

Volatilization Process of Toluene From Contaminated Soil Under Consideration of Co-Existence of Pore Water

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Abstract

The volatilization process of contaminants has been shown to play an important role in remediation of VOCs-contaminated soils. The aim of this paper is to study the volatilization characteristics of both toluene and water from soil, and to evaluate their interaction mechanism under different conditions. A test system is developed to measure mass loss of contaminants and water from soil by volatilization. It was found that basically the volatilization process could be divided into two stages in clay and one stage in sand. Two main influential mechanisms of water on the volatilization of toluene include molecules competitive adsorption and blocking of volatilization channels. In addition, the evaporation process of water was restricted by volatilization of toluene. Volatilization rate of toluene would increase with the increase of water content when the water content was low. But it would decrease with the increase of water content when the water content was higher than 15% in clay. In addition, there existed an optimal water content (15%) under which the maximum volatilization rate could be achieved.

1. Introduction

Leakages and spills of volatile organic compounds (VOCs), such as accidental spills from deliberate dumping, leaching of old landfills, underground storage tanks or abandoned gas stations, have posed serious threats to soil and groundwater resources(Alharbi et al. 2018, Ma et al. 2018, Zhao et al. 2019). Parts of leaked contaminants will enter into the atmosphere through volatilization process, and others will migrate into underground strata by infiltration. The migration and transformation processes of non-aqueous phase liquids (NAPLs) in the vadose zone are very complex, including adsorption, dissolution, biodegradation, diffusion, and especially volatilization(Comegna et al. 2019, Mobile et al. 2016, Schubert 2015). Part of VOCs in vadose zones migrates upward through pore channels of soil, and then volatilize into atmosphere(Kim &Corapcioglu 2003, Lu &Zhu 2012).

Volatilization has been shown to play an important role in VOCs vapor migration and transformation process in vadose zone. It depends not only on the soil conditions (e.g., water content and characteristic of soil) but also on the environmental conditions (e.g., wind speed, air relative humidity and temperature) and VOCs properties(Li et al. 2004, Okamoto et al. 2009, Tao et al. 2017). In recent years, Wei(Wei et al. 2018) and Chao(Chao et al. 2018) studied the importance of temperature and wind speed on volatilization process from soil. The higher the temperature and wind speed are, the greater the volatilization rate will be. Researchers also believed that the type of organic contaminants was one of the most important factors affecting the volatilization process(Bani-Hani et al. 2015, Galin et al. 1990). For example, diesel oil with high carbon content volatilizes more slowly than those with low carbon content(Ma et al. 2014). The concentrations of VOCs are often dozens or even hundreds of times higher than controlled values according to the national standard when the accidents of underground storage tanks and gas stations cause leakages(Alharbi et al. 2018, Makarenko &Budak 2017, Moshkovich et al. 2018). The change of contamination degree significantly influences the volatilization and migration process of VOCs. However, few studies have been done about the effect of initial concentration of VOCs on volatilization process from soil.

Due to the influence of groundwater supply and rainfall infiltration, the soil water content varies from time to time. Besides, the remediation process of contaminated site through different techniques such as soil vapor extraction and thermal remediation also can significantly change the distribution of water content in soil. Some researchers pointed out that the variation of water content would affect the volatilization and migration process of contaminants in soil(Gustin &Stamenkovic 2005). It was found that the increase of soil water content would decrease soil permeability, which then blocked the volatilization channels of VOCs and affected the volatilization process(Zhan et al. 2014). Similar results were reported by Zhang(2011) and Niu(2012). Furthermore, Poulsen(1998) indicated that there existed an competitive adsorption between water and VOCs molecules on the surface of soil particles. One possible explanation is that polar water molecules are more readily sorbed on soil particles than weakly polar toluene molecules(Tekrony &Ahlert 2001). Moreover, the process of volatilization of VOCs is accompanied simultaneously by the evaporation of water from soil. The interaction mechanism between volatilization of VOCs and evaporation of water is complex. Yet, some researchers(Song et al. 2018) often neglected the evaporation process of water and the interaction between water and VOCs. Thereby, little work is paid attention to the interaction between VOCs and water and respective volatilization process from soil.

In this paper, a test system is specially developed to measure the mass loss of toluene and pore water from soil by volatilization. Volatilization test is carried out under different water content and type of soil(clay and sand). Based on tested results, volatilization law and interaction between toluene and water are studied.

2. Materials And Methods

2.1 Materials

The clay used in this study was sampled from the depth of 1 ~ 3 m in Jiulonghu Campus of Southeast University, Nanjing city of China. The clay was first oven-dried under 105°C, and then crushed to particles passing the 2-mm mesh sieve. The sand samples used in the research were collected beside Yangtze river in Nanjing. The samples of sand were manually divided into coarse sand and fine sand in accordance with ASTM D2487(ASTM 2017). The basic physical properties and particle distribution of the clay and sand are given in Table 1 and Fig. 1. Median particle size (D_{50}) of coarse sand, fine sand and clay are 0.9, 0.19, and 0.026 mm, respectively. The value of coefficient of uniformity (C_u) and coefficient of curvature (C_c) are also shown in Fig. 1. Toluene (Analytical Reagent with purity > 99.5%) was obtained from CHRON CHEMICALS (Chengdu, Si Chuan Province). Distilled water was used for preparation of test samples. Table 2 summarizes the basic properties of toluene and water. Detailed water content, initial concentration of toluene and dry density of the prepared samples are shown in Table 3. First, clean sand or clay with different initial water content(w) were manually prepared, and then kept in sealed plastic bag for 24 hours to reach moisture equilibrium. After that, the samples were put into aluminium box whose height and diameter were 40 and 65 mm, respectively. The clay or sand was mixed in the aluminium box thoroughly. Dry density of the samples were basically controlled in the same value in all tests to study the

influence of various factors in volatilization process. At last, toluene was injected uniformly into the clay by a microinjector.

