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# Large variation in glyphosate mineralization in 21 different agricultural soils explained by soil properties



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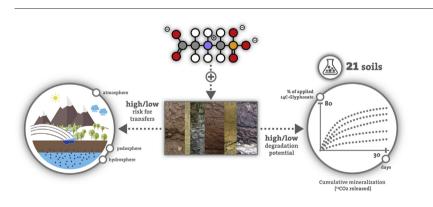
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#### HIGHLIGHTS

#### GRAPHICAL ABSTRACT

- Glyphosate mineralization ranged from 7 to 70% in 32 days, depending on soil composition.
- Glyphosate degradation in soils is strongly and mainly influenced by exchangeable acidity.
- A reliable parameter of soil potential to mineralize glyphosate is "NaOH-extractable residues".



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#### ABSTRACT

Glyphosate and its main metabolite aminomethylphosphonic acid (AMPA) have frequently been detected in surface water and groundwaters. Since adequate glyphosate mineralization in soil may reduce its losses to environment, improved understanding of site specific factors underlying pesticide mineralization in soils is needed. The aim of this study was to investigate the relationship between soil properties and glyphosate mineralization. To establish a sound basis for resilient correlations, the study was conducted with a large number of 21 agricultural soils, differing in a variety of soil parameters, such as soil texture, soil organic matter content, pH, exchangeable ions etc. The mineralization experiments were carried out with <sup>14</sup>C labelled glyphosate at a soil water tension of -15 kPa and at a soil density of 1.3 g cm $^{-3}$  at 20  $\pm$  1 °C for an incubation period of 32 days. The results showed that the mineralization of glyphosate in different agricultural soils varied to a great extent, from 7 to 70% of the amount initially applied. Glyphosate mineralization started immediately after application, the highest mineralization rates were observed within the first 4 days in most of the 21 soils. Multiple regression analysis revealed exchangeable acidity ( $H^+$  and  $AI^{3+}$ ), exchangeable  $Ca^{2+}$  ions and ammonium lactate extractable K to be the key soil parameters governing glyphosate mineralization in the examined soils. A highly significant negative correlation between mineralized glyphosate and NaOH-extractable residues (NaOH-ER) in soils strongly suggests that NaOH-ER could be used as a simple and reliable parameter for evaluating the glyphosate mineralization capacity. The NaOH-ER were composed of glyphosate, unknown <sup>14</sup>C-residues, and AMPA (12%–65%, 3%–34%, 0%–

\* Corresponding author at: Biotechnical Faculty, Center for Soil and Environmental Sci., University of Ljubljana, SI 1000 Ljubljana, Jamnikarjeva 101, Slovenia. E-mail address: marjetka.suhadolc@bf.uni-lj.si (M. Suhadolc). 11% of applied <sup>14</sup>C, respectively). Our results highlighted the influential role of soil exchangeable acidity, which should therefore be considered in pesticide risk assessments and management to limit efficiently the environmental transfers of glyphosate.

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#### 1. Introduction

Glyphosate [N-(phosphonomethyl)glycine] based herbicides (GlyBH) are the most widely used commercial formulations of pesticides worldwide (Jensen et al., 2009). GlyBH are applied to agricultural fields before planting the crop and/or pre- or post-harvest, in both conventional and reduced/no-till farming, to control the growth of annual and perennial weeds, both broad-leaved and grasses. Glyphosate was considered in the past to be harmless to the environment and to human health. However, in the recent years this evaluation was been questioned, with some studies associating its use with cancer and endocrine disruption in humans and acute and chronic toxicity to aquatic species. The European Union (EU) recently adopted a renewal of the approval of glyphosate for 5 years after intensive discussion (EFSA, 2015; IARC, 2015; Myers et al., 2016). It is important to note that numerous reviews concerning health effect of GlyBH report conflicting opinions, especially on the long-term effects of glyphosate, and on no-observedadverse effect levels (NOAEL) (US EPA, 1993; EC, 2002; US EPA, 2009; Antoniou et al., 2012; Mesnage et al., 2015; Niemann et al., 2015; Germany Rapporteur Member State, 2015).

There are also large discrepancies among studies evaluating the environmental fate of glyphosate after its use. Due to its high biodegradability (Landry et al., 2005; Grundmann et al., 2008) and strong adsorption in soil (Mamy et al., 2005), glyphosate has a weak (negligible) leaching potential (Borggaard and Gimsing, 2008; Klier et al., 2008). However, under some environmental conditions, for instance in soils with weak sorption and degradation capacity and in soils with colloidal flow in macropores, glyphosate can also be transported into groundwater (Strange-Hansen et al., 2004; Landry et al., 2005; Borggaard and Gimsing, 2008), thereby threatening drinking water quality. Glyphosate and its main metabolite aminomethylphosphonic acid (AMPA) have frequently been detected in surface and ground water in Norway, Sweden, Denmark and The Netherlands (Sørensen et al., 2006; Adriaanse et al., 2008; Keshteli et al., 2011). Similarly, a recent study by Silva et al. (2017) showed that glyphosate and AMPA were also detected at the highest frequencies in soil samples from the northern regions of the EU, indicating the importance of adapting pesticide use to site specific environmental conditions, which govern the fate of glyphosate, including degradation. Temperature and soil water content have already been reported to affect greatly the half-life of glyphosate and AMPA, ranging from a few days to several months, up to one or two years (EFSA, 2013; Bento et al., 2016).

