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Original Article

Distribution of Bisphenol A Diglycidyl Ether (BADGE) and Its Derivatives in Indoor Dust from Various Micro-environments in Hanoi, Vietnam

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Abstract: In this report, the method for the determination of BADGE and its derivatives in dust samples has been optimized. The recoveries of the surrogate standard (*d6*-BADGE) and target compounds (in matrix spiked) were in the ranges of 78.5–99.0% and 75.4–116% (SD < 10.0), respectively. Total concentrations of BADGEs in indoor dust ranged from no detection (ND) to 6640 ng/g (mean: 950 ng/g and median: 434 ng/g). The mean (median) concentrations of four BADGEs in indoor dust samples collected from public places, homes, laboratories, and offices were 1630 (830), 957 (434), 707 (375), and 344 (170) ng/g, respectively. Among BADGEs, BADGE.2H_2O was found at the highest levels with the ranges of ND–4880 ng/g (mean: 634, and median: 247) in indoor dust from all micro-environments. A significant correlation existed between the sum concentrations of two pairs: BADGE + BADGE.H_2O and BADGE.2H_2O + BADGE.HCl.H_2O. Based on the measured concentration of BADGEs in indoor dust, the estimated exposure doses to BADGEs through dust ingestion were respectively in ranges of 0.406-1.92 and 0.351–1.66 ng/kg-bw/d for women and men.

Keywords: Bisphenol; BADGEs, Indoor dust; Human exposure.

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1. Introduction

Bisphenol A diglycidyl ether (BADGE) is a major component of epoxy resins used in various commercial products since the 1940s [1, 2]. The annual global BADGE production was approximately 957,000 metric tons in 2003 [3]. BADGE is a reactive molecule and following contact with aqueous and acidic media, chlorinated and hydrated derivatives such as bisphenol A (2,3-dihydroxypropyl) glycidyl ether (BADGE.H_2O), bisphenol A (2,3-dihydroxypropyl) ether (BADGE.$2H_2O$), and bisphenol A (3-chloro-2-hydroxypropyl) (2,3-dihydroxypropyl) ether (BADGE.HCl.H_2O) can be formed. Zhang et al., (2022) showed that derivatives of BADGEs such as BADGE.H_2O, BADGE.$2H_2O$, and BADGE.HCl.H_2O was formed in food matrices during storage [4]. Overall, BADGE and its derivatives have been found in various materials such as food packages, coating in beverage cans, and household products as emerging plasticizers [5, 6].

Due to its widespread use in many different commercial products, BADGE and its derivatives are distributed in most environments such as dust [7, 8], sewage sludge [9], and wastewater [10]. They have even been found in foodstuffs [4, 6, 11]. Several studies have found the occurrence of BADEs and derivatives in biological samples such as urine [8, 12, 13] and blood [14, 15]. Furthermore, Bisphenol A bis (2,3-dihydroxypropyl) ether [BADGE·2H2O], a derivative of BADGEs, was found with high frequency in adipose (60%) and plasma (70%) samples [16]. Recently, BADGE·$2H_2O$ was measured in all serum samples of infants collected from a history of NICU (neonatal intensive care unit) hospitalization in Japan [17]. However, few studies deal with the occurrence of BADGEs in the indoor environment. Earlier research reported median concentrations of BADGEs in dust samples collected from Vietnam at the range of 23–1750 ng/g (median: 184 ng/g) [5]. Up to now, no further studies have reported the distribution of BADGEs in different micro-environments in Vietnam.

Various studies have shown that BADGEs formed toxic effects on laboratory animals such as estrogenicity [18], cytotoxicity [19], Caco-2 cells [20], and cell over-proliferation [21]. It has been reported that the genotoxic effect of BADGE was stronger than that of BADGE.H_2O, BADGE.HCl.H_2O and BADGE.$2H_2O$ [22]. Furthermore, Marqueno et al., (2019) presented evidence that BADGE and its derivatives have the potential to influence placental lipid handling and modulate placental CYP19 activity [23]. Several organizations such as the European Food Safety Authority (EFSA, 2004) and the U.S. Food and Drug Administration (FDA. 2005) recommended toxicity of BADGEs and derivatives and included them on the list of substances banned/restricted for use in the manufacturing process [3]. However, regulations on allowable concentrations of BADGEs in the environment are still very scarce. Therefore, research on the fate of these chemicals is necessary to help consolidate scientific evidence to plan appropriate policies for environmental protection.

In this present report, BAGDE and its three derivatives were determined in 59 indoor dust samples collected from various micro-environments in Hanoi, Vietnam. Based on the concentrations of BADGEs measured in dust, human exposure to these compounds via dust ingestion was estimated for adults.