Table 1: Basic properties of the tested soils

Parameters	Clay	Fine sand	Coarse sand
Specific gravity G_s	2.65	2.53	2.57
Maximum dry density (g/cm ³)	2.04	1.77	1.71
Plastic limit w_p (%)	18.2	/	/
Liqui limit w_L (%)	35.2	/	/
Plasticity index I_p	17.0	/	/

Table 2: Basic properties of toluene and water

Property	Toluene	Water
Vapor pressure (mmHg) (at 20°C)	22	17.54
Water Solubility (mg/L)	515	/
Relative density	0.867	1
Boiling point (°C)	110.6	100
Flash point (°C)	4.4	/

Table 3: Detailed water content, initial concentration and dry density of the prepared samples

Sample number	Type of soil	Target contaminant	Initial concentration IC (mg/kg)	Water content	Dry density (g/cm ³)
				w (%)	
#1	Clay	Toluene	5000	15	1.34
#2	Clay	No	/	15	1.34
#3	Fine sand	Toluene	5000	3	1.60
#4	Fine sand	No	/	3	1.60
#5	Coarse sand	Toluene	5000	3	1.46
#6	Coarse sand	No	/	3	1.46
#7	Clay	Toluene	5000	5	1.34
#8	Clay	Toluene	5000	10	1.32
#9	Clay	Toluene	5000	20	1.31
#10	Clay	Toluene	5000	25	1.27
#11	Fine sand	Toluene	5000	0	1.62
#12	Fine sand	Toluene	5000	1	1.61
#13	Fine sand	Toluene	5000	10	1.58

2.2 Experimental setup

The concentration of VOCs in soil is mainly measured by gas chromatography and portable VOCs analyzer(Li et al. 2020). The measuring procedure of VOCs concentration in soil by gas chromatograph usually requires long time due to transport, storage and laboratory testing(Yu et al. 2016). Given the continuous mass loss during the test, the gas chromatograph is time-consuming for measurement of the concentration of VOCs in soil. Portable VOCs detector is a semi-quantitative method to measure the concentration of VOCs in soil, which is widely used in field work(Heron et al. 2016). This method mainly reflects the concentration of VOCs in soil by measuring the mass loss through volatilization from soil. Currently, there is no test device that can continuously measure the mass loss of VOCs and water from soil simultaneously. In this paper, the volatilization experiments were conducted with a specially designed test system as shown in Fig. 2. Through this system, the relationship between the concentration in soil and the mass loss of toluene which volatilize into air can be established. Thus, the volatilization process

of toluene and water from soil is quantitatively analyzed. The whole system consists of sealed volatilization chamber, electronic balance, ventilation device and monitor device. Samples of contaminated soil were prepared separately in aluminium boxes, which were placed on electronic balance in sealed volatilization chamber. Electronic balance could accurately measure the mass loss of toluene and water in aluminium box. The monitor device of VOCs concentration was a portable VOCs detector, which used Photo Ionization Detector (PID) detection technology. In addition, the test device also measured variation of air temperature and humidity in sealed volatilization chamber through the thermohygrometer. Contaminated waste gas was finally treated by adsorption on activated carbon.

In this experiment, the total mass loss (M) of the contaminated soil is due to the overall volatilization of both toluene (m_1) and water (m_2). The mass loss of toluene by volatilization per unit time can be obtained by portable VOCs detector in sealed volatilization chamber. According to mass conservation law, the total mass loss (M) of soil from aluminium box is equal to the sum of the mass loss of toluene (m_1) and the mass loss of water (m_2) by volatilization. The total mass loss (M) was measured by electronic balance. The formula of toluene and water mass loss by volatilization are given as follows:

$$m_1 = \frac{C_T \times V \times M_r}{V_m} \quad (1)$$

$$m_2 = M - m_1 \quad (2)$$

Where C_T is the concentration of toluene in sealed volatilization chamber; V is volume of sealed volatilization chamber; M_r is relative molecular mass; V_m is the molar volume of gas at 20 °C. It is worth saying that the portable VOCs detector device measures the total VOCs content, which needs to be calibrated with isobutylene before measurement(Mizukoshi et al. 2010). For a specific VOCs, correction factor is used to adjust the sensitivity of PID to convert isobutylene equivalent concentrations to toluene equivalent concentrations. Toluene concentration is calculated using the follow equation(Mizukoshi et al. 2010):

$$C_T = C_i \times f \quad (3)$$

Where f is the correction factor for toluene(value is 0.5 (Mizukoshi et al. 2010)); C_i is the concentration of isobutylene, which is actually measured during the volatilization test.

