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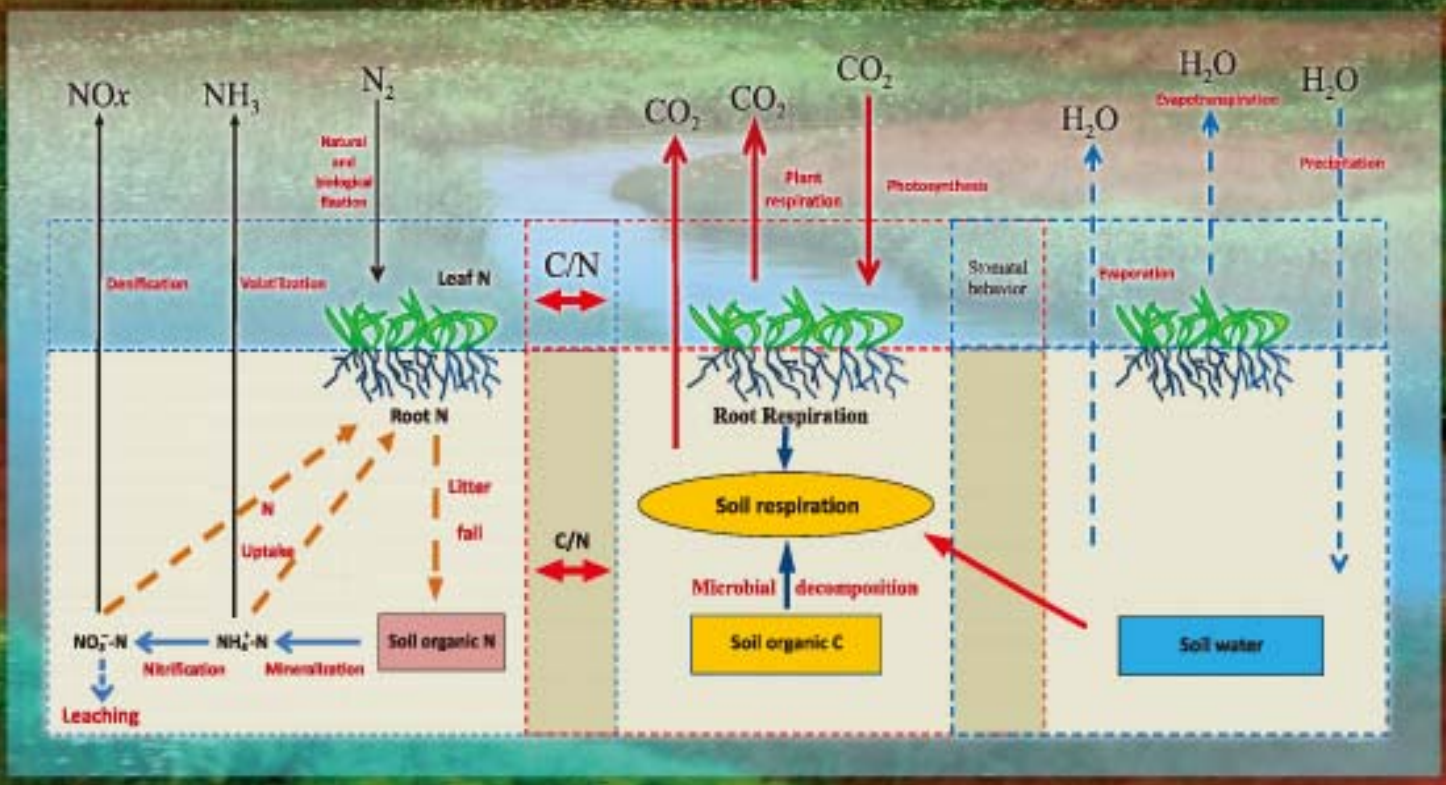


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# Minimization of methabenzthiazuron residues in leaching water using amended soils and photocatalytic treatment with $TiO_2$ and ZnO

