Sorption behavior of tetrabromobisphenol A in two soils with different characteristics

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ABSTRACT

Sorption of tetrabromobisphenol A (TBBPA) in soil influences its fate and transport in the environment. The sorption behaviors of TBBPA in two soils with different characteristics were investigated using batch equilibration experiments in the study, and the impacts of ionic strength and pH on the sorption were also evaluated. The results showed that the fast sorption rather than the slow sorption played a main role in the sorption process. The nonlinear sorption isotherms of TBBPA in the soils were observed and the Freundlich model could describe the sorption behavior of TBBPA well. The calculated K_F were 78.5 and 364.6 $(mg/kg)(mg/L)^{-n}$ for LN soil (loamy clay) and GX soil (silt loam), respectively. Soil organic matter (SOM) played a main role in the sorption of TBBPA, which contributed about 90% to the sorption in the soils. The desorption hysteresis of TBBPA was revealed in the single-cycle sorption and desorption experiments. The sorbed amount of TBBPA decreased with the increase in solution pH and increased with the increase in ionic strength. It was found that the effects of ionic strength on the sorption were mainly caused by the change of solution pH.

1. Introduction

In the last decades, brominated flame retardants (BFRs) have been widely used in industrial practice to improve the flame resistance of polymeric materials. Tetrabromobisphenol A [TBBPA; 4,4′-isopropylidenebis(2,6-dibromophenol); Fig. 1; CAS no. 79-94-7], the most widely used BFR, accounted for more than half of the usage of BFRs. The worldwide market demand for TBBPA was estimated at 120,000 tons in 1999 [1]. TBBPA is primarily used as a reactive flame retardant, which is covalently bound into epoxy and polycarbonate resins used in the manufacture of printed circuit boards and other electronic equipments [2]. It is also used as an additive flame retardant in acrylonitrile-butadiene-styrene (ABS) resins as well as in high impact polystyrene [2].

It has been demonstrated that TBBPA is toxic to aquatic life. The acute 48-h LC_{50} (lethal concentration to 50%) for Daphnia magna was reported to be 0.96 mg/L, and 96-h LC_{50} for fish ranged from 0.40 to 0.54 mg/L [2]. The acute oral toxicity of TBBPA for mammals is low [2]. However, results of many studies in vitro suggested that TBBPA induces cytotoxicity [3], immunotoxicity [4], hepatotoxicity [5], disruption of thyroid homeostasis [6,7] and has potential to disrupt estrogen signaling [8,9]. TBBPA can be released into the environment during the manufacture, use and disposal of prod-

ucts including TBBPA [10,11]. It was of concern since TBBPA has been widely found in the environment such as stream sediments, municipal wastewater, sewage sludge, soils and biota [1,12–16]. At a contaminated site in Israel, the concentration of TBBPA in the upper 20 cm of the soil layer was more than 50 mg/kg of soil, which can be transferred to the deeper soil profile and may contaminate groundwater [16].

Recently, majority of the current research has focused on the toxicity [3–9] and degradation of TBBPA in the environment [17–21]. Only a few mentioned the transport behavior of TBBPA in soil [16]. Sorption in soil/sediment is an important process for organic contaminants influencing transport, degradation and bioavailability of these compounds in the environment. There have been many reports on sorption behaviors of nonionizable contaminants [22] and ionizable contaminants with relative high aqueous solubility in soil [23,24], but reports on sorption behaviors of ionizable contaminants with low aqueous solubility are limited. TBBPA is a hydrophobic ionizable compound with low aqueous solubility (4.6 mg/L at 25 °C), high log K_{ow} (4.50) and two near neutral pK_a values (7.5 and 8.5, respectively) [2]. Therefore, the sorption behaviors of TBBPA are strongly pH dependent and seem more complicated. To our knowledge, there is no study reported the sorption behaviors of TBBPA in soil/sediment to date.

