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Adsorption of Volatile Organic Compounds on Soil and Prediction of Desorption Breakthroughs

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ABSTRACT

Vapor extraction of volatile organic compounds from soil was investigated by performing adsorption and desorption experiments of trichloroethylene, trichloroethane, and chlorobenzene on soil particles. The adsorption breakthrough curves were obtained using a dynamic response technique based on frontal analysis chromatography. The shape of the breakthrough curves indicated that the adsorption process was close to an ideal adsorption system of no mass transfer resistance, no axial dispersion, and infinitesimal width of mass transfer zone. The adsorption isotherms were BET Type I for trichloroethylene and trichloroethane, and BET Type II for chlorobenzene. Two types of desorption profiles were observed depending on the compounds, i.e., the continuously decreasing profiles of trichloroethylene and trichloroethane and the stepwise decreasing profiles of chlorobenzene. The desorption profiles of trichloroethylene and trichloroethane were simulated using a local equilibrium theory which indicated that desorption behavior was independent of the number of adsorption layers on soil. For chlorobenzene, the monolayer desorption was the rate-controlling step of the overall desorp-

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tion process. The effect of moisture on desorption efficiency was significant for chlorobenzene, which showed more unfavorable desorption behavior than trichloroethylene and trichloroethane.

INTRODUCTION

The remediation technologies proposed for decontamination of soil include destruction-based methods such as incineration and biodegradation, and separation-based methods such as thermal stripping, solvent extraction, and soil vapor extraction (1). Selection of a particular technology for soil cleanup depends on the type of contaminant, the configuration of the target site, and the quantity of soil treated. A small volume of soil contaminated with heavy metals, for example, can be excavated and treated by solvent extraction using a proper agent.

One of the major contaminants for soil together with leaching water is volatile organic compounds (VOCs), mainly from the leakage of underground storage tanks. Soil vapor extraction, also known as air stripping and soil venting, has been successfully implemented to remove VOCs from soils (2–6). The technology involves desorption of contaminants from soil particles to the soil gas-phase in the presence or absence of moisture. The advantages of vapor extraction of soil over the conventional technologies are: 1) it creates minimum site disturbance, 2) its cost is reasonable compared to excavation methods, 3) it effectively reduces the contaminant concentration in the vadose zone and minimizes the risk of further contaminant migration, 4) coupled with air-sparging, it can be applied to remediate saturated and unsaturated zones, and 5) it stimulates biodegradation, also called bioventing (7).

In this technology the removal of VOCs is performed by mechanically venting air through the soil layer and forcing the contaminants to desorb. The vapor extraction of soil can be considered to be a simple vaporization process combined with flow in porous media. The technology, however, involves the phenomenon of adsorption/desorption on soil particles. Therefore, an understanding of the thermodynamic and kinetic aspects of the sorption of VOCs is a necessary step in formulating the interaction existing between the contaminant and soil and in the development of the technology base.

The adsorption and desorption of VOCs on soil has been investigated extensively using a wide variety of soil matrices (8–10). The results of the studies, however, often show inconsistent conclusions which can be explained by the complex and heterogeneous nature of soil. Poe et al. (11) measured the adsorption isotherms of various VOCs on different types of dry soils using a static vapor adsorption apparatus. Thibaud et al. (12,

13) studied the adsorption of toluene and chlorobenzene on soil in the presence of water, and showed that the VOCs adsorption isotherms became progressively unfavorable as the relative humidity increased. Campagnolo and Akgerman (14) investigated the adsorption of *n*-hexane and nitrogen onto soil constituents and their mixtures, and found that in all cases the adsorption isotherms had the BET Type II sigmoid shape, identical to the isotherms on soils. The literature on VOCs adsorption was recently reviewed by Thibaud-Erkey et al. (15). Amali et al. (16) modeled the competitive adsorption of multicomponent volatile organic and water vapor on soils. Petersen et al. (17) examined the effects of differences in texture and soil-water content on vapor partition coefficients for trichloroethylene. Recently, Petersen et al. (18) studied the transient diffusion, adsorption, and emission of volatile organic vapors in soils with fluctuating low water contents.

