Determination of cyclic volatile methylsiloxanes in sludge samples collected from To Lich River, Ha Noi, Vietnam

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Abstract

Cyclic volatile methylsiloxanes (cVMSs) are synthesis chemicals used widely in consumer products such as building materials, households, electrical and medical devices, and personal care products. Due to being used in many applications, cVMSs are released into various micro-environments. Recently, the pollution level of cVMSs in the environment including sewage sludge has been gradually concerned. In this work, four typical cVMSs: hexamethylcyclotrisiloxane (D3), octamethylcyclotetrasiloxane (D4), decamethylcyclopentasiloxane (D5), and dodecamethylhexasiloxane (D6) were measured in 20 sludge samples collected from a sewage river (To Lich River) in Ha Noi metropolitan by using the gas chromatography-tandem mass spectrometry (GC-MS) method. The method detection limits (MDLs) and the method qualification limits (MQLs) of cVMSs in sewage sludge were a range of (1.20-3.00) ng/g-dw (dry weight) and (3.60-9.00) ng/g-dw, respectively. Recoveries of the surrogate compound (tetrakis-(trimethyl siloxane)-silane: M4Q) in blank and real samples were from (89.4 to 112) % (RSD: 9.2 %). The total concentrations of cVMSs in sewage sludge samples from To Lich River ranged from (260 to 13,800) ng/g-dw. Among the studied cVMSs, D5 was found at the highest levels in a range of (150-6,040) ng/g-dw. To our knowledge, this is the first report on the occurrence of cVMSs in sewage sludge samples in Ha Noi as well as Vietnam. The results indicate that the distribution of cVMSs in environments is significantly high and further studies should be conducted.

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cVMSs, D5, Environment pollution, Sewage sludge, Ha Noi metropolitan.

1 Introduction

Cyclic volatile methylsiloxanes (cVMSs), the organosilicon compounds, were first synthesized in 1863; however, cVMSs began their history in personal care products in the 1950s [4]. Since then, various cVMSs applications widely occur in human life, especially in personal care products such as shampoos, hair conditions, antiperspirants, etc. [6]. The noticeably increasing use of their applications caused the occurrence of cVMSs in different environments [16, 17, 21]. Tran et al. (2015) had a survey of 12 countries around the world and showed siloxane (including cVMSs and linear volatile methylsiloxanes: lVMSs) concentrations in indoor dust samples in Greece (2,790 ng/g), Korea (1,810 ng/g), Japan (1,500 ng/g), USA (1,220 ng/g), China (1,070 ng/g), Vietnam (206 ng/g) India (132 ng/g), and Pakistan (68.3 ng/g).

Although not enough evidence on the direct harms of cVMSs to human health, the adverse effects and toxicity of cVMSs on laboratory animals are of interest [12, 14]. In female rats, octamethylcyclotetrasiloxane (D4) was considered to inhibit ovulation, while decamethylcyclopentasiloxane (D5) caused liver enlargement, endometrial cancer [2,12]. However, dodecamethylcyclohexasiloxane (D6) did not meet the criteria for a toxic, persistent, and bioaccumulative compound [1].

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In Vietnam, there were only a few studies about cVMSs in personal care products, and air, dust, water environments [13, 18, 19]. In this study, the authors have investigated a method for determining cyclic volatile methylsiloxanes (cVMSs) including hexamethylcyclotrisiloxane (D3), D4, D5, and D6 in sewage sludge samples by gas chromatography tandem mass spectrometry (GC-MS). Then, the optimal method was applied to analyze and monitor the distribution of cVMSs in sludge samples collected from a sewage river (To Lich River) in Ha Noi metropolitan area, Vietnam.

