

Effects of phosphate on the adsorption of glyphosate on three different types of Chinese soils

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Abstract: Glyphosate (GPS) is a non-selective, post-mergence herbicide that is widely used throughout the world. Due to the similar molecular structures of glyphosate and phosphate, adsorption of glyphosate on soil is easily affected by coexisting phosphate, especially when phosphate is applied at a significant rate in farmland. This paper studied the effects of phosphate on the adsorption of glyphosate on three different types of Chinese soils including two variable charge soils and one permanent charge soil. The results indicated that Freundlich equations used to simulate glyphosate adsorption isotherms gave high correlation coefficients (0.990–0.998) with *K* values of 2751, 2451 and 166 for the zhuanhong soil (ZH soil, Laterite), red soil (RS, Udic Ferrisol) and Wushan paddy soil (WS soil, Anthrosol), respectively. The more the soil iron and aluminum oxides and clay contained, the more glyphosate adsorbed. The presence of phosphate significantly decreased the adsorption of glyphosate to the soils by competing with glyphosate for adsorption sites of soils. Meanwhile, the effects of phosphate on adsorption of glyphosate on the two variable charge soils were more significant than that on the permanent charge soil. When phosphate and glyphosate were added in the soils in different orders, the adsorption quantities of glyphosate on the soils were different, which followed GPS-soil > GPS-P-soil = GPS-soil-P > P-soil-GPS, meaning a complex interaction occurred among glyphosate, phosphate and the soils.

Keywords: soil; glyphosate; phosphate; competitive adsorption

Introduction

Glyphosate is a non-selective, post-mergence herbicide that is widely used in agronomic and vegetable crops, and in orchards. More than ten thousands of tons of glyphosate are consumed each year, which production is the third one among all chemical pesticides produced in China (Ouyang, 2000). Soil is one of the important sinks of chemical pollutants, and adsorption of glyphosate on soil will affect its mobility and degradation.

Glyphosate has three functional groups (amine, carboxylate and phosphonate) that can form strong coordination bonds with most metal ions to give bidentate and tridentate complexes (Glass, 1984; McBride, 1989; Undabeytia, 2002; Wang, 2004; Zhou, 2004). The sorption of glyphosate on soils includes specific and non-specific adsorptions. The phosphate moiety of glyphosate was responsible for its strong adsorption to soils and that phosphate capacity is related directly to glyphosate adsorption (Hance, 1976; Prata, 2003; Gimsing, 2004a; 2004b). Recently, Dideriksen and Stipp (Dideriksen, 2003) studied the mechanism of glyphosate adsorption to goethite with a molecular-scale atomic force microscopy.

The phosphate content in soils had a significant influence on the sorption of glyphosate (Jonge, 2001). With increasing phosphate content in soil, the adsorption of glyphosate on soils decreased. Gimsing *et al.* (Gimsing, 2001) studied the competitive adsorption of glyphosate and phosphate on soils. They found that phosphate had stronger affinity to soils than glyphosate. Phosphate can desorb glyphosate from soils, but glyphosate does not affect phosphate adsorption. The competition for adsorption sites of soil may imply that phosphate influences the mobility of glyphosate and enhance its transport from soil to surface water and ground water. Soil characteristics had a significant effect on the competitive adsorption of glyphosate and phosphate.

However, limited such studies were performed using variable charge soils, which contain large amounts of Fe and Al oxides facilitating glyphosate adsorption.

There are varieties of soil types distributed in China. In this paper, the adsorption of glyphosate on three types of soils including two variable charge soils and one permanent charge soil was investigated in the absence and presence of phosphate, and the effects of different adding orders of glyphosate and phosphate were considered to further understand their competitive adsorption processes.

