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Adsorption characteristic of bensulfuron-methyl at variable added Pb^{2+} concentrations on paddy soils

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Abstract

The combined pollution of heavy metal Pb^{2+} and bensulfuron-methyl (BSM), originating from chemical herbicides, in agro-ecological environments has become commonplace in southern China. The adsorption of BSM on three paddy soils in the presence of Pb^{2+} was examined using high-performance liquid chromatograph (HPLC). Results indicated that adsorption of BSM could accurately be described by a Freundlich isotherm equation with correlation constant (R) > 0.98, irrespective of the presence of spiked Pb^{2+}. Of the various factors influencing BSM sorption, soil pH appeared to be the most influential. The constant K_f of Freundlich isotherm equation tended to increase with increasing Pb^{2+} concentration in soil which indicated that the spiked of Pb^{2+} in paddy soils would promote the sorption of BSM. ΔG^0 of BSM in three paddy soils was less than 40 kJ/mol in all treatments, indicating the adsorption of BSM is mainly physical in nature. The elution of soil dissolved organic matter (DOM) enhanced the adsorption of BSM in paddy soils. The mechanisms involved in the promotion effects of the spiked Pb^{2+} on BSM adsorption might be the modified surface characteristics of paddy soil solids due to the soil acidification and the increase of soil organic matter concentration because of DOM binding.

Key words: bensulfuron-methyl; adsorption; paddy soils; Pb pollution

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Introduction

As one of sulfonylurea pesticides, the bensulfuron-methyl (BSM, 2-(4,6-dimethoxypyrimidin-2-carbamoylsulfamoyl)-*o*-toluic acid methyl ester) is widely used on paddy field, which could damage the following sensitive crops even there is only a trace amounts of residues in soil (Lin, 2000; Cavanna *et al*., 1998) and cause negative impact on aquatic biota (Sabater *et al*., 2002). Pb pollution is also widespread in agricultural soil through dry and wet depositions of air pollutants caused by the emission of industry waste gases and automobile exhaust, as well as domestic and industry wastes and agricultural chemicals (Zhou *et al*., 2004). The estimated annual accumulation rate for Pb was 0.2–1.0 mg/kg and the apparent pollution loading was 5–30 kg/hm^2 in surface paddy soils from the Tai Lake region, southern Jiangsu Province, China for the last decade (Li *et al*., 2005). The anthropogenic Pb was mainly accumulated in soil surface (Fernandez *et al*., 2008). Therefore, combined pollution of BSM and Pb became the growing concerns in the agro-ecological environment in southern China.

Adsorption is probably the most important mode of interaction between soil and pesticides and controls the concentration of the latter in the soil liquid phase (Gevao *et al*., 2000). There is a close relationship between the adsorption-desorption behaviors of sulfonylurea herbicides and the physical and chemical properties of soil (Zhang *et al*., 2007). As a consequence, great concern of BSM was focused on the investigation of adsorption characteristics and the affecting factors including soil composition and soil chemical properties (Cavanna *et al*., 1998; Si *et al*., 2003).

BSM is a weak acid, exiting predominantly in the anionic form in most agricultural soils. It has been generally accepted that the adsorption of BSM could be affected by soil pH value, organic matter and clay content of soil (Finocchiaro, 2005; Si *et al*., 2003; Chen *et al*., 1995; Beyer *et al*., 1988). However, the influence of dissolved organic matter (DOM) on the adsorption of BSM by soil has not been reported, while the growing attention has been given to the influence of DOM on the adsorption of pesticides by soil in recent years (Gao *et al*., 2007; Cox *et al*., 2000; Spark and Swift, 2002).

Adsorption of BSM might be attributed to ion exchange, ligand-exchange, and direct combination with the soil surface (McBride, 1994) and the metal adsorption onto hydrous solid is mainly a surface coordination process. It has been indicated that metal might have some influence on the adsorption of BSM to soil. The divalent ion of

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Pb would be specially adsorbed by many soil constituents and the process is strongly pH dependent (Forbes *et al*., 1976). Moreno *et al*. (2006) revealed that the added Pb is largely retained by crystalline iron oxides and the soil clay fraction. The speciation study demonstrated that most of the Pb in soil solutions is present as organo-Pb complexes (Sauvé *et al*., 1998). However, the knowledge of the effect of Pb^{2+} on adsorption of BSM to paddy soils is not sufficient in assessing their environmental impact.

