



Assessment of agro-industrial and composted organic wastes for reducing the potential leaching of triazine herbicide residues through the soil ☆☆☆



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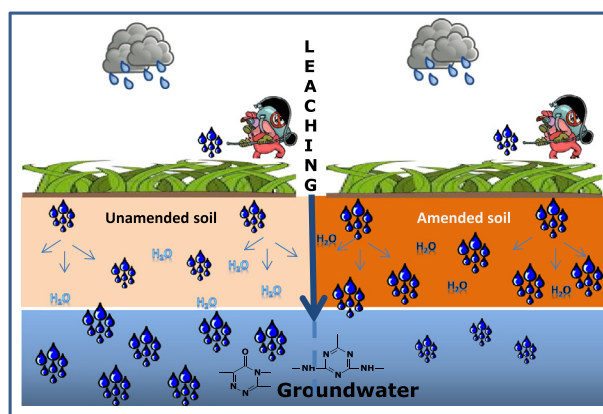
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HIGHLIGHTS

- The leaching potential of ten triazine herbicides through the soil was demonstrated using disturbed soil columns.
- Addition of agro-industrial and composted organic wastes strongly decreased the mobility of herbicides.
- Half-lives were markedly higher in un-amended than in amended soils.
- The used organic wastes could be useful in reducing groundwater pollution by triazine herbicides.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, we examined the effect of four different organic wastes – composted sheep manure (CSM), spent coffee grounds (SCG), composted pine bark (CPB) and coir (CR) – on the sorption, persistence and mobility of eight symmetrical and two asymmetrical-triazine herbicides: atrazine, propazine, simazine, terbutylazine (chlorotriazines), prometon (methoxytriazine), prometryn, simetryn, terbutryn (methylthiotriazines), metamitron and metribuzin (triazinones). The downward movement of herbicides was monitored using disturbed soil columns packed with a clay loam soil (Hipercalcic calcisol) under laboratory conditions. For un-amended and amended soils, the groundwater ubiquity score (GUS) was calculated for each herbicide on the

Abbreviations: BTC, breakthrough curve; CPB, composted pine bark; CR, coir; CSM, composted sheep manure; DEA, deethyl atrazine; DET, deethyl terbutylazine; DET, deisopropil atrazine; DOM, dissolved organic matter; ESI, electrospray ionization; GUS, groundwater ubiquity score; HT, hydroxy terbutylazine; K_d , distribution coefficient; K_{OC} , organic carbon normalized adsorption coefficient; LC-MS, liquid chromatography-mass spectrometry; LOD, limit of detection; LOQ, limit of quantitation; OA, organic amendment; OC, organic carbon; OECD, Organization for Economic Cooperation and Development; OM, organic matter; PV, pore volume; PVC, polyvinyl chloride column; RSD, relative standard deviation; SCG, spent coffee ground; XRD, X-ray diffraction.

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basis of its persistence (as $t_{1/2}$) and mobility (as K_{OC}). All herbicides showed medium/high leachability through the unamended soils. The addition of agro-industrial and composted organic wastes at a rate of 10% (w:w) strongly decreased the mobility of herbicides. Sorption coefficients normalized to the total soil organic carbon (K_{OC}) increased in the amended soils. These results suggest that used organic wastes could be used to enhance the retention and reduce the mobility of the studied herbicides in soil.

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1. Introduction

Pesticides help farmers and growers to produce good quality food at reasonable prices while controlling costs. At the same time, there may be undesirable side effects, which must be identified and, as far as possible, eliminated. All pesticides are subject to an approval procedure under EU legislation, and often under national legislation. Plant Protection Products Regulation 1107/2009 and the Sustainable Use Directive, 2009/128/EC are essential elements in this context. However, some risks are inevitable because the use of pesticides has an impact on the natural environment through spray drift, leaching and run-off into water, or even affect non-target organisms.

For a proper risk assessment of these chemicals, it is important to estimate their potential for transformation in soil and for movement (leaching) into deeper soil layers and eventually into groundwater. Leaching, the movement of water and dissolved chemicals through the soil, of pesticides into the groundwater from agricultural practices is receiving increasing attention in European countries because a high percentage of the drinking water is extracted from groundwater. For this reason, the EU has established the individual ($0.1 \mu\text{g L}^{-1}$) and total ($0.5 \mu\text{g L}^{-1}$) concentrations of pesticides in drinking water to safeguard consumers from harmful effects (Directive, 2006/118/EC).

Under certain conditions, some pesticides may leach to the groundwater after normal field applications. In this process, multiple factors such as the physicochemical properties of the agrochemicals used, soil hydrological processes and management, as well as soil properties (texture, clay content, organic matter and permeability), play a decisive role (Navarro et al., 2007). The hypotheses proposed to explain this rapid transport include preferential flow, co-transport with colloidal matter, and a combination of both processes (Arias-Estévez et al., 2008).

