



Contaminant characterization of odor in soil of typical pesticide-contaminated site with shallow groundwater

Tiantian Ye¹ · Zhenxing Wang¹ · Gang Liu¹ · Jianbiao Teng¹ · Chong Xu² · Lihong Liu¹ · Chenhui He¹ · Jianyu Chen¹

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Abstract

Odor emission from the soil of pesticide-contaminated sites is a prominent environmental problem in China, but there are very few researches about the component and spatial distribution of odorous substances in the soil of contaminated sites. In this paper, to investigate the odor pollution condition of an organophosphorus pesticide production site in a city of South China, the odor pollutants in the soil and soil gas were analyzed and the key odor-contributing substances were identified. Besides, the correlation between the concentrations of odorous substances in soil and soil gas was analyzed, and the measured results were compared with the predicted results by the linear model and DED model. An off-line soil gas sampling device was designed to collect the gas emitted from soil because the groundwater level in the site was too shallow to build a soil gas well. The key odor substances were screened from the detection results of soil gas via odor activity value (OAV) analysis, which revealed that the key odorous substances included benzene, ethylbenzene, ammonia, toluene, m,p-xylene, methyl sulfide, dimethyl disulfide, and formaldehyde. Furthermore, the spatial distribution of the odor substances in the soil of the pesticide-contaminated site was closely related to the layout and geologic structure of the site. The odor pollutants in soil were mainly distributed near the phosmet production workshop and the drainage ditch network. As for the deep distribution, the odorous substances were mainly enriched in the silty clay or clay layer (5.6–11 m), followed by the sludge layer (1–3.6 m). Finally, the predicted model (linear model and DED model) analysis suggested that the linear model was more suitable for predicting the concentration of odorous substances in the soil gas with the detection data of soil in this pesticide-contaminated site.

Keywords Odor · Soil · Pesticide-contaminated site · Shallow groundwater · Model

Introduction

There are lots of report about the characteristics, distribution, and health risks of soil pollutants in different contaminated sites (Liu et al. 2015; Mcneel et al. 1997; Morra et al. 2011; Vijgen et al. 2018). However, they mainly focus on investigating toxic pollutants with health risks, while paying less attention to soil odor pollution (Tao et al.

2022; Wang et al. 2023). In fact, offensive odors pose a direct threat for human health and welfare, and the odor pollution of contaminated sites has raised more and more public attention (Yang et al. 2015). Organic solvents used in pesticide production, such as acetone, formaldehyde, xylene, benzene, toluene, and ethylbenzene, can cause odorous gas emission not only during the production process but also after the factory was closed. Presently, the research on odor pollution is mainly focused on the emission from production of factories, sewage treatment plants, and landfill leachate (Blanes-Vidal et al. 2009; Dincer et al. 2006; Jiao et al. 2023). It is reported that the main odorous substrates emitted during the pesticide production included hydrogen sulfide, methyl mercaptan, sulfur dioxide, methyl sulfide, and dimethyl sulfide (Jiao et al. 2023). However, there are few research literatures on soil odor pollution of pesticide-contaminated sites (Meng et al. 2022; Yan et al. 2022; Zhao et al. 2021).

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✉ Gang Liu
liugang@scies.org

¹ Ministry of Ecology and Environment, South China
Institute of Environmental Sciences, Guangzhou,
People's Republic of China 510655

² Centre Testing International Pinbiao (Guangzhou) Co., Ltd,
Guangzhou, People's Republic of China 510700

Currently, there are two general approaches to odor measurement: olfactometric techniques for odor concentrations C_{OD} (OU_E/m^3) and analytical techniques for concentration measurements C (mg/m^3) (Abraham et al. 2020; Sivret et al. 2016). Odor concentration of a gaseous sample is the number of dilutions with neutral gas, at which 50% of the panelists can still detect odor in the air sample. Olfactometry is not limited by the type and concentration range of odorants and can quickly reflect people's intuitive perception of the odor. Although this method is simple and easy to operate, it has uncontrollable subjectivity by using human olfactory organs as identification carriers. Therefore, the stability of this method is doubtful (Zhao et al. 2015). Analytical techniques use the gas chromatography (GC) for detecting and analyzing the concentrations of substances in the gas, which is the most accurate and widely used chemical method. Analytical techniques are in principle more reliable than olfactometric techniques, but it cannot determine the perceivable odor intensity because there is great discrepancy among the odor detection thresholds (ODT) of different compounds (Davoli et al. 2003). To solve this problem, some studies proposed to identify the key odorants by calculating the odor activity value (OAV) of a single substance, the sum of OAVs for odorous mixtures (SOAV), and the odor contribution rate of each odorant (Sivret et al. 2016; Wu et al. 2017). OAV can better describe the properties of odor and has been widely used to evaluate the key odorants in gaseous mixtures cost-effectively (Wu et al. 2017). For example, Wu et al. measured the concentrations of individual compounds in the air of a waste disposal plant by analytical analysis and identified the key odor components by calculating the OAV values. The result found that a total of 28 odorants were detected and the H_2S , trimethylamine, phenol, limonene, ethylbenzene, styrene, and NH_3 were selected as key odorants for their OAV above 1 (Wu et al. 2015). The odor with complex compositions in the soil of pesticide-contaminated site was mainly derived from raw and materials, products, intermediate products, and degradation products in the production process. In order to economically and scientifically characterize odor of soil in the pesticide-contaminated site, the types and concentrations of odor mixtures in soil gas can be determined scientifically through combining analytical techniques and OAV.

Odor pollution in contaminated site can be determined by collecting soil gas and then analyzing the main components in the sample to determine the extent and scope of odor pollution. However, the depth of groundwater level in some area, such as the Pearl River Delta of South China, is generally shallow (about 1–2 m), which cannot meet the requirement of the standard for sampling of soil gas (ASTM D7758-17 2017). Meanwhile, the odorous substrates are often enriched in the clay of the saturated zone. Therefore,

it is urgent to propose a new sampling method to detect odor pollution in the contaminated site such as with shallow groundwater. Alternatively, soil gas concentrations can be predicted using mathematical model based on the soil concentration (Johnson et al. 1993; Pavlovic et al. 2018). In general, the measured soil gas concentration accurately reflects the actual level of soil pollution. However, due to the unique advantages of simplicity and low cost, models are widely used in field studies. Models used for studying the concentration of volatile organic compounds in soil and soil gas include the linear adsorption model and the dual equilibrium desorption (DED) model (Chen et al. 1999, 2010; Johnson et al. 1993; Zhang et al. 2018). For example, Zhang et al. used the linear adsorption model and the DED model to estimate the concentrations of dichloromethane and benzene in soil gas at a hazardous material storage warehouse site in Handan, Hebei Province, North China (Zhang et al. 2018). Therefore, using soil gas-measured concentration or model-predicted concentration for odor pollution in shallow groundwater level site is worth studying.