The experimental procedures are listed in the following steps: (i) Measure the initial total mass of aluminum box and contaminated soil, and then let the toluene and water volatilize from soil for 10 minutes; (ii) Measure the total mass of the aluminum box and contaminated soil, and then seal the aluminum box by sealed cap; (iii) Measure the concentration of toluene(C) in sealed volatilization chamber by portable VOCs detector; (iv) Open the sealed window and start ventilator to remove contaminated vapor of toluene and water in sealed volatilization chamber; (v) Close sealed window and open sealed cap, continue the volatilization for 10 minutes which includes the period of toluene

measurement; (vi) Repeat step (ii) until the end of volatilization process. The data was acquired every 10 minutes in the first 3 hours and every 30 minutes after 3 hours. The indoor air temperature and relative humidity were controlled during volatilization test. The air temperature was $20 \pm 2^\circ\text{C}$ and air humidity was $65.0 \pm 3\%$.

3. Results And Discussion

3.1 Volatilization characteristics of toluene and water

The curves of samples #1, #3, and #5 in Fig. 3 represent volatilization process of toluene, water and total mass loss by volatilization in clay, fine sand and corase sand, respectively. Volatilization rate is defined as the mass loss of toluene or water per unit area and unit time by volatilization. According to Fig. 3, it can be seen that the volatilization process of toluene can be divided into two stage: stage I and stage II. At stage I, volatilization rate of toluene linearly decreases with time. It shows a low volatilization rate at stage II. As can be seen from Fig. 3, the volatilization process of toluene only have stage I in coarse and fine sand. The volatilization process of toluene in clay has stage I and stage II. At stage II, the average volatilization rate of clay, fine sand and coarse sand are 667, 786 and 762 mg/(min·m²), respectively. Volatilization rate of toluene in clay is significantly lower than that in sand. It can be explained by the fact that sand has higher air permeability, and sand has well volatilization channel. In addition, the clay particles are smaller in size and have larger specific surface area, causing higher adsorption capacity(Song et al. 2018). At Stage II, the duration time of volatilization process for coarse sand and fine sand are shorter obviously, which can be ignored. Compared with clay, adsorption capacity of sand is pretty weak. Most of the toluene in sand has volatilized into air at stage II. So generally the volatilization process of stage II in sand will not occur. For clay, there obviously exists a low-speed volatilization process at stage II. The main reason for this phenomenon is that the clay has strong adsorption capacity for toluene, which weakens the volatile capacity of toluene. Under the same initial concentration, the proportion of adsorption phase(solid phase) toluene in clay is higher than that in sand, resulting in lower volatilization rate of toluene.

Figure 4 (a), (b) and (c) are the variation results of the mass proportion of volatilization of toluene and evaporation of water with time in clay, fine sand and coarse sand, respectively. It is found that the mass loss proportion of toluene by volatilization is much higher than that of water in early stage II. This is attributed to the fact that the vapor pressure of toluene is higher than that of water. Vapor pressures of toluene and water are 22.1 mmHg and 17.5 mmHg at 20°C , respectively(Hauxwell et al. 1968). With the increase of vapor pressure, there exists greater pressure difference at liquid-gas interface. So the toluene molecules are more easily to escape from liquid into pore space of soil, resulting in the improvement of volatilization process of toluene.

3.2 Effect of toluene on water

The curves of samples #2, #4, and #6 in Fig. 3 represent volatilization process of water without toluene in clay, fine sand and corase sand, respectively. As we can see from Fig. 3(a), volatilization rate of water for

sample #2 with time decrease slowly. It is noted, however, evaporation rate of water from samples (#1) presents rising curve process, when the volatilization rate of toluene decreases significantly. It is indicated that the volatilization of toluene can inhibit evaporation of water. A possible explanation is that polar water is more easily sorbed on clay than weakly polar toluene molecules(Tekrony & Ahlert 2001). Researchers(Smith et al. 1990, Unger et al. 1996) studied the adsorption of VOCs vapor to vadose zone soils from 0% water content to saturation. It was found that water effectively competed with VOCs for mineral surfaces and therefore suppressed VOCs adsorption considerably. In other words, the adsorption of toluene molecules are weaker than water molecules, resulting that the toluene molecules may mainly exist outside of water film. Therefore, higher concentration of toluene inhibits the evaporation of water.