The objective of this study was to investigate the behavior of adsorption and desorption of VOCs on soil specially obtained from a VOC-contaminated industrial site. The adsorption isotherms of VOCs on dry soil are determined by using a dynamic response technique based on frontal analysis chromatography, and the data are modeled by the BET theory. The vapor extraction is performed by desorbing the contaminants from loaded soil using a gas stream, nitrogen in this work, and the desorption profile is modeled by the local equilibrium theory. The effect of moisture in the vapor on the desorption behavior is also investigated.

EXPERIMENTAL METHODS

Dynamic Response Technique

A dynamic response technique based on frontal analysis chromatography is used to obtain the adsorption and desorption profiles of VOCs on soil particles. Details of the experimental technique were reviewed by Rhue and Rao (9). For the adsorption process the concentration of the VOC of interest in the vapor phase gives a positive step change at the inlet of a soil bed, and the responding effluent concentration is monitored as a function of time at the outlet of the bed. The adsorption is considered to be completed when the effluent concentration is equal to that of the inlet stream. The resulting breakthrough curve is used to obtain the amount of the VOC adsorbed on the soil using a mass balance. If m_i and m_o are the masses of a VOC entering and exiting the soil bed, respectively, then the mass of adsorbed VOC becomes

$$m_{\text{adsorbed}} = m_i - m_o \tag{1}$$

The mass of adsorption in time *t* is calculated using the inlet concentration

C_i, effluent concentration C, volume flow rate of vapor phase V, and molecular weight of VOC M:

$$m_{\text{adsorbed}} = MVC_i \left[\int_0^t \left(1 - \frac{C}{C_i} \right) dt \right] \tag{2}$$

Integrations of the breakthrough curves obtained at various inlet concentrations enable determination of the adsorption isotherm. After the adsorption process is completed, VOC-free gas is introduced at the inlet of the bed, and the effluent concentration is measured to obtain the desorption profile. Similarly, integration of the desorption curve gives the mass of desorption.

Adsorption and Desorption Experiment

The soil used in this study was obtained from the Panfex Plant site, located in north Texas. The soil was dried in the oven and screened to the desired size. The average particle diameter of soil was 230 μm, and the BET surface area of the particle was 3.885 m^2/g soil. For the experiments, 21.5 g of soil was charged in the soil bed (7.75 mm ID, 9.5 mm OD, 310.0 mm length). The soil bed was heated up to 150°C between each experiment to ensure complete desorption of the adsorbed species. VOCs investigated in this study were trichloroethylene (TCE), trichloroethane (TCA), and chlorobenzene. All the chemicals were purchased from Aldrich Co., Deerfield, IL, and used without further purification. Nitrogen was used as the gas-phase carrier for the VOCs in the adsorption process and to extract VOCs in the desorption process.

Figure 1 shows the experimental apparatus used in this study. Prior to the adsorption experiment, nitrogen, whose flow rate was controlled by mass flowmeter M1, was saturated with a VOC by bubbling it through the saturator S1. The flow rate of pure nitrogen was controlled by adjusting the mass flowmeter M2, resulting in the variation of VOC concentration of the inlet stream. Therefore, a vapor stream with the desired concentration of a contaminant could be introduced to the soil bed. During the above procedure for adjusting inlet concentration, the vapor stream passed through a bypass line to determine the inlet concentration. To start the adsorption, the stream was switched from the bypass to the soil bed which generated the positive step change of inlet concentration. The flow rate of the vapor stream flowing through the bed was maintained at 1.2 mL/s. The effluent stream was analyzed using an on-line sampling valve (Valco) and a gas chromatograph (SRI, model 8610) equipped with a flame ionization detector and a Poropaq P (Alltech) column. The adsorption experiment was completed when the effluent concentration became constant and equal to the inlet concentration.

