

## Adsorption of glyphosate and aminomethylphosphonic acid in soils

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*Received February 15, 2012; accepted October 26, 2012*

**A b s t r a c t.** The results showed that glyphosate is initially adsorbed mostly in the upper 2 cm. It is then transported and adsorbed after few days in deeper soil horizons with concomitant increasing content of its metabolite aminomethylphosphonic acid. Moreover, Fe-oxides seem to be a key parameter for glyphosate and aminomethylphosphonic adsorption in soils. This study confirmed previous studies: the analysis showed lower contents of dithionite-soluble and Fe-oxides for the Chernozem, with consequently lower adsorption of glyphosate and aminomethylphosphonic acid as compared with the Cambisol and the Stagnosol.

**K e y w o r d s:** adsorption, glyphosate, aminomethylphosphonic acid, soils

### INTRODUCTION

Soils play an important role in the regulation of contaminants in ecosystems. Identification and understanding of the mechanisms controlling the fate of chemicals as a source of environmental contamination, especially in soils and water are of major concern. A better understanding for the organic pollutant behaviour in soils is important for the improvement of environmental protection.

The behaviour of organic contaminants in soils is generally governed by a variety of physical, chemical and biological processes, including sorption-desorption, volatilization, chemical and biological degradation, uptake by plants, run-off, and leaching (Mamy *et al.*, 2005).

Organophosphonates are released into the environment in enormous quantities and, among the non-selective herbicides, glyphosate is applied at a volume corresponding with about 60% of the global sales (Candela *et al.*, 2007). Glyphosate is a polar, highly water-soluble substance that easily forms complexes with metals and binds tightly to soil components (Ghanem *et al.*, 2007; Gimsing *et al.*, 2004; Schnurer *et al.*, 2006).

Glyphosate (N-(phosphonomethyl)glycine) is the active compound in Roundup Max, a post-emergency non-selective broad spectrum herbicide widely applied in agricultural practice. Glyphosate itself is an acid, but it is commonly used as a salt, most commonly as isopropylammonium salt.

The persistence is typically up to 170 days, with a half-life time of 45-60 days (Peruzzo *et al.*, 2008). Some studies however, show a half-life time of years. The major degradation product of glyphosate is aminomethylphosphonic acid (AMPA), (Gimsing *et al.*, 2004; Locke and Zablotowicz, 2004; Peruzzo *et al.*, 2008).

The glyphosate fate and behaviour in soil are affected by different soil factors and processes, but depends also on interactions between herbicide and soil under the specific local conditions (Gimsing *et al.*, 2004; Locke and Zablotowicz, 2004; Soulas and Lagacherie, 2001). Thus, traces of this herbicide have been found in many surface- and groundwater systems, (Landry *et al.*, 2005; Peruzzo *et al.*, 2008). Consequently, farmers should strive for improved management practices in the use and release of these chemicals. This calls for considering the pesticide type and application rates as well as the characteristics of the application site (Soulas and Lagacherie, 2001).

One of the key problems for obtaining reliable results from field samples is to control the possible biodegradation of glyphosate during storage and preparation of the soil samples because the glyphosate content can be influenced by soil microbes.

The aim of this study was to investigate the behaviour of glyphosate and AMPA at different soils and time intervals after field application.

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## MATERIAL AND METHODS

The experiments were carried out at agricultural experimental fields, where different tillage systems: no-tillage (NT), direct drill, no plough, with a winter green vegetation cover and maize crop in spring, and conventional tillage (CT), plough with or without a winter green vegetation cover in 3 field replications are tested since 2007 (Kirchberg, Styria), 1999 (Pyhra and Pixendorf, Lower Austria).

Three soils under different climatic conditions and featuring different physico-mineral composition were investigated: a sandy stagnic Cambisol (WRB, 2006; Nestroy *et al.*, 2000) at Kirchberg (Styria) from tertiary carbonate free sediments, a loamy Stagnosol (WRB, 2006; Nestroy *et al.*, 2000) from carbonate free sediments (flysch, sandstone) at Pyhra (Lower Austria) and a Chernozem (WRB, 2006; Nestroy *et al.*, 2000) from loess at Pixendorf (Lower Austria). Moreover, these three soil types were selected because of their contrasting physico-chemico-mineralogical parameters *eg* texture, carbonate content, pH-value, and Fe-oxides for a better understanding of their influence on the glyphosate behaviour and extraction from soils. All three sites were under comparable tillage systems (no-tillage and conventional tillage) in long-term experiments (Klik *et al.*, 2010).