3.3 Effect of water content on volatilization of toluene

Figure 5 shows the mass loss of toluene due to volatilization over time in clay and fine sand under different water content, as well as the variation of volatilization rate with increase of water content in clay. As observed from the volatilization curve of toluene, the influence of water content on volatilization of toluene in clay can not be neglected. Researchers(Donaldson et al. 1992, Tekrony & Ahlert 2001, Wilson et al. 1994) found that there might exist competitive adsorption relationship between water molecules and toluene molecules for the binding sites on soil particle surface(Fig. 7). That is, compared with toluene molecules, water molecules present stronger polar. So it has stronger competitive adsorption capacity on the surface of clay particles(He et al. 2008, Poulsen et al. 1998). In other words, water molecules occupy part of the binding sites of toluene molecules(Fig. 7). As is shown in Fig. 7, more toluene molecules are desorbed from the soil binding sites and the volatilization process of toluene is promoted. Increases in water content after a dry period can cause VOCs fluxes that are many times higher than the average background level, which is called "wet-dog" effect(Petersen et al. 1996, Poulsen et al. 1998). This so-called "wet-dog" effect, where VOCs are released to the soil air during an increase of water content, has been reported by Petersen(1996) and Poulsen(1998). When the water content of soil increases, VOCs molecules are released to the pore spaces of soil with potentially large fluxes of VOCs to the atmosphere as a result(Poulsen et al. 1998). This is justified by the decrease of adsorption in the solid phase, related to reduce contact between the vapor phase contaminant and the soil matrix due to the presence of water(Alvim-Ferraz et al. 2006, Petersen et al. 1996). These results show that the increase of water content decreases the clay particles adsorption capacity to VOCs molecular, enhancing the volatilization of toluene. Sterrett (1989) obtained similar phenomena by studying soil vacuum extraction after rains at field sites with very dry soil. In Fig. 5(a), compared with 5% water content, the toluene volatilization rate at 10% and 15% increased 13% and 22%, respectively. This also proves the existence of such kind of competitive adsorption.

However, the effect of water content in clay on promoting volatilization only exists at the lower water content stage. The increase of clay water content will also reduce clay permeability(Niu et al. 2012, Qin et al. 2010). Figure 5 presents that the volatilization rate of toluene gradually increases with the water content before 15%, and then decreases significantly. It can be explained that water molecules will

occupy the pore space of clay, and high water content will reduce its air permeability. In Fig. 7, the volatilization channels of toluene from soil are blocked when water content is high, resulting in the reduction of volatilization of toluene. Figure 6 shows the elapsed time at toluene mass loss reaching 90% in samples with different water content. In Fig. 6, it took 150, 136, 107 and 210 minutes for the samples with 5%, 10%, 15% and 20% water content to reach the 90% removal rate, respectively. It shows that there exists an optimal water content of 15% under which the maximum volatilization rate can be reached. This conclusion can also be confirmed in Fig. 5 (a). When the water content reached 25%, the removal rate of toluene by volatilization reached 67%, and the removal rate of toluene by volatilization did not increase with the elapsed time. This means that the volatilization channels are severely blocked, and toluene molecules are trapped in clay.

Since adsorption of toluene and water on sand is weak, competitive adsorption can be neglected. The increase of water content will block the volatilization channels in sand to some degree. However, compared with clay, the effect of water content on volatilization process of toluene in sand is relatively small. As can be seen from Fig. 5, the change of water content in sand has less influence on mass loss of toluene by volatilization. In addition, the permeability of sand is much higher than that of clay (Islam et al. 2021, Taheri et al. 2018). For the two samples with water content of 1% and 10%, the time periods to reach 90% toluene removal rate are 63min and 81min, respectively (Fig. 6). The increase of water content will block the volatilization channels in sand. However, compared with clay, the effect of water content on volatilization process of toluene in sand is relatively small.

In summary, there are two main mechanisms for the effect of water content on volatilization process of toluene in clay. The first is the mechanism of competitive adsorption between toluene molecules and water molecules in clay. Water molecules have a competitive advantages than toluene molecules adsorbed on the soil surface. The higher water content is, the stronger volatilization process of toluene will be. The second is that the increase of water content decreases soil permeability and thus blocks the volatilization channels of toluene in clay. When water content is low, the mechanism of molecular competitive adsorption plays a dominant role, and the water content has little effect on the volatilization channels of toluene. But for the samples with different water content, the blocking mechanism of volatilization channels under the influence of water content plays an increasingly important role with the increase of water content. That is, volatilization rate of toluene decreases with the increasing of water content. As shown in Fig. 5(a) and Fig. 6, there exists an optimal water content of 15% under which the maximum volatilization rate can be reached. As discussed above, water content has an important effect on the volatilization process of toluene in clay. However, such kind of effect in sand is relatively weak.

4. Conclusion

In this paper, laboratory tests were conducted to study the volatilization process of toluene and water from soil as well as their interaction mechanism under different water content. The volatilization process of toluene in clay can be divided into two typical stages (I and II), but volatilization process of toluene in sand only contains stage I. The volatilization rate decreases linearly with time at stage I and maintains a

very low value when it reaches stage 3. The volatilization rate of toluene is obviously higher than that of water. Volatilization process of toluene inhibits the evaporation of water. In addition, the effects of water existence on volatilization process of toluene include two mechanisms. The increase of water content will promote the volatilization of toluene under relative low water content mainly due to their competitive adsorption on soil particles. However, the increase of water content will decrease the permeability of clay and then reduce the volatilization rate of toluene once the water content is relatively high. There exists an optimal water content of 15%, under which the maximum volatilization rate can be achieved.

Declarations

Authors' contributions This manuscript was contributed by all authors. All the authors have ratified the manuscript. Lu Liangliang conceived the presented idea and compiled the manuscript; Liu Zhibin reviewed the manuscript and contributed to the original draft preparation and editing. Li Shangan and Bai Mei collected data and information; Liu Zhibin and Fan Zhanhuang provided sources of funding. All authors have participated in the work of this manuscript and agreed to the publication of this paper in your journal.