The objectives of the present research were: (1) to obtain the adsorption isotherms of BSM on three contrasting paddy soils and its influential factors, (2) to investigate the influence of Pb^{2+} of the adsorption isotherms of BSM on the soils, and (3) to elucidate the possible mechanism of the adsorption of BSM in the presence Pb^{2+}. The study will ultimately provide basic data for evaluating the risk of the combined pollution of Pb^{2+} and BSM to soil and water resource.

1 Materials and methods

1.1 Chemicals

BSM was purchased from DuPont China (Shanghai, China) (purity: 98%) and used as the model compound in this study. The total standard solutions of BSM were also prepared in CH_3CN to calibrate the analytical instrument.

The molecular weight, solubility in water at 25°C (pH 5), $\log K_{ow}$ (pH 5) of BSM were 410.4 g/mol, 2.9 mg/L, and 155, respectively (Cavanna *et al*., 1998).

1.2 Soils

Three paddy soil samples were collected at a soil depth of 0–15 cm from Longyou, Pinghu and Hangzhou in Zhejiang Province. The paddy soils were paddy field on quaternary red soil (PQR), blue clayey paddy soil (BCP), and desalting muddy polder (DMP), respectively, and chosen based on contrasting physicochemical properties (Table 1). Soil samples were air-dried and passed through a 100-mesh sieve. Soil pH, soil organic matter (SOM), and cation exchange capacity (CEC) were tested according to the literature (Lu, 1999). SOM was determined by the potassium dichromate method. Particle size was determined according to the pipette method after dispersion in Na-pyrophosphate. The pH of soils was determined in a 1:2.5 soil to water ratio suspension by a digital pH meter.

To evaluate the impact of soil inherent dissolved organic matter (DOM) on adsorption of BSM by soils, the deionized water-eluted soil samples were also used (Gao *et al*., 2007). Certain amounts of control soils were placed into 100-mL glass tubes with 60 mL deionized water. The tubes were shaken on a rotating shaker for 4 h, and centrifuged at 4000 r/min for 30 min. Then the supernatant was decanted, and the soils were replenished with fresh deionized water. This process was repeated four times. Soil samples were then dried, grounded, and sieved through a 100-mesh sieve.

Table 1 Basic properties of the used paddy soils

Sample	PQR	BCP	DMP
Location	Longyou	Pinghu	Hangzhou
SOM (g/kg)	13.6	39.8	35.2
pH	5.0	6.1	7.4
CEC (cmol(+)/kg)	8.4	18.3	9.9
Clay (%)	26.0	46.4	29.0
Silt (%)	49.5	42.1	62.7
Sand (%)	24.5	1.5	8.3
Texture	Silty loam	Silty clay	Silty clay loam
Mineral composition	Kaolinite, illite, vermiculite	Kaolinite, illite, vermiculite	Illite

1.3 Spiking of soil with Pb^{2+}

In general, the Pb concentration in soil was in the range of 2–200 mg/kg (Liang *et al*., 2003). However, the highest Pb content can reach 1143 mg/kg in some soils (Chen, 1996). Thus, artificially contaminated soils were used in this study. The soils were spiked with Pb^{2+} at a rate of 100, 500, and 1000 mg/kg air-dried soil. The Pb^{2+} spiking process was conducted according to the method in the literature (Gao *et al*., 2003; Saison *et al*., 2004). First, analytical reagent $Pb(NO_3)_2$ was diluted in water. The $Pb(NO_3)_2$ solution was then added to 100 g of soil to reach a high Pb^{2+} concentration. Certain amounts of the dried and grounded soil samples spiked with Pb^{2+} were thoroughly mixed with the control soil. After sieving through a 100-mesh sieve, the contaminated soil samples with a series of final spiked concentrations of Pb^{2+} were obtained at 0, 100, 500, and 1000 mg/kg.