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ABSTRACT

In the present work, potential groundwater pollution by methabenzthiazuron (MTBU) and the effect of three different amendments (composted sheep manure, composted pine bark and spent coffee grounds) on its mobility were investigated under laboratory conditions. The efficiency of ZnO and $TiO_2$ suspensions in the photocatalytic degradation of MTBU in leaching water was also investigated. The relative and cumulative breakthrough curves were obtained from disturbed soil columns. The presence and/or addition of organic matter drastically reduced the movement of the herbicide. On other hand, photocatalytic experiments showed that the addition of ZnO and $TiO_2$ strongly enhances the degradation rate of this herbicide compared with the results of photolytic experiments under artificial light. ZnO appeared to be more effective in MTBU oxidation than $TiO_2$. The results obtained point to the interest of using organic wastes and heterogeneous photocatalysis for reducing the pollution of groundwater by pesticide drainage.

## Introduction

Banned pesticides and/or their degradation products have frequently been detected in groundwater (Kolpin et al., 2004). This possible occurrence of pesticides in groundwater can be caused by (1) sorption and degradation processes influenced by chemical properties of the pesticide, weather conditions and soil properties (Kreuger, 1998); (2) spills by inappropriate handling of pesticides and non-agricultural application of the same; (3) preferential flow, whereby part of the soil volume is bypassed (Flury, 1996; Vereecken, 2005); and (4) colloid-facilitated transport (Hesketh et al., 2001).

Methabenzthiazuron (MTBU, 1-(1,3-benzothiazol-2-yl)-1,3-dimethylurea) is a selective urea herbicide banned by European Union. It has been used to control broad-leaved weeds and grasses in cereals, legumes, garlic, peas and onions. MTBU inhibits photosynthetic electron transport (PS II). This herbicide is very toxic for aquatic organisms and may cause long-term adverse effects in the aquatic environment, since it is quite soluble in water (around 60 mg/L). It can be transferred to the aquatic environment by leaching, although it has even been detected in surface water (Irace-Guigand et al., 2004). For these reasons, the minimization of MTBU residues in leaching water is of prime importance.

The mobility of pesticides and therefore their risk of leaching are determined by the extent and strength of sorption reactions, which are governed by the chemical and physical properties of the soils and pesticides involved (Spark and Swift, 2002). The impact of different organic amendments on the transport of pesticides has been discussed in several studies (Cox et al., 1997; Dao, 1991; Guo et al., 1993; Sanchez-Camazano et al., 1996). The addition of animal manures or crop residues

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to agricultural soils is a common practice to increase soil organic matter and the nutrient content. In general, these additions lead to enhanced sorption and decreased mobility of pesticides in the soil profile (Briceño et al., 2007; Worrall et al., 2001). Moreover, the addition of organic amendment enhances pesticide biodegradation in polluted soils (Felsot and Dzantor, 1995; Johnson et al., 1997). However, some amendments produce dissolved organic matter, which decreases pesticide sorption and enhances their aqueous solubility and mobility through stable interactions in solution between the pesticide and the dissolved organic matter (Li et al., 2005).

On other hand, advanced oxidation processes, particularly heterogeneous photocatalysis using UV/$TiO_2$ or UV/ZnO systems, are effective and low cost processes to degrade pesticides in water and to mineralize some of them (Herrmann, 2005; Malato et al., 2009). These processes use UV light with wavelength energy $h\nu \geqslant E_g$ (band gap energy) to excite the semiconductor particles and to produce an excited high-energy state of electron and hole pairs. These can migrate to the surface of the semiconductor and initiate a wide range of redox reactions, which can lead to complete mineralization of a large variety of pesticides (Ahmed et al., 2011; Chnirheb et al., 2012). The most recent studies on MBTU photochemical behaviour were performed in aqueous solution (Alloui et al., 2011; Malouki et al., 2003, 2005). However, the effect of ZnO and $TiO_2$ for the removal of this phenylurea herbicide from water has not been studied.