2 Materials and methods

2.1 Materials

In this report, the cyclic volatile methylsiloxane (cVMS) standards (D3, D4, D5, and D6), and surrogate standard (M4Q) with 97 % purity was purchased of Sigma-Aldrich (St.Louis, MO, USA). Dichloromethane, *n*-hexane, and acetone (analytical solvents) were purchased from Merck KGaA (Darmstadt, Germany). Florisil, Silica gel and anhydrous sodium sulfate (used for column chromatography) were purchased from Merck KgaA (Darmstadt, Germany) and Sanchun Chemical Co (Gyeonggi-Do, Korea). All surrogate and target standards were dissolved in *n*-hexane.

2.2 Sampling

Sludge samples were collected during the period from September to November 2020 at three different locations with the distance between tow consecutive points from (4 to 8) km, in the To Lich River flowing throughout Ha Noi urban, Vietnam: Hoang Quoc Viet Bridge (n = 7), Khuong Dinh Bridge (n = 7) and Quang Bridge (n = 6). A steel shovel or bucket is used to collect surface sludge samples. The analyzed samples were taken in batches at least five days after the rainy day. Necessary information about the sample such as coordinates, location, the sampling time is carefully recorded. All sludge samples were wrapped in aluminum foil and afterward fixed in polypropylene bags. Before transported to the laboratory, samples were put away in cooler boxes. After that, samples were freeze-dried, sieved, and homogenized through a 150 µm sifter. At that point, samples were removed promptly for cVMSs experiments or put away at –20 °C in a fridge.

2.3 Sample preparation

Surrogate standard (M4Q: 200 ng) was spiked into 0.5 g of freeze-dried sludge samples. After equilibrated for 30 min at room temperature, the extraction was accomplished with a 5 mL combination of dichloromethane (DCM) and *n*-hexane (1:1, v:v) for 15 min in an orbital shaker (Eberbach Corp., Ann Arbor, MI, USA). Then, samples were centrifuged in Eppendorf Centrifuge 5804 machine (Hamburg, Germany) at 2,000 rpm for 10 min, and the supernatant was moved into a 15 mL glass tube. The extraction was repeated two times again. Then, a stream of nitrogen was used to concentrate the extracts to 3 mL. The following two procedures were performed to provide the optimal procedure for analysing cVMSs in sludge samples.

Procedure 1: The concentrated extracts were passed onto a chromatography column (loaded with glass fleece, 0.5 g anhydrous Na_2SO_4, 0.5 g alternative between Florisil (or silica gel) and 0.5 g anhydrous Na_2SO_4, respectively), which had been conditioned previously by 5 mL *n*-hexane. After that, 15 mL *n*-hexane was used for elution purpose. The eluted solutions were concentrated to 1 mL under a gentle stream of nitrogen and transferred into a GC vial for analysis.

Procedure 2: C18 solid-phase extraction (SPE) cartridge (500 mg/6 mL, Macherey-Nagel, Thermo Fisher Scientific Inc., USA) was conditioned with 5 mL *n*-hexane. Then 3 mL extract was loaded onto the C18- SPE cartridge, eluted with 12 mL *n*-hexane, and concentrated to 1 mL for instrumental analysis.

These procedures were based on prior studies [11, 13, 21] on the solvent and the extraction process in an effort to provide the most optimal method.

2.4 The optimal conditions of GC analysis

The appropriate chromatographic conditions of a gas chromatographic system (GC7890B) connected to a mass spectrometer detector (MS-5977A) of Agilent Technologies, USA used to analyze cyclic volatile methylsiloxanes are as follows: column DB-5MS capillary (30 m × 0.25 mm id × 0.25 µm), 99.99 % pure He carrier gas, injector temperature: 250 °C. The temperature of GC oven started from 40 °C (hold 2 min) to 220 °C at 20 °C/min, further increase to 280 °C at 5 °C/min (hold 10 min), finally increase to 300 ºC at the 8 °C/min and hold for 5 min. The MS detector was operated in SIM mode: ion fragment *m/z* 207 for quantification for D3, ion fragment *m/z* 281 for D4 and M4Q, *m/z* 355 for D5, and *m/z* 341 for D6 [18,19].