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Data availability The data is true and reliable.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical approval Not applicable

Consent to participate All authors agree to participate in the editing of the paper.

Consent to publish All authors agree to publish this manuscript in your journal.

Competing interests The authors declare that they have no competing interests.

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Figures

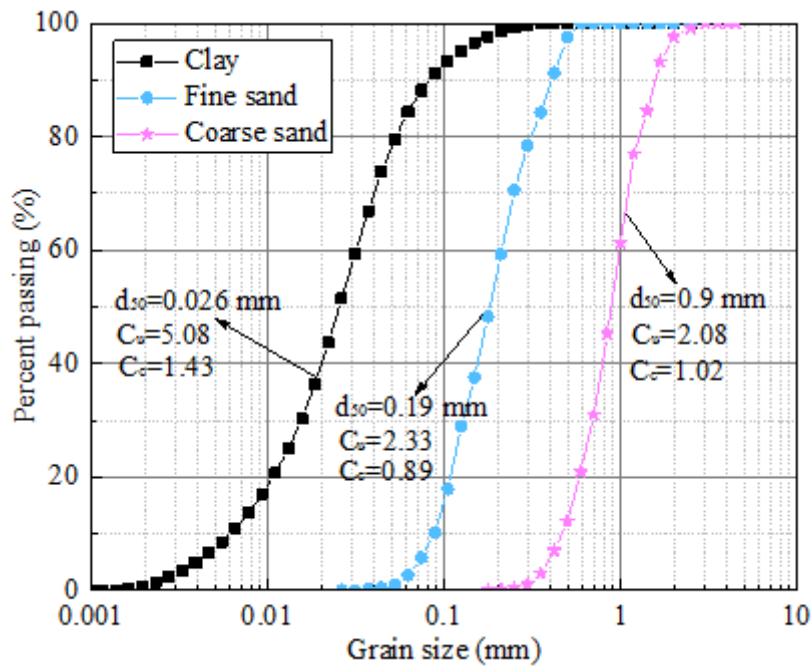


Figure 1

Particle size distribution of coarse sand, fine sand and clay.

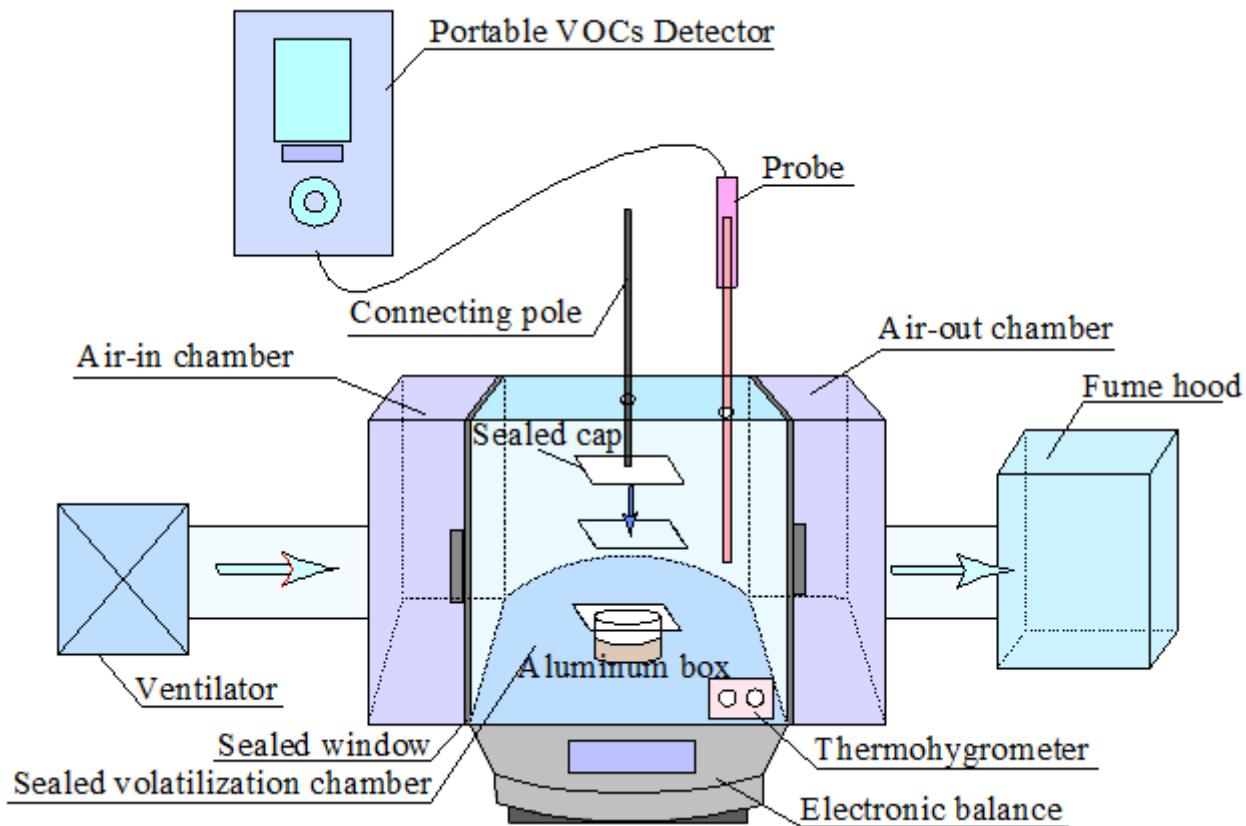


Figure 2

Schematic diagram of the toluene and water volatilization test system.