The aim of the present research work was to minimize the contamination of leaching water by MBTU in two ways: (1) preventing contamination by amending soil with composted sheep manure, composted pine bark and spent coffee grounds, and (2) decontamination by photocatalysis under artificial light irradiation using ZnO and $TiO_2$ as catalysts.

## 1 Materials and methods

### 1.1 Chemicals

Analytical grade MTBU (97%) was obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany). ZnO (99.9%, BET 10 $m^2/g$, < 70 μm) was purchased from Alfa Aesar (Karlsruhe, Germany). $TiO_2$ P25 Degussa (99.5%, BET 50 $m^2/g$, < 21 nm) were supplied by Nippon Aerosil Co., Ltd. (Osaka, Japan). Sodium peroxydisulfate (98%) was purchased from Panreac Química (Barcelona, Spain). Acetonitrile was supplied by Scharlau (Barcelona, Spain).

A stock solution (1000 μg/mL) of MTBU standard was prepared in acetonitrile, protected from light and stored at 5°C. An intermediate standard solution of the pesticide was prepared by dilution in the same solvent to obtain a concentration of 10 μg/mL. Several standard solutions, with concentrations of 0.5 to 200 μg/L, were injected to obtain the linearity of detector response.

### 1.2 Leaching study

#### 1.2.1 Soil and amendments for the leaching experiment

Four different agricultural soils were chosen for this study. Soil A (hypercalcic calcisol), soil B (calcaric regosol) Soil C (hypercalcic calcisol) and soil D (haplic calcisol) were brought from the Campo de Cartagena, Murcia (south-eastern Spain). Soil samples were collected from the surface (top 20 cm), air-dried, and passed through a 2 mm sieve. The experimental studies began as soon as possible (not more than four days after sampling, with the samples stored under cool conditions). The composition of the soils is shown in **Table 1**.

The main characteristics of the soil (TOC, pH, alkalinity, texture) 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-$K\alpha$ radiation at a scanning speed of 1° (2θ) after washing, disaggregation and dispersion of the sample.

As amendments, ecological compost sheep manure (CSM) [pH = 8.3 (1/20, *W/V*), electrical conductivity = 5.2 dS/m (1/20, *W/V*), total nitrogen (%) = 3.2, total carbon (%) = 27.5, total organic carbon (%) = 26.5], composted pine bark (CPB) [pH = 5.9 (1/20, *W/V*), electrical conductivity = 0.9 dS/m (1/20, *W/V*), total nitrogen (%) = 4.5, total carbon (%) = 31.4, total organic carbon (%) = 29.6] and coffee (*Coffea arabica*) wastes (spent coffee grounds, SCG) [pH = 5.8 (1/20, *W/V*), electrical conductivity = 0.5 dS/m (1/20, *W/V*), total nitrogen (%) = 7.4, total carbon (%) = 48.4, total organic carbon (%) = 47.7] were used.

**Table 1** Characteristics of the soils used in this study ($n$ = 3)

| Parameters | Soils | | | |
|---|---|---|---|---|
| | A | B | C | D |
| Clay (%) | 29.1 | 31.6 | 25.1 | 32.9 |
| Silt (%) | 33.4 | 47.3 | 51.4 | 30.2 |
| Sand (%) | 37.5 | 21.1 | 23.5 | 36.9 |
| Total nitrogen (%) | 0.2 | 0.8 | 0.4 | 0.5 |
| Total carbon (%) | 7.2 | 6.4 | 7.3 | 7.4 |
| Total organic carbon (%) | 0.9 | 1.2 | 1.1 | 1.8 |
| Organic matter (%) | 1.6 | 2.1 | 1.9 | 3.1 |
| pH ($H_2O$ 1/1, *W/V*) | 7.9 | 8.2 | 8.0 | 7.3 |
| Electric conductivity (dS/m) | 1.3 | 0.4 | 0.5 | 7.3 |
| Alkalinity (mg $CaCO_3$/kg) | 549 | 601 | 469 | 398 |
| Clay content (% weight) | | | | |
| Illite | 16.5 | 16.1 | 13.6 | 18.6 |
| Vermiculite | 11.9 | 12.4 | 13.4 | 12.9 |
| Kaolinite | 6.5 | 4.1 | 5.5 | 6.2 |
| Montmorillonite | 34.6 | 37.3 | 41.1 | 40.1 |
| Interstratified minerals | 30.5 | 30.1 | 26.4 | 22.2 |