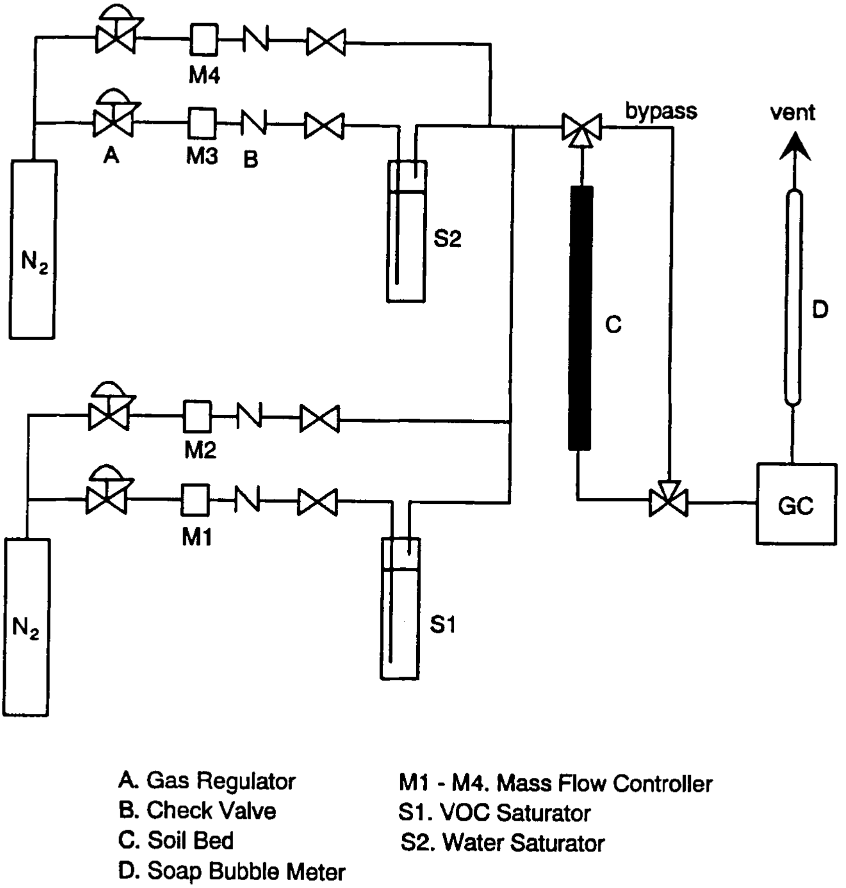

FIG. 1 Experimental apparatus for adsorption and desorption of VOCs on soil particles.

Two sets of desorption experiments were performed. The first one was desorption by a dry nitrogen stream and the second was wet desorption, i.e., desorption by a nitrogen stream of 100% relative humidity. For dry desorption, pure nitrogen was introduced to the VOC-saturated soil bed and the effluent concentration was measured until the detection limit was reached. For wet desorption, nitrogen was saturated with water by bubbling it through the saturator S2 and introducing it to the soil bed. The effect of moisture in vapor extraction was investigated by comparing the desorption of soils identically contaminated with dry nitrogen and with water-saturated nitrogen. The flow rates of pure and water-saturated nitrogen were controlled in the 1.2 to 1.3 mL/s range using mass flowmeters M4 and M3, respectively.

RESULTS AND DISCUSSION

BET Analysis of Adsorption Isotherm

The adsorption isotherm is represented as the mass of contaminant adsorbed per unit mass of soil X as a function of vapor concentration. The BET theory is a model isotherm to account for multilayer adsorption, and this model is used to extract the monolayer adsorption capacity and hence the specific surface area. A number of refinements to the BET model have been developed; however, the basic BET method remains the most widely used technique (19). Brunauer et al. (20) proposed an isotherm equation based on the assumption that multimolecular adsorption is induced by the same forces that produce condensation, and it occurs in case the number of adsorbed layers is limited. The equation is valid for the whole range of relative vapor concentrations and is known as the BET three-parameter equation:

$$\frac{X}{X_m} = \frac{cy}{1 - y} \frac{1 - (n + 1)y^n + ny^{n+1}}{1 + (c - 1)y - cy^{n+1}} \tag{3}$$

where y is the relative vapor concentration, X_m is the monolayer adsorption capacity (that is, the amount of contaminant required to complete the monolayer coverage per unit mass of soil), c is the BET constant, and n is the theoretical number of adsorbed layers.