In this study, organophosphorus pesticide (phosmet, dimethoate, butachlor, and trichlorfon) production site with shallow groundwater in the Pearl River Delta of South China was selected as a typical contamination site to analyze the distribution of odorous contaminants in soil. The objectives of this study were to (a) propose a set of experimental device to collect odor gas from soil; (b) screen the odorous substances in the soil and identify the key odorants responsible for the odor emitted from soil gas; (c) research the extent of odorous contamination, their spatial distribution, and the influencing factors; and (d) explore the applicability of two adsorption/desorption models in predicting the concentration of odor pollutants in soil gas. The results will provide valuable information to understand the pollution status of odorous contaminants in pesticide-contaminated site and provide reference for the investigation and restoration of the odor-contaminated soil.

Materials and methods

Site characterization

The typical pesticide factory site is located in Guangzhou, Guangdong Province, South China, where the depth of groundwater level is shallow (about 0.4–2.3 m). This pesticide factory was built in 1958 and produced various pesticides such as phosmet, dimethoate, butachlor, and trichlorfon. The plant was shut down in 1996, and the site was repurposed as a sports field and ground parking lot. The area of production workshops for dimethoate, butachlor, and trichlorfon were remediated, and buildings have been

built. Therefore, the area of this study is concentrated in the phosmet workshop area and other ancillary facilities, covering 23,264 m².

Previous investigation showed that the stratigraphy of the site can be generalized as backfill soil (1~3 m thickness), sludge soil (0~3.6 m thickness), silty clay or clay (1.6~6.6 m thickness), sandy (2.6~4.9 m thickness), and mudstone (2.00~3.80 m thickness). The overall distribution is shown in Fig. 1. The depth of groundwater level in this site is shallow (0.4~2.3 m), and the pollution distribution is in relatively deep soil. In some areas, the deep soil had a strong pungent odor, indicating that soil odor pollutants mainly existed in the saturated soil layer.

Sample collection and analysis

Odor screening sample collection

The possible pollutants of pesticide-contaminated site are extremely complex, involving not only various raw materials, solvents, intermediates, and products but also a large number of by-products, degradation products, and so on. Therefore, in order to screen the main odor pollutants in the soil, soil and soil gas pre-sampling were carried out for qualitative analysis. To collect the heavily contaminated soil samples, the drilled soil of each sampling point was first quickly tested by a photoionization detector (PID, with measurement range 0–2000 ppm) during the soil sampling process, and the soil samples with higher PID (PGM-7340, RAE Systems, USA) readings and obvious stench were collected. Then, the collected samples were subjected to GC/MS or GC (QP2020NX, Shimadzu, Japan) scan analysis to determine the detectable pollutants in the soil and soil gas. A total of 10 soil samples and 5 soil gas samples were collected in this stage (Fig. 2). In view of the low sensitivity of amines in GC, parallel soil gas samples were taken for ion chromatography (IC, Aquion, Dionex, USA) analysis to

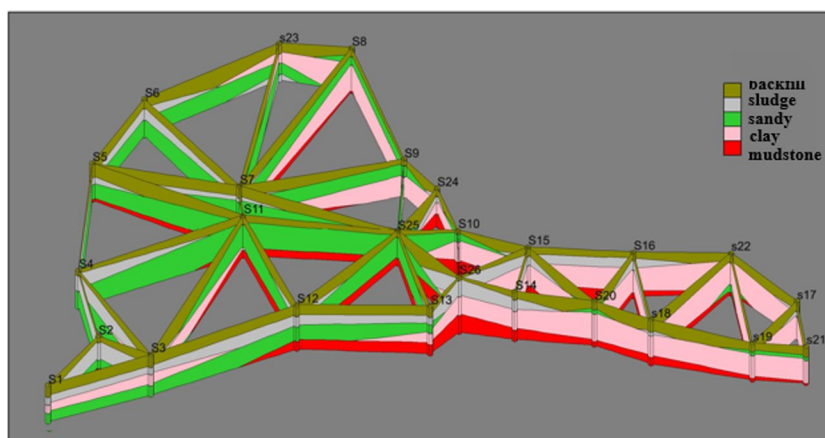
determination of ammonia, methylamine, dimethylamine, and trimethylamine in soil gas.

Odor investigation sample collection

Soil sampling Given the complexity of soil pollution, soil sampling is divided into two stages: preliminary sampling and supplementary sampling. The preliminary sampling stage identifies the general spatial distribution of odor pollution, while the supplementary sampling further determines the extent and boundary of odor pollution. In the preliminary sampling stage, the professional judgment method was used to arrange soil sampling points, mainly in potentially contaminated areas, including the phosmet workshop, the chloroacetic acid workshop, the fine workshop, the calcium plastic machine repair workshop, the sewage collection tank, and the drainage ditches. Among them, the phosmet production workshop, chloroacetic acid workshop, and fine workshop are used to the produce of pesticide phosmet, chloroacetic acid, and the phosphite, respectively. The calcium plastic workshop was built for storing raw materials before 1983, and after 1983, it was employed for the production of packaging boxes for pesticides. Those workshop locations at the production site are shown in Fig. 2 (blue font). Consideration is also given to pre-sampling soil pollution-heavy areas. Therefore, in this stage, soil samples of 26 soil points (S1–S26 in Fig. 2) were collected with a SH-30 model rig at different depths of the boreholes based on the distribution of soil layers. The final sampling depth was 9–15 m, and a total of 250 samples were collected.

On the basis of odor substance pollution analysis of preliminary investigation results, supplementary sampling was conducted around the points which exceeded the soil screening value recommended by the Ministry of Ecology and Environment of the People's Republic of China (MEEPRC). Then, 16 additional sampling boreholes (X1–X16 in Fig. 2) were added in this study, and a total of 162 samples were