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Composting of the manure was carried out in piles with periodical turning (indore method) over a period of 4 months. After composting, the compost was left untouched in static piles for 3 months to complete the maturation process. Aged pine bark is sometimes referred to as composted bark, although in the strictest sense, unless pine bark is amended with a nitrogen source, moistened and turned regularly (recommended procedures for composting leaf yard wastes and animal wastes), true composting may not fully occur. The stability of an organic material is frequently dependent upon its carbon to nitrogen ratio. For most organic potting components the ratio should be at least 30/1, however, even composted pine bark may not have such a low of ratio since most of the pine bark is lignin and not cellulose. For their part, spent coffee grounds, were extracted with hot water, air-dried and then refluxed with *n*-hexane to extract the oil from the coffee particles. Finally, the grounds were dried and sieved through a 0.3 mm mesh.

### 1.2.2 Downward movement of the pesticides through the soil columns

The experiment was performed according to the OECD guidelines (OECD, 2007). Downward movement of the pesticides was studied in polyvinyl chloride columns of 40 cm length × 4 cm i.d.. There were seven sets of columns: soil A (200 g), soil B (200 g), soil C (200 g), soil D (200 g), soil A (180 g) mixed with CSM (20 g), soil A (180 g) mixed with CPB (20 g), and soil A (180 g) mixed with SCG (20 g). The top 3 cm of the columns were filled with sea sand and the bottom 3 cm with sea sand plus nylon mesh with an effective pore diameter of 60 m to minimize the dead-end volume and prevent losses of soil during the experiment. Before the application of the compound, columns (three replications at room temperature, avoiding direct light) were conditioned with 0.01 mol/L $CaCl_2$ in distilled water to their maximal water holding capacity and then allowed to drain for 24 hr. The pore volume (PV) of the packed columns was estimated by the weight difference between water-saturated columns and dry columns. The calculated PVs (mL) of the soil columns after saturation were 70.1 ± 1.9 (soil A), 73.6 ± 2.2 (soil B), 71.4 ± 2.9 (soil C), 79.3 ± 1.6 (soil D), 89.0 ± 2.1 (soil A + CSM), 94.3 ± 3.1 (soil A + CPB) and 87.2 ± 2.8 (soil B + SCG). Following this, 1.0 mL of acetonitrile/water solution (10+90, *V/V*) containing 20 g of MTBU were added to the top of each column. Twenty four hours after pesticide application, the compounds were leached by adding 850 mL of 0.01 mol/L $CaCl_2$ over a period of 17 days with a peristaltic pump. $CaCl_2$ instead of water was used in order to minimize disruption of the soil mineral balance. The leachates (50 mL per day) were quantitatively collected at the bottom of the columns, and then filtered through a nylon membrane filter (0.45 μm). After this time, the columns were opened and the soil separated into two segments of approximately 10 cm each.

### 1.2.3 Determination of $K_d$ values

The $K_d$ (sorption distribution coefficient) value was considered a measure of herbicide adsorption capacity by the unamended and amended soils. The sorption of MTBU for both unamended and amended soils was determined using a batch equilibrium method. Triplicate 5 g soil samples were equilibrated with 20 mL of an aqueous solution of MTBU at an initial concentration ($C_i$) of 25 ng/mL for 24 hr. The amount adsorbed was considered to be the difference between that initially present in the solution ($C_i$) and that remaining after equilibration ($C_e$). $K_d$ values were obtained from the relationship between $C_s$ and $C_e$ ($K_d = C_s/C_e$).