However, among soil properties, the organic carbon (OC) content is the single factor that has maximum influence on pesticide adsorption and mobility in soil. The application of OC in the form of manure, sludge, or crop residues is a common soil management practice followed in wide areas of SE of Spain where soil degradation, adverse climatic conditions, high temperature and high evapotranspiration are responsible for the scant vegetal growth and consequent lack of organic remains that would improve the soil nutrient status (Bastida et al., 2008). Soils of low OC content have a low capacity for retarding pesticide mobility, since the soil organic matter (OM), especially humic substances, is the primary adsorbent for pesticides (Cox et al., 2000). At present, the addition of organic amendment (OA) to soils is being studied to understand any effect it may be on pesticide sorption and movement through the soil profile in order to minimize the risk of water pollution associated with rapid run-off and leaching. However, pesticide behavior in amended soils has been reported to have different responses and a variety of influences has been recognized (Briceño et al., 2007).

The main benefit concerning the sorption of pesticides to OM is that it generally restricts leaching. However, decreased leaching may not only be due to the presence of additional OM in the amended soil, but also to structural changes in the porosity induced by the increased OC content (Worrall et al., 2001). Forming part of the OA added, dissolved organic matter (DOM) is incorporated, which affects pesticide sorption and movement (Huang and Lee, 2001). Competition between pesticides and DOM molecules for sorption sites and pesticide–DOM interactions may both account for enhanced pesticide leaching (Velarde et al., 2007). The extent and nature of this interaction depend on such factors as molecular weight and the polarity of the pesticides involved (Spark

and Swift, 2002). Moreover, the addition of OA to soil normally results in an increase in the microbiological activity (Ros et al., 2003), and as a consequence, OA enhances pesticide biodegradation in polluted soils.

More specially, triazine compounds have been among the most widely used pesticides in recent decades (LeBaron et al., 2008). As a general rule, they are weak bases with low water solubility and they persist a relatively long time in the soil, where their main degradation process is microbial (Roberts and Hutson, 1999). The decrease in triazine usage in Europe is chiefly due to legal provisions limiting the use of these products. The banning of triazines in the EU was precipitated mainly by the fact that water sources can be seriously contaminated by some of them. Atrazine, simazine and terbutryn have been classified as priority substances by the European Parliament and the Council of the European Union in water policy Directive, 2013/39/JE. Triazine parent compounds and their metabolites have been detected in surface and ground waters in several European countries and USA (Kolpin et al., 1996; Arias-Estévez et al., 2008; Koskinen and Banks, 2008).

The use of OA increases the OM content of the soil, enhancing triazine sorption, and thereby decreasing its availability for transport. Soil amended with sludge, urban waste compost, composted straw, fly ash, olive oil mill waste or wood residues has been shown to increase triazine sorption as compared with unamended soil (Cox et al., 1997; Celis et al., 1998; Houot et al., 1998; Majumdar and Singh, 2007; Cabrera et al., 2008; Delgado-Moreno and Peña, 2009; Jiang et al., 2011; Grenni et al., 2012; Gámiz et al., 2012). With this aim, we compared the effect of different farming (composted sheep manure), agro-industrial (spent coffee grounds and coir) and forestry (composted pine bark) wastes on the leaching of eight triazine and two triazinone herbicides through the soil by using disturbed soil columns.

2. Materials and methods

2.1. Chemicals

Herbicide analytical standards ($\geq 98\%$) were obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany). The main physicochemical properties of the active ingredients are shown in Table 1. Experimental values of the octanol/water partition coefficient (K_{OW}), soil/organic partition coefficient (K_{OC}), aqueous solubility (S_w), aqueous hydrolysis, and Groundwater Ubiquity Score (GUS) index were taken from The Pesticide Properties DataBase (AERU, 2014) and KOCWIN™ included in the EPI Suite v4.10 program (US EPA, 2012).

Stocks solutions ($1000 \mu\text{g mL}^{-1}$) of each herbicide standard were prepared in acetonitrile, protected from light and stored at 5°C . Several standard solutions, with concentrations of $0.5\text{--}200 \mu\text{g L}^{-1}$, were injected to obtain the linearity of detector response and the detection limits (LOD) of the herbicides studied. Pesticide grade acetonitrile and dichloromethane were supplied by Scharlab (Barcelona, Spain).

2.2. Soil and amendments

The soil (Hipercalcic calcisol) chosen for this study was a clay loam soil (bulk density 1.35 g cm^{-3}) from Campo de Cartagena (south-eastern Spain). Soil samples were collected from the surface (top 20 cm), air-dried, and passed through a 2 mm sieve. The main characteristics of the soil were determined by standard soil analysis techniques (Reeuwijk, 2002). The clay content was determined by means of a powder X-ray diffractometer (XRD) on a Philips PW 1700 using Cu-K α radiation at a

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