



Behaviour of glyphosate in a reservoir and the surrounding agricultural soils



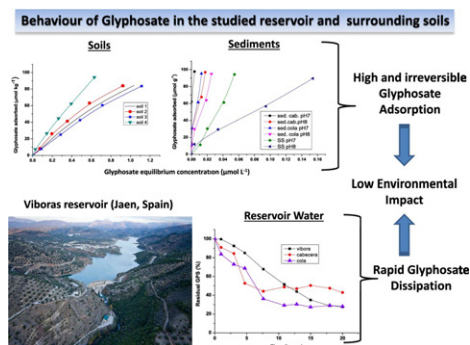
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HIGHLIGHTS

- High GPS adsorption to surrounding soils due to amorphous oxides and clay minerals
- Extremely high adsorption capacity of reservoir sediments for GPS
- GPS desorption from soils and sediments almost irreversible
- High levels of amorphous oxides act as photosensitizers for GPS water dissipation.
- High GPS sorption on sediments and dissipation in water reduce its environmental impact.

GRAPHICAL ABSTRACT



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ABSTRACT

Glyphosate (GPS) is an herbicide currently used on olive crops in Spain, and can be transported to the nearby reservoirs currently used for human consumption. The purpose of this work was to study the behaviour and environmental fate of GPS in water and sediments of the Vibora Reservoir, its tributary river, and the surrounding agricultural soils to assess the risk of water pollution of this reservoir. The adsorption of GPS by different matrices was as follows: heading of the reservoir sediment (Cabecera) > tail sediment (Cola) > soils > Vibora sediment. The highest amount of oxides (especially Fe oxides) was observed in sediments from Cabecera and Cola whereas the lowest values were recorded on Vibora sediment. Results indicate that the highest GPS adsorption is due to the amorphous oxides and the edge sites of the clay minerals. Glyphosate adsorption increased with decreasing pH from 8 to 7. The desorption percentage of GPS from the four soils studied ranged only from 0.40 to 1.22%. Desorption was almost irreversible for Cabecera and Cola sediments, with values between 0 and 1.1%. Conversely, Vibora sediment presented about 20% desorption, probably due to its coarse texture and lower levels of amorphous oxides. Hockey-stick first-order kinetics was the best descriptor for water glyphosate dissipation at the Cabecera and Cola locations, and simple first-order kinetic for the water from the tributary Vibora River. The half-lives (DT50) were between 6.3 and 11.0 days. The rapid degradation of GPS in surface waters and its practically irreversible sorption on these soils and sediments implies that GPS use in similar agricultural areas is of very low environmental risk. This manuscript also outlines the importance of the presence of photo-sensitizers in waters in the degradation routes of GPS in reservoirs.

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1. Introduction

Monitoring surveys carried out in United States and Europe have indicated that the use of pesticides in agricultural and non-agricultural areas have yielded contamination of surface and groundwater through runoff and soil leaching (Aparicio et al., 2013; Hildebrandt et al., 2008). Many researchers have found a strong correlation between water pollution due to pesticides and agricultural practices in the surrounding areas (Palma et al., 2009; Ricart et al., 2010). Water quality is an important issue in the European Union. The EU has prescribed the maximum concentration of $0.1 \mu\text{g L}^{-1}$ for each pesticide and of $0.5 \mu\text{g L}^{-1}$ for the whole in drinking water (Directive 2000/60/EC). Most contaminants released into aquatic environments will be incorporated in sediments, and these sediments might later act as the major source of contaminants released to water and biota.

Glyphosate [*N*-(phosphonomethyl) glycine] (GPS), is a broad-spectrum, post-emergence, non-selective herbicide and very soluble in water (12 g L^{-1}). It is the world's biggest-selling chemical used for weed control in agricultural, silvicultural and urban environments (Benbrook, 2016), and it is currently used on olive crops in Spain (Cañero et al., 2011). Some authors such as Giesy et al. (2000) and Howe et al. (2004) indicated that GPS is relatively safe environmentally, although nowadays some researchers indicated risks to humans and the environment stemming from applications of glyphosate (Myers et al., 2016). For this reason, the European Union, whose authorization procedure for pesticides is the strictest in the world, has approved a limited extension of the current approval of glyphosate only until the end of 2017, while studies about its carcinogenicity are carried out (European Commission, 2016). In relation to its behaviour in soil, some investigations showed possible leaching and toxicity problems with its use (Veiga et al., 2001). Other authors have also shown that soil adsorption and degradation of glyphosate exhibit great variation depending on soil composition and properties (Gimsing et al., 2004a; Gimsing et al., 2004b; Mamy et al., 2005). Therefore, it is important to study the behaviour and fate of GPS in soils and sediments, including adsorption, dissipation and leaching. GPS is a polar, amphoteric compound that binds strongly to soil. GPS have a soil half-life that range between 2 and 215 days and in aquatic medium is of 2 to 91 days (Battaglin et al., 2014). Predicting the behaviour and transport of an herbicide in soil and sediments involves understanding its adsorption characteristics. Therefore, adsorption coefficients obtained from batch experiments are an important tool for understanding the processes and the prediction of the contaminant behaviour (Wauchope et al., 2002).