### 1.3 Photoreaction setup

The photocatalytic and photolytic experiments were performed in a 2 L cylindrical glass (250 mm long, 100 mm diameter) photochemical reactor (SBS, Barcelona, Spain) equipped with a magnetic stirring bar, and an 8 W low pressure mercury lamp. The intensity of the light was 8.5 mW/cm$^2$ in the wavelength range 300–460 nm (based on the manufacturer's data). The study was carried out in batch recirculation mode. The reactant solution containing the semiconductor powder (200 mg/L) and $Na_2S_2O_8$ (200 mg/L) was circulated at a flow rate of 600 mL/min. The reaction system was periodically bubbled with air, continuously stirred to achieve a homogeneous suspension and thermostated by circulating water to keep the temperature at (23 ± 1)°C during 2 hr of irradiation. The diagram of the experimental system used is shown in **Fig. 1**.

The water used in the photodegradation studies was obtained from two lysimeters (3.5 m × 4 m × 1 m) from an experimental greenhouse located in Campo de

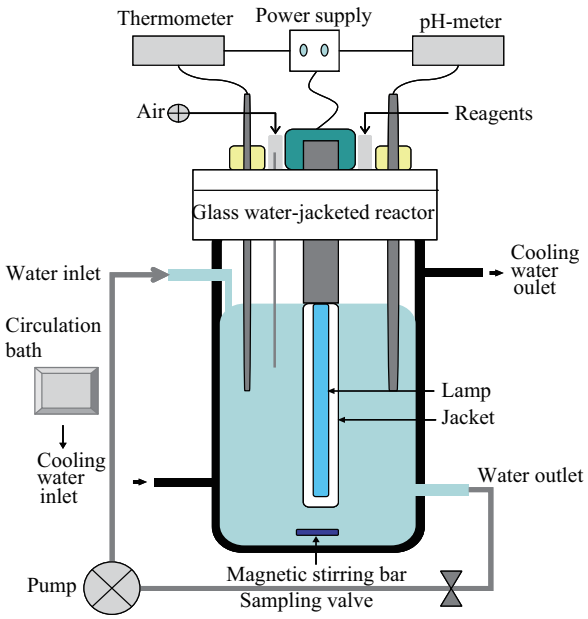


**Fig. 1** Schematic drawing of the experimental setup.

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Cartagena (SE Spain). A clay loam soil (soil A) was used. The soil was irrigated every four days by three driplines (45 min per day and 50 mL/min per emitter). About 8 L per day were collected from each lysimeter. The water had the following characteristics: pH = 8.41, EC = 4.32 dS/m, TOC = 130 mg/L, $NO_3^-$ = 547 mg/L, and $NO_2^-$ = 0.12 mg/L. The leaching water (100 L) was collected and transported to a storage tank. Initially, 2 L of this water containing 100 μg/L of MTBU were homogenized by shaking for 20 min to constant concentration in the dark. After this time the appropriate amount of catalyst and oxidant ($Na_2S_2O_8$) were added to the reaction solution. The respective mixtures were maintained for 20 min in the dark, prior to illumination, in order to achieve maximum adsorption of the herbicides onto semiconductor surface. Several samples from 0 to 120 min were taken during the illumination period. A parallel blank assay, without semiconductor and oxidant (photolytic test) was carried out. In all cases, assays were replicated three times.

