock standards were diluted with methanol to prepare the working mixed standard solutions and then stored at - 20 °C in a freezer. Methanol (LC-MS-grade) and ammonium acetate (CAS No. 631-61-8, 1 mol/L, HPLC-grade) was obtained from Kanto Chemical and Fujifilm Wako Pure Chemical Corporation (Osaka, Japan), respectively. Tap water was purified (LC-MS-grade) using an Elga Purelab Chorus 1 (Analytical Research; Veolia Water, Tokyo, Japan). Millex-LG syringe filters (0.2 μm pore size, Ø4 mm) were obtained from Merck Millipore (Darmstadt, Germany). All solvents used in this study including hexane (Hex), acetone (Ace), and ethyl acetate (EtAc) were purchased from Kanto Chemical Co. (Tokyo, Japan) and they were of analytical grade.

Glassware was firstly cleaned and washed with hot chromic acid (including 5 % potassium dichromate in sulfuric acid solution). After carefully rinsing with deionized water and acetone, the glassware was dried at 250 ºC for 5 hours prior to usage.

2.2. Sample collection

Indoor dust samples (n = 10) were collected from 6 districts of Hanoi including Long Bien district (N1, N2, N3), Hoan Kiem district (N4, N5, N6), Ba Dinh district (N7), Cau Giay district (N8), Tu Liem district (N9) and Thanh Xuan district (N10). Dust samples were collected in June 2018 according to a previous protocol of Wang [30]. The indoor dust samples were taken in the living room from the surface of furniture; under the table, sofa; on top of the cabinet, refrigerator, air conditioner, and fan blades by the vacuum cleaner which was fitted with nylon socks inside its nozzle. This fraction of dust was formed mainly by the accumulation of fine particles deposited from indoor air over time and can be representative of the dust accidentally ingested by humans [30]. Individual samples were wrapped in aluminum foil and sealed in polyethylene zip bags for preserving from humidity, light, and other external factors that might change its composition. After being transported to the laboratory, all the indoor dust samples were sieved through a 250 μm sieve and were stored at - 20 °C until analysis.

2.3. Sample extraction and analysis

The extraction procedure was carried out according to the method described by Doan *et al*. [31]. In brief, 0.5 g dust and spiked with a mixture of surrogate solution (4 μg/mL, 50 μL) prior to sonication-extraction for 15 min with 20 mL methanol. The extract was then centrifuged at 2000 rpm for 20 min, and the supernatant was transferred to a 50 mL pear-shaped evaporation flask. This extraction procedure was repeated twice (15 mL methanol each). The extracts were combined and concentrated by rotary evaporation to approximately 2 - 3 mL and then transferred to a vial for further evaporation to 0.4 mL using a gentle nitrogen stream. Mixed internal standard solution (4 μg/mL, 50 μL) were then added, and the mixture was reconstituted to 500 μL with methanol. The final extract was filtered with a syringe filter (Millex-LG, 4 mm, Merck Millipore, Darmstadt, Germany) prior to LC-QTOF-MS-SWATH analysis.

The identification and quantitation method for imidacloprid and thiamethoxam using LC-QTOF-MS-SWATH were described in detail by Kadokami and Ueno [32]. Instruments and conditions of LC-QTOF-MS-SWATH are listed in Table 1.

Table 1. LC-QTOF-MS-SWATH conditions.

Name	Parameter
Instrument	Sciex X500R QTOF system
Column	GL Science ODS-4 HP (150 mm, 2.1 mm, 3 μm)
Column temp.	40 °C
Flow rate	0.3 mL/min
Mobile phase	(A) 5 mM CH_3COONH_4 in H_2O (B) 5 mM CH_3COONH_4 in CH_3OH
Gradient profile	time, min 0 30 - 40 40.01 - 50 (B) % 5 95 5
Injection volume	2 μL
Ion source	TurbolonSpray
Ionization	ESI-positive
Measurement mode	Swath
TOF-MS (scan range)	50 - 1000 Da, 0.1 s
TOF MS/MS	50 - 1000 Da, 22 ranges, 0.07 s each
Collision energy ramp	20 - 50 V
Mass resolution	30 000
Total cycle time	1.768 s

The method detection level (MDL) and the level of quantitation (LOQ) of each target were determined according to the relationship between the instrument detection level (IDL), the MDL and the LOQ (IDL:MDL:LOQ = 1:4:10) [33]. Both IDLs of imidacloprid and thiamethoxam were 1, the MDL and LOQ for two insecticides were 0.004 and 0.01 μg/g, respectively.

