



Solarization and biosolarization using organic wastes for the bioremediation of soil polluted with terbuthylazine and linuron residues



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ABSTRACT

Strategies for remediation of polluted soils are needed to accelerate the degradation and natural attenuation of pesticides. This study was conducted to assess the effect of solarization (S) and biosolarization (BS) during the summer season using organic wastes (composted sheep manure and sugar beet vinasse) for the bioremediation of soil containing residues of terbuthylazine and linuron. The results showed that both S and BS enhanced herbicide dissipation rates compared with the non-disinfected control, an effect which was attributed to the increased soil temperature and organic matter. Linuron showed similar behavior under S and BS conditions. However, terbuthylazine was degraded to a greater extent in the biosolarization experiment using sugar beet vinasse than in the both the solarization and biosolarization experiments using composted sheep manure treatments. The main organic intermediates detected during the degradation of terbuthylazine and linuron were identified, enabling the main steps of degradation to be proposed. The results confirm that both S and BS techniques can be considered as a remediation tools for polluted soils containing these herbicides.

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1. Introduction

Methyl bromide, widely used as a broad-spectrum soil fumigant during the last half century, has been banned since 2005, leading to the proposal of both chemical and non-chemical alternatives to control soilborne pests (Rodríguez-Kábana, 1997). Among non-chemical alternatives to methyl bromide, solarization, biofumigation and biosolarization are three attractive methods currently used in different crops in the Mediterranean Basin (Rodríguez-Kábana, 1997; Ros et al., 2008). Soil solarisation, which originated during the 1970s, involves covering the moist soil by a thin clear plastic film, which is heated by solar radiation for several weeks (Katan and DeVay, 1991). To be effective, this technique requires warm sunny days, limiting the timing and location of its use (Sosnowski et al., 2009). Soil biofumigation refers to the suppression of selective soil-borne organisms by toxic volatile compounds liberated in the biodecomposition of organic matter (Matthiessen and Kirkegaard, 2006). Organic amendments used for soil-borne

pathogen control are extremely heterogeneous, and include vegetal and animal manure along with agro-industrial by-products (Piedra Buena et al., 2007). Applying organic amendments to soil not only increases the total organic carbon (TOC) content and its different fractions but also has a series of effects on microbial proliferation and activity. Generally, such amendments improve microbial development and its activity. Accordingly, the characteristics of different organic materials may have a differential effect on the soil microbiota and strongly influence the microbial use of the C contained in these materials, which may, in turn, affect the preferential development of those groups of microbes best adapted to the amendment in question (Bastida et al., 2008). The approach of combining soil solarization and the application of organic matter has been defined as biosolarization (Ros et al., 2008). These non-chemical alternatives to methyl bromide have produced good results, especially in low-input and organic farming systems (Klein et al., 2012).

On the other hand, pesticides are still heavily used in agriculture, horticulture and other fields. Terbuthylazine and linuron – triazine and phenylurea herbicides, respectively – are used as broad-spectrum pre-emergence and post-emergence agents for the control of weeds in many agricultural crops. These compounds are

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primarily absorbed through the roots and are photosynthetic electron transport inhibitors at the photosystem II (Tomlin, 2006). In addition, both terbuthylazine and linuron, together with their degradation products, are toxic and persistent in the soil.

The widespread use of pesticides can lead to the presence of residues of these products and their metabolites in the soil. Under certain conditions, some pesticides may leach to groundwater or be absorbed by plants following normal field applications (Navarro et al., 2007). The toxicity of these agrochemicals means that soil remediation strategies are necessary in order to protect consumers from exposure to unacceptable levels of pesticide residues in food and water. The use of zerovalent iron, phytoremediation, ozonization, soil solarization and soil biosolarization have been proposed for the treatment of pesticide-polluted soils (Fenoll et al., 2011a; Navarro et al., 2009; Pavel and Gavrilescu, 2008). In particular, soil solarization and soil biosolarization are two efficient techniques that accelerate the degradation and natural attenuation of pesticide residues in soils (Fenoll et al., 2011a, 2010a, 2010b, 2011b, 2010a, 2010b). However, little effort has been devoted to evaluating the effect of solarization and biosolarization using different amendments on terbuthylazine and linuron degradation in soil. For this reason, the aim of this work was to study the use of solarization and biosolarization using two different organic amendments (composted sheep manure and sugar beet vinasse) for the remediation of soils containing residues of terbuthylazine and linuron. The experiment was carried out in greenhouses, under the climatic conditions of Murcia (south-east Spain) and the main degradation products of these herbicides were monitored over a three month period.

2. Materials and methods

2.1. Herbicides and reagents

The following analytical standards were purchased from Dr. Ehrenstorfer (Augsburg, Germany): Terbuthylazine (99.5% purity), linuron (99% purity), atrazine-desisopropyl (98.5% purity), atrazine-desethyl (96% purity), terbuthylazine-desethyl (99% purity), terbuthylazine-2-hydroxy (98.5% purity) 1-(3,4-dichlorophenyl)-3-methylurea (99% purity), 1-(3,4-dichlorophenyl)urea (99% purity) 1-(3,4-dichlorophenyl)-3-methoxyurea (99% purity) and 3,4-dichloroaniline (99% purity). The main physical and chemical characteristics of the herbicides used are shown in Table 1. The experimental values of the octanol/water partition coefficient (K_{OW}), soil/organic partition coefficient (K_{OC}), Henry's law constant and aqueous solubility taken were taken from The Pesticide Properties DataBase (AERU, 2013).