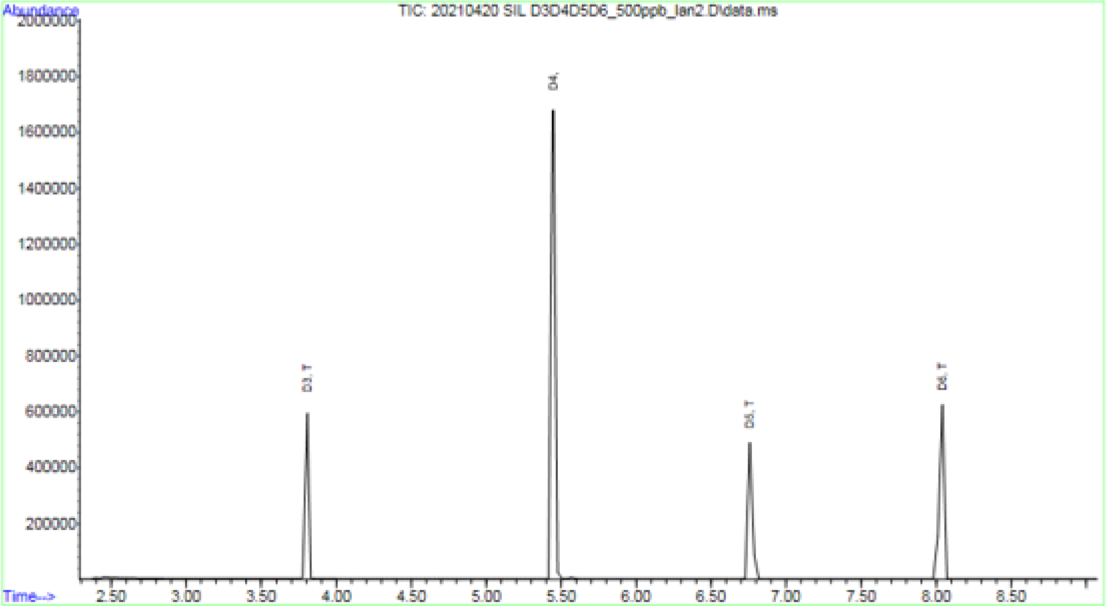

Fig. 1 The chromatogram of four cVMSs: D3, D4, D5, and D6 at 500 ng/L.

3 Results and discussion

3.1 Validate analytical procedures

To evaluate the effectiveness of chromatographic column separation, M4Q was used as a surrogate standard for recovery calculation. Calculation of recovery based on cVMSs standards is not feasible because the initial cVMSs concentrations of the environmental samples are unknown; thus, substitution by M4Q, a rare occurrence in the environment and essential characteristics cVMSs properties is required. The sludge that has undergone the extraction processes is kept, then left to dry naturally and considered as a blank sample. Then, the experiments are carried out according to the above two procedures comparing the extraction process. The recovery was calculated based on the formula below:

$$R\ (\%) = \frac{S_x}{S_o} \times 100$$

Where, S_x is the signal (peak area) of M4Q obtained by the GC analysis through the experimental procedure meanwhile S_o is the average peak area of M4Q obtained by the GC analysis when measuring directly the surrogate solution (200 ng of M4Q dissolved in *n*-hexane). Table 1 presents the recoveries of M4Q in blank samples by various kind of SPE column in this study.

Table 1 Recoveries of the surrogate standard (M4Q)

Number of experiments	**Recoveries (%) of M4Q**		
	Florisil	**Silica gel**	**C18**
1st	80.2	78.2	90.7
2nd	93.4	64.5	95.1
3rd	87.6	59.2	89.4
4th	79.8	63.1	92.2
5th	92.1	77.6	112
6th	83.4	55.4	107
7th	94.5	71.5	91.5
Mean	**87.3**	**67.1**	**96.8**
RSD (%)	**7.2**	**13.3**	**9.2**

As can be seen in the Table 1, the average recoveries of M4Q in blank samples were (67.1 ± 13.3) % and (87.3 ± 7.2) % for silica gel and Florisil, respectively. However, the highest mean recoveries were calculated (96.8 ± 9.2) % via procedure using C18; therefore, procedure two was choose to apply for cVMSs analysis in sewage sludge in this work.