1.4 Adsorption experiments

Batch equilibrium experiments were conducted to evaluate the adsorption of BSM on the three paddy soils. The sieved soils (0.3 g) were weighed into 25-mL glass centrifuge tubes with polytetrafluoroethylene screw caps, followed by 15 mL of different BSM-spiked level background solutions containing 0.1 mol/L $CaCl_2$ (pH 5) solutions with 0.05% NaN_3 to inhibit aerobic biodegradation. The ratio of soil to solution was 1:5 to achieve 30%–70% of total solute adsorbed. The tubes were shaken for 24 h at (25 ± 1)°C and 250 r/min in the dark on a gyratory shaker. The solution and soil were separated by centrifugation at 4500 r/min for 15 min. The supernatant was filtered through a syringe of 0.22-μm Millipore membrane (ANPEL Co., Ltd., China, Φ13 mm), and the filtrate was analyzed for residual BSM concentration by high-performance liquid chromatograph (HPLC). Tubes without soil were prepared as controls for each batch experiment to monitor the loss of BSM. The control series did not indicate any significant BSM loss caused by photochemical degradation, volatilization, or adsorption to vials during the course of the experiment. Three replications were conducted in each treatment.

HPLC analysis was carried out on a Waters Alliance 2695-2487 HPLC system fitted with a reverse phase C-18 column (3.9 × 150 mm, Waters, USA). The flow rate was 0.6 mL/min. The mobile phase was composed by 0.5% glacial acetic acid (30%) and methanol (70%), and the injection volume was 10 μL. The detection wavelength of phenanthrene was 240 nm. Chromatography was performed at 30°C. The retention time of BSM amounts was about 4.5 min in this condition.

1.5 Data analysis

The data were subjected to two-way ANOVA with related procedure in software package Statistical Package for the Social Science (SPSS) and the data processing system (DPS) on the computer.

2 Results and discussion

2.1 Adsorption isotherm of BSM on paddy soils

The adsorption isotherms of BSM on the three tested paddy soils was well described with correlation constants (R) greater than 0.93 (Fig. 1 and Table 2). The equilibrium could be well described quantitatively by Freundlich equation (Eq. (1)):

$$C_s = K_f C_e^{1/n} \quad (1)$$

where, C_s is the concentration of BSM adsorbed on the solid phase, C_e is the concentration in solution of BSM at equilibrium, and K_f and n are empirical constants which are related to the adsorption phenomenon. The constant n is Freundlich exponent related to adsorption intensity (dimensionless) (Hamdaoui and Naffrechoux, 2007), and it may be used as an index of linearity (decreasing with increasing nonlinearity) (Si *et al*., 2003; He *et al*., 2006). The constant n for the three types of paddy soils BCP, DMP, PQR in the absence of Pb^{2+} was 0.96, 1.03, and 1.27, respectively (Table 2).

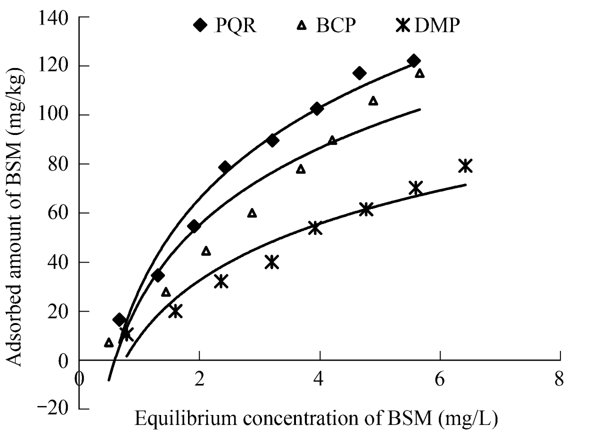

Fig. 1 Adsorption isotherms of BSM on three paddy soil without Pb^{2+} addition.

Table 2 Freundlich parameters for adsorption isotherm and free energy (ΔG^0) for BSM adsorption on three paddy soils spiked with various amounts of Pb^{2+} at 25°C

Soil type	Pb^{2+} content (mg/kg)	K_f	n	R	K_{OM}	ΔG^0 (kJ/mol)
PQR	0	33.1	1.27	0.9831	2429	19.3
	100	34.4	1.53	0.9976	2522	19.4
	500	36.7	1.90	0.9967	2692	19.6
	1000	38.7	2.42	0.9959	2840	19.7
BCP	0	19.7	0.96	0.9985	494.4	15.4
	100	23.4	1.19	0.9932	586.3	15.8
	500	29.5	1.43	0.9916	741.5	16.4
	1000	31.4	1.64	0.9965	788.2	16.5
DMP	0	13.56	1.03	0.9970	396.1	14.8
	100	13.9	1.04	0.9971	397.6	14.8
	500	14.5	1.08	0.9859	412.6	14.9
	1000	14.9	1.11	0.9892	426.1	15.0

$K_{OM} = K_f$/OM.