The overall objective of this study was to investigate the sorption behaviors of TBBPA in two Chinese soils with different characteristics. The sorption and desorption isotherms of TBBPA were obtained. In addition, the effects of ionic strength and pH on the

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Fig. 1. Chemical structure of TBBPA.
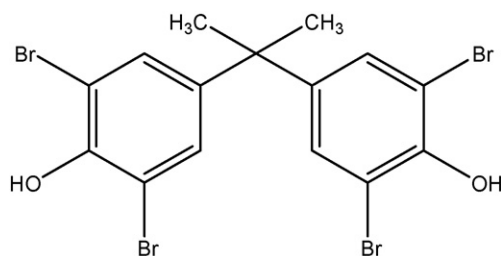

Fig. 2. Sorption equilibrium time of TBBPA in the soils.

sorption of TBBPA were also evaluated. The results would provide a better understanding of environmental behaviors of TBBPA in soil and contribute to risk assessment and fate modeling of TBBPA in the environment.

2. Materials and methods

2.1. Materials

Tetrabromobisphenol A was obtained from Aldrich chemicals with a purity >97%. Methanol (HPLC grade) was obtained from Tedia Company (USA). All other chemicals were of analytical grade.

Two surface (0–20 cm) soil samples were collected from Liaoning (LN) and Guangxi (GX) provinces, China, respectively. The soil samples were air-dried and passed through a 0.25-mm sieve. Selected properties of the soils are given in Table 1. Soil organic carbon (OC) was determined by the method of dichromate oxidation, and soil cation exchange capacity (CEC) was determined by the method of NH_4Ac exchange [25]. Small quantities of soil samples were combusted at 600 °C for 24 h under air in a muffle furnace to remove organic matter in the soils [24,26]. The combusted soils were acidified with 1 M HCl to remove carbonates and analyzed with a Heraeus CHN-O-Rapid Elemental Analyzer (Germany) at 1030 °C. The organic carbon contents of the combusted soils were below 0.3% (detection limit). The original and combusted soil samples were stored in glass vessels for sorption experiments.

2.2. Sorption and desorption experiments

Sorption experiments were conducted using batch equilibration technique in 40 mL PTFE-lined screw cap glass tubes. The background solution was 0.01 M $CaCl_2$ to maintain a constant ionic strength and 100 mg/L NaN_3 to inhibit microbial activity. The soil samples were weighted (300 mg for LN soil and 100 mg for GX soil, respectively) into the glass tubes and 30 mL background solution was added into each tube. The initial concentration of TBBPA added into the tubes ranged from 0.05 to 1.50 mg/L. Due to the low aqueous solubility, TBBPA were mixed at high concentration in methanol before being added to the background solution. Methanol concentration was always less than 0.1% of total solution volume to avoid cosolvent effects. The tubes were shaken at 150 rpm for 48 h at 25 °C. After mixing, the tubes were centrifuged for 10 min at 4000 rpm, and 1.0 mL of the supernatant was removed into a sampling vial for analysis. Simultaneously, the control experiments containing solutes without soil were also carried out to evaluate the loss of TBBPA. It was found that the loss of TBBPA from pho-
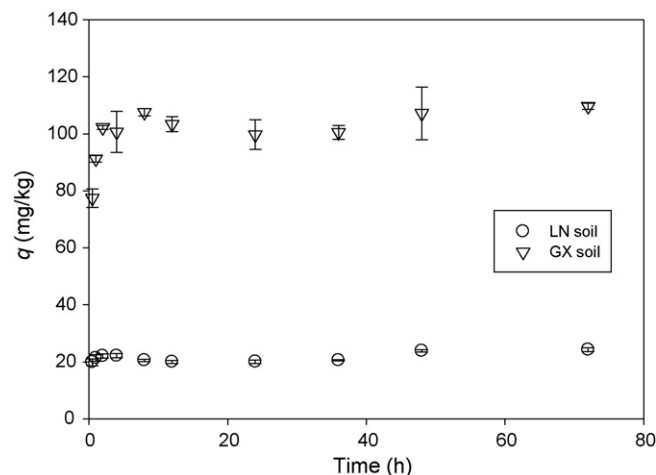
tochemical decomposition, volatilization and sorption to the tube was negligible.