Fig. 1 Block geological profile stereogram



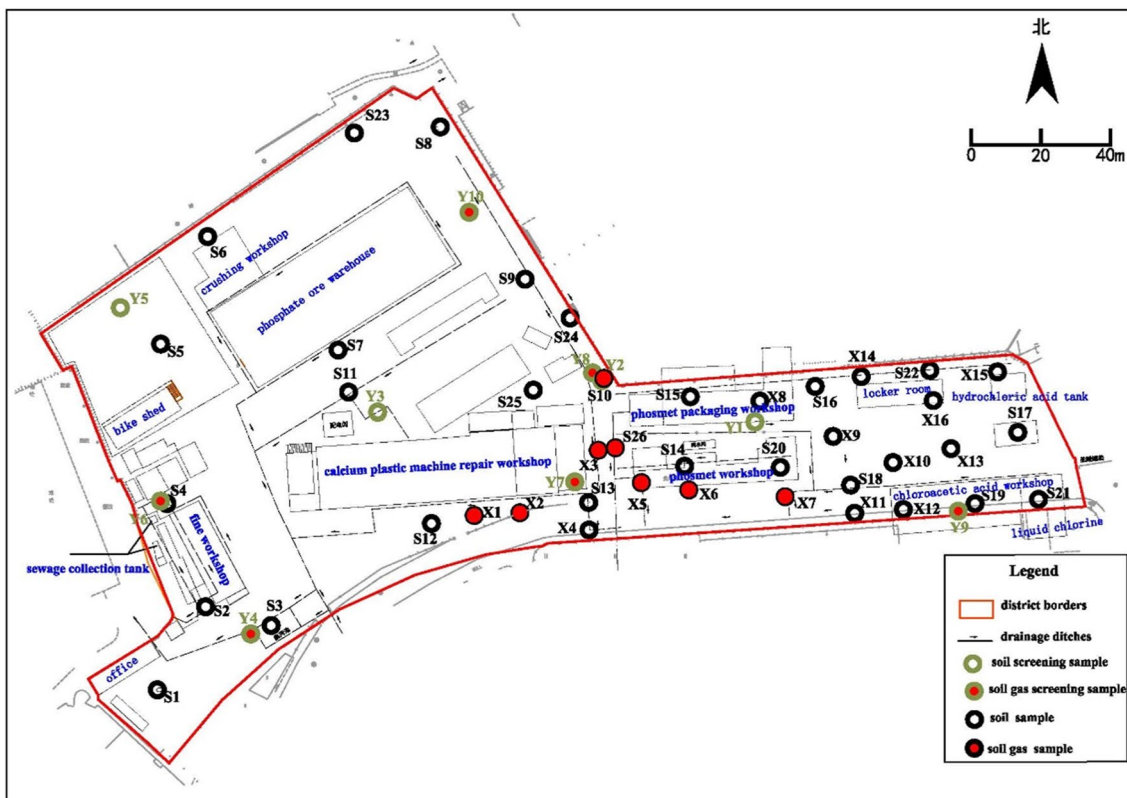


Fig. 2 Distribution of soil and soil gas sampling points of the pesticide-contaminated site

collected. All soil samples were collected and preserved according to GB/T 36197–2018 (PRC 2018).

Soil gas sampling As a typical area with shallow groundwater level, the odor pollutants in the study area mainly were distributed in the saturated soil layer, which cannot meet the requirements for standard soil gas sampling (ASTM D7663-12 2018). As the odor pollution mainly spreads into the air through volatilization, a soil volatilization gas sampling device was invented in this study to collect soil gas from the saturated soil, and then, the concentration of odor substances in the soil gas was detected to characterize the odor pollution in the soil. The soil gas collection method used in this study was the static chamber method, which involves directly drilling to collect a certain amount of odor-contaminated saturated soil into a static chamber. The odor pollutants in the soil will then volatilize into the gas in the chamber. Three outlets are provided on the chamber, each of which is connected to a pump. The pumps are used to control the flow rate of the three outlets. The outlets are connected to absorbent solutions, adsorption tubes, and gas cylinder, for the measurement of ammonia odor, VOC odor, and sulfur odor substances, respectively. The schematic diagram of the sampling device is shown in Fig. 3. Considering the vertical distribution of soil gas, samples of different depths can be

collected at each borehole according to the actual situation. For soil with large readings of PID or pungent odor, 1 kg was collected in the static chamber for soil gas sampling, and two parallel samples were collected for each sample. A total of 14 soil gas samples were collected in this study based on the odor distribution on site.

Analysis methods

The concentration of the odorous substances in soil samples was measured by Environmental Protection Agency (EPA) methods 8260C (EPA 2006) or HJ605 (PRC 2011). The volatile organic components in soil gas samples were analyzed using GC according to the HJ 583–2010 (PRC 2010). Methyl sulfide and dimethyl disulfide in soil gas samples were determined by GC according to GB/T 14678–1993 (PRC 1993). Formaldehyde in soil gas samples was tested by spectrophotometric method according to GB/T 15516–1995 (PRC 1995). Ammonia in soil gas samples was measured using ion chromatography according to HJ 1076–2019 (PRC 2019). All testing followed the requirements of the China Inspection Body and Laboratory Mandatory Approval (CMA) (Li et al. 2023). The quality control and assurance of testing were guaranteed with certified reference material, duplicates, and method blanks.

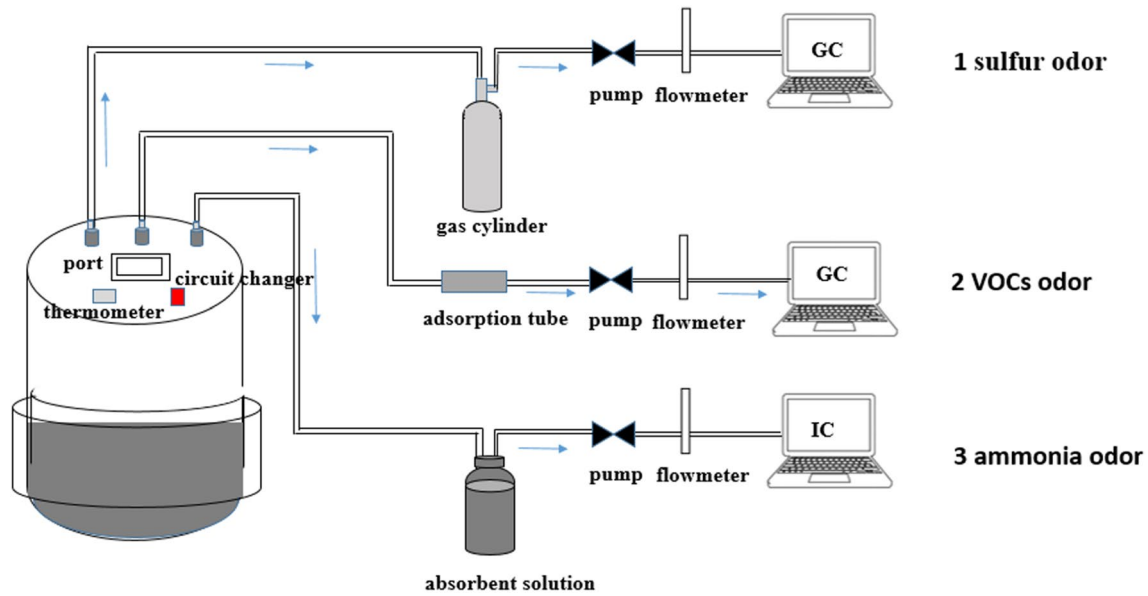


Fig. 3 Soil gas sampling device diagram

Key odor identifying methods

It is necessary to identify the key odorous substances with a significant contribution to the odor from the gas mixture for the treatment and restoration of pesticide-contaminated sites. Studies showed that the substances that produce strong odors are not necessarily those with the highest concentration, but with the highest odor activity value (OAV) (Blazy et al. 2015). The greater the OAV, the greater the contribution of the substance to the odor. The OAV can be calculated using Eq. (1), and the odor contribution (P_i) of a single substance gives the relative importance of the olfactory perception (Chai et al. 2022). Odorous substances with OAV equal to or above one are identified as substances with a significant contribution to the odor, referred to as the key odorous substances (Wu et al. 2015).

$$OAV_i = C_i / OT_i \quad (1)$$

$$SOAV = \sum_{i=1}^n OAV_i \quad (2)$$

$$P_i = OAV_i / SOAV \quad (3)$$

where OAV_i is the odor activity value of a single odorous substance, C_i is the concentration of the odorous substance i (mg/m^3), OT_i is the olfactory threshold value of the odorous substance i (mg/m^3), $SOAV$ is the total odor activity value of all odorous substances, and P_i is the odor contribution rate of the odorous substance i (%).