The soils investigated are representative for the main agricultural regions of Austria, where Cambisols – 50%, Chernozems – 18%, and Stagnosols cover approximately – 10% of the agricultural land use of Austria (Haslmayr, 2010).

The Roundup Max application was performed at all three sites according to the common agricultural practice *ie* 4 l Roundup Max (450 g glyphosate / l Roundup Max) were dissolved in 200 l of water and applied per ha (2 % herbicide solution). This corresponds to an application of 1 800 g glyphosate ha<sup>-1</sup> or 180 mg glyphosate m<sup>-2</sup>. The application was carried out at sunny and not windy weather at the NT-plots.

Soil bulk samples from all plots (NT and CT) were taken for physico-chemico-mineralogical analysis at each site at two soil depths (0-5 and 5-20 cm), collected from 10 different points/field replication. The samples were air-dried and sieved at 2 mm size (fine earth). Moreover, for further physical analysis undisturbed samples (cylinders with 200 cm<sup>3</sup>) were taken separated from each NT and CT field replication at 5-15 cm soil depth each in 5 repetitions.

In order to investigate the fate of glyphosate and AMPA in depth and time after Roundup Max application, soil bulk samples were taken at different time intervals after application at 10 points within each NT-field replication (pooled than to one sample per site) as follows:

Kirchberg:

- immediately after the Roundup Max application, at 0-2 cm soil depth;
- 3 days after application at 0-2 and 2-5 cm soil depth;
- 12 days after application at 0-2, 2-5, and 5-10 cm soil depth;

Pyhra:

- immediately after the Roundup Max application, at 0-2 cm soil depth;
- 28 days after application at 0-2, 2-5, and 5-10 cm soil depth;

Pixendorf:

- immediately after the Roundup Max application, at 0-2 cm soil depth;
- 3 days after application at 0-2 and 2-5 cm soil depth;
- 10 days after application at 0-2, 2-5, and 5-10 cm soil depth.

After each soil sampling soil samples were immediately transported to the laboratory in cooling boxes. In the laboratory all samples were stored at -18 °C until measurements. All physical, chemical and mineralogical analyses were carried out according to the standard methods.

## RESULTS AND DISCUSSION

The investigated soils show their distinguished formation through very different textures. The Chernozem shows the development from loess with typical high silt content. The Stagnosol has a heterogeneous texture, typical for a loamy soil and the high amount of silt and clay explains the water stagnation of this soil. The Cambisol is a sandy soil, with over 50% mass of sand fraction.

There is a distinguished difference between the CT-plots and the NT-plots. NT-plots show higher bulk density and lower total porosity than (CT) plots. This compaction is known from the literature and is due to a natural settlement of particles free from tillage practices. As a consequence, there is a loss of coarse pores which leads to a slightly diminished available water capacity (Table 1).

The silty Chernozem is slightly alkaline with a medium carbonate content. The siliceous Stagnosol and Cambisol are weakly acidic, according to their formation conditions. The contents of soil organic matter decrease with soil depth (Table 2).

Pedogenic Fe-(Al)-oxides are important indicators for *eg* weathering intensity and redox processes in soils. They play also a major role as absorbers for glyphosate in soils. The dithionite-soluble Fe(Fe<sub>d</sub>) indicates the total amount of pedogenic formed Fe-oxides, that means organically bound, amorphous and well crystallized. This Fe-extraction does not allow any conclusion about the type of Fe-oxide (goethite is however the most relevant Fe-oxide in Central Europe).

The Chernozem features a low content, the Stagnosol a medium content and the Cambisol a high content of Fe-oxides (Table 3). From this, the expected sorption capacity for glyphosate and AMPA increases respectively from the Chernozem, over the Stagnosol to the higher weathered Cambisol. All sites show a low Fe-oxides/ dithionite-soluble Fe(Fe<sub>o</sub>/Fe<sub>d</sub>) ratio which indicates that in all soils investigated the most relevant amount of pedogenic Fe-oxides is well crystallized (Table 3).