Stock solutions (200 $\mu\text{g mL}^{-1}$) of each pesticide standard were prepared by dissolving 10 mg of the pesticide in 50 mL of acetonitrile. These were then stored in the dark at 4 ± 1 °C. Working standard solutions were prepared freshly by dilution in the same solvents. The acetonitrile for herbicide residue analysis was supplied by Scharlab (Barcelona, Spain).

2.2. Greenhouse procedures and sampling

Several hundred 6-L (16 cm diameter x 30 cm depth) pots were filled with 4.5 kg of a clay-loam soil (32.9% clay, 30.2% silt, 36.9% sand; bulk density = 1.33 g cm^{-3}). The soil had a pH (H_2O 1:1) of 7.3, 3.1% organic matter content and electrical conductivity of 7.3 dS m^{-1} . The pots were placed in a greenhouse located in Torre-Pacheco (Murcia, SE Spain) during the summer season (July–September, 2012). The treatments consisted of solarization and biosolarization techniques carried out on the soil previously contaminated with the two herbicides. There were four sets of containers: Non-mulched soil (control, C) ii) soil covered with low-density polyethylene film (LDPE) (solarized, S), iii) soil plus composted sheep manure covered with LDPE (biosolarized, BS1) and iii) soil plus sugar beet vinasse, which is used for covered with LDPE (biosolarized, BS2) (25 pots per treatment). Sugar beet vinasse is a by-product of the sugar and alcohol industries, and is used for organic fertilization. First, the manure was applied to the BS1 pots at a rate of 90 g pot^{-1} (according to the rate recommended for the control of soilborne fungi) and then thoroughly mixed with the soil. In BS1 pots, only 4.41 kg of soil were used to avoid a 'dilution effect' following the application of organic matter. The organic matter used for BS1 was a composted sheep manure (pH = 7.76, electrical conductivity = 13.24 dS m^{-1} , organic matter = 373.2 g kg^{-1} , total N = 9.3 g kg^{-1} , P = 10.8 g kg^{-1} and K = 29.7 g kg^{-1}). Next, all the pots (C, S, BS1 and BS2) were spiked with commercial formulations of the herbicides being studied: Cuña (50% terbuthylazine from Inagra) and Linukey (45% linuron from Key). For this, 750 mL of a solution containing 375 mg of each active ingredient was applied to 375 kg of soil in the case of C, S and BS2, while 250 mL of a solution containing 125 mg of each active substance was applied to 125 kg of amended soil in the case of BS1. These amounts were applied with a sprayer (Matabi) with an adjustable nozzle size of 1 mm, and then thoroughly mixed with the soil. The organic amendment used for BS2 was a sugar beet vinasse (Azucarera del Ebro, pH = 5.82, electrical conductivity = 3.53 dS m^{-1} , organic matter = 592.3 g kg^{-1} , total N = 33.3 g kg^{-1} , P = 0.42 g kg^{-1} and K = 35.1 g kg^{-1}). All the pots (C, S, BS1 and BS2) were irrigated to field capacity. Vinasse was added to water and applied to the BS2 pots at a dose of 18 mL pot^{-1} (according to the rate recommended

Table 1
Analytical conditions and main physical–chemical properties of the studied herbicides.

Compound	Analytical conditions		Properties ^a				
	Retention time (min)	Quantifier transition; qualifier transition (m/z)	MW	Log K_{OW}	H	S_W	Log K_{OC}
Terbuthylazine	25.06	230 → 132; 230 → 174	229.7	3.4	3.24×10^{-03}	6.6	2.50
Atrazine-desisopropyl	12.91	174 → 104; 174 → 132					
Terbuthylazine-2-hydroxy	13.66	212 → 156; 212 → 114					
Atrazine-desethyl	15.91	188 → 146; 188 → 104					
Terbuthylazine-desethyl	20.25	202 → 110; 202 → 146					
Linuron	25.51	249 → 182; 249 → 160	249.1	3.0	2.0×10^{-4}	63.8	2.53
1-(3,4-Dichlorophenyl)-3-methylurea	21.51	219 → 127; 219 → 162					
1-(3,4-Dichlorophenyl)-3-methoxyurea	22.59	235 → 161; 235 → 168					
1-(3,4-Dichlorophenyl)urea	20.13	205 → 127; 205 → 162					
3,4-Dichloroaniline	23.75	162 → 127; 162 → 109					

^a Molecular weight (MW); octanol/water partition coefficient (K_{OW}), Henry's law constant (H, $\text{Pa m}^3 \text{ mol}^{-1}$ at 25 °C), water solubility (S_W , mg L^{-1}), soil/organic partition coefficient (K_{OC} , mL g^{-1}).

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