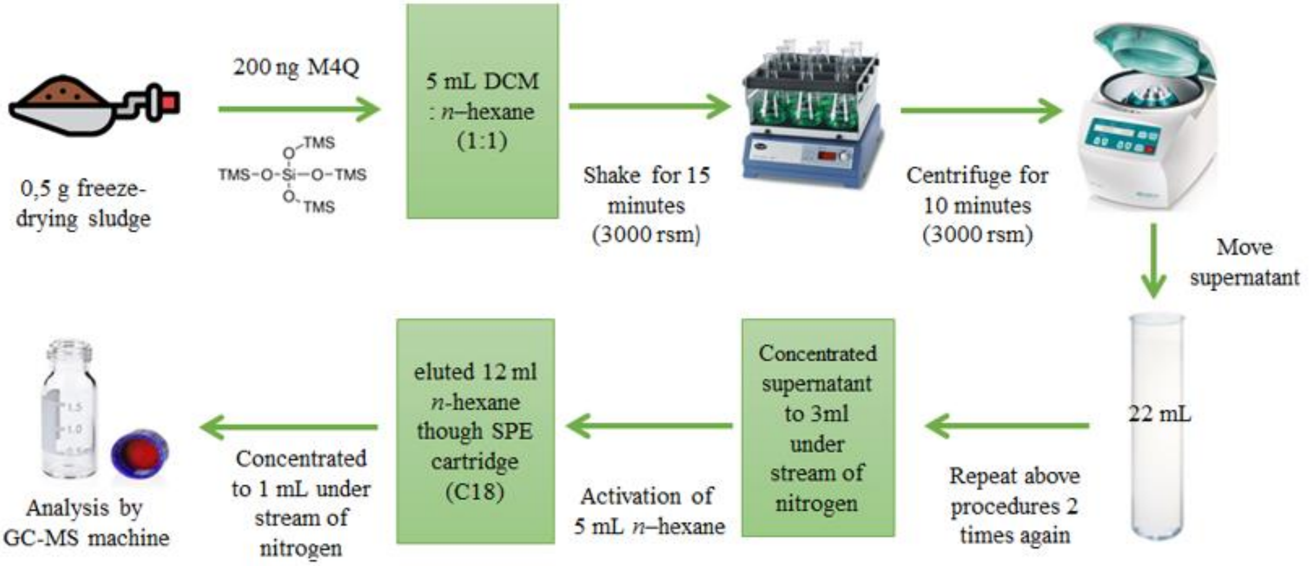

Fig. 2 The analytical procedure of cVMSs in sludge samples

To avoid sample contamination, hair and skincare products are prohibited in the laboratory due to the prevalence of siloxane in personal care products. Furthermore, the glassware used instead of plastic or silicone was rinsed with double-distilled water, acetone, DCM and *n*-hexane then calcined at 400 °C and kept at 100 °C before use. In order to minimize instrumental background levels, *n*-hexane was injected into the GC several times before performing the analysis by the GC-MC method.

Moreover, the concentrations of D4, D5, and D6 in blank samples were detected at (2.5-4.0) ng/g-dw, (3.5-6.0) ng/g-dw, and (2.5-4.5) ng/g-dw, respectively. Meanwhile, D3 was not found in blanks. To reduce trace contamination, the reported cVMSs concentrations of all sludge samples were determined by subtracting the contribution of the blank to the measured cVMSs concentration. The method detection limits (MDLs) and the method qualification limits (MQLs) of cVMSs in sewage sludge ranged from (1.20-3.00) ng/g-dw and (3.60-9.00) ng/g-dw, respectively. The calibration curve for individual cVMSs was linear over a concentration range of (1.0 to 1,000) ng/mL (8 points of calibration curve with $R^2 \geq 0.995$).