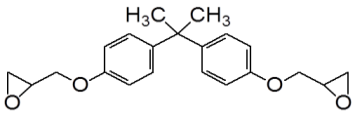
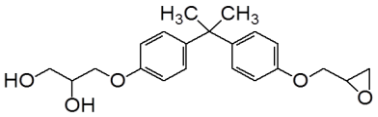
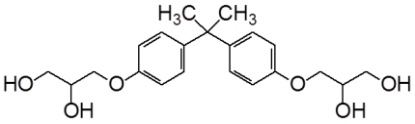
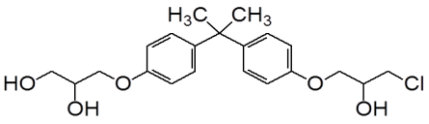
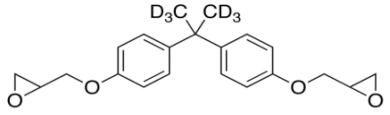
2. Materials and Methods

2.1. Chemicals

In this work, solvents [acetone, *n*-hexane, acetonitrile (ACN), methanol (MeOH), formic acid (analytical grade), and double-distilled deionized water (LC–MS grade)] and ammonium acetate (LC–MS grade) were supplied by Merck KGaA (Darmstadt, Germany). Bisphenol A diglycidyl ether (BADGE with a purity ≥95%), bisphenol A (2,3-dihydroxypropyl) glycidyl ether (BADGE.H_2O, ≥95%), bisphenol A (3-chloro-

2-hydroxypropyl) (2,3-dihydroxypropyl) ether (BADGE.HCl.H_2O, ≥95%), and bisphenol A (2,3-dihydroxypropyl) ether (BADGE.2H_2O, ≥97%) were purchased from Sigma-Aldrich (Table 1). *d*6-BADGE (99%) was purchased from Cambridge Isotope Laboratories (Andover, MA, USA) and used as a surrogate standard. The native and surrogate standard solutions were prepared in methanol. Working standard solutions at seven levels of 1, 2, 5, 20, 50, 100, and 200 ng/mL by diluting the mixed standard solutions.

Table 1. Molecular formula, structures, and molecular weight of BADGEs in this study

Chemicals	Abbreviation	Molecular Formula	Molecular Structure	Mol weight (g/mol)
Bisphenol A diglycidyl ether	BADGE	$C_{21}H_{24}O_4$		340.41
Bisphenol A (2,3-dihydroxypropyl) glycidyl ether	BADGE.H_2O	$C_{21}H_{26}O_5$		358.43
Bisphenol A (2,3-dihydroxypropyl) ether	BADGE.2H_2O	$C_{21}H_{28}O_6$		376.44
Bisphenol A (3-chloro-2-hydroxypropyl)(2,3-dihydroxypropyl) ether	BADGE.HCl.H_2O	$C_{21}H_{27}ClO_5$		394.89
Bisphenol A diglycidyl ether (deuterium)	*d*6-BADGE	$C_{21}H_{24}O_4$		346.45

2.2. Instrumental Analysis

BADGEs were measured by a liquid chromatography-tandem quadrupole mass spectrometry system (LC-MS/MS-8040, Shimadzu) equipped with a binary pump and an autosampler. Ten microliters of the sample extracts were injected into an analytical column (Betasil® C18, 100 X 2.1 mm column; Thermo Electron Corp., Waltham, MA, USA), which was connected to a Javelin guard column (Betasil® C18, 20 X 2.1 mm column; Thermo Electron Corp.). The mobile phase comprised methanol (A) and 10% methanol in Milli-Q water that contained 2 mM (0.15 g/L) ammonium acetate (B) at a flow rate of 300 µL/min. The proportion of methanol was linearly increased from 20% to 75% in 5 min, then increased to 95% in 3 min and held for 8 min, and then reverted to 20% methanol in 1 min and held for 5 min. The MS/MS was operated in multiple reaction monitoring (MRM) negative and positive ionization modes for parabens and BADGEs, respectively. The MRM transitions were set at 358 > 191 for BADGEs, 376 > 209 for BADGE.H_2O, 394 > 209 for BADGE.2H_2O, 412 > 227 for BADGE.HCl.H_2O, and 364 > 197 for d_6-BADGE. The details were described in previous studies [5, 10].

2.3. Sample Collection

In this work, 59 indoor dust samples were collected during February-August 2023 in Hanoi metropolitan areas. The samples were divided into 4 categories including homes (living rooms and bedrooms, n = 17), public places (supermarkets/shops, garages, and bus stations, n = 16), laboratories (n = 13), and offices (n = 13). Dust samples were from the floor and on the surface of household items by using a vacuum cleaner or sweeping with a broom. Dust samples were placed in glass tubes and then stored at 4 ºC until analysis.