Adsorption isotherms and model development

Linear adsorption model

The linear adsorption model assumes that VOCs in soil are linearly and reversibly adsorbed. Organic matter is in a three-phase dynamic equilibrium in soil, and its model equation is obtained by normalizing the linear adsorption of organic matter onto soil particles (Eq. (4)) and Henry's law (Eq. (5)) (Karickhoff et al. 1979; Park 1999). If an NAPL phase is not present at the site, the mass of pollutants in the soil matrix gas, solid, and liquid phases satisfies the relationship in Eq. (6). The soil gas calculation formula can be obtained by combining the above equation, as shown in Eq. (7).

$$q_e = C_e \times f_{oc} \times K_{oc} \quad (4)$$

$$C_{sg} = C_e \times H \quad (5)$$

$$C_s = q_e \times \rho_b + C_{sg} \times \theta_{as} + C \times \theta_{ws} \quad (6)$$

$$C_{sg} = C_s \times \frac{H \times \rho_b}{\theta_{ws} + H \times \theta_{as} + \rho_b \times K_{oc} \times f_{oc}} \quad (7)$$

where q_e and C_e represent the solid phase concentration and liquid phase concentration of soil particles, respectively, when organic matter is adsorbed in equilibrium; C_s is the concentration of odorous substances in soil (mg/kg); C_{sg} is the concentration of odorous substances in soil gas (mg/L); ρ_b is the dry bulk density of soil (g/cm^3); K_{oc} is the organic

carbon–water partition coefficient (L/kg); f_{oc} is the organic carbon content in soil, dimensionless, and can be calculated by soil organic matter content (f_{om}), $f_{oc} = f_{om}/1.72$ (Sabljic 1989); θ_{ws} represents the volumetric air content, dimensionless; and θ_{as} represents the volumetric water content, dimensionless.

DED model

A large number of studies showed that the adsorption of pollutants in soil is not always linear (Weber et al. 2001; Weber and Huang 1996). The DED model assumes that adsorption consists of two parts. The first part is reversible adsorption, which usually occurs at high concentrations of pollutants, and in this part, the amount of adsorption is linearly related to the concentration of pollutants in the water phase. The second part is irreversible adsorption, which usually occurs at low concentrations of pollutants, and in this part, the amount of adsorption is represented as a Langmuir type in relation to the concentration of pollutants in the water phase (Kan et al. 1998). Assuming that adsorption is the linear sum of the first and second parts, the soil adsorption in the DED model can be calculated as follows:

$$q_e = q^{first} + q^{second} \tag{8}$$

$$q^{first} = C_e \times f_{oc} \times K_{oc}^{first} \tag{9}$$

$$q^{second} = \frac{K_{oc}^{second} \times f_{oc} \times f \times q_{max}^{second} \times C_e}{f \times q_{max}^{second} + K_{oc}^{second} \times f_{oc} \times C_e} \tag{10}$$

where C is the concentration of pollutants in the water phase (mg/L); K_{oc}^{first} and K_{oc}^{second} are the organic carbon–water partition coefficients for the first and second parts (L/kg), where the value of K_{oc}^{first} equals K_{oc} and K_{oc}^{second} can be calculated using Eq. (11) (Chen et al. 2010); K_{ow} is the partition coefficient of n-octanol/water (L/L); C_{sat} is the solubility of the pollutant (mg/L); f represents the fraction of the second compartment that is saturated upon exposure, $0 \leq f \leq 1$, and literature suggests taking $f = 1$ (Chen et al. 1999, 2010); and q_{max}^{second} is the maximum adsorption capacity of the second part (mg/kg), calculated by Eq. (12). Combined with Eqs. (8)–(10), the total adsorption of VOCs by soil solid particles under the DED model can be obtained, and the relationship formula between C_s and C_{sg} is finally obtained. For convenience of representation, a coefficient A and functions $F(C_s)$ and $G(C_s)$ of C_s are first established, as shown in Eqs. (13)–(15). And the expression for calculating soil gas C_{sg} from soil concentration C_s under the DED model is derived as Eq. (16).

$$\log K_{oc}^{second} = 5.92 + 0.16 \tag{11}$$

$$q_{max}^{second} = f_{oc} \times (K_{ow} \times C_{sat})^{0.534} \tag{12}$$

$$A = K_{oc}^{second} \times f_{oc} \times (\theta_{ws} + H \times \theta_{as}) + K_{oc}^{first} \times K_{oc}^{second} \times f_{oc}^2 \times \rho_b \tag{13}$$

$$F(C_s) = f \times f_{oc} \times \rho_b \times q_{max}^{second} \times (K_{oc}^{first} + K_{oc}^{second}) + f \times q_{max}^{second} \times (\theta_{ws} + H \times \theta_{as}) - K_{oc}^{second} \times f_{oc} \times \rho_b \times C_s \tag{14}$$

$$G(C_s) = -f \times q_{max}^{second} \times \rho_b \times C_s \tag{15}$$

$$C_{sg} = H \times \frac{\sqrt{F^2 - 4A \times G} - F}{2A} \tag{16}$$

Result and discussion

Key odorous substances in soil gas

During the odor pollutant screening sample collection, 10 soil samples and 5 soil gas samples were collected for qualitative analysis. A total of 119 kinds of VOCs and 139 kinds of semi-volatile organic compounds (SVOCs) were detected in the soil samples by GC/MS scanning. These organic substances were compared with the list of odorous substances proposed by the Japan Odor Environment

Association in 1988 (Nagata 1988), and the detected odorous substances in the soil included benzene, toluene, o-xylene, m,p-xylene, ethylbenzene, carbon disulfide, dimethyl disulfide, and methyl sulfide. Meanwhile, the results of the GC/MS analysis of the soil gas samples showed that the odorous substances included benzene, toluene, o-xylene, m,p-xylene, methyl sulfide, dimethyl disulfide, formaldehyde, carbon disulfide, and ammonia. In addition, IC analysis showed that the soil gas contained ammonia. Therefore, the soil gas of the pesticide-contaminated site in this study contained 10 odorous substances including benzene, toluene, o-xylene, m,p-xylene, methyl sulfide, dimethyl disulfide, formaldehyde, carbon disulfide, and ammonia.