Some analytical methods from previous studies are shown in Table 2. Most of the studies used extraction solvents such as *n*-hexane, dichloromethane, and acetone [7-10, 23]. These are considered suitable solvents for the extraction of cVMSs in sludge. Among those, the study of Lee et al. (2014) reported a method with good recovery (81-143) % and quantitative limit (0.03-23.0) ng/g-dw. In overall, this study yielded a higher recovery rates than that in some previous reports [7, 10], and the method qualification limits (MQLs) of cVMSs are slightly higher than those reported in some studies [7, 10, 23].

Table 2 Comparison of various methods for analysis of cVMSs

Country	Sample preparation	Sample weight/Extraction method	Instrument	MQL (ng/g-dw)	Recovery (%)	Reference
China	Freeze-dried	0.2 g Ultrasonic extraction	GC-MS	< 3	75-93	[23]
China	Freeze-dried	0.1 g Solid-liquid extraction,	GC-MS	0.4-2.2	69-104	[10]

		followed by solid phase extraction				
China	Freeze-dried	1 g Solid-liquid extraction	GC-MS	200-2000	80.3 ± 10.2	[9]
South Korea	Freeze-dried	0.5 g Solid-liquid extraction	GC-MS	0.03-23.0	81-143	[8]
South Korea	Freeze-dried	2.5 g Solid-liquid extraction	GC-MS	0.21-3.02	54-69	[7]
Vietnam	Freeze-dried	0.5 g Solid-liquid extraction, followed by solid phase extraction	GC-MS	3.60-9.00	89.4-112	This study

3.2 Concentrations of cVMSs in sludge samples collected in To Lich River.

The levels of cVMSs in sludge collected from To Lich River in Vietnam are presented in Fig. 3. The total concentrations of cVMSs ranged from (260 to 13,800) ng/g-dw (mean/median: (3,520/2,690) ng/g-dw). In addition, the concentration of cVMSs fluctuates along the river, being lowest level at Hoang Quoc Viet Bridge (2,910/1,810 ng/g-dw) and highest level at Khuong Dinh Bridge (4,480/2,890 ng/g-dw).

To Lich River is currently acting as a waste receiving river in the inner city of Ha Noi. Hoang Quoc Viet Bridge is an upstream sampling location, where the cVMSs measured concentrations in the collected sludge samples were lower than that found in the other locations because of the flowing influence. However, the highest of cVMSs concentration found in sludge collected from the Khuong Dinh Bridge where higher the population density, had higher cVMSs concentration due to popular cVMSs in personal care products [6].

The concentrations of each cVMSs found at different locations in the river have fluctuated, with the lowest for D3 (min/max: nd (not detection)/230 ng/g-dw) and the highest for D5 (200/8,033). The total concentrations of the four substances ranged from (2,800 to 13,800) ng/g, indicating cVMSs in the sludge samples.