There are many papers that describe the factors that control the adsorption of glyphosate on soils and their components (Gimsing et al., 2004a; Morillo et al., 2000). However, there is no agreement between the different authors. Glyphosate is a polyprotic acid and forms, within the pH range of 4–8 found in most soils, mono- and divalent anions with high affinity for, in particular, trivalent cations such as Al^{3+} and Fe^{3+} (Barja and Dos Santos Afonso, 2005). GPS can mainly be adsorbed onto variable-charge surfaces and much less onto permanent-charge (negative) sites on layer silicates (Morillo et al., 1997).

Some authors have indicated that adsorption depended mainly on cation exchange capacity (CEC) and clay content (Glass, 1987; de Santana et al., 2006; Da Cruz et al., 2007). However, others authors have postulated that iron and aluminium amorphous oxides or organic matter content had the major influence (Morillo et al., 2000; Maqueda et al., 2002; Prata et al., 2005). Some studies have shown that soil adsorption and degradation of GPS exhibit great variation depending on soil composition and properties (Sørensen et al., 2006; Gimsing et al., 2007). In addition, the effect of pH on adsorption of GPS in soils and clay minerals is crucial (Morillo et al., 2000; Sheals et al., 2002; Gimsing et al., 2004a; Pessagno et al., 2008). Soil organic matter (SOM) seems to play a controversial and dual role in soil adsorption of GPS. Some authors indicated a negative correlation with GPS adsorption; however, Yu and Zhou (2005) found a great influence of the OM

on GPS adsorption. In addition, Maqueda et al. (1998) showed a high GPS adsorption by a natural fulvic acid complex and Piccolo et al. (1996) reported very high adsorption by 4 different purified humus samples, explained by the formation of hydrogen bonding between humus and GPS.

Nevertheless, there is a general agreement in the literature that the adsorption of GPS in soil is governed by the mineral rather than the organic phase (Sheals et al., 2002; da Cruz et al., 2007; Gimsing et al., 2007; Rampazzo et al., 2013). The main soil adsorption sites are found on surfaces of aluminium and iron oxides, especially adsorption by goethite ($\alpha\text{-FeOOH}$) (Maqueda et al., 2002; Dideriksen and Stipp, 2003), poorly ordered aluminium silicates (allophane/imogolite) and edges of layer silicates. Soils enriched with these variable-charge minerals have been demonstrated to be effective GPS sorbents (Gimsing and Borggaard, 2007), whereas soils dominated by permanent charge minerals such as illite, smectite and vermiculite adsorb less GPS (De Jonge et al., 2001). The layer silicates can contribute with the OH groups on the octahedral layer.

GPS has a limited risk of leaching to ground water because it is inactivated by strong adsorption in soils and degrades relatively rapidly (Busse et al., 2001; Borggaard and Gimsing, 2008). However, long-term use of GPS may cause surface, ground water and sediments pollution (Kogan et al., 2003; Stewart et al., 2014). The presence of GPS in natural waters is mainly due to off-site movement via surface waters (Landry et al., 2005; Ibañez et al., 2006; Battaglin et al., 2014). Once GPS has reached the reservoir, it can suffer degradation in water or can be adsorbed to sediments. Microbial degradation of GPS is an important dissipation pathway in surface waters, whereas photolytic decomposition and chemical degradation are comparatively minor (Degehardt et al., 2012).

The purpose of this work was to study the behaviour and environmental fate of GPS to assess the risk of water pollution of a reservoir taking into account three environmental compartments: water and sediments of the Vibora Reservoir and its tributary river, and their surrounding agricultural soils. As far as we know, most of the previous studies carried out in reservoirs were restricted only to water. Vibora Reservoir is located in a semi-arid region where the main economic activity is the agriculture focused on the cultivation of olive trees. The herbicide GPS was selected because is one of the most used pesticides in olive tree orchards in Spain. The crop yields are strongly dependent on the use of pesticide because of climatic and soil conditions, with a potential risk of water contamination. The herbicides applied to olive crops can be transported to the nearby reservoir currently used for human consumption. Specific attention was given to the adsorption behaviour of GPS in the different media and its dissipation in the aquatic environment, for understanding the dynamic of herbicide contamination of surface waters.

2. Materials and methods

2.1. Pesticide

High purity glyphosate (98% purity) was used in adsorption and dissipation experiments. The herbicide was purchase from Dr. Ehrenstorfer GmbH (Augsburg, Germany). GPS is a non-residual herbicide, with solubility in water of 12 g l^{-1} at $25 \text{ }^\circ\text{C}$. GPS has a zwitterion structure depending on the pH.

2.2. Sampling site

Vibora Reservoir is located at the Vibora riverbed in the province of Jaen, in the south of Spain ($37^\circ 38'8''\text{N}$ $3^\circ 59'36''\text{W}$). It is a water reservoir for drinking water and fishing and has a capacity of 19 hm^3 . It has a tributary river with the same name, Vibora. In its surrounding environment there are olive trees planting. The province of Jaen is the region with higher olive production concentration in the world and made

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