Knowledge of the adsorption-desorption parameters was essential to estimate the persistence and mobility of BSM in soil system. K_f is a constant indicative of the relative adsorption capacity of the adsorbent (Hamdaoui and Naffrechoux, 2007; Coquet, 2002; Liang, 2004). Hence, the adsorptive capabilities of three paddy soils for BSM decreased in the sequence: PQR (K_f = 33.10) > BCP (K_f = 19.70) > DMP (K_f = 13.56). In contrast, the pH of the three soil types was in the sequence: PQR < BCP < DMP (Table 1). Obviously, adsorption of BSM on soil decreased with increasing pH value, which was in accordance with the result obtained by Si *et al*. (2003) that there existed a significant negative correlation between the pH value and the adsorption of BSM on soil. BSM is a weak acid with a pK_a of 5.2, and at a higher pH aqueous phase existed as anions. A decrease in pH would promote the existence of BSM as neutral forms, which had higher hydrophobicity and could easily be adsorbed on the surface of soils (Liao and Xie, 2007).

It was reported that the physical and chemical properties of soils, including SOM, clay content, and pH were the dominating factors during the adsorption-desorption of the adsorption of BSM (Finocchiaro *et al*., 2005; Si *et al*., 2003; Chen *et al*., 1995). BSM mobility increased with increasing soil pH and decreasing SOM (Beyer *et al*., 1988; Finocchiaro *et al*., 2005; Si *et al*., 2003). Table 1 shows that the SOM content in BCP soil was 65.8% and 11.6% greater than that in PQR and in DMP, respectively. Clearly, the adsorption of BSM on soils did not increase with the increase of SOM in the present study. Soil organic matter was a solid phase with pH-dependent functional groups (Gevao *et al*., 2000). Additionally, the charge properties of clay, charge density of clay, and the existence of the state of pesticide were influenced by soil pH. Therefore, a possible explanation for this result might be that soil pH was one of the controlling key factors (Chen *et al*., 1995) and overshadowed the influence of other factors such as organic matter and clay content on the adsorption of BSM.

2.2 Adsorption characteristics of BSM on paddy soils in the presence of Pb^{2+}

The adsorption of BSM on the 3 paddy soils in the presence of Pb^{2+} could also be well described by Freundlich model quantitatively (R > 0.98). The addition of Pb^{2+} to the paddy soils increased K_f values. For instance, K_f in PQR, BCP, and DMP spiked with Pb^{2+} was 1.26 times, 2.01 times, and 1.08 times that of the control unspiked soil, respectively, when the added Pb^{2+} concentration in soil reached 1000 mg/kg. Moreover, the constant K_f tended to increase with increasing Pb^{2+} concentration in soil (Table 2). It was indicated that the presence of Pb^{2+} in paddy soils promoted the adsorption of BSM, and the higher contents of Pb^{2+} would generally lead to a stronger adsorption of BSM on soils. These results showed that external Pb^{2+}

in soils would enhance the retention of BSM, and thus effectively retard BSM from entering the aqueous phase.

The parameter n in the three paddy soils in the presence of Pb^{2+} were also changed. When the added Pb^{2+} concentration in soil reached 1000 mg/kg, n in PQR and BCP increased to 2.42 and 1.64, respectively. The parameter n in PQR showed a more significant variation, which increased from 1.27 in the control soil to 2.42 in the soil spiked with Pb^{2+} at 1000 mg/kg.

2.3 Mechanism of the enhanced adsorption of BSM on paddy soils in the presence of Pb^{2+}

The adsorption of BSM in the paddy soils increased with increasing content of Pb^{2+} addition. The report about mechanism of extraneous Pb^{2+} affecting BSM adsorption in paddy soil was scarce.

First, the standard free energy change (ΔG^0) of BSM in paddy soils was studied. Based on Gibbs Equation, the standard molar adsorptive ΔG^0 is calculated by Eq. (2) (Yang *et al*., 1995):

$$\Delta G^0 = -RT\ln K_{\mathrm{OM}} \tag{2}$$

where, R is universal gas constant and T is absolute temperature, and K_{OM} =100 × K_{f}/OM%. The value of 40 kJ/mol was considered as a threshold for identifying the physical and chemical mechanisms of adsorption, and physical adsorption mainly was involved below the threshold (Carter *et al*., 1995). Whatever Pb^{2+} was added to the soil, the ΔG^0 of BSM in three paddy soils was less than 40 kJ/mol (Table 2). The results indicate that the adsorption of BSM on the paddy soils was mainly a physical process, even with the presence of Pb^{2+}. The adsorption of BSM on the paddy soils also unspontaneous process for the positive value of ΔG^0.