Quality assurance and quality control (QA/QC): In order to remove contaminations before use, all glassware was cleaned with acetone twice. Quality assurance and quality control were performed by analyzing the method blanks samples (n = 3), spiked samples (n = 3). The extraction procedure and the analysis were applied by the same method as used for indoor dust samples. As a result, imidacloprid and thiamethoxam were not detected in all blank samples. The average recovery rates of imidacloprid and thiamethoxam were 92 and 94 %, respectively. This analytical method is effective for the analysis of imidacloprid and thiamethoxam in indoor dust samples.

Statistical analysis: The statistical analyses were performed using Microsoft Excel Professional Plus 2013. During this analysis, imidacloprid and thiamethoxam concentrations below the MDL, LOQ were assigned a value of zero for data analysis.

2.4. Health risk assessment

It is necessary to contribute additional data on toxicity and chronic exposure capability of the individual chemicals to make the diagnosis of risks to human health [29]. As a result, in order to assess the exposure of imidacloprid and thiamethoxam in indoor dust to human health including adults and children in Ha Noi, Viet Nam; the daily Intake Doses (ID) (ng/kg-bw/day) of imidacloprid and thiamethoxam due to dust ingestion are estimated according to the following equation [29, 34].

$$IDi = (Ci \times IR \times FT \times AF) / BW \quad (1)$$

where, IDi is the daily intake dose of chemical i (ng/kg-bw/day); Ci is the measured concentration (ng/g); IR is the daily dust ingestion rate; FT is fractions of time spent indoor;

AF is the absorption fraction, BW is the average body weight (kg); FT is fractions of time spent indoor; AF is the absorption fraction, BW is the average body weight (kg).

The Hazard Quotient (HQ) is a measurement of the potentially adverse effect of an individual pesticide to human health. The Hazard Quotient (HQ) and the non-cancer hazard index (HI) were performed using the following equation [29, 34]:

$$HQi = IDi / RfDi \quad (2)$$

$$HI = \Sigma HI = \Sigma (IDi / RfDi) \quad (3)$$

where RfDi is the corresponding reference dose of chemical i (ng/kg-bw/day). If the values of HQ, and HI were ≤ 1 which reported considered acceptable risk levels, whereas HQ, and HI values >1 indicating daily accumulation will potentially cause adverse health effects [35].

3. RESULTS AND DISCUSSION

3.1. Distribution and concentration of imidacloprid and thiamethoxam in indoor dust

The concentration of imidacloprid and thiamethoxam in dust samples at different sampling sites was shown in Figure 1. At sampling sites insecticide contamination in indoor dust was gradually revealed. The similarity between the study sites were that at the sampling sites with high concentrations of imidacloprid (N2, N8, and N10), the concentration of measured thiamethoxam was also at the top. In particular, N10 was the location where the highest concentration of imidacloprid was recorded and also reported the highest thiamethoxam content. In contrast, thiamethoxam was hardly detected at the study sites including N5, N6, N7. Coincidentally, the concentrations of imidacloprid measured at those points were also the lowest. Imidacloprid was discovered in all indoor dust samples with an average concentration of 0.079 µg/g (ranging from 0.028 to 0.216 µg/g). Meanwhile, the average concentration of thiamethoxam was revealed lower than imidacloprid at 0.013 µg/g (ranging from 0.01 to 0.027 µg/g). In addition, the concentration of imidacloprid in indoor dust samples were much higher than that of thiamethoxam.