### 1.4 Analytical determinations

Water and soil samples were extracted and analyzed according to the procedure described by Fenoll et al. (2011a, 2012b). The separation, identification and quantification of MTBU were carried out using an HPLC system (consisting of vacuum degasser, autosampler and a binary pump) (Agilent Series 1100, Agilent Technologies, USA) equipped with a reversed phase C8 analytical column of 150 mm × 4.6 mm and 5 m particle size (Zorbax Eclipse XDB-C8) and a G6410A triple quadrupole mass spectrometer from Agilent equipped with an ESI interface operating in positive ion mode. Blank samples were used to establish the limit of detection (LOD) and the limit of quantification (LOQ). Both, LODs and LOQs were calculated from the signal-to-noise ratio 3 and 10, respectively. The calibration samples were analyzed by spiking MTBU at 0.5–150 μg/L into water samples in five replicates. The correlation coefficient was found to be > 0.99 and the limits of quantification in soil and water were 0.1 ng/g and 0.04 μg/L, respectively.

### 1.5 Statistical analysis

Curve fitting was carried out and statistical data were obtained using SigmaPlot version 12.0 statistical software (Systat, Software Inc., USA).

## 2 Results and discussion

### 2.1 Leaching and persistence of MTBU on unamended and amended soils

The distribution from soil and water for MTBU applied to soil and amended soil columns is shown in **Fig. 2**. The

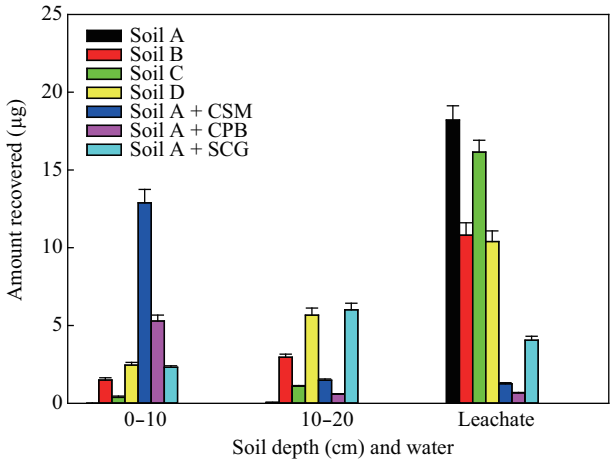


**Fig. 2** Distribution of MTBU applied to unamended and amended soil columns. The error bars denote standard deviation.

greatest pore water volumes observed in soil D, with a high organic matter content, and in soil A with organic amendment, may be attributed to the beneficial effect of organic matter on the pore size distribution by decreasing the larger pores and increasing the smaller ones. Similar results were found by Cox et al. (2001), who suggested that insoluble organic matter cements and aggregates soil particles, blocking large conducting pores. In the case of the unamended soils, total recoveries of MTBU from soil and water were in the range 76%–93% for soil B and soil D, and varied from 33% for soil A + CPB to 78% for soil A + CSM in the case of the amended soils. The difference between initial and recovered amounts can be attributed to biochemical degradation during the experiment. According to Roberts and Hutson (1998), metabolism is influenced by competitive pathways of *N*-demethylation and hydroxylation of the side-chain methyl group.

MTBU was found in leachates in different proportions. Thus, MTBU was found in unamended soils in percentages higher than 50% while in amended soils 3%–20% of the total mass fraction applied to the column was recovered from leachates in soil A + CPB and soil A + SCG. Finally MTBU was found in the bottom layer of the unamended soils in proportions ranging from 0.2% to 28% while in the top fraction of soil it was recovered in proportions of 0.1%–12% (lower than in the bottom layer). For the amended soil, the amount recovered ranged from 29.4% to 71.8% of the initial mass applied to the columns in soil A + CPB and soil A + CSM, respectively. The highest proportions were recovered from the upper layer (0–10 cm) with the exception of soil A + SCG, where 30% of its initial amount was found in the lower layer (10–20 cm).