Desorption was evaluated using decant and refill technique after the completion of the sorption experiments [27]. After the 1 mL aliquot was withdrawn, as a part of sorption procedure, 25 mL of the remaining supernatant was discarded and replaced by 26 mL fresh background solution (dilution). Following dilution, the tubes were mixed for the same time as used in the sorption experiments. After mixing, the tubes were centrifuged and 1.0 mL of the supernatant was collected for analysis.

Kinetic experiments were conducted according to a method similar to that of sorption experiments. The difference was that the mixing time of TBBPA and soil in the tubes were various intervals from 0.5 to 72 h and the initial concentration of TBBPA added was 0.5 mg/L. The sorption samples were prepared in triplicate and the means were shown in the figures.

2.3. Effects of ionic strength and pH on the sorption of TBBPA

The sorption isotherms of TBBPA at different ionic strengths were obtained according to the similar procedure of sorption experiments. The difference was that the background solutions used in the experiments were 100 mg/L NaN_3 and different concentrations of $CaCl_2$. The concentrations of $CaCl_2$ in the background solutions were 0.001, 0.01 and 0.1 M, respectively. Sorption experiments at different ionic strengths were also conducted at different pH values by addition of 0.5 M HCl or 0.5 M NaOH as needed to the background solutions. The initial concentration of TBBPA added was 0.4 mg/L. After shaken and centrifugation, the pH values of the supernatants were measured using a 320-S pH meter (Mettler-Toledo Instruments (Shanghai) Co., Ltd., China).

2.4. Analytical technique

The samples were analyzed by a high performance liquid chromatography (HPLC) equipped with a UV detector with a wavelength at 210 nm (Agilent 1100). The column was a 4.6 mm × 250 mm model (Agilent ZORBAX SB-C18). The mobile phase was mixture of 85:15 (v:v) methanol and 0.2% (volume ratio) acetic acid aqueous

Table 1
Selected physical and chemical properties of the soils

Soil	pH (s:w = 1:2.5)	OC (g/kg)	CEC (cmol$_{(+)}$/kg)	Sand (wt %)	Silt (wt %)	Clay (wt %)	Texture
LN soil	7.81	13.7	22.6	51.3	21.8	26.9	Loamy clay
GX soil	7.11	21.3	9.50	41.3	45.9	12.8	Silt loam

Table 2
The Freundlich model parameters of sorption and desorption isotherms

Soil	Sorption			Desorption		
	$K_F \pm$ S.E.[a] $(mg/kg)(mg/L)^{-n}$	$n \pm$ S.E.	R^2	$K_F \pm$ S.E. $(mg/kg)(mg/L)^{-n}$	$n \pm$ S.E.	R^2
LN soil	78.5 ± 1.36	0.748 ± 0.019	0.9975	108.6 ± 2.35	0.851 ± 0.014	0.9990
GX soil	364.6 ± 9.42	0.756 ± 0.023	0.9964	484.6 ± 29.2	0.741 ± 0.033	0.9922

[a] Standard errors.

solution at a flow rate of 1 mL/min. TBBPA sorption was calculated from the difference between the total amounts of TBBPA initially added to the solution and the amounts remaining in the solution at equilibrium.