2.4. Sample Preparation

The dust samples were treated by the procedure previously described elsewhere [5, 10] with some modifications. Three hundred milligrams of dust were accurately weighed and spiked with 50 ng *d*6-BADGE, as a surrogate standard. The spiked dust samples were equilibrated for 30 min at room temperature. The samples were extracted with 3 mL of methanol/water mixture (2:1, v/v) by shaking in an oscillator shaker (Eberbach Corp., Ann Arbor, MI, USA) for 30 min, and the mixture was centrifuged (Eppendorf Centrifuge 5804, Hamburg, Germany) at 4000 rpm for 5 min. The supernatant was transferred into a glass tube. The extraction was repeated twice. The combined extracts were concentrated to ~3 mL under a gentle nitrogen stream. The extract was diluted to 10 mL with 0.2% formic acid (pH 2.5). The extract was purified by passage through Oasis MCX® cartridges (60 mg/cm^3; Waters Corp., Milford, MA, USA), preconditioned with 5 mL of methanol and 5 mL of water. The diluted sample extract was loaded, followed by passage of 10 mL methanol/water (1:3, v/v) and 5 mL of water. After drying the cartridge with nitrogen, the target compounds were eluted with 7 mL of methanol. The extract was concentrated under a gentle stream of nitrogen to 1 mL. The final solution was transferred to a 1.5 mL vial for analysis by liquid chromatography-tandem mass spectrometry (LC/MS/MS).

2.5. Quality Assurance and Quality Control

Several efforts were done to minimize contamination of target substances in the analytical procedure. All glassware was rinsed thoroughly with acetone, toluene, and hexane to and baked at 400 ºC for 3 hours. Solvents were used directly from the container.

Table 2. Recoveries of BADGEs and *d6*-BADGE in the procedural blanks (%)

Experiment	BADGE	BADGE.H_2O	BADGE.$2H_2O$	BADGE.HCl.H_2O	*d6*-BADGE
1st	89.6	88.5	104	95.4	105
2nd	92.6	98.8	100	101	95.7
3rd	98.7	105	110	97.2	92.9
4th	98.4	87.9	95.6	98.0	98.3
5th	92.0	101	85.5	99.2	89.9
6th	85.5	91.9	88.2	97.7	89.0
7th	78.9	85.5	88.0	90.3	105
Mean	90.8	94.1	95.9	95.6	96.5
SD	*7.02*	*7.50*	*9.23*	*7.35*	*6.60*

For BADGEs, procedural blanks (n = 8) contained trace levels of BADGE.H_2O (0.7 ng/g) and BADGE.$2H_2O$ (1 ng/g). These values were subtracted from sample concentrations. Fifty nanograms of d_6-BADGE (for BADGEs) were spiked into procedural blanks and every sample. Recoveries of surrogate standards in procedural blanks and samples ranged from 89.0 to 105% and 78.5 to 99.0%, respectively. Recoveries of target

analytes (four BADGEs, spiked at 20 ng of each) in procedural blanks and spiked matrices were in ranges of 78.9–105% and 75.4–116%, respectively (Table 2). The method quantitation limit (MQL) was calculated based on the instrumental quantitation limit (as 10-times signal-to-noise ratio), Final volume of concentrated solution (1 mL), sample mass (300 mg), and recoveries. The MQL for BADGE and its derivatives were in ranges of 1.0-1.5 ng/g. Instrumental calibration was verified by the injection of 7-point calibration standards (at concentrations ranging from 1.0 to 200 ng/mL for BADGEs), and R^2 was ≥ 0.99.

2.6. Statistical Analysis and Risk Assessment

Minitab 18® Statistical Software (Minitab Inc.) was used for statistical analysis in this report. Pearson correlation analysis and principal component analysis (PCA) were conducted on the whole dataset to assess correlations and potential sources of BADGEs. The level of statistical significance was set at $p < 0.05$. A value of one-half of the method detection limits (MDLs) was used in statistical analysis as concentrations below the MDLs.