Table 3 shows the mineral distribution of the soils under investigation.

**Table 1.** Bulk density (BD), total porosity ( $P_{total}$ ) (as sum of all pores), pore size distribution and available field capacity (aFC) the investigated soils at the CT and NT plots

Site	Soil type (WRB)	Plot	Depth (cm)	DB ( $Mg\ m^{-3}$ )	$P_{total}$	Pore size distribution (% vol.)				aFC (mmWC)
						cP	ncP	mP	fP	
Pixendorf	Chernozem	CT	5-15	1.51	43.2	12	5	15	11	20
		NT		1.62	38.8	9	4	15	12	19
Kirchberg	Cambisol	CT	5-15	1.62	38.8	8	6	15	9	21
		NT		1.74	34.3	5	4	16	9	20
Pyhra	Stagnosol	CT	5-15	1.47	44.5	10	6	16	12	21
		NT		1.65	39.2	7	4	15	13	19

cP – coarse pores ( $> 50\ \mu m$ ), ncP – narrow coarse pores ( $50-10\ \mu m$ ), mP – medium pores ( $10-0.2\ \mu m$ ), fP – fine pores ( $< 0.2\ \mu m$ ), aFC – available field capacity in mm water column (WC) as the sum of narrow coarse pores and medium pores, whereas 1% vol. pore space = 1mm WC.

**Table 2.** General chemical parameters of the investigated soils

Site	Soil type (WRB)	Depth (cm)	pH		$CaCO_3$	$C_{anorg}$	$C_{org}$	OM	pH $CaCl_2$	C/N
			NT	CT						
Pixendorf	Chernozem	0-5	0.13	3.7	14.7	1.80	1.86	3.2	7.3	14.8
		5-20	0.11	3.1	15.4	1.85	1.25	2.2	7.3	11.4
Kirchberg	Cambisol	0-5	0.13	1.7	<0.5	0.0	1.73	3.0	5.7	13.3
		5-20	0.11	1.5	<0.5	0.0	1.51	2.6	5.8	13.7
Pyhra	Stagnosol	0-5	0.12	1.6	<0.5	0.0	1.61	2.8	5.7	13.4
		5-20	0.10	1.2	<0.5	0.0	1.17	2.0	5.6	12.2

**Table 3.** Fe-oxide distribution in the investigated soil

Site	Soil type (WRB)	Depth (cm)	$Fe$ distribution ( $mg\ kg^{-1}$ )			$Fe_o/Fe_d$
			$Fe_p$	$Fe_o$	$Fe_d$	
Pixendorf	Chernozem	0-5	37	983	7 970	0.12
		5-20	39	1 040	8 378	0.12
Kirchberg	Cambisol	0-5	530	3 422	14 843	0.23
		5-20	569	3 726	15 032	0.25
Pyhra	Stagnosol	0-5	550	3 241	9 959	0.32
		5-20	538	3 215	9 918	0.32

$Fe_p$  – organic bound Fe-oxides, pyrophosphate-soluble,  $Fe_o$  – ‘amorphous’ (weakly crystallized) Fe-oxides, oxalate-soluble,  $Fe_d$  – well crystallized Fe-oxides, dithionite-soluble.

**Table 4.** Semiquantitative total mineral distribution (% mass) in the investigated soil

Site	Soil type (WRB)	Depth (cm)	Mineral distribution (%)					
			Micas	Quartz	Feldspars	Calcite	Dolomite	Pyrite
Pixendorf	Chernozem	0-20	47	24	12	10	7	0
Kirchberg	Cambisol	0-20	42	31	27	0	0	0
Pyhra	Stagnosol	0-20	45	37	18	0	0	0