3. Results and discussion

3.1. Kinetics of sorption

Fig. 2 displayed the sorption kinetics of TBBPA in the soils. TBBPA sorption in the two soils showed a fast step within 2 h and followed by a slow step. Sorption of TBBPA was extremely rapid during the first 30 min. The sorbed amount of TBBPA at 30 min was 85.5% and 72.4% of maximal sorbed amount for LN soil and GX soil, respectively. The sorption within 2 h exhibited relatively fast after the initial 30 min immediate rapid sorption, the sorbed amount of TBBPA at 2 h accounted for 91.0% and 93.2% of every maximal sorbed amount, respectively. After the fast sorption step, the desorption began to play an important role, and the sorbed amount of TBBPA changed slightly. The sorption reached apparent equilibrium at approximately 48 h. The results revealed that the fast sorption rather than the slow sorption played a main role in the sorption process. The fast sorption of TBBPA was probably attributed to adsorption of the solute to mineral surface [28] or partition into a rubbery domain of the soil organic matter (SOM) [29], while the slow sorption was resulted from gradual diffusion of the solute into soil organic matter matrices and soil micropores [30]. The results also suggested that the dissolution or partition domain of organic matter in the soils may play a major role in TBBPA sorption. According to the results, 48 h was selected as the sorption equilibrium time for all the samples in the sorption and desorption experiments.
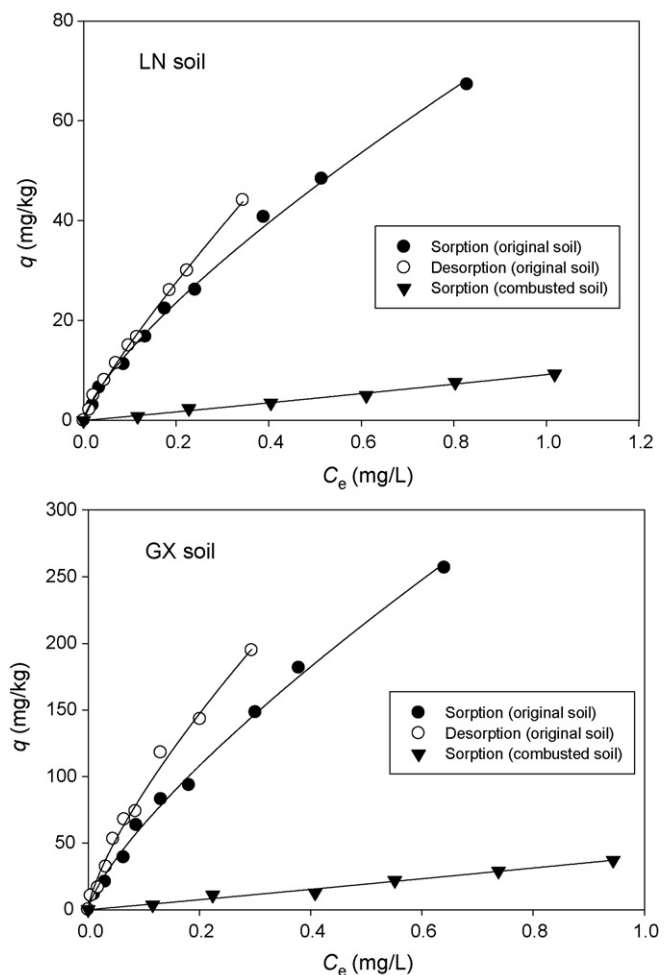
3.2. Sorption isotherms

The sorption isotherms of TBBPA in the tested soils were shown in Fig. 3. The experimental data were fitted to the Freundlich model,

$$q = K_F C_e^n \tag{1}$$

where q is the sorbed amount in the soil (mg/kg), C_e is the equilibrium concentration in the solution (mg/L), K_F is the Freundlich affinity coefficient $(mg/kg)(mg/L)^{-n}$, and n is an empirical exponent which indicated isotherm nonlinearity. The parameters of the Freundlich model were listed in Table 2. The experimental data of TBBPA sorbed in the two soils were fitted well to the Freundlich model with $R^2 > 0.99$, the exponents, n, in the Freundlich model were all less than 1, which suggested that the isotherms were nonlinear and the sorption behavior of TBBPA in the soils could be described by the Freundlich model well. The nonlinear sorption of TBBPA was mainly attributed to the heterogeneity of soil organic matter. Previous research has shown that SOM may comprise two principle types of heterogeneous organic domains, a condensed organic domain and a highly amorphous domain [29]. The sorption of TBBPA in the SOM consists of site-specific and capacity-limited adsorption in a condensed organic domain and nonspecific partition in a highly amorphous domain [31]. Adsorption is nonlinear while partition is linear, which results in the nonlinearity of the

overall sorption. The Freundlich parameter K_F can be taken as a relative indicator of sorption capacity. The calculated K_F were 78.5 and 364.6 $(mg/kg)(mg/L)^{-n}$ for LN soil and GX soil, respectively.