The study by Ouyang *et al*. (2003) concluded that the adsorption of BSM on goethite occurred through carbonyl group and sulfonyl group of BSM with hydrate groups on the surface of goethite, where the adsorption was hydroxyl conducted by means of charge-dipole interaction (mainly hydrogen bonding). There was hydrophobic interaction between BSM and the surface of soil (Liao and Xie, 2007).

The retention (adsorption) of Pb by solid phases in soil is governed by various mechanisms including adsorption (ion exchange, specific adsorption) onto surface active minerals, diffusion in mineral structures, precipitation as carbonates and hydroxides (McLean and Eledsoe, 1992; Reed and Cline, 1994). Specific chemical interaction was the major mechanism responsible for the adsorption process of Pb^{2+} adsorption onto soil (Weng, 2004). Obviously, the adsorption mechanisms of Pb^{2+} and BSM on soils were different.

In this work, Pb^{2+} was spiked to the soil ahead of the BSM adsorption experiment, which might result in the modification of some soil properties. Therefore, the investigation on the modified properties of soils caused by the spiked Pb^{2+} may be a possible and effective way to elucidate the effect of external Pb^{2+} on the adsorption of BSM on paddy soils.

First of all, soil pH was affected by external Pb^{2+} loading. It was slightly decreased with increasing Pb^{2+} loading. Result from the study of Li *et al*. (2007) indicated that the addition of Pb^{2+} at 1200 mg/kg decreased soil pH by 0.4 and 0.3 units, respectively, for the Inceptisol and Ultisol, as compared with the control, indicating that Pb^{2+} contamination can cause soil acidification. The enhanced acidification of the soil by Pb^{2+} pollution is likely related to the exchange of surface H^+ and/or Al^{3+} by the added Pb^{2+} (Yang *et al*., 2006). It had been established that the adsorption of BSM on soil increased with decreasing pH. It was therefore concluded that the promotion effect of the spiked Pb^{2+} on the adsorption of BSM might attribute to the soil acidification.

As an important active component in the soil-ecosystem, the presence of DOM tended to reduce the adsorption of herbicide due to DOM-herbicide interactions and/or competition for adsorption sites on soil particles (Cox *et al*., 2000) and could enhance the aqueous solubility of organic pollutants to such an extent that transport through soil can be significantly influenced (Haberhauer *et al*., 2002). It was then speculated that the spiked Pb^{2+} in soil would also modify the quantity of DOM in soil and thereby play an important role in enhancing the adsorption of BSM on soil. In the present study, K_{f} values of BSM isotherm adsorption increased in all three paddy soils while the inherent DOM was removed from the soil solids (Fig. 2, Table 3). Taking PQR as an example, the K_{f} value (37.2) for BSM adsorption by eluted soil was accordingly greater than the K_{f} value (33.1) by the control soil ($p < 0.01$). The results indicate that the elution of soil inherent DOM enhances the adsorption of BSM in tested paddy soils. Consequently, it was considered that the adsorption of BSM on soil solids would be inhibited by the presence of soil DOM in a similar manner.

In addition, the increment of K_{f} values of PQR, BCP, and DMP resulted from DOM elution were 4.1, 9.8, and 1.3, respectively (Table 3), correlated positively with the SOM contents in the paddy soils. The association between SOM and adsorption of BSM on soils was then concerned by speculating that the elution of soil inherent DOM would result in the decrease of SOM content and thus reduce adsorption of BSM.