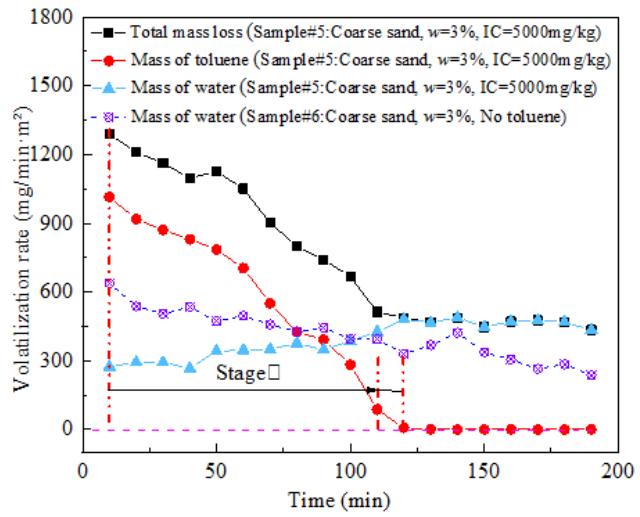
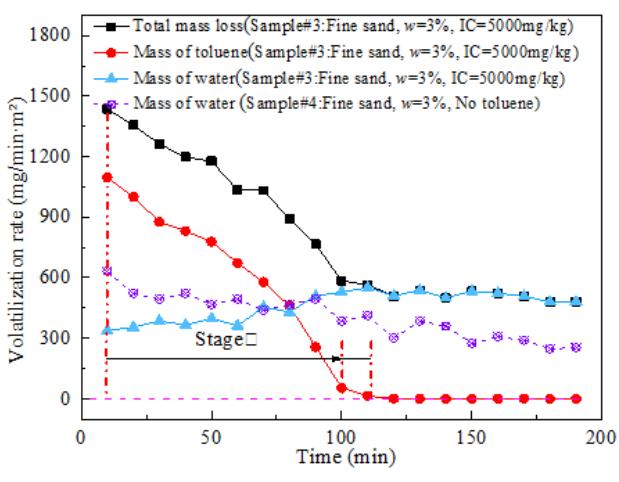
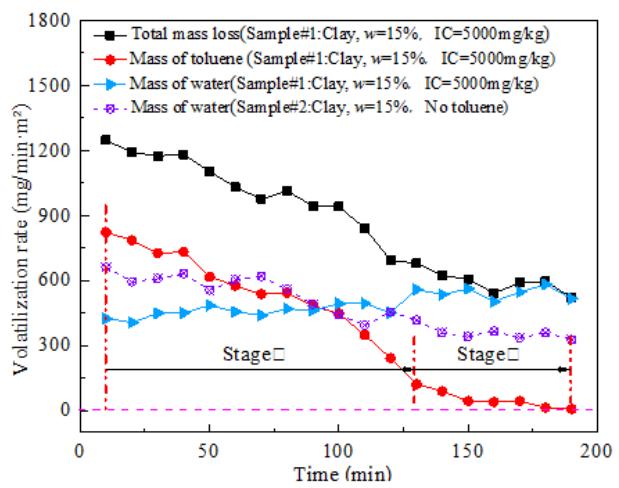


Figure 3

Volatilization rate variation of toluene and water with time(w and IC are water content and initial concentration in soil, respectively)