3. Results and Discussion

3.1. Concentration of BADGEs in Indoor Dust Samples

BADGE and its three derivatives were investigated in indoor dust samples from Hanoi, Vietnam. Total concentrations of ΣBADGEs in indoor dust from various micro-environments ranged from ND (no detection) to 6640 ng/g (mean: 950 and median: 434) (Table 3). Overall, the levels of BADGEs found in this study were higher (3 times) than the previous report in Vietnam in 2016 with a median value of 184 ng/g [5]. These results suggest a trend of increasing pollution levels of BADGEs in the environment in Vietnam. In comparison, the concentrations levels of ΣBADGEs in dust from Vietnam were 2-3 times lower than those from various locations such as the USA (1350 ng/g), China (1410 ng/g), South Korea (2380 ng/g), and Japan (2020 ng/g), respectively [15].

Table 3. Concentrations of BADGEs in indoor dust samples from Hanoi, Vietnam

Categories	Statistics	BADGE	BADGE.H_2O	BADGE.$2H_2O$	BADGE.HCl.H_2O	ΣBADGEs
Homes (n=17)	Range	ND–161	ND–242	ND–4880	ND–1355	ND–6640
	Mean	40.0	80.6	649	187	957
	Median	15.9	75.5	247	39.7	434
	DF (%)	*64.5*	*88.2*	*94.1*	*88.2*	-
Public places (n=16)	Range	ND–395	ND–562	ND–4700	ND–1350	ND–6580
	Mean	84.6	181	1130	231	1630
	Median	28.9	134	576	103	830
	DF (%)	*68.8*	*87.5*	*93.8*	*81.3*	-
Laboratories (n=13)	Range	ND–154	ND–321	ND–1320	ND–391	ND–2190
	Mean	47.8	106	430	124	708
	Median	26.4	77.8	246	37.6	375
	DF (%)	*76.9*	*84.6*	*92l3*	*84.6*	-
Offices (n=13)	Range	ND–76.8	ND–138	ND–624	ND–313	ND–1190
	Mean	21.7	47.6	202	74.8	345
	Median	13.4	34.7	124	47.8	171
	DF (%)	*53.8*	*69.2*	*84.6*	*69.2*	-

Overall (n=59)	Range	ND–395	ND–562	ND–4880	ND–1350	ND–6640
	Mean	50.3	106	634	161	950
	Median	22.9	65.9	246	39.7	434
	DF (%)	*66.1*	*83.1*	*91.5*	*83.1*	-
DF: Detection Frequency; ND: No Detection; n: (values in parentheses) refer to the number of samples						

The mean (median) concentrations of ΣBADGEs in dust samples collected from homes, public places, laboratories, and offices were 957 (434), 1630 (829), 708 (375), and 345 (171) ng/g, respectively. The difference between mean and median concentration values shows that the levels of BADGE pollution in indoor dust samples are very different. Among BADGEs, BADGE.2H$_2$O was the predominant compound found in dust with a detection rate of 91.5%, followed by BADGE.HCl.H$_2$O and BADGE (detection rate: 83.1%). Furthermore, the distribution profile of BADGEs in indoor dust from Vietnam was similar to that reported for the USA, China, South Korea, and Japan [15].

3.2. Distributions of BADGEs in Indoor Dust

In this study, the Pearson correlation was analyzed for pairs of BADGEs. Accordingly, the correlation coefficient (r) of pairs BADGE versus (vs) BADGE.H$_2$O, BADGE.H$_2$O vs BADGE.2H$_2$O, and BADGE.2H$_2$O vs BADGE.HCl.H$_2$O were 0.860, 0.739, and 0.891, respectively. Especially, there existed a significant correlation (r = 0.747, p < 0.005) between BADGE.2H$_2$O + BADGE.HCl.H$_2$O (sum concentrations) and BADGE + BADGE.H$_2$O were investigated (Figure 2). which suggests similarity in sources of release for these compounds.

Concentrations of BADGEs in dust samples were significantly different among the four micro-environments (p < 0.005). The highest concentration of the sum of the four BADGEs was found in indoor dust from public places with a range of ND–6580 ng/g (mean: 1630 ng/g) followed by homes (range: ND–6640, mean: 957 ng/g), laboratories (range: ND–2190, mean: 708 ng/g), and offices (range: ND–1190, mean: 345 ng/g). In general, the distribution level of BADGEs in micro-environments including homes, laboratories, and offices in Hanoi tends to increase compared to previous research [5].

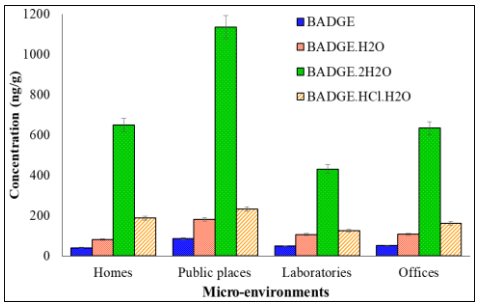


Figure 1. Distributions of BADGEs in indoor dust samples collected from various micro-environments.