Combustion at 600 °C was employed to remove organic carbon with only those contained minerals. The sorption isotherms of TBBPA in the combusted soils were displayed in Fig. 3. The pH values of the equilibrium solution for the combusted LN soil and GX soil were 7.38 and 7.00, respectively, which were similar to that for the corresponding original soils. As shown in Fig. 3, the isotherms also could be fitted by the Freundlich model well ($R^2 > 0.98$), the calculated K_F values were 9.17 and 39.4 $(mg/kg)(mg/L)^{-n}$, and n values were 1.05 and 1.04 for the combusted LN soil and GX soil, respectively. The sorbed amount of TBBPA in the two combusted soils decreased as about 90% of that in the original soils, which suggested that the SOM plays a main role in the sorption of TBBPA. The calculated n values were close to unity, which meant the sorption isotherms in the combusted soils are more linear than that in the original soils. It also suggested that the nonlinearity of TBBPA sorption is mainly caused by the heterogeneity of the SOM. The sorbed

Fig. 3. Sorption and desorption isotherms of TBBPA in the original and combusted soils.

amount of TBBPA in the combusted GX soil was greater than that in the combusted LN soil, which implied other factors, e.g., soil mineral and pH, may affect TBBPA sorption in addition to the SOM.

3.3. Desorption behavior of TBBPA in soils

The nonlinear desorption isotherms in the two soils were observed in the study (Fig. 3). The Freundlich model could fit the desorption isotherms well, and the parameters were shown in Table 2. The apparent sorption–desorption hysteresis was quantified for each sorbent–solute–solution system using the Hysteresis Index (HI) defined by Huang et al. [32],

$$\text{Hysteresis Index} = \left. \frac{q^d - q^s}{q^s} \right| T, C_e \tag{2}$$

where q^s and q^d are soil sorbed sorbates for the single-cycle sorption and desorption experiments, respectively, and the subscripts T and C_e specify conditions of constant temperature and residual solution phase concentration. A zero or negative value of HI indicates that sorption–desorption hysteresis is insignificant. HI values at 25 °C and three different equilibrium concentrations (C_e = 0.10, 0.20 and 0.30 mg/L) were calculated using the Freundlich model parameters of sorption and desorption isotherms listed in Table 2. The calculated HI values were 0.09, 0.17 and 0.22 for LN soil, and 0.38, 0.36 and 0.35 for GX soil, respectively. The HI values in the soils at the three equilibrium concentrations were all greater than zero, which indicated the occurrence of sorption–desorption hysteresis of TBBPA. The desorption hysteresis was probably attributed to slow desorption and entrapment of sorbed TBBPA within the condensed SOM and inorganic matrices [33,34], and irreversible binding to specific sorption sites [35]. The desorption hysteresis suggested that the adsorption also plays an important role in the overall sorption of TBBPA in the soils besides the partition.

3.4. Effects of ionic strength and pH on the sorption of TBBPA

According to the previous studies, ionic strength significantly affected the sorption of bisphenol A (BPA) to sediment/soil [24,36]. TBBPA has the similar structure to BPA, so the effect of ionic strength on the sorption of TBBPA should not be neglected. The nonlinear sorption isotherms of TBBPA in the tested soils at three ionic strengths (Ca^{2+}) were shown in Fig. 4, and the Freundlich model fitted the isotherms well ($R^2 > 0.99$). The sorbed amount of TBBPA in the two soils increased with the increase in ionic strength. When the Ca^{2+} increase from 0.001 to 0.01 M and 0.1 M, the K_F values increased by 177% and 315% for LN soil, and by 28.5% and 70.2% for GX soil, respectively.