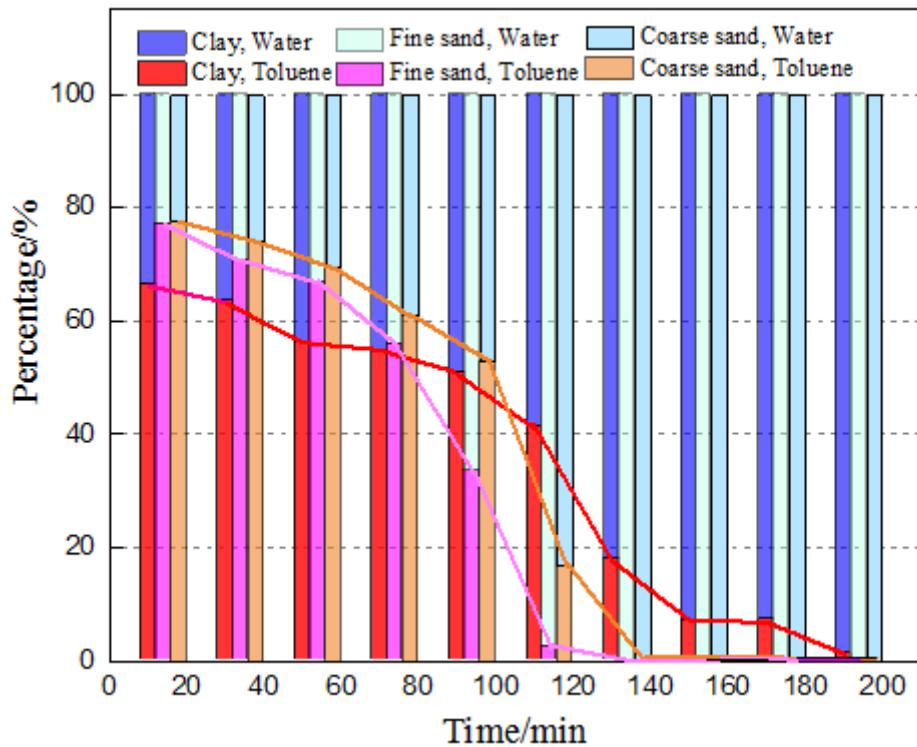


Figure 4

The mass proportion of toluene and water during volatilization (a) Clay of samples #1; (b) Fine sand of samples #3; (c) Coarse sand of samples #5.

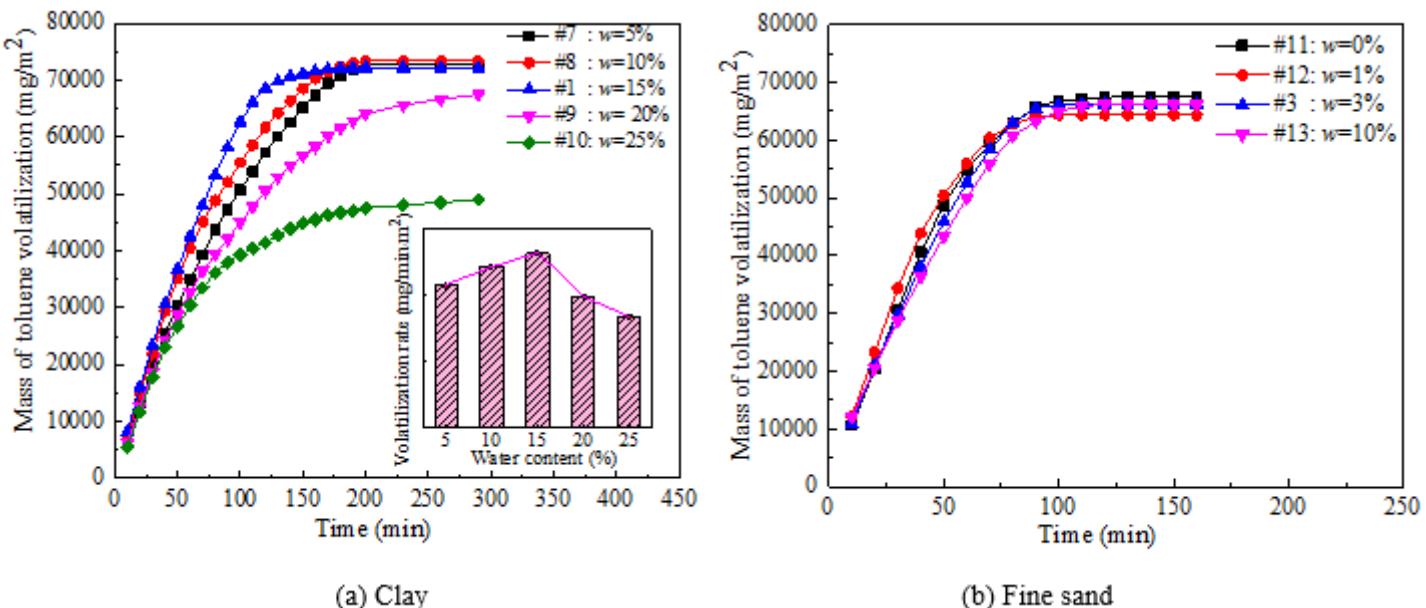


Figure 5

The volatilization of toluene under different water content.

