



# Soil spreading of liquid olive mill processing wastes impacts leaching of adsorbed terbuthylazine



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## HIGHLIGHTS

- OMWW is frequently spread on agricultural land for disposal.
- OMWW effect on leaching of adsorbed terbuthylazine was tested in four soils.
- Terbuthylazine solubility in OMWW is significantly higher than in water.
- Terbuthylazine leaching from OMWW-treated soils is lower than control treatment.
- Low soil organic carbon and clay contribute to reduced terbuthylazine leaching.

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## ABSTRACT

Olive mill waste water (OMWW) is a major byproduct of the three phase olive oil production process. OMWW has high acidity (pH ~ 4–5), high salt content (EC ~ 5–10 mS cm<sup>-1</sup>), extremely high biological and chemical oxygen demand (BOD and COD up to 100,000 and 220,000 mg L<sup>-1</sup>, respectively), and also high concentrations of organic compounds such as phenols and polyphenols. As a result, OMWW cannot be freely discharged into domestic wastewater treatment plants, but on-site treatment is very expensive and not sufficiently effective. Uses for OMWW such as agricultural recycling and co-composting were found to be impractical or expensive. Thus, OMWW is frequently spread on agricultural land for disposal. However, excessive or uncontrolled spreading of such organic-rich and saline wastewater could have many deleterious effects on soil quality, including salinization, phytotoxicity, or contaminant movement. The impact of OMWW on the leaching of adsorbed terbuthylazine, a soil-applied herbicide, was tested in four soils of varying physical and chemical properties. Although terbuthylazine solubility in OMWW is significantly higher than in water, leaching of adsorbed terbuthylazine from OMWW-treated soils was less than from control treatments. Low soil organic carbon and clay contents were major factors that contributed to reduced terbuthylazine leaching after soil treatment with OMWW.

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## 1. Introduction

The rise in global quality of life over the last two decades, specifically in the Mediterranean countries, and Israel among them, has increased the demand for olive oil. While many olive oil production plants in the Mediterranean area use a two phase production process, the three phase process is still very popular (Laor et al., 2007). Olive mill waste water (OMWW) is a major byproduct

of the three phase olive oil production process. About 50,000 to 100,000 m<sup>3</sup> of OMWW are produced in Israel in alternating years, and based on the current planting activities, the amount is expected to grow significantly in the coming decade (Laor et al., 2007).

OMWW has numerous detrimental characteristics such as high acidity (pH ~ 4–5), high salt content (EC ~ 5–10 mS cm<sup>-1</sup>), extremely high biological and chemical oxygen demand (BOD and COD up to 100,000 and 220,000 mg L<sup>-1</sup>, respectively), and also high concentrations of organic compounds such as phenols and polyphenols (Azbar et al., 2004; Aviani et al., 2009), carbohydrates, polyalcohols, tannins and pectins (Poerschmann et al., 2013a,

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2013b). Due to the high organic load and toxicity, OMWW cannot be freely discharged into domestic wastewater treatment plants (WWTP), but treatment at the production site is very expensive and not sufficiently effective in reducing the organic load (Rozzi and Malpei, 1996). Other solutions such as agricultural recycling and co-composting the OMWW were suggested and tested (Laor and Raviv, 2009), but found thus far to be impractical or expensive. As a result, the Israel Ministry of Environment allows spreading of OMWW on agricultural land, as in other countries such as Italy (Rinaldi et al., 2003).

While being an inexpensive solution, excessive or uncontrolled OMWW spreading may negatively impact soil quality by causing soil salinization (Paredes et al., 1987; Aqeel and Hameed, 2007), phytotoxicity (Mekki et al., 2006; Saadi et al., 2007), or soil water repellency (Graber et al., 2009). It may also endanger the quality of underlying aquifers due to the downward transport of OMWW constituents such as phenolic substances (Mekki et al., 2007; Moraetis et al., 2011) and other high BOD organic material (Spandre and Dellomonaco, 1996; Saadi et al., 2007; S'habou et al., 2009).

In addition to these recognized problems, OMWW is spread on agricultural soil which may also be treated with herbicides and other pesticides for plant protection. This may be problematic because application of solutions rich in dissolved organic matter (DOM) has been seen to mobilize some pesticides and enhance their downward transport (Graber et al., 1995; Celis et al., 1998), while adding solid organic matter residues can increase binding of pesticides and reduce their efficacy (Cabrera et al., 2011). Mobilization and subsequent leaching of soil-adsorbed pesticides may cause pollution of ground and surface water resources (Graber et al., 2001a).

There is very little information on the effect of spreading liquid OMWW on the mobility of soil-adsorbed pesticides. Most previous studies found that amendment of soil by either highly concentrated liquid residues (above 10% dry matter) (Cox et al., 1997) or solid residues of olive oil production (olive pomace) increased sorption of pesticides and reduced their leaching and downward mobility (Albarrán et al., 2003, 2004; Delgado-Moreno et al., 2007; López-Piñero et al., 2010). However, studies of pesticide release and mobility in the presence of dissolved organic matter (DOM) from organic wastes, amendments and treated wastewater, have frequently reported a reduction in pesticide sorption (Graber et al., 1995, 1997; Businelli, 1997; Barriuso and Houot, 1998; Graber et al., 2001b; Wallach et al., 2005; Wallach and Graber, 2007) and an increase in pesticide downward transport.