The sorption of ionizable organic compounds increased with the increase in Ca^{2+} concentration may be attributed to several reasons such as formatting neutral ion pairs, screening the negative charge of soil surface, decreasing the solubility of the compounds by salting out effect and inhibiting the release of organic matter [36–40]. Moreover, the solution pH values decreased at higher Ca^{2+} concentration caused by the exchange of H$^+$ on the soil surface by Ca^{2+} (Fig. 4), which also contributed to the increase in sorption. It has been demonstrated that the sorption of ionizable compounds in soil is strongly influenced by pH [41–43]. In order to know if pH was the only variable controlling the change of TBBPA sorption at different Ca^{2+} concentrations, TBBPA sorption in the soils at different equilibrium solution pH values was studied at the three different Ca^{2+} ionic strengths. The experimental results were shown in Fig. 5. At the same pH, there is no significant difference among the sorbed amount of TBBPA at the three ionic strengths, which suggested that the increase in sorption is mainly caused by the decrease in solution pH.
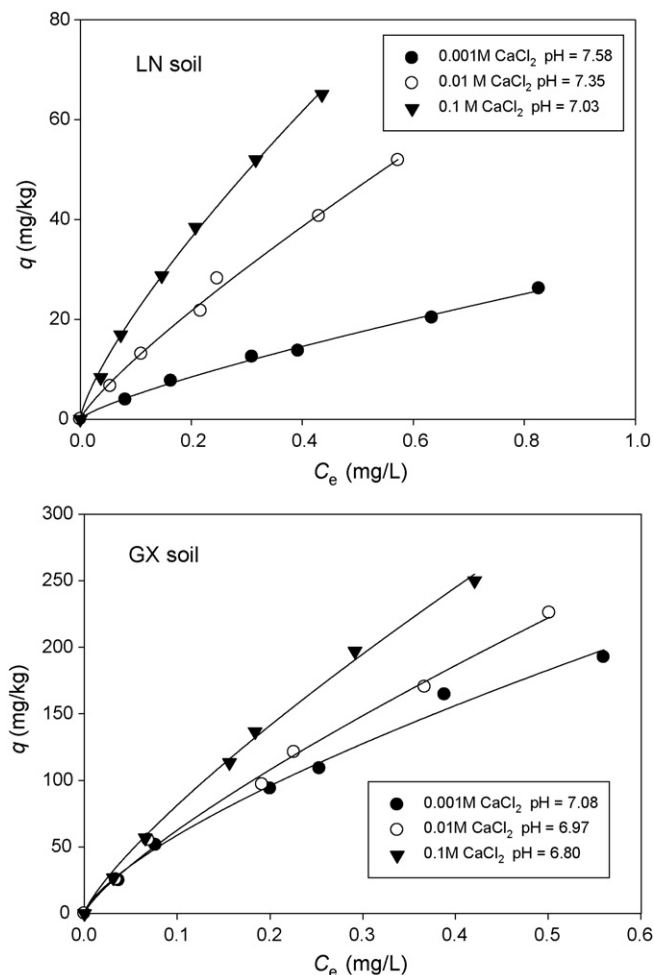

Fig. 4. Sorption isotherms of TBBPA in the soils at different ionic strengths.

As an ionizable compound having the pK_{a1} and pK_{a2} of 7.5 and 8.5 [2], respectively, the fractions of existed species of TBBPA depending on the solution pH can be calculated by the following formulas,

$$\alpha_{\text{TBBPA}} = \frac{1}{1 + 10^{(\text{pH}-\text{p}K_{a1})} + 10^{(2\text{pH}-\text{p}K_{a1}-\text{p}K_{a2})}} \tag{3}$$

$$\alpha_{\text{TBBPA}^-} = \frac{1}{1 + 10^{(\text{p}K_{a1}-\text{pH})} + 10^{(\text{pH}-\text{p}K_{a2})}} \tag{4}$$

$$\alpha_{\text{TBBPA}^{2-}} = \frac{1}{1 + 10^{(\text{p}K_{a2}-\text{pH})} + 10^{(\text{p}K_{a1}+\text{p}K_{a2}-2\text{pH})}} \tag{5}$$