Actually, adsorption of pesticides by soils was closely related to the SOM content (Hutson and Roberts, 1990). When the organic carbon in the medium was more than 5.00 mg/kg, SOM was the dominant material for the adsorption of organic compounds (Jaynes and Boyd, 1991). It also had been reported that the partitioning into SOM was the main mechanism for the adsorption of organic

Table 3 Freundlich parameters for isotherm adsorption of BSM on the three paddy soils affected by DOM

Soil type	Condition	K_{f}	n	R
PQR	Control	33.1	1.27	0.9914
	DOM-free	37.2	1.11	0.9853
BCP	Control	19.7	0.96	0.9975
	DOM-free	29.5	0.89	0.9919
DMP	Control	13.6	1.03	0.9983
	DOM-free	14.9	1.01	0.9976

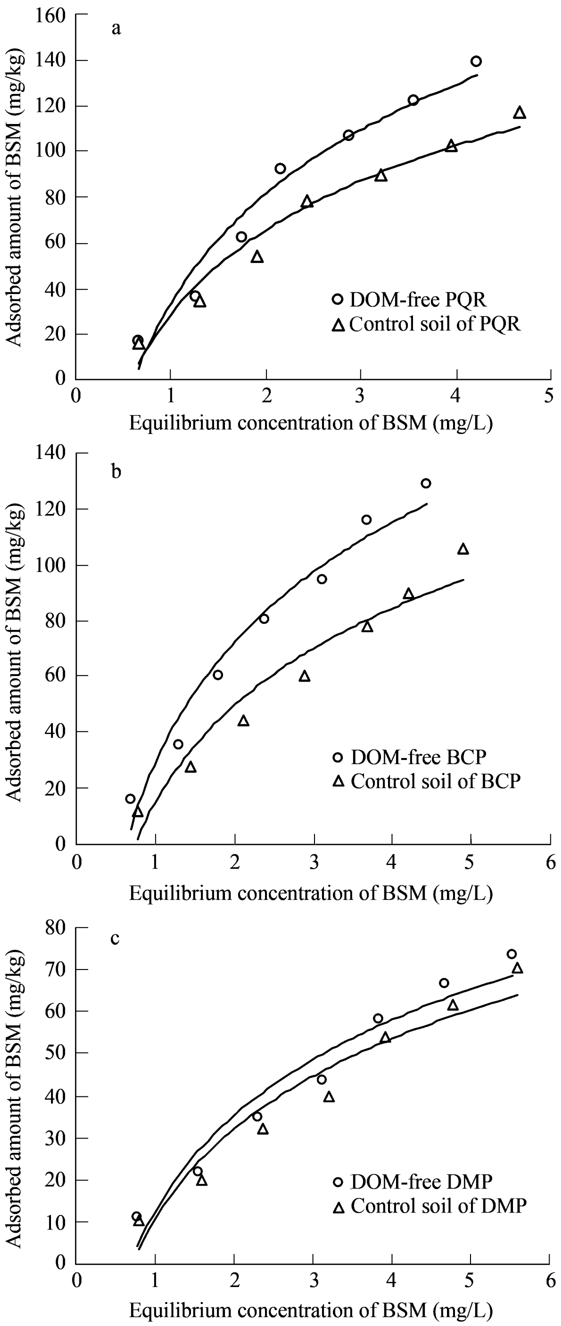

Fig. 2 Adsorption isotherms of BSM on DOM-free PQR (a), BCP (b) and DMP (c).

contaminant on soils (Hwang and Cutright, 2002; Gao *et al*., 2007).

When the paddy soil was spiked with Pb^{2+}, there is a markable increase in the adsorption of BSM by paddy soils based on the observed K_f values. Heavy metals would possibly influence the adsorption of organic pollutants in soil by affecting DOM concentration, as suggested by Gao *et al*. (2003). The metallic cations had been known could form a complex with functional groups of organic molecules in solution, and this leads to the formation of "bridges" between soil solid surface and DOM in aqueous phase (Gao *et al*., 2003; Saison *et al*., 2004). Due to Pb's chemical characteristics (relatively high electronegativity, low hydrolysis constant, small hydrated radius, and electronic structure), Pb^{2+} absorbed on solid phase has a greater affinity for most functional groups of DOM including carboxylic and phenolic groups (McBride, 1994). As a result, the spiked Pb^{2+} in soil tended to reduce the DOM concentration in the equilibrium solution, and the reduced part of DOM in the equilibrium solution could be adsorbed by the solid phase in soil, which would lead to an increase of SOM content and thus strengthen the adsorption of BSM on soils.

Consequently, the mechanisms involved in the promotion effects of the spiked Pb^{2+} on adsorption of BSM might be the modified surface characteristics of paddy soil solids due to the soil acidification and the increase of SOM content because of DOM binding.

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