Among the BADGEs, BADGE.2H$_2$O was measured at the highest concentrations, ranging from no detection (ND) to 4880 ng/g (mean: 634 and median: 247 ng/g), followed by BADGE.H$_2$O (106 and 65.9 ng/g), and BADGE.HCl.H$_2$O (161 and 39.7 ng/g). In contrast, the concentrations of BADGE were 12 times lower than the concentration found for BADGE.2H$_2$O. The pattern of BADGEs from this investigation is also consistent with previous research that reported BADGE.2H$_2$O accounted for the highest component with a mean value of 274 ng/g, followed by BADGE.H$_2$O (mean: 52.3 ng/g) and BADGE.HCl.H$_2$O (38.3 ng/g) [5]. These results lead to the hypothesis that these pairs of substances have the same dispersal origin. However, the distribution of chemicals in the environment depends on many factors such as environmental conditions and photodegradability. BADGE.2H$_2$O and BADGE.HCl.H$_2$O are stable hydrolysis derivatives of BADGE. BADGE.H$_2$O is formed by the reaction of BADGE with moisture and

humidity in the atmosphere [15]. The toxicity of hydrated products of BADGE was reported to be significantly higher than that of BADGE itself [18]. Therefore, more in-depth studies are needed to clarify this issue.

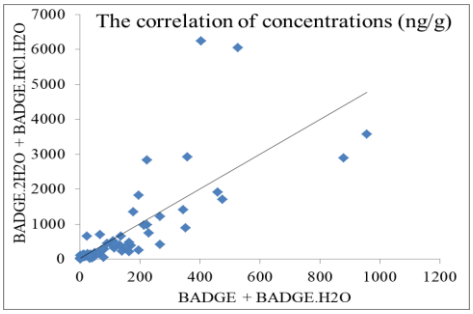


Figure 2. Correlations of BADGE concentrations in indoor dust samples.

3.3. Estimated Human Exposure to BADGEs via Dust Ingestion

The risk assessment of human exposure to chemicals through dust ingestion has been reported in several previous studies [5, 24]. The estimated exposure dose (EED), which has the unit of ng/kg-bw/d, was calculated by the equation (*):

$$EED = C_{dust}.f/M \quad (*)$$

Where C_{dust} is the concentration of target compounds in dust (ng/g), f is the dust ingestion rate (g/d), and M is the body weight (kg). The mean dust ingestion rate was 0.06 g/d for adults [5]. The average body weight (bw) for Vietnamese adults was applied as: women (51 kg) and men (59 kg) [25].

Generally, the exposure doses to BADGEs through dust ingestion for women were higher than for men (Table 4). However, the estimated exposure dose values are higher for women than for men due to the lower mean body weight of women (51 kg) compared to men (59 kg). In comparison, the estimated exposure doses of Vietnamese adults to BADGEs through dust ingestion were similar to those reported for Chinese (0.66–3.22 ng/kg-bw/d) and three times lower than those reported for Koreans (1.38–6.77 ng/kg-bw/d) and Japanese (1.42–6.45 ng/kg-bw/d) [15].

Table 4. Estimated exposure doses (ng/kg-bw/d) of BADGEs via dust ingestion for women/men

Micro-environments	BADGE	BADGE.H_2O	BADGE.$2H_2O$	BADGE.HCl.H_2O	ΣBADGEs
Homes	0.0471/0.0407	0.0947/0.0819	0.764/0.661	0.220/0.190	1.13/0.974
Public places	0.0995/0.086	0.213/0.184	1.34/1.15	0.273/0.236	1.92/1.66
Laboratories	0.0562/0.0486	0.124/0.107	0.506/0.438	0.146/0.126	0.832/0.720
Offices	0.0256/0.0221	0.056/0.0484	0.238/0.206	0.088/0.076	0.406/0.351

4. Conclusions

The occurrence of BADGE and its hydrolysis derivatives in indoor dust samples collected from various micro-environments in Hanoi, Vietnam has been investigated. Total concentrations of BADGEs in indoor dust ranged from ND to 6640 ng/g (mean: 950 ng/g and median: 434 ng/g). Concentrations of BADGEs in dust samples were significantly different among the four micro-environments. The highest concentration of the sum of the four BADGEs was found in indoor dust samples from public places, followed by homes, laboratories, and offices. Based on the measured mean concentration of BADGEs, we estimated human exposure doses to these compounds through indoor dust ingestion for adults. The exposure doses to BADGEs for women and men were in ranges of 0.406–1.92 and 0.351–1.660 ng/kg-bw/d, respectively.

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