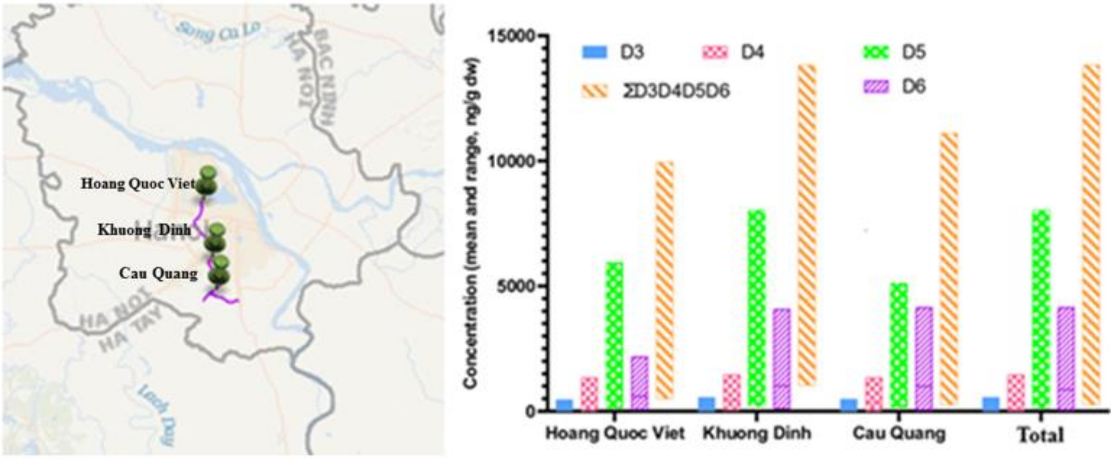

Fig. 3 The sampling sites and cVMSs concentrations in sludge collected from To Lich River

3.3 Composition profiles of cVMSs in sludge samples collected in To Lich River

The relationship of the distribution component of each analyzed cVMSs with the total concentration is shown in Fig. 4. Among the cVMSs, D5 is the dominant substance, accounting for more than 64 % in Khuong Dinh Bridge and 54 % in Quang Bridge. In contrast, D3 was the least dominant compound with less than 7 % of the total concentration in all sampling sites. CVMSs in aquatic environments tend to absorb suspended particulate matter or sludge/sediment. Some studies indicated that D5 concentrations were the most dominant via cVMSs in aquatic; D3 concentration, in contrast, was the lowest [13, 15]. In addition, D5 is relatively persistent in sludge with a half-life time up to (1,200-2,700) days in anaerobic sediments [3].

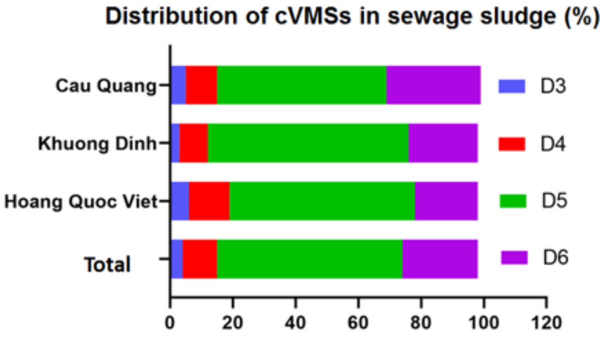

Fig. 4 The relative composition of cVMSs concentration in sludge samples
collected from To Lich River

In general, cVMSs concentrations in sludge samples from this study were compared with some previous studies, the results shown the similar to sludge samples collected from industrial wastewater treatment plants in Korea [8]. However, when comparing with those reported in sludge from wastewater treatment plants in Northeastern China [24] and from sediment collected from may countries such Canada, South Korea, and China in previous studies [5, 7, 21, 25] the author indicated that cVMSs concentration was considerably higher. These results suggest that the occurrence of cVMSs in sewage sludge samples were in To Lich River, Vietnam.

3.4. Correlation of cVMSs in sludge samples collected from To Lich River

The assessment of concentration correlation between cVMSs indicates the source of the dispersion in the environment. The Pearson correlation is the correlation coefficient to measure the how strong relationship between two variables. The correlation of cVMS concentrations in the To Lich River is considered and presented in Table 3. The pairs of concentrations D5 via D6, D4 versus D6, and D4 versus D5 have high positive correlation coefficients with 0.89, 0.89 and 0.77, respectively. The high correlation between D4, D5, and D6 showed the relationship between application the various personal care products, silicone products and cVMSs percentage in PCPs [6, 19, 20]. However, the concentration correlation between D3 and D4, D5 and D6 were approximately 0.4. Although lack of information on D3, such as decomposition time in the sludge environment, however, unlike substances D4, D5, and D6 are all present in many daily products and D3 often appears in industrial products.