1 Materials and methods

1.1 Soils and chemicals

Three surface soils (0–20) including a zhuanhong soil (ZH soil, Laterite), a red soil (RS, Udic Ferrisol) and a Wushan paddy soil (WS soil, Anthrosol) were sampled from Changshu County in Jiangsu Province, Yintan County in Jiangxi Province, and Xuwen County in Hainan Province, respectively. The dominant minerals of the WS soil contain hydromica, vermiculite and montmorillonite, and the dominant minerals of the ZH soil and RS contain kaolinite, gibbsite and hematite. These soil samples were air-dried, passed through a 60 mesh screen, and stored in glass bottles for further use. Some physical and chemical characteristics of these three soils were listed in Table 1. Soil organic matter (OM) was determined by the method of dichromate oxidation, the content of soil total Fe and Al oxides were determined by digestion of HF-HClO₄-HCl, soil cation exchange capacity (CEC) was determined by the method of NH₄Ac exchange, and the clay content is determined by the sucker method (Lu, 1999). The WS soil has higher contents of organic matters and CEC than the other two soils, but the ZH soil and RS have higher contents of total Fe and Al oxides and clay than the WS soil.

Glyphosate, as a non-residual herbicide, was purchased from Sigma Co. All other chemicals are of analytical grade.

Deionized water was used for all experiments.

Table 1 Some physical and chemical characteristics of soils

Soil	pH	CEC, cmol/kg	O.M., %	AlO, %	FeO, %	Clay content, %
ZH	4.58	11.4	1.42	29.9	17.5	75.6
RS	4.95	14.1	0.76	17.4	6.57	56.2
WS	7.20	23.6	4.57	15.4	5.55	34.3

1.2 Glyphosate adsorption isotherms on the soils in the absence and presence of phosphate

Glyphosate adsorption isotherms on the soils with and without phosphate were performed by adding 5.0 ml of 0.01 mol/L NaCl solution with different concentrations (0–1.00 mmol/L) of glyphosate in each polyethylene centrifuge tube containing 0.2 g soil. The solution glyphosate concentration series were 0, 0.125, 0.25, 0.375, 0.5, 0.625, 0.75 and 1.00 mmol/L. When phosphate was considered as a coexisting chemical, 0.5 or 1.0 mmol/L phosphate was respectively contained in the above NaCl solutions in each series of solution glyphosate concentrations. The final solution volume was 20 ml. The centrifuge tubes were continuously shaken for 20 h at 25 °C, and then centrifuged and filtrated through a filter membrane. The glyphosate concentration in the centrifuged solution was determined by HPLC. Phosphate was determined by method of molybdenum-stibium-ascorbic acid (Lu, 1999). The amount of glyphosate and phosphate adsorbed was calculated from the differences between its solution concentrations before and after equilibrium. All solution pHs after equilibrium were measured by a pH meter (PH-3B, Shanghai Leici Instrumental Factory, China). All experiments were performed in two replicates.

1.3 Effects of pH on glyphosate adsorption on soils in the absence and presence of phosphate with different adding orders

Glyphosate-soil: 0.2 g soil was weighed in every 50 ml centrifuge tube, and 5.0 ml of 0.01 mol/L NaCl solution containing 2.0 mmol/L glyphosate was added. And then, different volumes of 0.01 mol/L NaOH or 0.01 mol/L HCl solution were added in each tube to adjust solution pH varying from 3 to 9. In following, 0.01 mol/L NaCl solution was supplied into each tube and the final solution volume was 20 ml. The centrifuge tubes were continuously shaken for 20 h at 25 °C, and then centrifuged and filtrated through a filter membrane. The glyphosate concentration in the centrifuged solution was determined by HPLC. The amount of glyphosate adsorbed was calculated from the difference between its solution concentrations before and after equilibrium. All solution pHs after equilibrium were measured by a pH meter.

Glyphosate-P-soil: 0.2 g soil was weighed in every 50 ml centrifuge tube. 5.0 ml of 2.0 mmol/L glyphosate and 5.0 ml of 2.0 mmol/L P containing 0.01 mol/L NaCl as electrolyte were premixed for 1 h and then added into the above centrifuge tube containing 0.2 g soil. And then, different volume of 0.01 mol/L NaOH or 0.01 mol/L HCl solution was added in each tube to adjust solution pH varying from 3 to 9. In following, 0.01 mol/L NaCl solution was supplied into each tube and the final solution volume was 20 ml. The centrifuge tubes were continuously shaken for 20 h at 25 °C and were centrifuged and filtrated through a filter membrane. The glyphosate concentration in the centrifuged solution was determined by HPLC. The amount of glyphosate

adsorbed was calculated from the difference between its solution concentrations before and after equilibrium. All solution pHs after equilibrium were measured by a pH meter.