Studies have also shown a great variation in glyphosate degradation depending on soil properties (Smith and Aubin, 1993; Cheah et al., 1998; Gimsing et al., 2004a; Mamy et al., 2005; Sørensen et al., 2006; Bonfleur et al., 2011), which can be ascribed to soil microbiological and chemical characteristics (Albers et al., 2009; Kim et al., 2011). For instance, correlations have been found between glyphosate mineralization and soil microbial parameters, such as soil microbial biomass (Von Wiren-Lehr et al., 1997) or population size of Pseudomonas spp. bacteria in soil (Gimsing et al., 2004a). It has also been reported that glyphosate degradation is negatively correlated to the adsorption capacity of the soil (Nomura and Hilton, 1977; Moshier and Penner, 1978; Sørensen et al., 2006). Actually, all soil parameters that have been identified as having an impact on glyphosate adsorption such as pH, clay content, as well as phosphate, copper, iron oxides and aluminium oxides contents (Morillo et al., 2000, De Jonge and de Jonge, 1999; Autio et al., 2004; Gimsing et al., 2004b; Mamy et al., 2005), should have a contrary effect on glyphosate mineralization; in other words strong retention of glyphosate to the soil matrix possibly reduces its availability for the degradation. Several studies have in fact confirmed correlations between increased glyphosate sorption (caused by increased contents of copper, iron and aluminium oxides or decreased pH and phosphate content) and decreased mineralization/degradation (Sprankle et al., 1975; Moshier and Penner, 1978; Morillo et al., 2000; Gimsing et al., 2004a). Unfortunately, despite major scientific efforts, clear patterns between soil properties and glyphosate degradation have not emerged. Comparison between these degradation-correlation studies is difficult due to the high variation of experimental conditions among different studies; in addition, experiments were in general conducted under artificial conditions, especially with regard to soil water content and soil density. In most of these degradation studies, only one, up to few soils were investigated, so it cannot be excluded that the established correlations were found by a chance.

To avoid inconclusive results and to establish a sound basis for resilient correlations, we conducted a glyphosate degradation study with more than twenty different soils under comparable conditions: at identical soil bulk density of 1.3 g cm<sup>-3</sup> at temperature of  $20 \pm 1$  °C, and a soil water content at the relevant optimum for pesticide mineralization in all studied soils, which is at a water potential of -15 kPa (Schroll et al., 2006). The agricultural soils used in our study were characterized by (i) large variations in soil texture (sand content 8%–86%) and organic matter content (1.2%–4.5%) (ii) fairly diverse soil–water retention curves and (iii) differing pH values (5.0–7.1) and exchangeable cations (Table 1). The main aim of this study was to elucidate the impact of soil parameters on glyphosate degradation, thereby explaining the different behaviour of glyphosate and AMPA formation in different soils.

#### 2. Materials and methods

#### 2.1. Soil samples

The study was conducted with 21 agricultural soils from different sites in Germany and Slovenia, differing in their composition (Table 1, Fig. S1). Selected fields had not been treated with glyphosate before (at least 5 years). All soils were taken on arable fields from the upper Ap horizon (0–30 cm), sieved (<2 mm) after sampling, homogenized and stored at 4 °C in the dark before use. Prior to the degradation experiments, the soils were moistened to a water potential close to -15 kPa, compacted to a soil density of 1.3 g cm<sup>-3</sup> and equilibrated at room temperature (20  $\pm$  1 °C) for 2 weeks.

#### 2.2. Soil characterisation

For soil analyses, the samples were air-dried and sieved to 2 mm (ISO 11464, 2006). Soil texture was determined by the pipette method (ISO 11277, 2009). Soil pH was measured in a 1/2.5 (w/v) ratio of soil and 0.01 M CaCl<sub>2</sub> suspension (ISO 10390, 2005). Total soil organic carbon (TOC) and total N (TN) were measured by dry combustion (ISO 10694, 1996; ISO 13878, 1987) using an elemental analyser (Elementar vario MAX instrument, Germany). Organic matter was calculated from the content of organic carbon using a conversion factor of 1.724. Ammonium lactate extraction was used for assessing plant available forms of P and K (AL-P and AL-K) (ÖNORM L 1087, 1993). Cation exchange capacity (CEC) was determined as the sum of exchangeable base cations and exchangeable acidity (expressed as H<sup>+</sup> and Al<sup>3+</sup> cations) (Soil Survey laboratory methods manual, 2004). For exchangeable base cations with

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