Pesticides interact with soil minerals or organic components or both. Soils having more than about 0.5% organic matter (OM) will sorb pesticides depending on the OM content but not its type (Hayes, 1970; Arienzo and Buondonno, 1993; Bekbölet et al., 1999), while in OM-poor soils, sorption of pesticides may be dominated by the inorganic clay component of the soil (Welhouse and Bleam, 1993; Baskaran et al., 1996). Leaching and enhanced transport of pesticides as a result of externally-applied DOM depend on the same processes (Businelli, 1997; Gao et al., 1997; Barriuso and Houot, 1998; Cox et al., 2000, 2007; Li et al., 2005). Usually it was found that when the interaction of pesticides with the added soluble OM is significant relative to their interactions with solid soil organic matter (SOM), transport of the pesticides through the soil may be enhanced by complexing with the mobile soluble OM as it moves through the soil profile. The concentration of applied DOM would therefore influence the concentration of pesticides in drainage waters, surface waters and ground waters associated with a particular soil (Cox et al., 2000; Spark and Swift, 2002). Another mechanism leading to enhanced leaching of pesticides is through competitive adsorption of applied DOM in soil surface, and

specifically in soil clay (Cox et al., 2000).

The objective of this work was to examine if OMWW spreading on agricultural soils would influence desorption and leaching of a common and important soil-applied herbicide, terbuthylazine (N-tert-butyl-6-chloro-N'-ethyl-1,3,5-triazine-2,4-diamine), which is a pre- and post-emergence herbicide from the s-triazine family that was chosen as the model compound. Terbuthylazine is in widespread use for controlling certain broadleaf weeds and annual grasses in corn in many parts of the world, where it replaces atrazine.

Terbuthylazine has an octanol-water distribution coefficient ( $K_{ow}$ ) of  $2.51 \times 10^3$  and a water solubility of  $8.5 \text{ mg L}^{-1}$  at  $20^\circ\text{C}$  (Tomlin and Council, 2001). As such, it generally has higher adsorption to soil compared to other s-triazines in both OM-amended and non-amended soils (Dousset et al., 1994). Terbuthylazine sorption to soil depends on soil type and organic carbon amendment (enhanced sorption to poor soils amended with organic carbon) (Dousset et al., 1994; Sluszný et al., 1999; Alister et al., 2011; Fenoll et al., 2014). Three main questions were addressed: (1) does the soluble organic matter from OMWW form complexes with free terbuthylazine, and does it, when applied to soil with bound terbuthylazine, enhance herbicide leaching; (2) what, if any, is the contribution of the inorganic salts fraction of OMWW on soil-terbuthylazine interactions (i.e., is there a "salting-out" effect); and (3) what is the combined contribution of soil type and chemical characteristics on the impact of OMWW on leaching of adsorbed terbuthylazine.

## 2. Materials and methods

### 2.1. Herbicide

Analytical grade terbuthylazine, (N-tert-butyl-6-chloro-N'-ethyl-1,3,5-triazine-2,4-diamine), cas# 5915-41-3, 99.8% purity, was generously donated by ADAMA Agricultural Solutions Ltd (Tel Aviv, Israel).

### 2.2. Amendment solutions

#### 2.2.1. Olive mill wastewater (OMWW)

Olive mill wastewater (OMWW) was provided by the Bnei-Darom olive oil production plant. The bulk OMWW solution was frozen in 1 L bottles and remained frozen until use, at which time it was thawed and passed through a 20–25  $\mu\text{m}$  cellulose paper filter (Whatman 41) to remove large particulate matter. The filtrate of each 1 L batch was characterized in triplicate for pH, electrical conductivity (EC), dissolved organic carbon (DOC),  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Na}^+$ ,  $\text{K}^+$ , and soluble  $\text{PO}_4^{3-}$ .

#### 2.2.2. Salt solution and control

A salt solution having a composition in terms of calcium ( $\text{Ca}^{2+}$ ), magnesium ( $\text{Mg}^{2+}$ ), sodium ( $\text{Na}^+$ ), potassium ( $\text{K}^+$ ) and phosphate ( $\text{PO}_4^{3-}$ ) concentrations similar to that of the OMWW was prepared using calcium chloride dehydrate salt (Merck KGaA Darmstadt, Germany), magnesium chloride salt (Merck KGaA Darmstadt, Germany), sodium chloride salt (Sigma-Aldrich, St. Louis MO, USA), potassium di-hydrogen phosphate salt (Merck KGaA Darmstadt, Germany) and potassium chloride salt (Merck KGaA Darmstadt, Germany). Millipore de-ionized water was used for all preparations and as a control treatment.

### 2.3. Terbuthylazine solubility

Twenty mg of terbuthylazine were suspended with 10 mL amendment solutions (OMWW, salt solution and DW) in amber

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