Glyphosate-soil-P: 0.2 g soil was weighed in every 50 ml centrifuge tube, and 5.0 ml of 0.01 mol/L NaCl solution containing 2.0 mmol/L glyphosate was added. And then, different volumes of 0.01 mol/L NaOH or 0.01 mol/L HCl solution were added in each tube to adjust solution pH varying from 3 to 9. In following, 0.01 mol/L NaCl solution was supplied into each tube and the final solution volume was 15 ml. The centrifuge tubes were continuously shaken for 10 h at 25 °C. In following 5.0 ml of 0.01 mol/L NaCl solution with 2.0 mmol/L P was added in above centrifuge tube, which were shaken for 10 h at 25 °C again. The equilibrium solution was then centrifuged and filtrated through a filter membrane. The glyphosate concentration in the centrifuged solution was determined by HPLC. The amount of glyphosate adsorbed was calculated from the difference between its solution concentrations before and after equilibrium. All solution pH after equilibrium were measured by a pH meter.

P-soil-glyphosate: 0.2 g soil was weighed in every 50 ml centrifuge tube, and 5.0 ml of 0.01 mol/L NaCl solution containing 2.0 mmol/L P was added. And then, different volume of 0.01 mol/L NaOH or 0.01 mol/L HCl solution was added in each tube to adjust solution pH varying from 3 to 9. In following, 0.01 mol/L NaCl solution was supplied into each tube and the final solution volume was 15 ml. The centrifuge tubes were continuously shaken for 10 h at 25 °C. In following, 5.0 ml of 0.01 mmol/L NaCl solution with 2.0 mmol/L glyphosate was added. The centrifuge tubes were shaken for 10 h at 25 °C again. The equilibrium solution was then centrifuged and filtrated through a filter membrane. The glyphosate concentration in the centrifuged solution was determined by HPLC. The amount of glyphosate adsorbed was calculated from the difference between its solution concentrations before and after equilibrium. All solution pHs after equilibrium were measured by a pH meter.

1.4 Chemical analysis of glyphosate and phosphate

The glyphosate concentration in the centrifuged solution was determined by an Agilent 1100 series HPLC (Agilent Co., USA), using a 4.6 × 150 mm Zorbax ODS column. At first, 0.50 ml supernatant content of glyphosate was derived with 130 mmol/L *p*-toluenesulphonyl chloride in acetonitrile (1:1 by volume) for 5 min at 50 °C. Derivatized samples (20 µl) were then injected in the chromatography column equilibrated with 50 mmol/L sodium phosphate (pH 2.3), containing 15% (V/V) acetonitrile. Elution proceeded at a flow rate of 1.0 ml/min, monitoring the eluate at 240 nm to analyze the concentration of glyphosate (Kawai, 1991; Zhou, 2004).

2 Results and discussion

2.1 Adsorption isotherms of glyphosate on the soils in the absence and presence of phosphate

Fig. 1 shows the adsorption isotherms of glyphosate on the three soils in the absence and presence of phosphate with solution phosphate concentrations of 0, 0.5 and 1.0 mmol/L, respectively. It indicated that in all cases glyphosate adsorption quantities on the soils increased with increasing glyphosate concentrations in the equilibrium solutions. The

ZH soil and RS as variable charge soils had higher adsorption capacity for glyphosate than the WS soil as a permanent charge one. The contents of iron oxides (Table 1) in the ZH soil, RS and WS soil were 17.48%, 6.57% and 5.55%, respectively, and the contents of aluminum oxides in the ZH soil, RS, and WS soil were 29.85%, 17.37% and

15.40%, respectively. So, the ZH soil and WS soil have the highest and lowest contents of iron and aluminum oxides, respectively. Previous results also showed that adsorption of glyphosate on soil was highly related to the soil iron and aluminum oxides and clay contents (Piccolo, 1994; Zhou, 2004).