To further investigate the sources of odor in the soil and the contribution of odorous substances, 14 soil gas samples were collected for quantitative analysis. The OAV_i of each substance were calculated using the average

concentration of odorous substances in the soil gas samples and their odor threshold. The results are shown in Table 1. The average concentrations of benzene, ethylbenzene, ammonia, toluene, m,p-xylene, methyl sulfide, dimethyl disulfide, formaldehyde, o-xylene, and carbon disulfide in the soil gas were 306.16, 2.52, 3.44, 3.60, 3.80, 0.03, 0.02, 1.19, 1.09, and 0.25 mg/m³, respectively. The corresponding OAV_i values were 32.54, 28.01, 14.93, 9.01, 8.83, 3.71, 2.06, 1.77, 0.83, and 0.43. Therefore, the key odorous substances with OAV_i greater than one were benzene, ethylbenzene, ammonia, toluene, m,p-xylene, methyl sulfide, dimethyl disulfide, and formaldehyde. The cumulative contribution of benzene, ethylbenzene, ammonia, toluene, and m,p-xylene accounted for more than 90% of the total odor contribution. Most odorous contaminants came from raw materials or intermediate products in the pesticide production process, such as benzene, ethylbenzene, ammonia, toluene, m,p-xylene, and formaldehyde. Other contaminants came from by-products or degradation products, such as methyl sulfide and dimethyl disulfide. The types and sources of key odorous substances in this study are highly consistent with the Yan et al.'s research which explored the odorous substances in the soil. The results showed that the raw and auxiliary materials used in the production process are the main source of odorous substances in the soil, and the key odor substances were dimethyl sulfide, dimethyl disulfide, and toluene, trichloroethylene of organochlorine and organophosphorus pesticide-contaminated site (Yan et al. 2022). For the remediation the odorous substances detected in the soil, sulfur-containing and nitrogen-containing compounds usually have reducibility and are easily oxidized, which can be treated through chemical oxidation technologies. The BTEX (benzene, toluene, ethylbenzene, and m,p-xylene) is usually relatively stable and requires the use of Fenton oxidation technology. The ammonia, formaldehyde, and

carbon disulfide with highly volatile, room temperature desorption technology can be used for treatment.

The concentration contribution PC_i of the odorous chemical in each soil gas sample and the odor contribution P_i of each chemical are shown in Fig. 4. It is demonstrated that the trend of odor activity value contribution is very different from that of concentration contribution. Most of the odorous substances in soil gas are benzene, and the concentration of benzene is very high in all 12 samples, accounting for more than 70% of the pollutants in soil gas. However, due to the high odor threshold of benzene of 9.41 mg/m³, its contribution to odor was only 5 to 60%, with an average contribution rate of 32.54%. For sulfur compounds (methyl sulfide and dimethyl disulfide) which contain typical odor-causing groups (sulfur-containing groups) (Jiao et al. 2023), the maximum concentration contribution of methyl sulfide (C_{max} = 0.049 mg/m³) was 2.17% but with an maximum contribution rate of 37.26%, and the maximum concentration contribution of dimethyl disulfide (C_{max} = 0.033 mg/m³) was 2.36% with an maximum contribution rate of 25.99%. Although their concentrations in soil gas were relatively low, their contribution to odor cannot be ignored. Thus, it is accurate and convenient to employ OAV to evaluate the contribution of each odorous substances.

Spatial distribution of odorous substances in soil

To reveal the horizontal distribution characteristics of the key odorous substances in the soil, Surfer 14 with kriging interpolation method was used to draw the spatial distribution of the key odor species (benzene, toluene, xylene, m,p-dimethylbenzene, methyl sulfide, dimethyl disulfide, formaldehyde) (Fig. 5) (Martínez-González et al. 2022). In view of that ammonia in the soil generally exists in the form of NH₃ and NH₄⁺, under certain conditions, NH₄⁺ can be converted into NH₃ volatilization. It is difficult to detect the concentration of ammonia in soil accurately, so the spatial

Table 1 Result of odor contribution to different odorous substances in soil gas

Compound	Measured (mg/m ³)	Odor threshold (mg/m ³)	OAV _i	P _i (%)	ΣP _i (%)
Benzene	306.16	9.41	32.54	31.86	31.86
Ethylbenzene	2.52	0.09	28.01	27.43	59.30
Ammonia	3.44	0.23	14.93	14.63	73.92
Toluene	3.60	0.40	9.01	8.82	82.75
m,p-Xylene	3.80	0.43	8.83	8.64	91.39
Methyl sulfide	0.03	0.01	3.71	3.63	95.02
Dimethyl disulfide	0.02	0.01	2.06	2.01	97.04
Formaldehyde	1.19	0.67	1.77	1.74	98.77
o-Xylene	1.09	1.32	0.83	0.81	99.58
Carbon disulfide	0.25	0.58	0.43	0.42	100.00
			102.11	1.00	

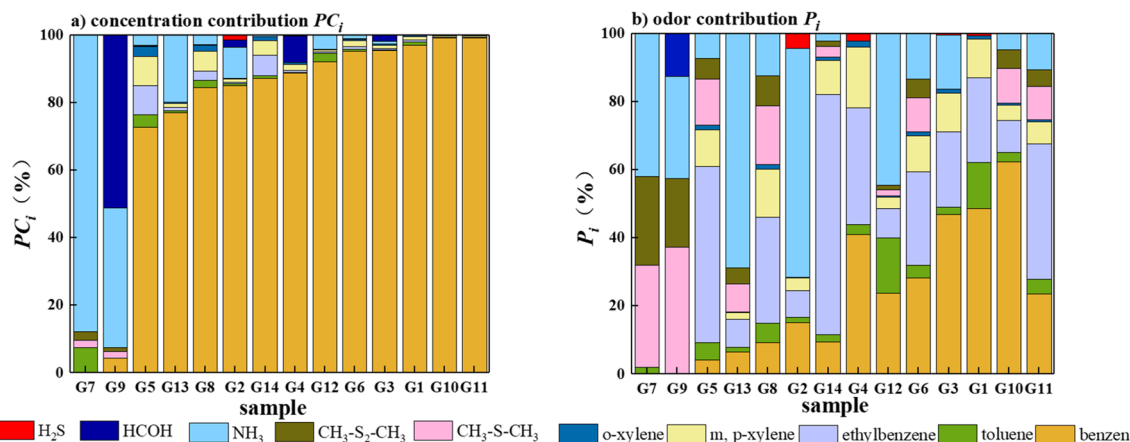


Fig. 4 Percentage of concentration contribution PC_i for the total chemical concentrations (a) and percentage of odor contribution P_i for the calculated odor concentration SOAV (b) of each odorous chemical in the soil gas

distribution of ammonia in soil is not included here. The statistical results of the key odorous substance content data are shown in Table 2. The maximum content values of benzene, toluene, xylene, m,p-dimethylbenzene, methyl sulfide, dimethyl disulfide, and formaldehyde in the soil were 3018.73, 163.65, 248.71, 121.81, 51.30, 88.70, and 46.18 mg/kg, respectively. The standard deviations were 460.24, 25.01, 37.86, 19.23, 9.19, 13.86, and 8.09 mg/kg, respectively. It can be seen that the content of odorous substances in different sampling points varied greatly, which showed that the distribution of pollutants in this site is extremely uneven.

As shown in Fig. 5, the key odorous substances were mainly present near the phosmet production workshop and the drainage ditch. Comparatively, the odorous substance content was low or even not detected in the area surrounding the calcium plastic machine repair workshop, the phosphate warehouse, and the phosphate ore yard. Overall, the raw and auxiliary materials and their degradation products in the production process are the main sources of odorous substances in the soil, and the horizontal distribution of the pollution has a significant correlation with the production process and layout of the pesticide-contaminated site. For example, formaldehyde is the main raw material in the production process of phosmet. The BTEX (benzene, toluene, ethylbenzene, and xylene) are the main solvent in the production process of pesticide such as dimethoate, butachlor, and trichlorfon. Moreover, the dimethyl sulfide and dimethyl disulfide in soil are mainly distributed in the phosmet production workshop and its downstream drainage ditch. The workshop has been used to produce products such as phosphorus pentasulfide that are the main raw materials in the production process. The phosphorus pentasulfide could enter the soil environment through leakage and then gradually convert into dimethyl sulfide and dimethyl disulfide through chemical and biological degradation (Zhang et al. 2013).