where α was the fraction of certain species. TBBPA mainly existed in neutral species as pH was below 6.0, then with the pH increasing the anionic species is becoming dominant. When the pH increased from 6.0 to 8.5, the neutral species decreased rapidly from 96.9% to 4.76%. As shown in Fig. 5, the sorbed amount of TBBPA in the soils decreased with the increase in solution pH. It was found that the sorbed amount of TBBPA was positively correlated with the fraction of neutral species in each soil ($R^2 > 0.97$, $n = 45$), which indicated that the neutral species of TBBPA rather than the anionic species plays a main role in the sorption of TBBPA in the soils. Similar result has been reported by Chen et al. that the sorption of 2,4-dichlorphenol neutral species was much higher than that of anionic species on HDTMA-modified loess soil [44]. In addition, the soils may be negatively charged at alkaline pH. The enhancement of the electrostatic repulsion between the anionic species of TBBPA and the negatively
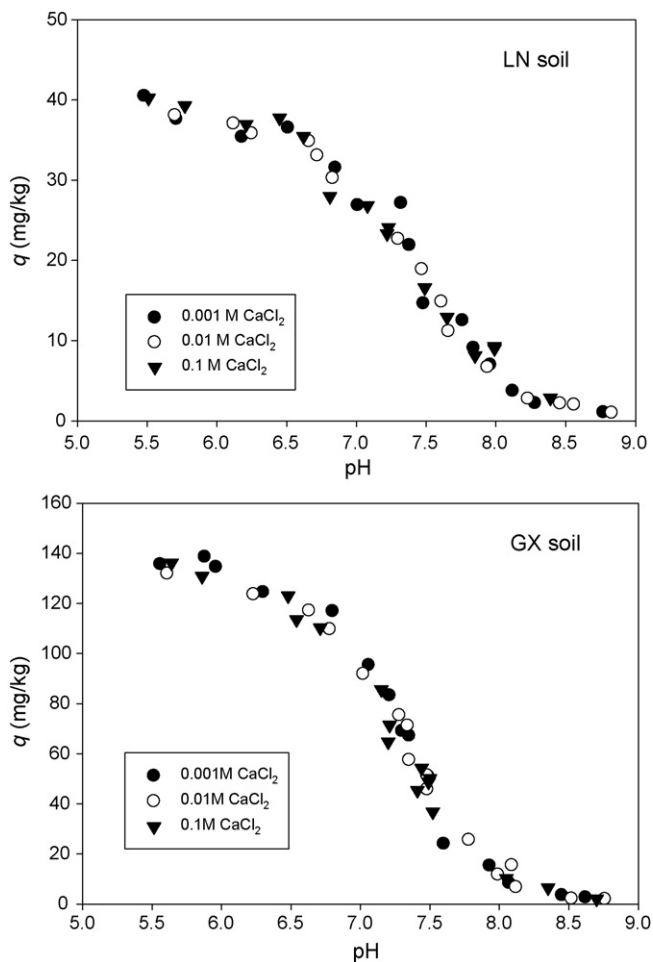
Fig. 5. Effects of pH on TBBPA sorption in the soils at different ionic strengths.

charged surface of the soils likely resulted in the decrease in the sorbed amount of TBBPA. Of course, such a sort of mechanism hypothesis needs the further investigation in the future.

4. Conclusions

Sorption and desorption behaviors of TBBPA in two soils were investigated in the study. The fast sorption was found to play a main role in the sorption process of TBBPA in the soils. The nonlinear sorption isotherms and desorption hysteresis were observed, and the Freundlich model could describe the sorption behavior of TBBPA well. The calculated K_F were 78.5 and 364.6 $(mg/kg)(mg/L)^{-n}$ for LN soil and GX soil, respectively. It was found that TBBPA sorption was affected by solution pH and ionic strength. The sorbed amount of TBBPA increased with the decrease in solution pH and the increase in ionic strength, and the effects of ionic strength on the sorption were mainly caused by the change of solution pH. Soil organic matter and the neutral species of TBBPA contributed mostly to the sorption, which indicated that the sorption of TBBPA is primarily controlled by sorption of the neutral species in the soil organic matter. The results would provide a better understanding of the transport and fate of TBBPA in the environment.

Acknowledgement

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