All investigated soils are characterized by the dominance of layer silicates, mostly micas. Quartz is detectable in all soils, so feldspars, but less in the carbonatic Chernozem. Carbonates (calcite and dolomite) are represented in the loess Chernozem as expected, whereas the siliceous Stagnosol and Cambisol are free of carbonates according to their genetical formation (Table 4). The soils show a distinguished high content of expandable clay minerals (smectite and vermiculite), especially in the Stagnosol where they amount up to > 50%, which explains the stagnic characteristics of that soil. Illite was found in all soils but with higher amounts in the Cambisol and Stagnosol. Kaolinite is a typical indicator for acidic conditions and reaches the highest contents in the acidic Cambisol. Chlorite was detected in all soils, but it must be distinguished into a 'primary' chlorite as a mineral of loess in the Chernozem, and a 'secondary' chlorite (pedogenetically formed) in the Stagnosol and in the Cambisol. In all soils traces of mixed layers minerals were found (Table 5).

Many results in the literature show that Fe-oxides play an important role in the soil retention of glyphosate. From the investigated Fe-oxides (ferrihydrite, hematite and goethite) ferrihydrite had the highest impact on the adsorption process of glyphosate (Mentler *et al.*, 2007).

The variation of the KD-values in different soils is significant (Mentler *et al.*, 2007) and seems to depend mainly on the Fe-oxide content (Table 6).

The results of the investigations of the Chernozem are shown in Fig. 1.

At the first sampling after field application of Roundup Max about 30% of the applied glyphosate amount was detected in the upper 0-2 cm. The main part of the herbicide adheres at the green plant cover and at first does not enter the soil surface.

After 3 days the glyphosate content decreased in the topsoil and was transported and adsorbed in the next horizon (2-5 cm) with concomitant increase of the AMPA content. After 10 days the glyphosate content was higher than immediately after application.

For this behaviour following hypothesis can be possible:

- plant-adsorbed glyphosate is released to the topsoil after partly decomposition of the weeds;
- during the time between second and third soil sampling about 10 mm precipitation fell down, this may have washed glyphosate from plant leaves out.

The increase of AMPA 3 days after application of Roundup Max shows the very quick degradation of glyphosate to AMPA. This degradation could probably take place already in the Roundup Max package, this would explain the fact that AMPA was detected immediately after the Roundup Max application.

The results of the investigations of the Stagnosol are shown in Fig. 2.

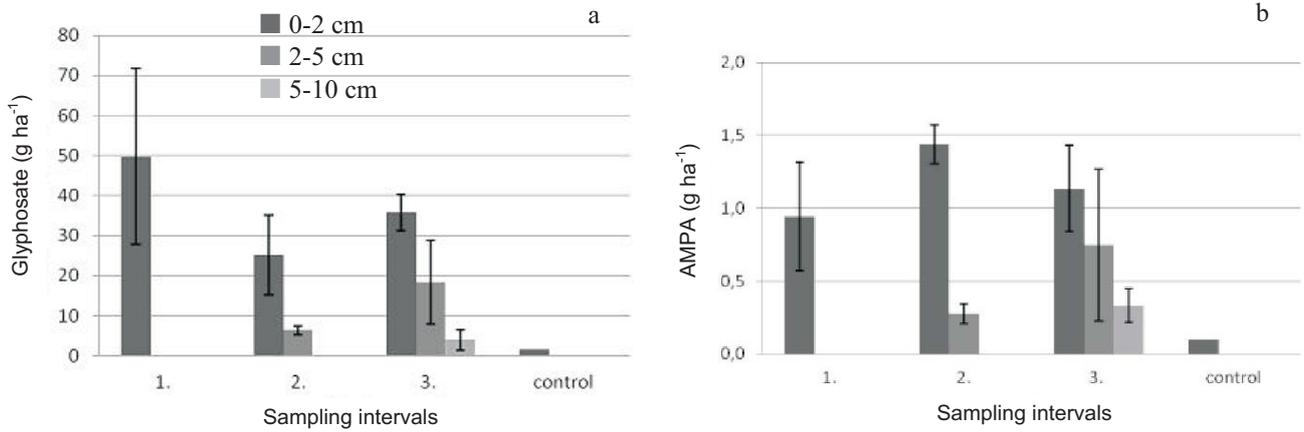
Most of the applied glyphosate was transported and adsorbed in deeper horizons after 28 days. The reference glyphosate and AMPA values in Figs 3 and 4 refer the amount of both substances before application *eg* the residues of the previous application (normally 2 years before). That means that in the Stagnosol glyphosate is transported downwards within 2 years and probably bound to deeper soil layers.