In addition, the soil odor pollution in the south is more severe than that in the north, which is mainly due to the impact of adjacent land plots. The surroundings of the site include the following: to the west of the site are small streams and residential areas; to the east of the site are the temporary warehouse, garbage dump, etc.; to the north side of the site are the ore raw material warehouse, phosphorus warehouse, phosphorus trichloride workshop, bucket washing yard, etc.; and to the south side of the site are the previously used workshops for methylamine, dimethoate, sulfide, and butachlor which have now been converted into residential buildings. Therefore, the southern side of the site is severely affected and polluted by the adjacent former methylamine workshop, dimethoate workshop, sulfide workshop, parka workshop, and butachlor workshop on the south side of the site.

Figure 6 illustrates the vertical distributions of odorous substances in the soil profiles of boreholes S10, S18, S26, X6, X7, and X10, which were located in the area surrounding the phosmet production workshop and the drainage ditch. During site sampling, the odor of the soil in these holes was very obvious. In particular, S10 with a strong odor showed very high benzene concentration levels, among which the highest concentration (304.73 mg/kg) was at the depth of 1.8–2.4 m. As shown in Fig. 1S, the relationship between benzene concentration and depth at borehole S10 shows two concentration peaks at depths of 1.8–2.4 m and 7.0–8.0 m, with concentrations of 304.73 mg/kg and 294.99 mg/kg, respectively. Consistent with the pollution characteristics of other boreholes, the odorous substances were generally concentrated in the sludge layer (1–3.6 m) and the silty clay or clay layer (5.6–11 m) of the site. The clay layer is the main aquiclude with a relatively compact soil structure and measured permeability coefficient between 2.88×10^{-7} and 9.79×10^{-6} cm/s (Table S2). This layer can

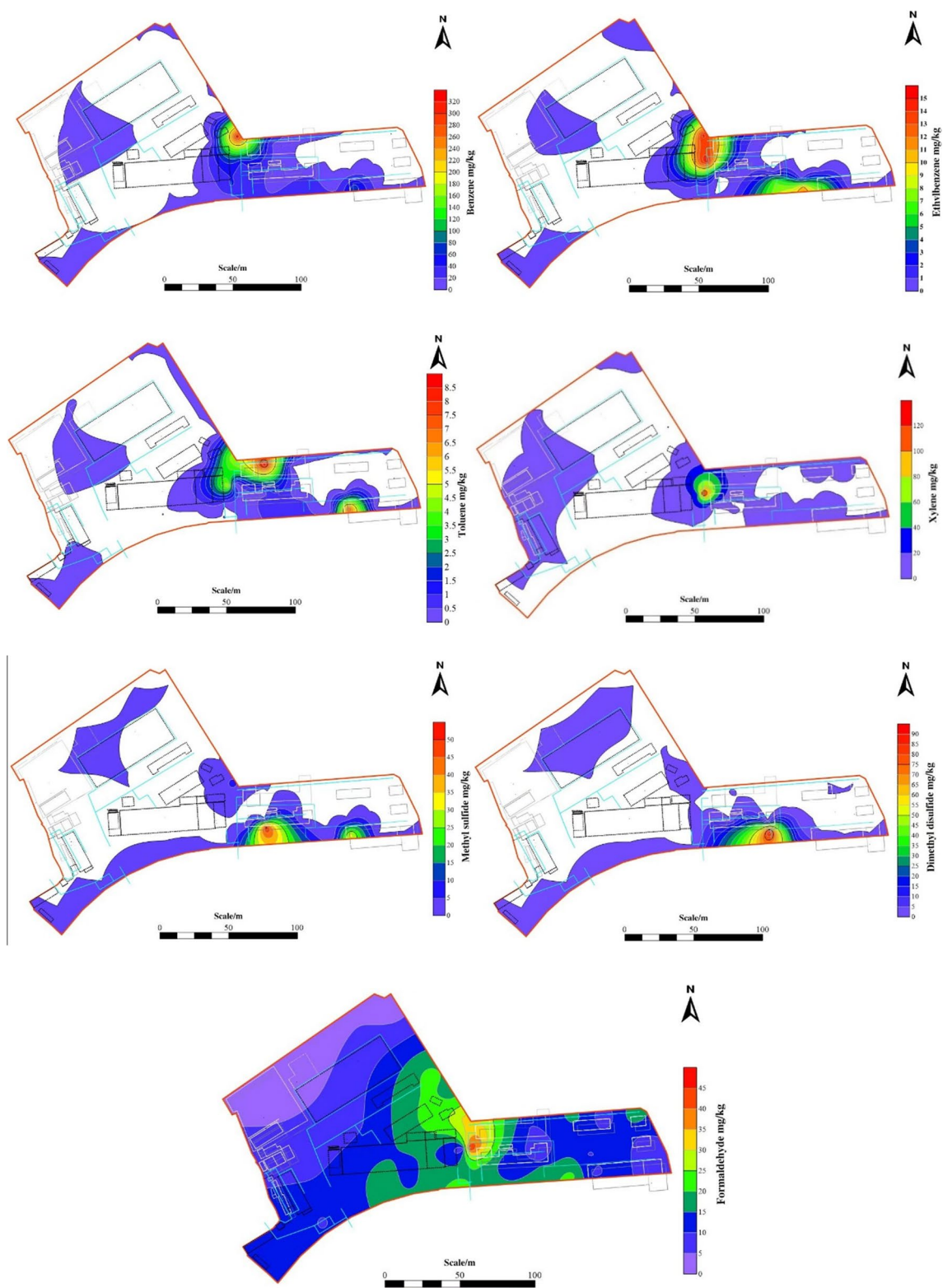


Fig. 5 Horizontal distribution of key odorous substances in the soil of the pesticide-contaminated site

Table 2 Descriptive statistics of main odorous substance contents in the soil of the pesticide-contaminated site (mg/kg)

Key odorous substances	C_{\max}	C_{mean}	C_{\min}	Detection rate	Standard deviation
Benzene	3018.73	87.91	0	55.20%	460.24
Ethylbenzene	163.65	5.60	0	30.51%	25.01
Toluene	248.71	6.56	0	45.76%	37.86
m,p-Xylene	121.81	5.42	0	33.41%	19.23
Methyl sulfide	51.30	2.40	0	17.68%	9.19
Dimethyl disulfide	88.70	3.36	0	30.27%	13.86
Formaldehyde	46.18	14.03	1.48	100%	8.09

block the vertical migration of odorous substances through soil capillary force and adsorption, resulting in the accumulation of pollutants. The sludge layer has a high organic matter content and a lower permeability coefficient, ranging from 2.18×10^{-8} to 8.13×10^{-6} cm/s (Table S2), which also inhibited the migration and diffusion of pollutants to some extent. In contrast, due to the loose structure of the sandy layer, odorous substances can diffuse and migrate in it. The vertical distribution generally showed a trend of increasing first and then decreasing. This result is consistent with the existing view (Liu et al. 2015; Yan et al. 2022). For example, Liu et al. explored the DDT vertical migration in pesticide-producing sites, and the result showed that DDT migrated more slowly due to poor permeability in silty clay or clay (Liu et al. 2015).