Figure 2 shows a typical adsorption breakthrough curve of trichloroethylene at 24°C. The ordinate is the ratio of effluent concentration C to the concentration of the saturated vapor stream C_{sat}, and the abscissa is the volume of vapor passing through the soil bed. Figure 2 is a record of effluent concentration responding to the two positive step changes of inlet concentration. For the first step change, the inlet concentration was changed from zero to $0.14C_{sat}$. After the breakthrough was completed, the second step change was made from $0.14C_{sat}$ to $0.28C_{sat}$. The breakthrough curve is very steep and sharply increases from initial concentrations to final stabilizing concentrations, where the vapor and soil phases reach equilibrium. The shape of the breakthrough curve indicates that the adsorption of trichloroethylene is close to an ideal adsorption system of no mass transfer resistance, no axial dispersion, and infinitesimal width of mass transfer zone. The shapes of the breakthrough curves of trichloroethane and chlorobenzene were about the same in the inlet concentration ranges up to $0.36C_{sat}$. The similar shape of the three curves implies the substantially identical adsorption behavior of the three compounds in the low concentration range. Figure 3 shows the breakthrough curve of chlorobenzene for the two-step changes of inlet concentrations from zero to $0.67C_{sat}$ and to $0.91C_{sat}$. The extended shape of the curve indicates the

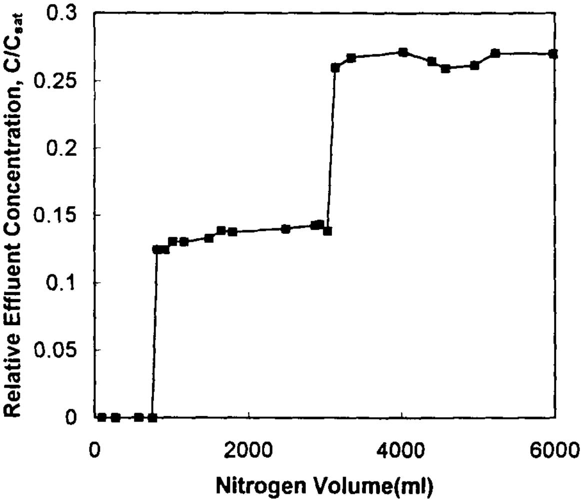

FIG. 2 Adsorption breakthrough curve of trichloroethylene at 24°C, responding to the two-step changes of inlet concentration from zero to $0.14C_{sat}$ and to $0.28C_{sat}$.

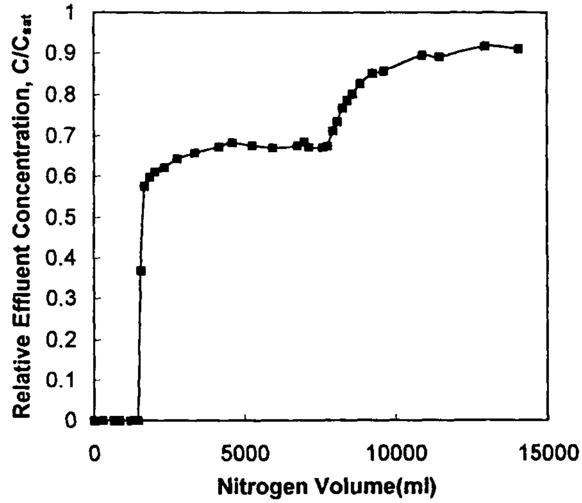

FIG. 3 Adsorption breakthrough curve of chlorobenzene at 24°C, responding to the two-step changes of inlet concentration from zero to $0.67C_{sat}$ and to $0.91C_{sat}$.

broad mass transfer zone in the bed due to the increased mass transfer resistance in the high concentration range, where the major adsorption occurs in multilayer form. Trichloroethane exhibited similar behavior. We believe that the major mass transfer resistance is the intraparticle diffusion.

