



# Phosphate and glyphosate sorption in soils following long-term phosphate applications



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## ABSTRACT

Phosphate and glyphosate molecules compete for sorption sites in soil. The objective of this study was to quantify the impact of Olsen P concentrations in two contrasting soils on phosphate and glyphosate sorption. Soils were a sandy clay loam soil rich in iron oxides (SCL-Fe<sub>2</sub>O<sub>3</sub>) and a clay loam soil rich in calcium carbonates (CL-CaCO<sub>3</sub>). The phosphate Freundlich sorption coefficient (K<sub>f</sub>) ranged from 3 to 68 L<sup>1/n</sup> mg<sup>1-1/n</sup> kg<sup>-1</sup> in the SCL-Fe<sub>2</sub>O<sub>3</sub> and from 21 to 76 L<sup>1/n</sup> mg<sup>1-1/n</sup> kg<sup>-1</sup> in the CL-CaCO<sub>3</sub>. Glyphosate sorption coefficient (K<sub>d</sub>) ranged from 293 to 1173 L kg<sup>-1</sup> in the SCL-Fe<sub>2</sub>O<sub>3</sub> but only 99 to 141 L kg<sup>-1</sup> in the CL-CaCO<sub>3</sub>. Glyphosate K<sub>d</sub> and phosphate K<sub>f</sub> values decreased significantly with increasing Olsen P concentrations in both soils. Glyphosate K<sub>d</sub> values were further significantly reduced when phosphate was added to the slurry solutions, but phosphate K<sub>f</sub> values were not impacted by the presence of glyphosate in solutions. We conclude that annual phosphate fertilizer applications leave phosphate concentrations in Prairie soils to the extent that soils have a lesser capacity to retain glyphosate and phosphate that are subsequently applied, but glyphosate residues will not influence phosphate sorption.

## 1. Introduction

Sorption is an important process that influences the transport and availability of nutrients and pesticides in soil (Duke et al., 2012; Hiller et al., 2012; Ige et al., 2005). Phosphate is an essential nutrient for crop growth and annually applied to agricultural soils (Gomes et al., 2015). Glyphosate [*N*-(phosphonomethyl) glycine] is a broad spectrum, non-selective systemic herbicide and among the most widely used pesticides in the world (Duke and Powles, 2009; FOEE, 2013).

Amorphous Fe and Al oxides in soil are the preferred sorption sites for phosphate and glyphosate molecules (Barja and Afonso, 2005; Gimsing et al., 2004; Piccolo et al., 1994). Phosphate is preferentially sorbed by Fe-oxides because the phosphate molecule (0.25 nm) is smaller than the glyphosate molecule (0.43 nm) (Gimsing et al., 2007; Gimsing and Borggaard, 2001), but glyphosate is retained by Fe-oxides through both phosphonic acid and carboxylic acid moieties (Gimsing and Borggaard, 2001; Sprankle et al., 1975; Tévez and Afonso, 2015). Phosphate readily forms complexes with Ca<sup>2+</sup> in calcareous soils (Busman et al., 2009) and glyphosate can form stable complexes with Ca<sup>2+</sup> in solution and sorb (Glass, 1987) or form weak bonds with exchangeable Ca<sup>2+</sup> associated with clays (de Jonge et al., 2001). Glyphosate can also be sorbed onto clay minerals (de Jonge et al., 2001), divalent cations of inter layer clay minerals (Piccolo et al., 1994) and

organic matter (Morillo et al., 2000).

Sorption of phosphate and glyphosate by soil is typically determined using soil slurries in batch-equilibrium experiments (de Jonge et al., 2001; Gimsing et al., 2004; Gimsing and Borggaard, 2002; Ige et al., 2005). Phosphate and glyphosate molecules compete for sorption sites in soil and adding phosphate and glyphosate to soil slurries at the same time can influence sorption of the individual chemicals (de Jonge and de Jonge, 1999). For example, in a range of batch equilibrium studies, glyphosate sorption by soil was less when phosphate was added with glyphosate (Gimsing et al., 2007; Gimsing and Borggaard, 2007; Kaniserry et al., 2015). Based on desorption experiments using batch equilibrium, two studies have shown that phosphate in soil has the potential to increase glyphosate desorption (Laitinen et al., 2008; Prata et al., 2003). In these studies, phosphate was applied to soil at 14 and 28 days prior to the start of batch equilibrium experiments, and glyphosate desorption increased by 1 to 13%, relative to soils that had not received phosphate. A recent study generated rainfall runoff at one day after glyphosate applications on a silty clay loam soil and showed that glyphosate concentrations in runoff were greater for plots that had also received phosphate applications (Sasal et al., 2015).