**Table 5.** Semiquantitative clay mineral distribution (mass %) in the investigated soil

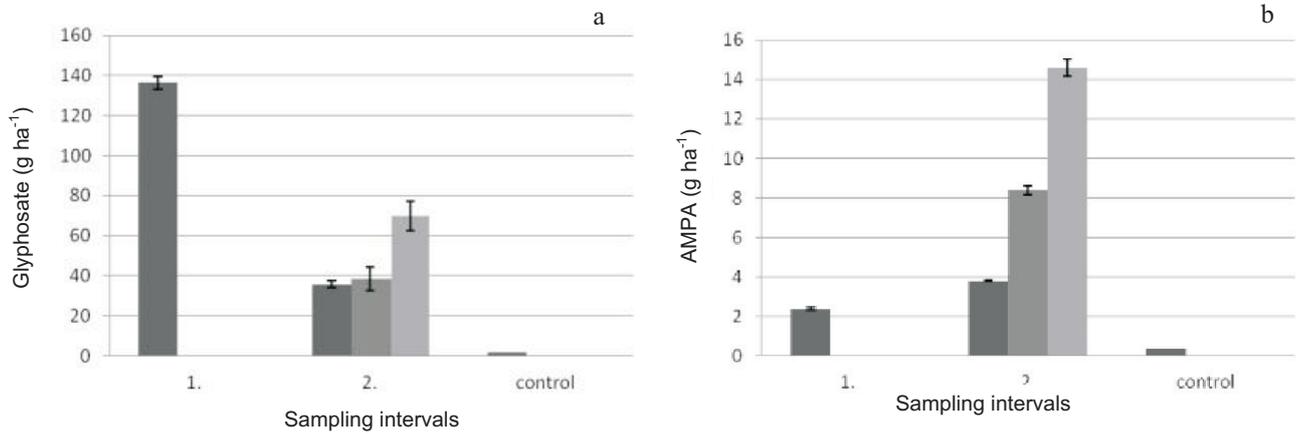
Site	Soil type (WRB)	Depth (cm)	Smectite	Vermiculite	Illite	Clay minerals (%)		
						Kaolinite	Chlorite	Mixed layers
Pixendorf	Chernozem	0-20	20	11	49	10	10	Tr.
Kirchberg	Cambisol	0-20	17	8	48	24	3	Tr.
Pyhra	Stagnosol	0-20	27	28	34	8	3	Tr.

**Table 6.** Soil properties and KD-values for glyphosate for different soils and silica sand based on literature data (after Mentler *et al.*, 2007)