Relative and cumulative breakthrough curves for MTBU applied to soil columns are shown in **Fig. 3**. For unamended soils, the maximum amount leached was observed at 1.4 PV, while in the case of amended soils the highest potential leaching was about 8.5 PV. At the end of the experiment, when 10 PV had been leached, the upward tendency of the

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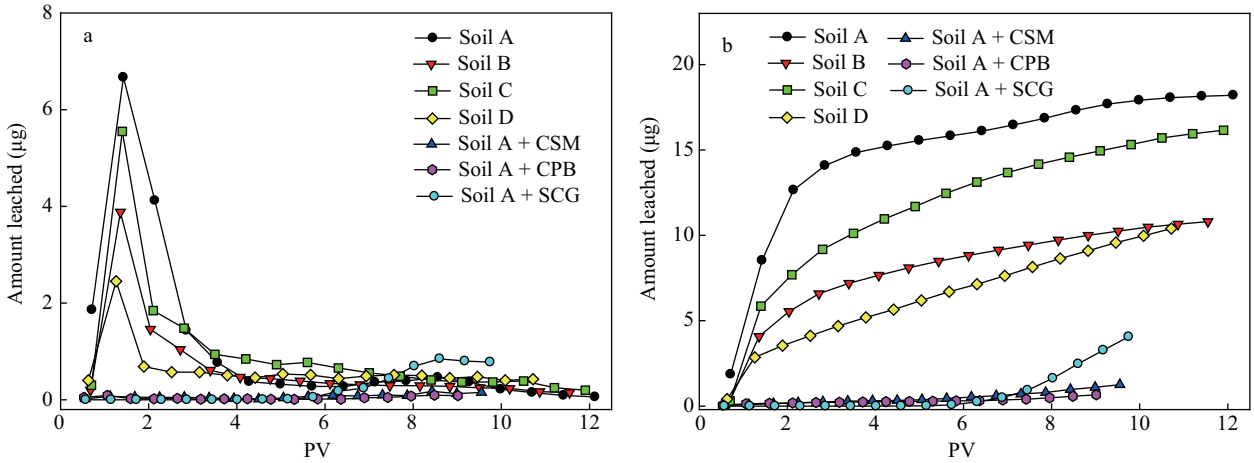


**Fig. 3** Relative (a) and cumulative (b) breakthrough curves of MTBU applied to unamended and amended soil columns. Error bars have been omitted in the figure for better observation.

curves indicated a certain interaction with the organic and inorganic soil colloids contrary to the behavior observed in soil A. As can be seen in **Table 1**, soil D showed higher content of mature organic matter (3.1%) than soil A (1.6%), soil B (2.1%) and soil C (1.9%). Additionally, soil B, C and D contained about 40% of montmorillonite in the clay, an expanding 2:1-type silicate clay with very high total specific surface area (800 $m^2/g$). According to the Groundwater Ubiquity Score Index (Gustafson, 1989) and taking into account the total amount of MTBU found in the leachates and its high water solubility (60 mg/L), this herbicide can be considered to have a high leaching potential in the conditions used. As a consequence, this compound must be considered a potential risk for the pollution of groundwater.

The presence and/or addition of organic matter drastically reduced the movement of MTBU. The $K_d$ (sorption distribution coefficient) obtained for MTBU in different unamended soils indicated their tendency to migrate in soil. The $K_d$ obtained for MTBU in soil A, B, C and D, were 2.1, 13.5, 6.6 and 15.6 mL/g, respectively. Thus, the leachability of MTBU was markedly lower in soil of a similar texture but high OM content (3.1%). Similar results have previously been reported for other pesticides (Fenoll et al., 2011b). According to Albarran et al., (2004), the addition of organic matter enhances the retention of pesticides by increasing soil organic carbon content. However, the environmental effect of amendment on pesticide transport is ambiguous because dissolved organic matter could facilitate the leaching of some pesticides by enhancing their water solubility through dissolved organic matter-pesticide associations (Li et al., 2005). For amended soils, the $K_d$ obtained for MTBU in soil A + CSM, soil A + CPB and soil A + SCG were 52.6, 56.0 and 14.3 mL/g, respectively. These differences can be attributed to the structure (specific surface, particle size, etc) of the substrate material (de Wilde at al. 2008). Thus, the decrease in MTBU leaching obtained by using SCG and CPB can also be attributed to its high lignocellulosic biomass content (cellulose, hemicellulose and lignin) or high lignin content, respectively. Lignocellulosic materials are commonly used as starting material for preparing activated carbon, a compound with a high adsorption capacity (Temdrara et al., 2008). A lignocellulosic substrate from the agro-industry was shown to act as a low-cost and effective adsorbent for the removal pesticides from soil (Karanasios et al., 2010) and wastewaters (Boudesocque et al., 2008; Fenoll et al., 2011c).