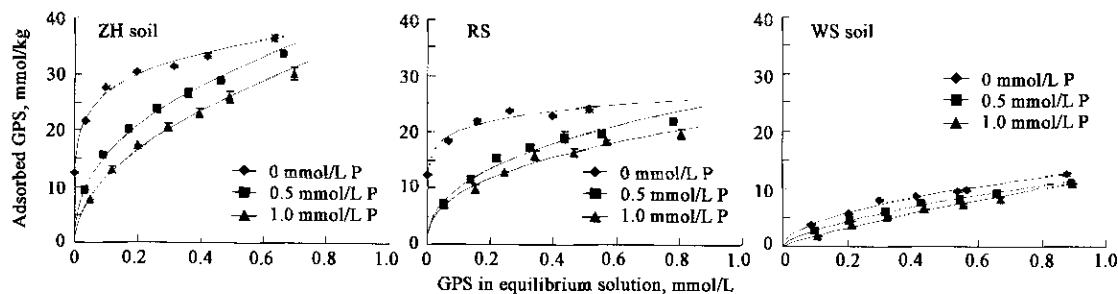


Fig. 1 Glyphosate (GPS) adsorption isotherms on three soils in the absence and presence of phosphate

Although the WS soil has the highest organic matter content (4.57%) and CEC (23.63 mmol/kg) (Table 1), glyphosate adsorption on this soil was the lowest. It suggests that soil Fe and Al oxides play more important roles on adsorption of glyphosate than soil CEC and organic matter, although soil CEC and soil organic matter were also responsible for the adsorption of glyphosate on soil (Morillo, 2002).

When phosphate coexisted with glyphosate in the equilibrium solution, adsorption of glyphosate on the soils decreased significantly (Fig. 1). Phosphate competed with glyphosate for adsorption sites of soils because of their similar functional groups. It was previously reported that the phosphate content in the soil samples taken from long-term field experiments that received different addition of phosphorus had a significant influence on the adsorption of glyphosate (Jonge, 2001). With 0.5 mol/L bicarbonate extractable P increasing from 6.2 to 58.7 mg/kg in the loamy sand and 9.1 to 87.4 mg/kg in the coarse sand, the Freundlich adsorption coefficient of glyphosate on the soils decreased from 215 to 106 and from 154 to 83.5, respectively. Glyphosate is bound to soils through the phosphonic acid moiety and competes with inorganic phosphate for adsorption sites. Addition of 98 or 196 kg/hm² of phosphate decreased glyphosate inactivation in the soil (Sprinkle, 1975). As the initial level of phosphate increased, adsorption of glyphosate decreased (Dion, 2001). Addition of as little as 5.3 mmol/L of phosphate caused a significant decrease for adsorption of glyphosate. But as initial level of glyphosate increased, the effect of phosphate tended to have less impact on glyphosate adsorption. A molecular-scale atomic force microscopy study showed that both phosphonic and carboxylic groups of glyphosate specifically adsorbed in a 1:2 ratio with singly coordinated hydroxyl groups of goethite, but phosphate specially adsorbed in 1:1 ratio with the singly coordinated hydroxyl groups (Dideriksen, 2003).

Freundlich equation was applied to simulate glyphosate adsorption isotherms on the soils, and the results show in Table 2. These equations fitted the adsorption isotherms of glyphosate on the three soils very well with high correlation coefficients (0.990–0.998). The coefficient K is an

indicator of sorption strength, which were 2751, 2451 and 166 for the ZH soil, RS and WS soil, respectively. In the presence of phosphate, the Freundlich adsorption coefficient K significantly decreased with increasing phosphate concentration. When 0.5 mmol/L phosphate was added, the coefficient K decreased to 783, 501 and 84 on the ZH soil, RS, and WS soil, respectively. The more the phosphate added, the more the coefficient K decreased. Meanwhile, the effect of phosphate on adsorption of glyphosate on the ZH soil and RS as variable charge soils were more significant than that on the WS soil as a permanent charge one, similar to the results reported by Gimsing *et al.* (Gimsing, 2002).