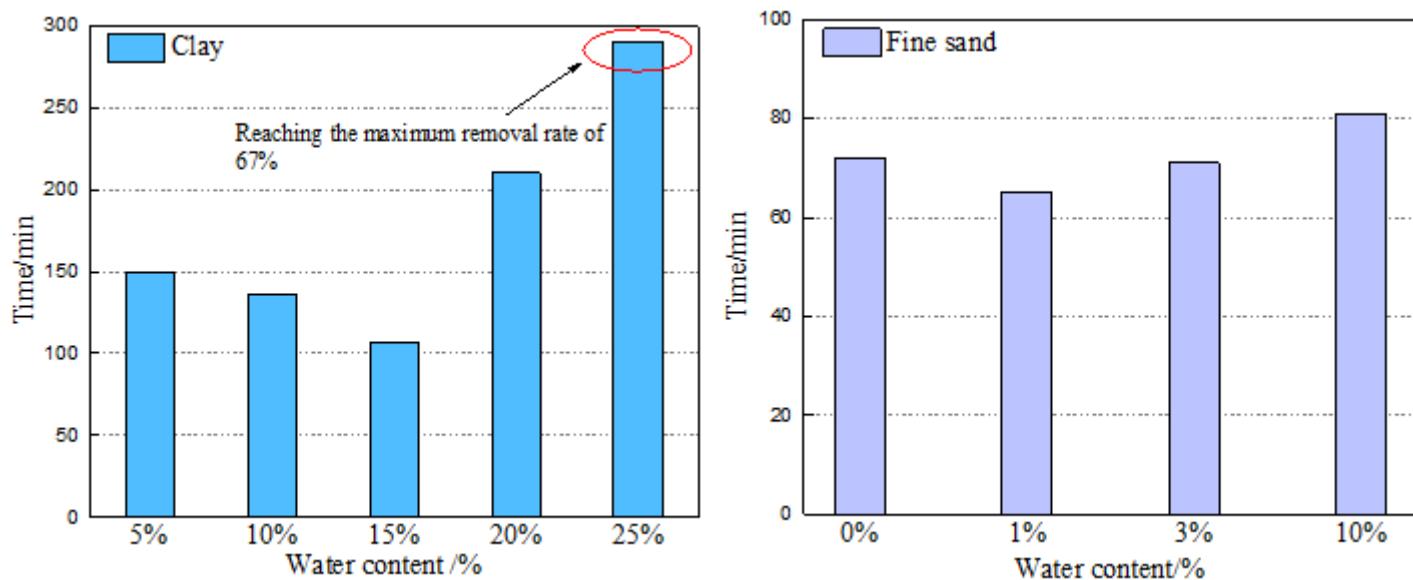


Figure 6

The volatilization elapsed time under different water content with 90% toluene removal rate.

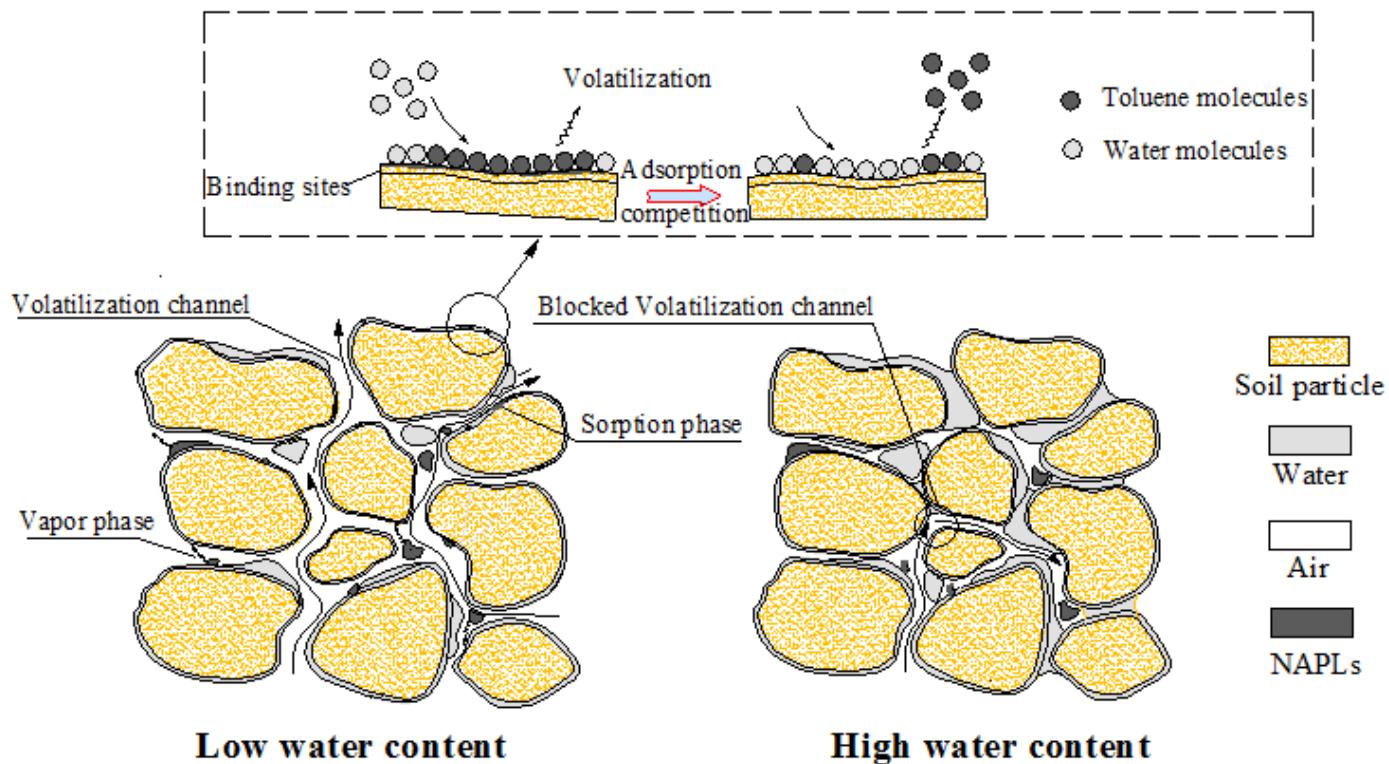


Figure 7

Sketch of NAPLs volatilization process from soil under different water content