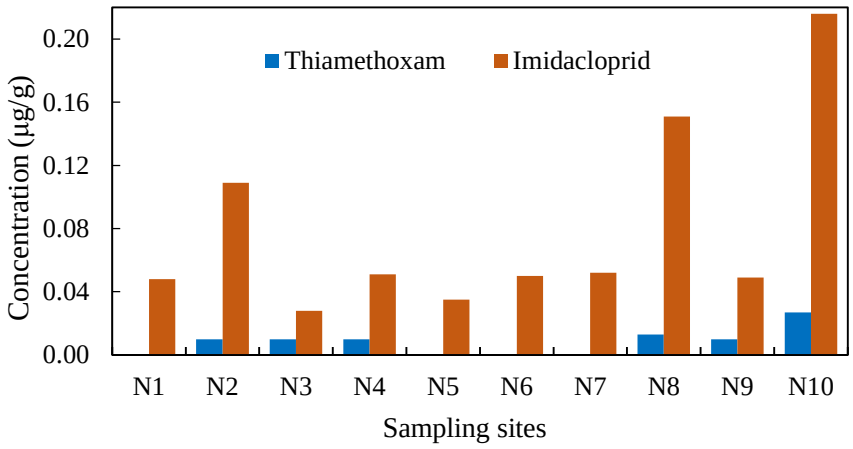

Figure 1. Concentration and distribution of imidacloprid and thiamethoxam in indoor dust in Ha Noi.

In general, imidacloprid was found in all indoor dust samples accounted for a high proportion of the total concentration (85.5 %). Meanwhile, thiamethoxam was found in only

60 % of the samples with a low percentage of the total concentration (14.5 %). Occurrence and concentrations of imidacloprid and thiamethoxam in indoor dust varied considerably among the surveyed districts. In which, Thanh Xuan and Cau Giay districts with high concentrations of imidacloprid and thiamethoxam ranked first in the total number of districts. The cause may be the use of insecticides at flower markets (Nga Tu So market - Thanh Xuan district and Nghia Do park - Cau Giay district) could increase the dispersion of imidacloprid and thiamethoxam into the air. Especially, synthetic pesticides could be sprayed at night to control pests in the fields in suburban districts of Ha Noi [36]. However, there should be research for a long time about the effects of environmental conditions for persistence of imidacloprid and thiamethoxam in indoor dust in Ha Noi.

3.2. Comparison of imidacloprid and thiamethoxam status in indoor dust

The comparison of imidacloprid and thiamethoxam existence between Viet Nam and various countries would be difficult due to the lack of research on these insecticides in Viet Nam as well as worldwide scale. Therefore, the comparison of this study aims to provide additional data to assist managers in making decisions regarding the future use of neonicotinoid pesticides in agricultural environments. The average imidacloprid concentration of 0.079 µg/g in this study was much lower than those reported in previous studies conducted in Italy (mean 1.584 µg/g in indoor dust from Italy), and significantly higher compared to China where frequently detected in indoor environments and 0.00146, 0.00526 and 0.00724 µg/g in Taiyuan (2016), Wuhan (2018), and Shenzhen, China (2019), respectively [37, 38]. Meanwhile, the lowest concentration of thiamethoxam measured in 3 cities in China was less than the method limits of detection and the highest concentration was 0.0921 µg/g detected in Wuhan in 2018 [37]; much higher than the highest measured in this study of 0.027 µg/g. From the comparison, it could be reported that imidacloprid and thiamethoxam contamination may be negligible in Hanoi.

3.3. Risk assessment to human health

The estimation of ID and HQ of imidacloprid and thiamethoxam for adults and children living in Hanoi used the concentration of these compounds. The various exposure scenarios were calculated using median and 95^{th} percentile (high-end exposure) concentrations for both mean dust ingestion rates (20 mg/day for adults and 50 mg/day for children) and high dust ingestion rates (50 mg/day for adults and 200 mg/day for children) with assuming 100 % absorption of contaminants from ingested dust [39]. The percentage of time spent indoors was assumed to be 86 and 64 % for children and adults, respectively [40]. In this study, the chronic dietary (cRfD) provided by USEPA were used as the reference dose when indoor dust was ingested [41, 42]. The average weight of adults and children in this calculation was assumed to be 60 kg and 18 kg, respectively [44]. The daily exposures to imidacloprid and thiamethoxam from indoor dust ingestion for adults and children were presented in Table 2. The estimated ID values of imidacloprid and thiamethoxam ranged 1.08×10^{-2} - 9.96×10^{-2} ng/kg-bw/day and 2.13×10^{-3} - 1.10×10^{-2} ng/kg-bw/day for adults; meanwhile, the IDs fluctuated in the ranges of 1.21×10^{-1} - 3.19×10^{-2} ng/kg-bw/day and 2.39×10^{-2} - 1.98×10^{-1} ng/kg-bw/day for children, respectively.