Recently, concerns have been raised about the possibility of glyphosate increasing the transport of phosphate to surface water thereby increasing the risk for lake eutrophication (Barrera, 2016). In batch

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equilibrium studies, when glyphosate was added at approximately 1000 to 1500 kg glyphosate ha<sup>-1</sup> rate, glyphosate additions to soils were able to release some phosphate in acidic soil particularly in soils high in Al and Fe-oxides (Gimsing et al., 2004). Additions of the same amount of glyphosate as Gimsing et al. (2004), also released phosphate molecules bound to synthesized Al-oxides and but this release was not observed with synthesized Fe-oxides (Gimsing and Borggaard, 2001, 2002). Generally, farmers apply phosphate fertilizer at 20 to 40 kg P ha<sup>-1</sup> and ~1 kg glyphosate ha<sup>-1</sup> on their crop farms (Akinremi and Grant, 2014; Benbrook, 2016). Thus, these previous studies showed that glyphosate has the potential to release phosphate but the concentrations of glyphosate used were much greater than environmentally relevant. It is unknown whether glyphosate can influence the retention of phosphate in calcareous soils. In our study, we utilized a soil high in Fe-oxides and a soil high in calcium carbonates and, for these soils, we were interested in testing whether there is any potential effect of glyphosate on phosphate sorption. Like previous studies that focused on acidic soils and Al and Fe-oxides (Gimsing et al., 2004; Gimsing and Borggaard, 2001, 2002), we utilized an amount of glyphosate that was much larger than relevant field doses.

Batch equilibrium studies of glyphosate or phosphate sorption typically use either 0.01 M CaCl<sub>2</sub> or 0.01 M KCl as background electrolyte solutions (Bhattacharyya et al., 2015; de Jonge et al., 2001; Gimsing and Borggaard, 2001, 2007; Okada et al., 2016). The batch equilibrium process does not distinguish between sorption and precipitation (Siebsen, 1981; Akinremi, 1990; Muhammad, 1992). At pH 7, the background electrolyte solution strongly influenced the sorption of phosphate by Fe-oxides because with 0.01 M CaCl<sub>2</sub>, phosphate formed complexes with Ca<sup>2+</sup> and precipitated, while this did not occur with 0.01 M or 0.1 M KCl (Gimsing and Borggaard, 2001). However, under the same conditions, the background electrolyte solution had little influence on glyphosate sorption by Fe-oxides (Gimsing and Borggaard, 2001).

Long-term phosphate fertilizer applications result in the build-up of phosphate in agricultural soils (de Jonge et al., 2001). Crops only utilize a small portion of the phosphate applied because sorption limits the availability of phosphate for plant uptake in soil (Holford, 1997). Long-term phosphate-fertilizer applications to sandy clay loam and loamy sand soils in India reduced the ability of the soils to retain phosphate molecules, as determined by batch equilibrium experiments (Bhattacharyya et al., 2015; Varinderpal-Singh and Brar, 2006). Long-term phosphate fertilizer applications to a sandy clay loam soil in Canada and coarse sand and sandy loam soils in Denmark resulted in fertilized soils showing significantly lesser glyphosate sorption than untreated soils, also as determined by batch equilibrium experiments (de Jonge et al., 2001; Munira et al., 2016; Munira & Farenhorst, 2017). The combination of fresh phosphate applications to soils containing field-aged phosphate and the impact of these phosphate concentrations on glyphosate sorption has received no attention except for Munira et al. (2016).

It is unknown whether increasing field-aged-phosphate concentrations have the same impact on reducing phosphate sorption as they have on reducing glyphosate sorption. In this study, we used a soil high in Fe-oxides (pH 4.7 to 5) versus a soil high in calcium carbonates (pH 7.3 to 7.5) that were both subjected to long-term phosphate fertilizer applications at 20, 40 and 80 kg P ha<sup>-1</sup> resulting in a range of Olsen P concentrations due to the build-up of phosphate in soil during eight years of annual applications. Using 0.01 M KCl and 0.01 M CaCl<sub>2</sub> as background electrolyte solutions, the objective of this study was to examine the impact of 1) field-aged phosphate concentrations and a commercially available glyphosate formulation on phosphate sorption in soils rich in iron oxides or calcium carbonates and 2) field-aged phosphate concentrations in combination with fresh phosphate on glyphosate sorption in these two contrasting soils.