The breakthrough curves obtained at various inlet concentrations were analyzed by Eq. (2) to calculate m_{adsorbed}, which was divided by the weight of soil in the bed to give the adsorbed amount per unit mass of soil, X. The experimental data of trichloroethylene, trichloroethane, and chlorobenzene were used to model the adsorption isotherm equation, Eq. (3). Figures 4 to 6 show the experimental and the modeled adsorption isotherms of the three compounds. The shapes of the isotherms correspond to the BET Type I isotherms for trichloroethylene and trichloroethane, and to the BET Type II isotherm for chlorobenzene. Although many investigators (15) observed Type II isotherms for most VOCs on different sorbents, our data indicate Type I for trichloroethylene and trichloroethane. It should be noted that our data extend to a C/C_{sat} of 0.8, and the trend indicates that the isotherm may bend upward. However, for the data range studied, the observed isotherm is better represented as Type I. The adsorption isotherms can be extrapolated to the saturation adsorption capacities X_a, which are obtained when the inlet vapor stream is saturated with

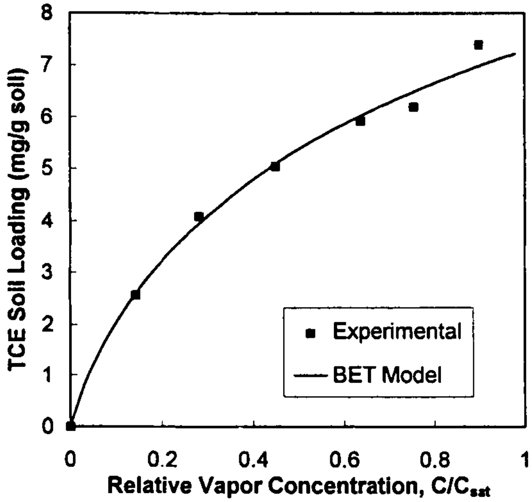

FIG. 4 Adsorption isotherm of trichloroethylene on soil at 24°C. The line corresponds to the BET Type I isotherm.

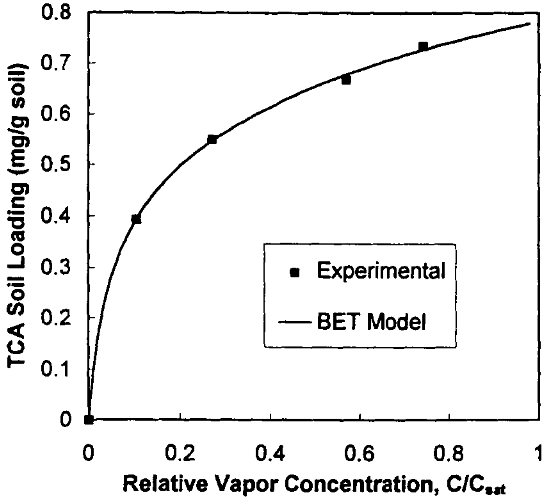

FIG. 5 Adsorption isotherm of trichloroethane on soil at 24°C. The line corresponds to the BET Type I isotherm.

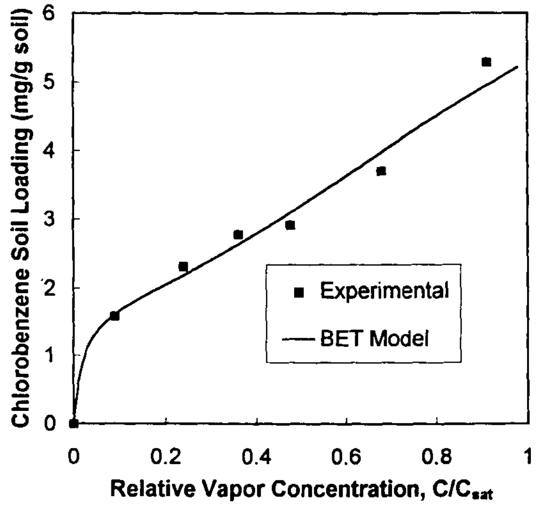

FIG. 6 Adsorption isotherm of chlorobenzene on soil at 24°C. The line corresponds to the BET Type II isotherm.

a contaminant and reaches equilibrium with the soil. X_a corresponds to the maximum amount of contaminant that can be adsorbed per unit mass of soil. Table 1 shows the calculated values of the isotherm parameters of Eq. (3) and the saturation adsorption capacity X_a for each compound. The saturation adsorption capacity exceeded the monolayer adsorption capacity by a factor of 1.59 to 2.99 for all the compounds. The saturation adsorption capacity increased in the order of trichloroethane, chlorobenzene, and trichloroethylene.