The higher ID values of imidacloprid for both children and adults compared to thiamethoxam may be a consequence of the uncontrolled use of the imidacloprid-based insecticide than the thiamethoxam-based insecticide. This hypothesis is supported by the presence of imidacloprid, which was found more frequently and in significantly higher concentrations than that of thiamethoxam. The value of ID of two insecticides in children were

much exceeded due to their higher air ingestion rate for physical development and lower body weight combined with higher metabolic rate compared to adults indicating the potential harm of insecticides is much higher for children than for adults [24]. However, for both adults and children, the ID values even in the worst case with a high dust exposure dosage (50 and 200 mg/day) were determined much lower than their cRfD.

Table 2. Daily intake doses (ID) of imidacloprid and thiamethoxam (ng/kg-bw/day) via ingestion exposure and Hazard Quotients (HQ) for detected imidacloprid and thiamethoxam in chronic health risk assessment.

Insecticides	**cRfD**[a]	**Adults**				
		Mean exposure[b]			High-end exposure[c]	
		ID	HQ		ID	HQ
Imidacloprid	0.057	1.08×10^{-2}	1.89×10^{-7}		9.96×10^{-2}	1.75×10^{-6}
Thiamethoxam	0.012	2.13×10^{-3}	1.78×10^{-7}		1.10×10^{-2}	9.20×10^{-7}
HI			3.67×10^{-7}			2.67×10^{-6}
Insecticides	**cRfD**[a]	**Children**				
		Mean exposure[b]			High-end exposure[c]	
		ID	HQ		ID	HQ
Imidacloprid	0.057	1.21×10^{-1}	2.12×10^{-6}		1.78	3.13×10^{-5}
Thiamethoxam	0.012	2.39×10^{-2}	1.99×10^{-6}		1.98×10^{-1}	1.65×10^{-5}
HI			4.11×10^{-6}			4.78×10^{-5}

[a] The chronic dietary reference doses (cRfD) (ng/kg-bw/day) are provided by USHHS.

[b] Mean concentrations in indoor dust were used for calculating exposure.

[c] 95th percentile (P95) indoor dust concentrations used for calculating exposure.

All estimated HQ and HI values of the imidacloprid and thiamethoxam insecticides were less than 1 for both of adults and children indicating the potential adverse effects to human health through direct dust ingestion of imidacloprid and thiamethoxam existing in indoor dust in Hanoi are negligible. However, due to their chronic toxicity and long-term exposure (initiatively and passively) to these compounds [43], this study recommends a reduction in use of insecticides within the inner area of Ha Noi possibly.

This is the initial study to investigate the presence of imidacloprid and thiamethoxam insecticides in indoor dust in Hanoi. Since the number of samples was limited therefore the exposure of imidacloprid and thiamethoxam insecticides in indoor dust to public health was not yet fully clarified. Further in-depth studies with a larger number of indoor dust samples are needed for more accurate evaluation.

4. CONCLUSIONS

This is an initial study to investigate the distribution and concentration of imidacloprid and thiamethoxam in indoor dust from 6 districts of Ha Noi. The results indicated that the concentration of imidacloprid was found in all indoor dust samples with a mean concentration of

0.079 µg/g (range 0.028 - 0.216 µg/g) while thiamethoxam only appeared in 6 out of 10 samples with a average concentration of 0.013 µg/g (range 0.010 - 0.27 µg/g). The existence of imidacloprid and thiamethoxam in indoor dust reported the use of neonicotinoid pesticides in the surveyed areas or the vicinity was relatively high. Imidacloprid and thiamethoxam in indoor dust could be derived from their widespread use in flower protection (public parks, flower markets) and pest control (agriculture). All values of HQ were less than 1, and the risk to human health was considered negligibly. However, more comprehensive studies should be performed to get an overview of the presence and exposure of imidacloprid and thiamethoxam to human health in Viet Nam.

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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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