Table 2 Freundlich equations and their correlation coefficients corresponding to glyphosate adsorption isotherms on three soils in the absence and presence of phosphate

Soil	Phosphate, mmol/L	Simulated Equation ($Q = K C^n$)	Correlation coefficients (r)
ZH soil	0	$Q = 2751 C^{0.1726}$	0.997***
	0.5	$Q = 783.3 C^{0.4265}$	0.997***
	1.0	$Q = 455.9 C^{0.5193}$	0.998***
RS	0	$Q = 2451 C^{0.1193}$	0.990***
	0.5	$Q = 501.0 C^{0.4331}$	0.992***
	1.0	$Q = 473.0 C^{0.4101}$	0.990***
WS soil	0	$Q = 166.2 C^{0.5122}$	0.996***
	0.5	$Q = 84.00 C^{0.6251}$	0.993***
	1.0	$Q = 31.19 C^{0.818}$	0.990***

Notes: C , glyphosate in equilibrium solution, mg/L; Q , adsorption quantity of glyphosate on soil, mg/kg; *** significant at 0.001 probability levels

No intentional control of solution pH was performed for the batch experiments of glyphosate adsorption isotherms on the soils. Table 3 shows the equilibrium solution pH after different concentrations of phosphate were added. When phosphate coexisted with glyphosate in the soils, it decreased the solution pH, especially at low concentration of glyphosate. Glyphosate is a weak acid with functional groups of amine, carboxylate and phosphonate. When the concentration of glyphosate increased, its buffer ability to counteract with solution acid and alkali increased, led to little effect on the solution pH by low concentration of P addition. In addition, the pH change in the WS soil was more significant than that in the ZH soil and RS.

Soil solution pH generally has a strong influence on the adsorption of glyphosate on soils (McConnell, 1985). Morillo

et al. (Morillo, 2002) and Zhou *et al.* (Zhou, 2004) studied the adsorption of glyphosate on soils in different equilibrium solution pH values, and found that the higher the solution pH is, the less glyphosate adsorbed on soils or minerals. Recently, Gimsing *et al.* (2004a) concluded that soil pH seemed to be the only important factor in determining the amount of glyphosate that could be adsorbed by the soils, and other factors such as organic carbon, the clay content and the mineralogy of the clay fraction had no effect on glyphosate adsorption. However, as shown in Fig. 1, the adsorption of glyphosate varied with different soils even if the equilibrium solution pH was the same.

Table 3 Equilibrium solution pH of different concentrations of glyphosate equilibrating with three soils in the absence and presence of phosphate

Soil GPS, mmol/L	Phosphate, mmol/L								
	ZH soil			RS			WS soil		
	0	0.50	1.0	0	0.50	1.0	0	0.50	1.0
	Solution pH								
0	5.73	5.50	5.44	4.20	4.04	3.97	6.79	6.59	6.36
0.125	5.34	5.20	5.16	3.78	3.73	3.68	6.36	6.13	5.98
0.25	4.95	4.86	4.81	3.50	3.45	3.42	6.00	5.82	5.77
0.375	4.36	4.50	4.46	3.25	3.22	3.22	5.58	5.46	5.39
0.50	4.09	4.12	4.18	3.07	3.06	3.06	5.29	5.24	5.16
0.625	3.83	3.84	3.96	2.93	2.93	2.94	5.07	5.02	4.96
0.75	3.67	3.63	3.75	2.82	2.83	2.84	4.89	4.84	4.82
1.0	3.31	3.34	3.38	2.64	2.66	2.67	4.62	4.60	4.49

Although the decreased equilibrium solution pH in the presence of phosphate, favored glyphosate adsorption, adsorption quantity of glyphosate decreased in this case because phosphate had stronger competitive absorption ability on soil surface than glyphosate. The results are the same for all the three types of soils with different characteristics.

Fig. 2 indicates the effects of glyphosate on the adsorption of phosphate on soils. Adsorption quantities of phosphate under the same initial concentration on the RS and ZH soil were the same, but were higher than that on the WS soil. The presence of glyphosate in the concentration range of 0—1.0 mmol/L did not affect the adsorption of phosphate on the soils, which further indicated that glyphosate had weaker adsorption ability than phosphate.