Local Equilibrium Theory for Desorption Profile

The local equilibrium theory (LET) is a simplified model which neglects all mass transfer resistances. As shown by the breakthrough curve of trichloroethylene, the system is close to an ideal adsorption system of negligible mass transfer limitation. Therefore, the theory is employed to model the desorption profile from the adsorption isotherm. The theory is based on the assumptions of 1) isothermal plug flow system, 2) negligible mass transfer resistances, 3) constant fluid properties, 4) no radial gradients, and 5) constant velocity. For such a system the differential fluid phase mass balance for the adsorbate leads to the equation

$$v \frac{\partial C}{\partial z} + \frac{\partial C}{\partial t} + \left(\frac{1 - \epsilon}{\epsilon}\right) \frac{\partial q}{\partial t} = 0 \tag{4}$$

where C is adsorbate concentration in the gas phase, v is interstitial gas velocity, ϵ is void fraction of bed, and q is adsorbate concentration in the soil particles. Using the methods of characteristic, Eq. (4) can be solved to obtain the concentration profile as a function of time (19):

$$t = \frac{L}{v}\left[1 + \left(\frac{1 - \epsilon}{\epsilon}\right) \frac{dq}{dC}\right] \tag{5}$$

The derivative dq/dC corresponds to the slope of the adsorption isotherm of each compound at a particular time t. The simulation was carried out

TABLE 1

Result of BET Analysis of Adsorption Isotherms at 24°C

	X_m (mg/g soil)	n	c	X_a (mg/g soil)	X_a/X_m
Trichloroethylene	4.11	2.71	7.36	7.28	1.77
Trichloroethane	0.49	2.21	22.12	0.78	1.59
Chlorobenzene	1.77	4.97	48.67	5.29	2.99

using Eq. (5) and the isotherm functionality with parameters in Table 1. The desorption profile of trichloroethylene was simulated and compared with experimental data, as shown in Fig. 7. The ordinate is the effluent concentration C, normalized by initial effluent concentration C_0. The soils loaded with the different initial contaminant concentrations were used, and the effluent concentrations were recorded as a function of nitrogen volume passed. It was observed that the normalized effluent concentration profiles collapse on almost a single curve regardless of the initial soil loading. It should be noted that the initial loading of 4.09 mg/g is less than the monolayer adsorption capacity, and 7.42 mg/g is close to the theoretical saturation adsorption capacity (see Table 1). This proves that the desorption behavior of trichloroethylene and trichloroethane is rather independent of the initial number of adsorption layers.

The desorption profile of chlorobenzene, however, exhibited different types of behavior depending on the initial loading, as shown in Fig. 8. The profiles at high initial loadings exhibit two down steps. The first down step is sharp, followed by a plateau, and then by a second step exhibiting a tail. The BET Type II isotherms that have an inflection point (also called the "knee" of the isotherm) are favorable to adsorption before the

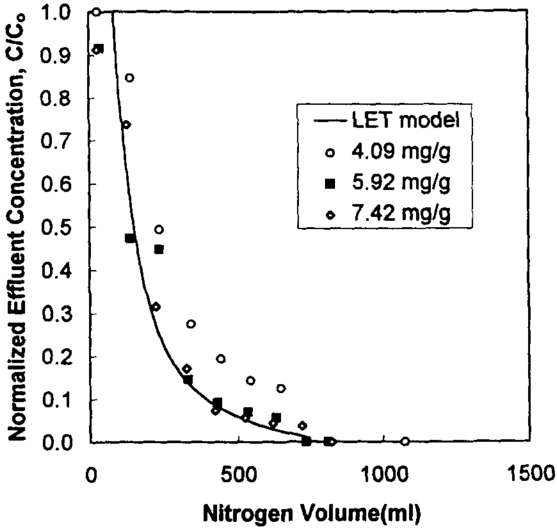

FIG. 7 Desorption profiles of trichloroethylene at various initial soil loadings. The concentration is normalized by the initial effluent concentration. The line corresponds to the LET simulation.