Over the decades, the odorous substances have penetrated from the surface layer to deep layer of soil, and the maximum contamination depth of soil reached 13 m. The depth of groundwater level in this site is shallow (0.40–2.31 m). When there is no anthropogenic disturbance, the odorous substances in contaminated saturated soil could threaten human health through vapor intrusion (McHugh et al. 2017). Meanwhile, due to the low content of vapor intrusion, the concentration of odor may not be enough to reach the human olfactory threshold. When the contaminated soil is excavated, a large amount of odorous substances will evaporate into the air and can produce strong pungent odors. Therefore, it is necessary to detect concentration of the key odorants in saturated soil layers, to provide reference for the effective control of odorous substances in the remediation process of pesticide-contaminated site. Especially for highly toxic BTEX (benzene, toluene, ethylbenzene, and xylene) or sulfides compounds, targeted protective measures could be taken to prevent soil gas poisoning accidents.

Relationship of odorous substances in soil and in soil gas

According to the soil gas detection results, most of the odorous substances in the site were benzene (more than 70% in soil gas). Therefore, benzene was selected as the representative contaminant to analyze the accuracy of the linear model and

the DED model in predicting the concentration of odorous substances in soil gas, and ethylbenzene was selected as a comparative study. The data pairs of the detected soil accounted for approximately 55.20% of the total and were mainly concentrated around the phosmet production workshop and the drainage ditch, and then, the data pairs with soil concentration below the detection limit were removed. It has been reported that different soil matrices have escape behaviors that differ substantially; the soil matrix type affects the fit (Zhang et al. 2019). Considering that the odorous substances were generally concentrated in the sludge layer and the silty clay or clay layer, so in this study, the soil samples corresponding to soil gas are basically clay or sludge soil (Table S1). Figure 7a shows the correlation between the concentration of benzene in soil gas (C_{sg}) and the concentration of benzene in soil (C_{s}), and the correlation coefficient R^2 can reach 0.93. Figure S2 shows the correlation of ethylbenzene in soil gas and soil, and the correlation coefficient R_e^2 can reach 0.92. Overall, whether it is benzene or ethylbenzene, as the concentration of benzene C_{s} increases, the measured C_{sg} also increases.

Figure 7b shows the logarithmic relationship between C_{sg} and C_{s} . The black dots represent the measured concentration of benzene in soil gas in this study, the blue dots represent the concentration predicted by the linear model, and the red dots represent the concentration predicted by the DED model. The dashed red line represents the soil gas odor threshold of benzene. Figure 7b clearly shows that only two of the 14 odorous soil gas samples did not exceed the benzene odor threshold (9.41 mg/m^3), and the maximum concentration was 2570.0 mg/m^3 , 273.1 times higher than the odor threshold. The soil gas concentrations predicted by the linear model are higher than the measured values. In contrast, the results of the DED model are generally lower than the measured results, which agrees with existing research results (Smith et al. 1990). It is worth noting that the results of laboratory simulation researches indicated that the DED model can better predict the concentration of VOCs in soil gas than the linear model (Kan et al. 1997, 1998, 2002). Reasons for this could be that the soil characteristics (such as soil moisture content, organic matter content, and volumetric air content) may influence the application of the DED model to the field (Zhang et al. 2019). For example, Zhang et al. collected 128 sets of soil and soil gas

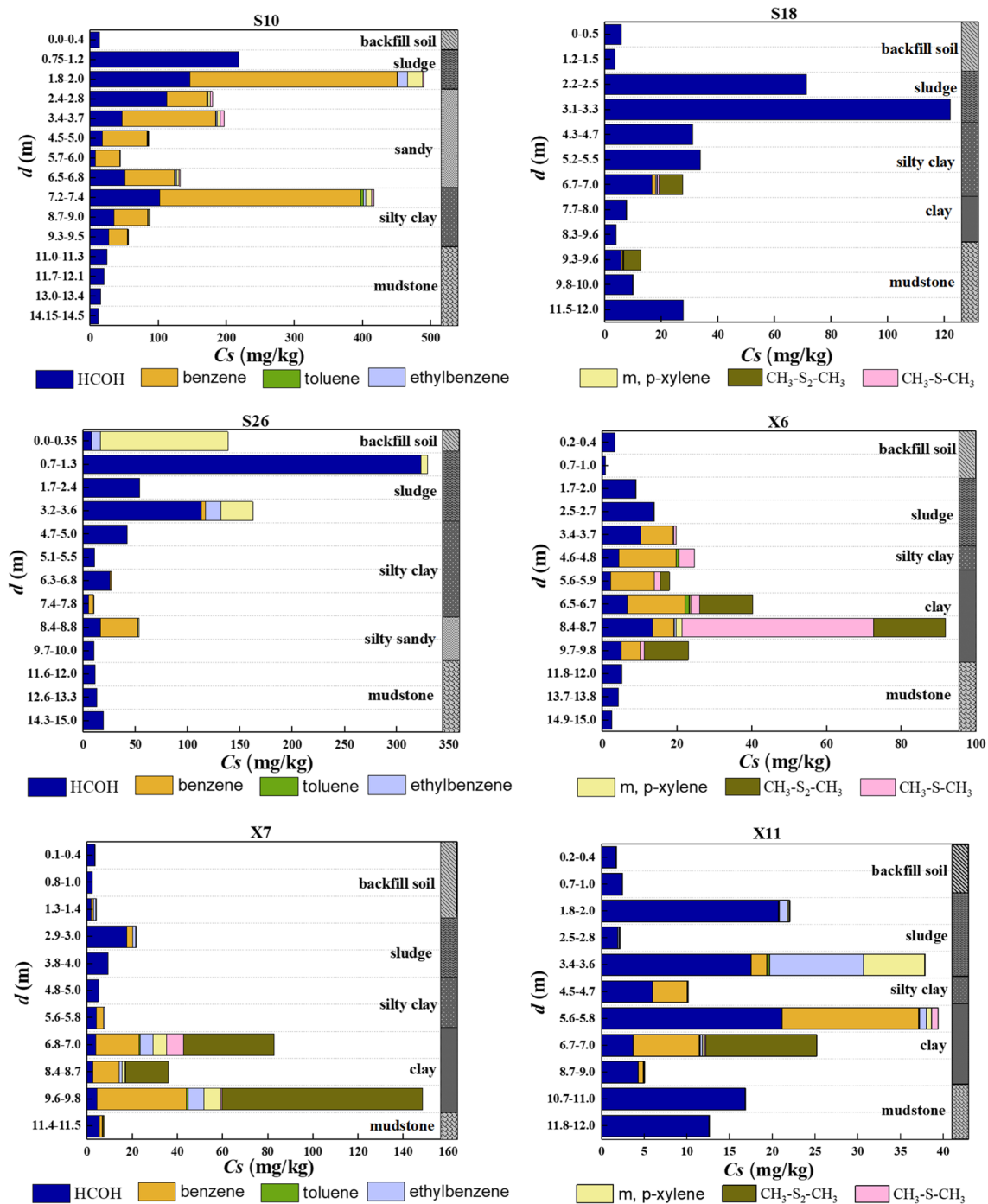


Fig. 6 Vertical distribution of odorous substances at pesticide-contaminated site

samples from 5 contaminated sites and then found that linear model results are closer to the measured soil gas concentrations, and the DED model may underestimate risks (Zhang et al. 2019). Therefore, the applicability of linear model and DED model differs under laboratory and field conditions, and the linear model was more suitable to predict the concentration of soil gas in contaminated site.