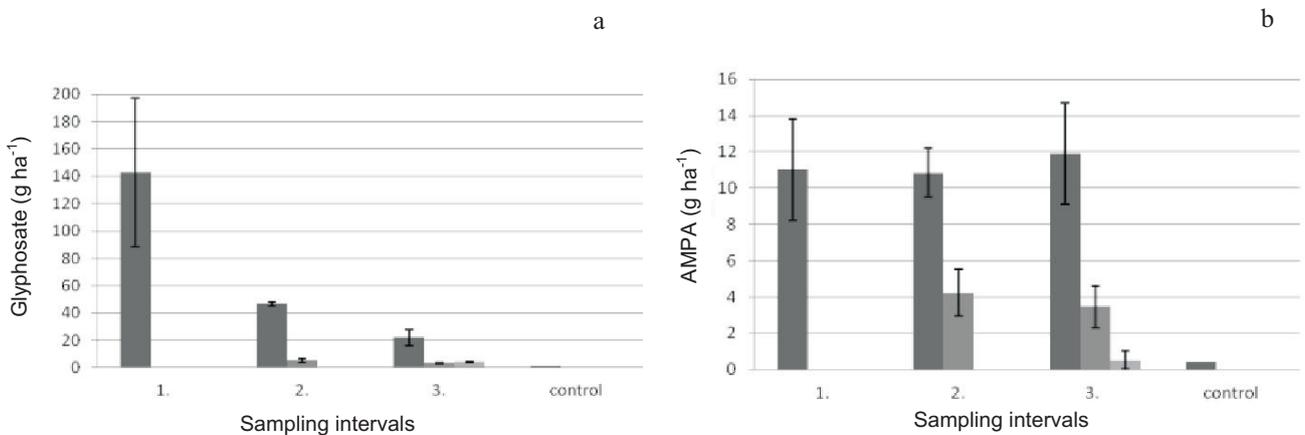
References	KD-value (l kg <sup>-1</sup> )	pH-value	Clay	C <sub>org</sub> (% mass)	Fe	Location
Mentler <i>et al.</i> , 2007	13.8-29.3	5.8	17.2	3.5	2.2	Pyhra
Mentler <i>et al.</i> , 2007	188-299	5.2	18.8	6.7	2.1	Eurosoil 7
Mentler <i>et al.</i> , 2007	1.5-2.9	6.4	<0.1	<0.01	<0.01	Silica sand
Sorensen <i>et al.</i> , 2006	271-385	4.3-5.6	2-4	0.1-4.9	0.01-0.05	Fladerne Beak
Mamy <i>et al.</i> , 2005	13.2-31.1	8.2-8.5	8.8-9.5	1.3-2	0.16-0.19	Chalons



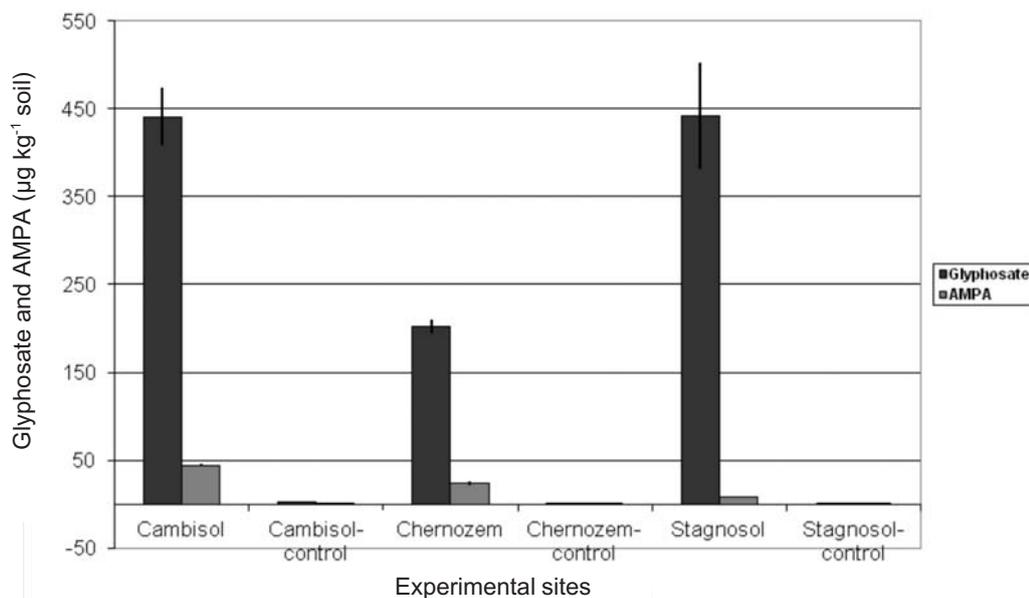
**Fig.1.** Content of: a – glyphosate and b – AMPA in Chernozem at different time intervals and soil depths, sampling: 1. – immediately after application, 2. – 3 days after application, 3. – 10 days after application; control – residues before application.



**Fig. 2.** Content of: a – glyphosate and b – AMPA in Stagnosol at different time intervals and soil depths, sampling: 1. – immediately after application, 2. – 28 days after application; control – residues before application.



**Fig. 3.** Content of: a – glyphosate and b – AMP contents in Cambisol at different time intervals and soil depths, sampling: 1. – immediately after application, 2. – 3 days after application, 3. – 12 days after application; control – residues before application.