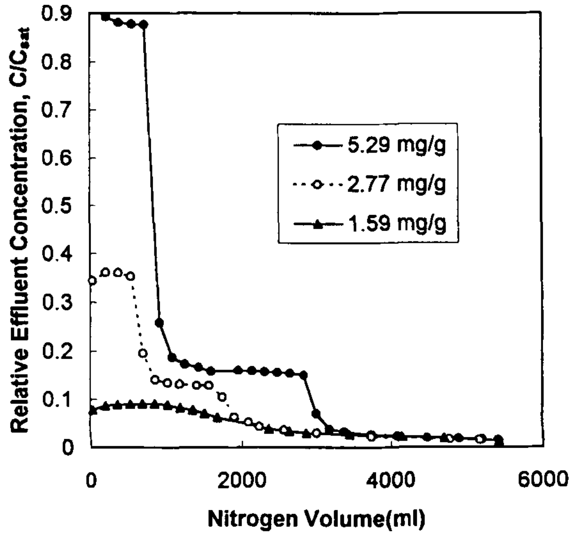

FIG. 8 Desorption profiles of chlorobenzene at various initial soil loadings.

inflection point (low concentrations) and unfavorable to adsorption after the inflection point (high concentrations). The inflection point is usually considered to indicate the completion of monolayer coverage. Type II isotherms that have inflection points are usually accompanied by a hysteresis phenomenon, in which case the desorption branch of the isotherm is not identical to the adsorption branch. Hysteresis appearing in the multilayer range of physisorption isotherms is usually associated with capillary condensation in a mesoporous structure. If desorption is started before the saturation pressure is reached, the desorption hysteresis is called the "secondary curve" or the desorption "scanning curve."

Numerous studies have shown the existence of hysteresis and/or scanning curves for systems such as nitrogen on alumina (21, 22), water on titania (23), silica gels (24, 25), cereals (26), and clays (27), and carbon tetrachloride on silica gel (28). Farrell and Reinhard showed the presence of hysteresis for the aqueous sorption of trichloroethylene on Santa Clara solids (5). Ritter and Yang (29) demonstrated that if the isotherm includes a hysteresis loop, the desorption profile for the elution of a bed saturated with an inlet concentration above the hysteresis loop closure exhibits a middle plateau. They related the length and steepness of the step to the shape of the hysteresis loop. In particular, the step occurred at the partial pressure corresponding to closure of the hysteresis. They only considered desorption from complete saturation, therefore, following the hysteresis loop. However, if the onset of desorption occurs prior to completion of the adsorption to saturation pressure, the corresponding desorption profile should follow a scanning curve instead of the hysteresis loop. Thibaud-Erkey et al. (30) recently showed that the plateau is indeed due to desorption hysteresis and developed a mathematical model for prediction of the desorption profiles that exhibit such plateaus. They showed that the desorption and adsorption branches of the isotherm combine at about the inflection point. The equilibrium is determined at low initial loadings by the adsorption branch of the adsorption/desorption isotherm and hence no plateau is observed.

For chlorobenzene, 1.59 mg/g loading (Fig. 8) corresponds to a point before the inflection point of the isotherm (Fig. 6), whereas both 2.77 and 5.29 mg/g loadings correspond to points beyond the inflection point of the isotherm. The adsorption isotherms of trichloroethylene and trichloroethane (Figs. 4 and 5) do not exhibit an inflection point (Type I isotherms), and it is highly unlikely that they exhibit desorption hysteresis, hence the desorption profiles do not have a plateau (Fig. 7).

Effect of Moisture on the Desorption Behavior

The effect of moisture on the desorption of trichloroethylene and trichloroethane was quite different from that of chlorobenzene. Figure 9 shows the typical desorption profiles of trichloroethylene and chlorobenzene obtained from the desorption of identically contaminated soil by dry nitrogen and water-saturated nitrogen. Table 2 shows the percent contaminant removals in dry and wet desorption after the same volume of nitrogen is used. For example, when the initial loading of trichloroethylene was 5.92 mg/g and 630 mL of dry and water-saturated nitrogen was passed through the bed, the removal was 43.2 and 46.5%, respectively. For trichloroethylene and trichloroethane, the percent removal improved a little in wet desorptions while a large increase was observed for chlorobenzene. The increased desorption is due to the adsorptive displacement of the contaminant by water molecules. The result shows that the presence of moisture significantly enhances desorption when the interaction of compound molecule and soil surface is strong as in desorption of chlorobenzene, while it has a minor effect on desorption of trichloroethylene and trichloroethane. In Fig. 9(b), the first down step in the desorption profile corresponds to multilayer desorption of chlorobenzene, and the two profiles for dry and wet desorption follow identical patterns. The desorption step after the middle plateau corresponds to the desorption of monolayer coverage of chlorobenzene, which is the rate-controlling step of the overall