Table 3 Correlation of cVMSs (D3, D4, D5, and D6) in sewage sludge in To Lich River

	D3	**D4**	**D5**
D4	$r = 0.44$; $p = 0.063$		
D5	$r = 0.42$; $p = 0.062$	$r = 0.77$; $p = 0.000$	
D6	$r = 0.47$; $p = 0.036$	$r = 0.89$; $p = 0.000$	$r = 0.85$; $p = 0.000$

4 Conclusion

In this study, the optimization of the method for the determination of some cyclic volatile methylsiloxanes by gas chromatography coupled to mass spectrometry was performed. The results showed that the method detection limits (MDLs) and the method quantitative limits (MDLs) of cVMSs in sludge samples were in ranges of (1.20-3.00) ng/g-dw and (3.60-9.00) ng/g-dw, respectively. The recoveries of surrogate standard (M4Q) in blank procedures ranged from (89.4 to 112.0) %. The procedure is applied to determine the concentration of cVMSs in sludge samples collected at To Lich River flowing Ha Noi metropolitan area. Total concentrations of cVMSs measured in sewage sludge samples collected from To Lich River ranged from (260 to 13,800) ng/g-dw. Among studied CVMSs, D5 was found at the highest concentrations, followed by D6, D4, and D3.

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Xác định methylsiloxan dễ bay hơi tuần hoàn trong mẫu bùn thải sông Tô Lịch, Hà Nội, Việt Nam

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Tóm tắt Các methylsiloxan dễ bay hơi tuần hoàn (cVMSs) là các hóa chất tổng hợp, được sử dụng rộng rãi trong các sản phẩm tiêu dùng như vật liệu xây dựng, hộ gia đình, thiết bị điện, y tế, cũng như các sản phẩm chăm sóc cá nhân. Do được sử dụng trong nhiều ứng dụng, cVMS được giải phóng vào nhiều môi trường vi mô khác nhau. Thời gian gần đây, mức độ ô nhiễm của cVMSs trong môi trường, trong đó có bùn thải, đang dần được quan tâm. Trong nghiên cứu này, bốn cVMS điển hình: hexamethylcyclotrisiloxane (D3), octamethylcyclotetrasiloxane (D4), decamethylcyclopentasiloxane (D5) và dodecamethylhexasiloxane (D6) được đo trong 20 mẫu bùn được lấy từ nước thải sông Tô Lịch ở Hà Nội bằng cách sử dụng phương pháp sắc kí khí − khối phổ (GC-MS). Giới hạn phát hiện của phương pháp (MDL) và giới hạn chất lượng của phương pháp (MQL) của cVMS trong bùn thải lần lượt nằm trong khoảng (1,20-3,00) ng/g-dw (trọng lượng khô) và (3,60-9,00) ng/g-dw. Độ thu hồi của hợp chất thay thế (tetrakis-(trimethyl siloxane)-silane: M4Q) trong mẫu trắng và mẫu thực là từ (89,4-112) % (RSD: 9,2 %). Tổng nồng độ cVMSs trong các mẫu bùn thải từ sông Tô Lịch dao động từ (260-13.800) ng/g-dw. Trong số các cVMS được nghiên cứu, D5 được tìm thấy ở mức cao nhất trong khoảng (150-6.040) ng/g-dw. Theo hiểu biết của chúng tôi, đây là báo cáo đầu tiên về sự xuất hiện của cVMS trong các mẫu bùn thải tại Hà Nội, cũng như tại Việt Nam. Kết quả chỉ ra rằng sự phân bố cVMS trong môi trường cao đáng kể và cần tiến hành các nghiên cứu sâu hơn.

Từ khóa cVMSs, D5, ô nhiễm môi trường, bùn thải, đô thị Hà Nội.