Limitation and application

Previous researches have pointed out that soil moisture greatly affects the vapor exposure concentration of VOCs in the environment (Chiou and Shoup 1985). On the one hand, soil moisture greatly affects the partitioning of VOCs in the soil environment; on the other hand, soil moisture

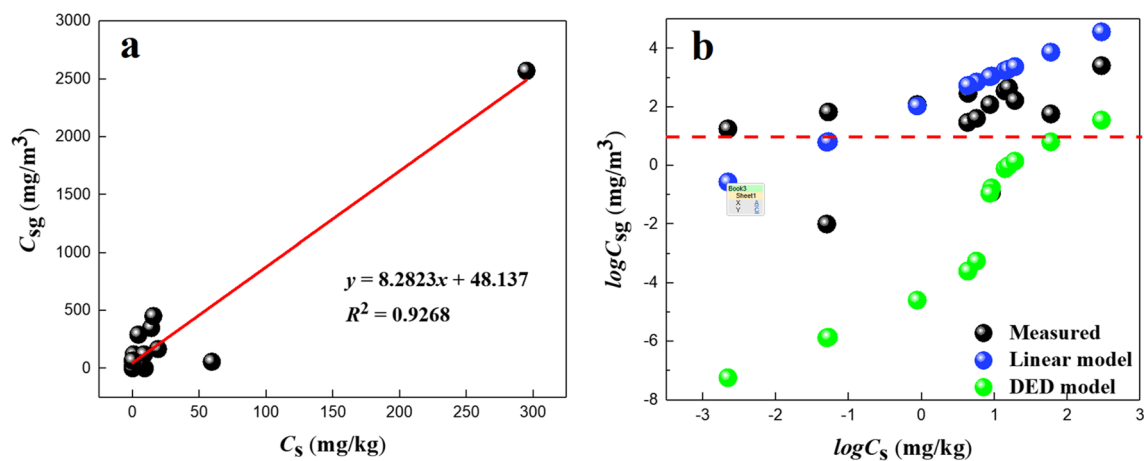


Fig. 7 **a** Relationship between concentration of benzene in soil and in soil gas. **b** Based on the measured soil concentrations of the points where benzene was detected in the soil, soil gas concentrations were predicted by the linear model and the DED model

affects the VOC migration (Zhang et al. 2022). According to the study of Zhang et al., the experimental results indicate that the vapor exposure concentration decreases gradually to 0 when soil relative saturation is above 0.8 (Zhang et al. 2022). Therefore, in the absence of anthropogenic disturbance, the presence of groundwater in the saturated soil results in very low vapor exposure concentrations of odorous substances in the soil to the gaseous environment (McHugh et al. 2017). Once the saturated soil is exposed to the air due to excavation, the soil moisture is significantly reduced and the VOCs through the pathways of vapor phase diffusion and advection into the air. Then, the vapor concentrations of odorous substances increase sharply, producing unpleasant gases that pose a threat to human health. Therefore, it is very meaningful to use the saturated soil volatile gas sampling device proposed in this study to simulate the diffusion and advection of odor pollutants into the air concentration of saturated soil under excavation exposure environmental conditions. However, due to the small volume of the sampling device and the limited amount of soil samples collected, the detection concentration of soil gas samples collected by this device is not equivalent to the concentration of odor pollutants in the air when the soil excavation of the site is exposed to the air. Certainly, the concentration of odor pollutants in the air caused by soil excavation is also a subject worth further longitudinal study.

This study suggests that the measured concentration for odorous compounds volatilized from soil can be used to estimate the potential odor impact of the saturated layer soil in the pesticide-contaminated site. Moreover, models could be used to evaluate the odor concentrations in soil gas from those in soil. The two approaches of odor investigation, chemical analysis of saturated soil gas or prediction by the model, should be used combined in practical

work. According to this paper and existing literature, the models have a good fit under strict conditions in laboratories (Chen et al. 2010; Kan et al. 1998; Zhang et al. 2019), but with a poor fit to the experimental data in field conditions. Overall, it is best to use chemical analysis of saturated soil gas to measure polluted concentration for odorous compounds.

Considering the pollutants that each model is suitable for simulating may be different, the performance results of the ethylbenzene with the linear and DED model are shown in Figure S2. By comparing the performance of two models for different odorants, the linear model results are generally closer to the measured soil gas concentrations than the DED model. Moreover, the results of the linear model are mostly greater than the measured results, but the results of the DED model are lower than the measured results. Due to the sensitivity and complexity of odor pollution, the DED model with lower prediction results is not recommended for predicting odor pollution concentration of soil gas in pesticide-contaminated sites which are often heavily polluted. In addition, before quantitatively evaluating the odor pollution, qualitative analysis of the composition of pollutants in soil gas should be conducted first to determine the possible pollution area and depth. Researches showed that contamination depth of soil has a significant correlation with the production duration, soil permeability, and properties of contaminants. The pesticide production sites that had been in production for > 30 years have a depth of contamination of > 10 m, while those produced for < 20 years have a depth of contamination of < 8 m (Li et al. 2023). Therefore, the saturated soil layer of the pesticide production site that has been production for a long time is highly likely to have odor pollution, where the soil gas sampling or prediction of odor pollution concentration in gas by the linear model should be conducted.

Conclusions

In this study, odor gas samples from a typical pesticide-contaminated site with shallow groundwater were collected by designed sampling device and measured by GC and IC. The results showed that the odorous substances included benzene, toluene, xylene, methylbenzene, ethylbenzene, methylene sulfide, dimethyl disulfide, formaldehyde, carbon disulfide, and ammonia. Evaluation of the OAV revealed that benzene (32.54%), toluene (28.01%), o-xylene (14.93%), m,p-xylene (9.01%), ethylbenzene (8.83%), carbon disulfide (3.71%), dimethyl disulfide (2.06%), and methyl sulfide (1.77%) contributed the most of the odor at the pesticide-contaminated site. Finally, soil gas concentrations predicted by the linear model and the DED model were compared with measured values, and the result showed that the prediction result of linear model was more accurate. Moreover, the DED model with low prediction result is not recommended to predict the concentration of soil gas for sensitive pesticide-contaminated sites.

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Author contribution T. Y. designed and performed the experiment and wrote the manuscript. J. T., C. X., L. L., and C. H. helped in the pot experiments and analyzed the samples. Z. W. and J. C. provided advice on the design of the research. G. L. provided advice on the design of the research, analyzed the data, and edited the manuscript.

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Data availability The datasets used and analyzed during the current study are available from the corresponding author.

Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare no competing interests.

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