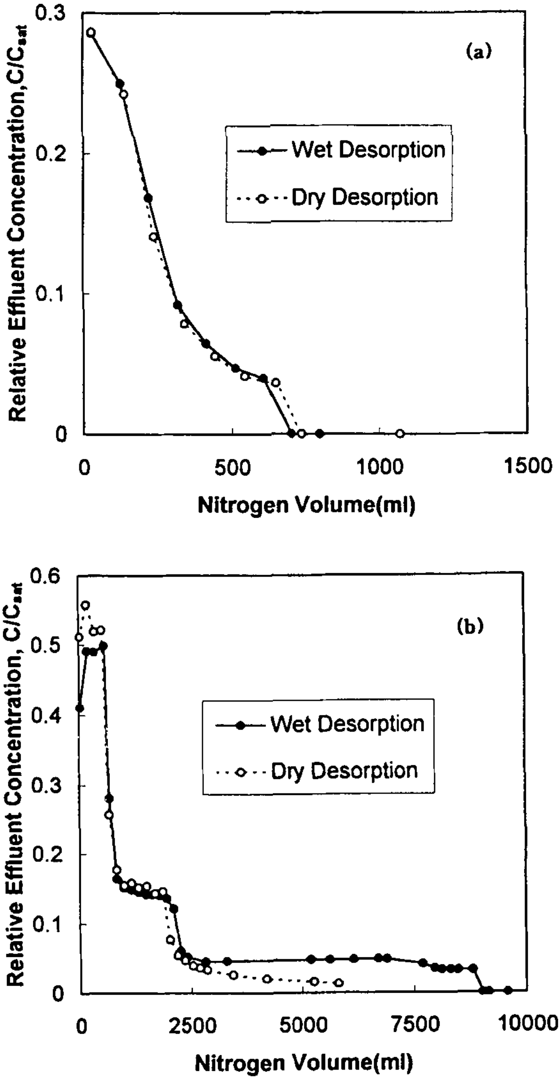

FIG. 9 Comparison of desorption profiles obtained by using dry and water-saturated nitrogen streams for (a) trichloroethylene and (b) chlorobenzene.

TABLE 2
Percentage VOC Removal by Dry and Wet Desorption after the Same Volume of Nitrogen Was Used

	Initial loading (mg/g)	Dry desorption (%)	Wet desorption (%)
Trichloroethylene	4.09	43.7	44.6
	5.92	43.2	46.5
	7.42	52.5	69.0
Trichloroethane	0.55	40.0	42.6
	0.66	38.8	42.1
Chlorobenzene	1.59	49.3	
	2.77	55.1	
	2.71		91.5
	5.29	67.1	

process. The results show that the presence of moisture accelerates monolayer desorption extensively. The present finding is consistent with the result of Thibaud et al. (13). They found that the effect of moisture on desorption could be predicted by considering the behavior of the adsorption isotherms to be a function of relative humidity in the gas phase. They showed that the dry isotherms of chlorobenzene were favorable to adsorption and, thus, unfavorable to desorption because, as the relative humidity increased, the isotherm became more favorable to desorption.

In conclusion, the adsorption isotherms provided fundamental equilibrium data between VOCs and soil particles, and showed that the process involved monolayer adsorption followed by multilayer adsorption. The vapor extraction process of VOCs from soil was investigated by performing desorption using dry and water-saturated nitrogen streams. Two types of desorption profiles were observed depending on the organic compounds, mainly due to the differences in the shapes of their adsorption isotherms. The effect of moisture on desorption efficiency was more significant for chlorobenzene than for trichloroethylene and trichloroethane.

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