**Fig. 4.** Glyphosate and AMPA contents in the investigated soils at 0-2 cm soil depth. The control values show the glyphosate contents before application (eg the residual traces, next to zero, from the previous application).

The results of the investigations of the Cambisol are shown in Fig. 3.

The Cambisol features the best potential adsorption capacity for glyphosate with about 16 000 mg Fe<sub>d</sub> kg<sup>-1</sup> soil and about 3 500 mg Fe<sub>o</sub> kg<sup>-1</sup> soil (Table 4), but previous studies with rain simulation experiments (Rampazzo Todorovic *et al.*, 2010) showed that site can be strongly influenced by erosion processes if the infiltration rate for rainfall is reduced by soil crusting.

This is the reason why glyphosate strongly decrease in the upper soil horizons but does not accumulate in deeper horizon. A considerable amount of the applied glyphosate may be transported downslope with runoff (Rampazzo Todorovic *et al.*, 2010).

Moreover, the degradation from glyphosate to its metabolite AMPA is visible by the increase of AMPA with time.

The three investigated soils, the most representative agricultural soils in Austria, differ through major contrasting chemico-mineralogical characteristics (Tables 3-6). The glyphosate and AMPA contents in the upper horizon of the three analyzed soils are shown in Fig. 4.

The results presented in Fig. 4 show distinguished contents of glyphosate and AMPA in different soils according to their chemical-mineralogical adsorption properties.

Based on previous studies, we expected that glyphosate and AMPA would be more strongly adsorbed in soils with a higher Fe-oxide (especially Fe<sub>d</sub>) content (Mentler *et al.*, 2007; Pessagno *et al.*, 2008). Our results show that both the Cambisol and the Stagnosol, with a higher pedogenic Fe-oxide content, 15 000 and 10 000 mg Fe<sub>d</sub> kg<sup>-1</sup> soil, respectively, (Table 4), adsorbed a distinctly higher quantity of

glyphosate and AMPA than the Chernozem which had a distinctly lower Fe-oxide content (7 900 mg Fe<sub>d</sub> kg<sup>-1</sup> soil, Table 4), and a KD-value about 10 times lower than the Cambisol (Klik *et al.*, 2010).

Moreover, the weakly weathered Chernozem consequently shows a low content of amorphous (Fe<sub>o</sub>) Fe-oxides (973 mg Fe<sub>o</sub> kg<sup>-1</sup> soil) with respect to the more highly weathered Cambisol (3 402 mg Fe<sub>o</sub> kg<sup>-1</sup> soil) and Stagnosol (3 279 mg Fe<sub>o</sub> kg<sup>-1</sup> soil) (Table 4). Higher content of pedogenic Fe-oxides (Fe<sub>d</sub>) (Barja *et al.*, 2001; Gimsing *et al.*, 2004; Morillo *et al.*, 2000; Zhou *et al.*, 2004) and even higher contents of amorphous Fe-oxides (Fe<sub>o</sub>) lead to higher sorption of glyphosate and AMPA, probably due to a larger and more reactive surface area of amorphous Fe-oxides. Thus, Fe-oxides in general seem to be a key parameter for glyphosate and AMPA adsorption in soils. Our study confirmed this: the analysis showed lower contents of Fe<sub>d</sub> and Fe<sub>o</sub> for the Chernozem, with consequently lower adsorption of glyphosate and AMPA compared with the Cambisol and the Stagnosol.

## CONCLUSIONS

1. No-tillage plots show a higher bulk density and a lower total porosity than conventionally tilled plots as due to a natural settlement of particles free from tillage practices.

2. Shortly after Roundup Max application only a part of the applied glyphosate amount enter the upper 0-2 cm and is then transported and adsorbed in deeper horizons with time with concomitant increase of the aminomethylphosphonic acid content.

3. The results showed distinguished contents of glyphosate and aminomethylphosphonic acid in different soils at the same soil depth, according to their chemical-mineralogical adsorption properties, especially Fe-oxides (Fe<sub>d</sub> and Fe<sub>o</sub>).

4. Thus, iron-oxides in general seem to be a key parameter for glyphosate and aminomethylphosphonic acid adsorption in soils.

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