2.2 Adsorption of glyphosate on the soils with different adding orders and solution pH

In order to further understand the competitive adsorption between glyphosate and phosphate, the adsorption behaviors of glyphosate and phosphate with different adding orders and solution pHs were investigated, and the results are shown in

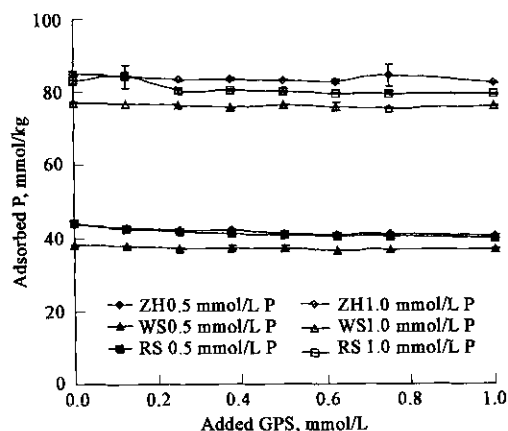


Fig.2 Adsorption of phosphate on soils in the absence and presence of glyphosate

Fig.3. It showed that the presence of phosphate significantly decreased glyphosate adsorption on the three types of soils in a wide pH range. But, different adding orders affected their interaction. When glyphosate was equilibrated with the soils first, the higher adsorption quantity of glyphosate was obtained. However, when phosphate was equilibrated with the soil first and then with glyphosate, it had the less adsorption quantity.

Phosphate strongly adsorbed on the adsorption sites of soil when it was first added into the equilibrium solution, because the affinity between soil and phosphate was much stronger than that with glyphosate. Glyphosate almost cannot desorb phosphate from soil. On the contrary, phosphate can desorb glyphosate from soil. Gimsing and Borggaard (Gimsing, 2001) studied the competitive adsorption of glyphosate and phosphate on goethite, and also found that only a negligible amount of glyphosate adsorbed in the presence of phosphate, but phosphate was adsorbed strongly in the presence of glyphosate.

In addition, for all three types of soils, the adding orders by glyphosate adsorption in the soils first and then by phosphate gave similar adsorption quantity as by the mixture of glyphosate and phosphate to adsorb in the soils. It is proposed that although glyphosate has weaker adsorption strength than phosphate, its adsorption rate is quicker than phosphate (Gimsing, 2004b). So, even phosphate existed with glyphosate in the equilibrium solution at the same time, its adsorption quantity kept almost the same with that by adding glyphosate to the soil solution first and then by phosphate.

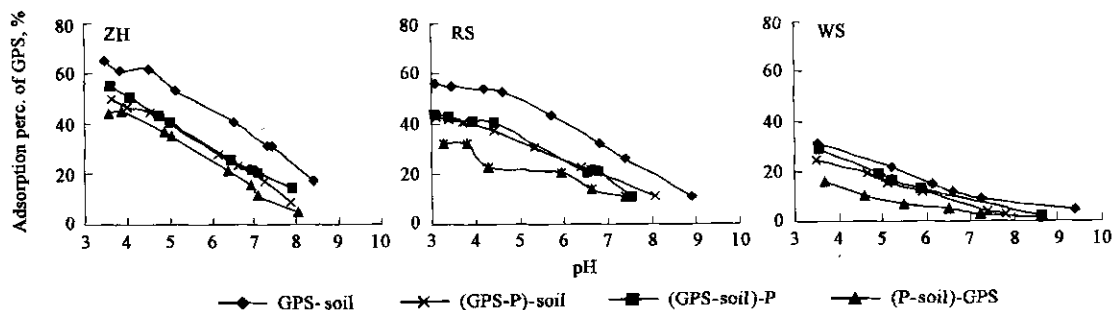


Fig.3 Effects of equilibrium solution pH on glyphosate adsorption on soils in the presence and absence of phosphate with different equilibrium orders ($C_{\text{glyphosate}} : C_{\text{phosphate}} = 1$)

3 Conclusions

Adsorption of glyphosate on soils was related to soil characteristics. The soils with higher contents of Fe and Al oxides and clay adsorbed more glyphosate, which can decrease the environmental risk of glyphosate after application. Phosphate, which has higher affinity to soils than glyphosate, competed with glyphosate for the adsorption site of soils when they coexisted. Applying glyphosate in soil containing higher content of phosphate will possibly increase the environmental risk of glyphosate transferring from soil to groundwater and surface water.

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