## 2. Materials and methods

### 2.1. Chemicals

Chemicals used were analytical grade glyphosate (99.9% purity) from Sigma-Aldrich Co., St. Louis, MO; [phosphonomethyl-<sup>14</sup>C]glyphosate (99% radiochemical purity; specific activity 50 mCi/mmol) from American Radiolabeled Chemicals Inc., St. Louis, MO; Roundup Ultra2® (49% active ingredient and 51% other ingredients, CAS No. 70901-12-1) from Monsanto Chemical Company; and analytical grade potassium dihydrogen phosphate (KH<sub>2</sub>PO<sub>4</sub>) (99% chemical purity), potassium chloride (100% chemical purity) and calcium chloride, dihydrate (> 95% chemical purity) from Fisher Scientific, Fair Lawn, NJ.

### 2.2. Soil characteristics and experimental design

This study utilized soil samples (0–15 cm) obtained from long-term experimental plots under a durum wheat and flax rotation near Carman (49° 29.7' N, 98° 2.4' W) and near Forrest (50° 1.2' N, 99° 53.3' W) Manitoba, Canada. Soil profiles at both sites were classified based on the Canadian System of Soil Classification as Orthic Black Chernozems, which is equivalent to the Udic Boroll subgroup in the U.S. Soil Taxonomy (Soil Classification Working Group, 1998). The experimental design at each site was a randomized complete block design with four mono ammonium phosphate fertilizer treatments and four replicates plots. Treatments were a control (no phosphate applications), and plots receiving annual applications of mono ammonium phosphate fertilizers at 20, 40, and 80 kg P ha<sup>-1</sup>, (Grant et al., 2013) or 20P, 40P, and 80P, respectively, from 2002 to 2009. For all plots that received mono ammonium phosphate, 20 kg P ha<sup>-1</sup> was placed near the seed to enhance fertilizer use efficiency, a common practice in Canadian Prairie agriculture. For the 40 and 80 kg P ha<sup>-1</sup> treatments, to avoid seedling toxicity, the additional mono ammonium phosphate was broadcast and then incorporated. From 2010 to 2013, the rotation was continued but no phosphate was applied. Application of urea fertilizer differed by year. Generally, durum wheat received 90 kg N ha<sup>-1</sup> and flax 50 kg N ha<sup>-1</sup>.

From each plot, composite samples were collected in spring, 2013 using a Dutch auger with ten (Carman) to eight (Forrest) samples per plot and cleaning the auger between plots. Soil samples were air-dried and sieved (< 2 mm) prior to soil property analyses and sorption experiments. The Carman soil has a sandy clay loam texture and is relatively high in iron oxides (SCL-Fe<sub>2</sub>O<sub>3</sub>), whereas the Forrest soil has a clay loam texture and is relatively high in calcium carbonates (CL-CaCO<sub>3</sub>) (Table 1). Available phosphate was extracted using the Olsen (0.5 M NaHCO<sub>3</sub>, pH 8.5) phosphorus test. 2 g of air dried soil and 40 mL of 0.5 N NaHCO<sub>3</sub> solution was mixed in a 50 mL Erlenmeyer flask. Flasks (duplicates) were shaken horizontally (200 excursions min<sup>-1</sup>). Equilibrium solutions were filtered through Whatman No. 2 filter paper and phosphate concentrations were determined colorimetrically (Frank et al., 2011).

### 2.3. Phosphate sorption

Phosphate sorption was determined by batch equilibrium using either 0.01 M CaCl<sub>2</sub> or 0.01 M KCl as the background electrolyte. Batch equilibrium procedures followed standard protocols using a soil/solution ratio of 1:10 and an equilibrium time of 24 h (Ige et al., 2005). Two experiments were conducted utilizing soil samples: (1) from all plots at each site to quantify the effect of Olsen P concentrations on phosphate sorption in soil and (2) from control and 80P plots at each site to quantify the effect of Roundup Ultra2 additions to soil slurries on phosphate sorption in soil.

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