### 2.2 Photocatalytic activity and kinetics

The comparative degradation of MTBU in water using the selected photocatalysts (photocatalysis) or not (photolysis) during the irradiation time (120 min) is shown in **Fig. 4**. In the absence of a catalyst, the photolytic oxidation of MTBU occurred at a very slow rate and 84% remained after 120 min. In the presence of the semiconductor oxides, the residual levels of MTBU for ZnO and $TiO_2$ at the end of the experiment were 0.1 and 0.3 g/L, respectively.

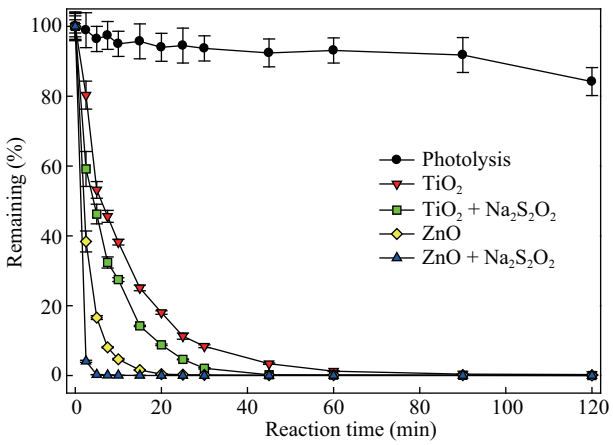


**Fig. 4** Degradation kinetics of MTBU in water by photolysis, and heterogeneous photocatalysis with $TiO_2$, $TiO_2/Na_2S_2O_8$, ZnO and $ZnO/Na_2S_2O_8$ during the photoperiod.

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In this experiment, the effect of photocatalyst and $Na_2S_2O_8$ concentrations on the oxidation of MTBU was studied using several concentrations (from 50 to 250 mg/L) of catalyst (ZnO and $TiO_2$) and $Na_2S_2O_8$. Significant differences were observed in the reaction rate when the concentration of both semiconductors was increased from 0 to 250 mg/L, while increased concentrations of $Na_2S_2O_8$ led to an increase in the reaction rate for this herbicide until it reached a plateau. The optimum value for catalyst was found to be 200 mg/L for both ZnO and $TiO_2$, while the optimum value for oxidant was 200 mg/L for both ZnO and $TiO_2$. When the influence of the initial pH value on the initial photooxidation rate of this compound for the $TiO_2$ and ZnO suspensions was studied, the initial pH values of 6 to 9 had a practically insignificant effect for $TiO_2$ and ZnO, since MTBU is a non-ionized compound at those pHs. Thus, the pH of the water should not be adjusted. As photodegradation progressed, there was a weak decrease in pH (0.4 and 1.3 units for ZnO/$Na_2S_2O_8$ and $TiO_2$/$Na_2S_2O_8$, respectively). At acidic pH, ZnO can react with acids to produce the corresponding salt and at alkali pH, it can react with a base to form $Zn(OH)_2$. Consequently, the toxicity in the illuminated ZnO system can be magnified by the increase of $Zn^{2+}$ in the solution. As regards temperature, photocatalytic systems do not require heating and operate at room temperature because of photonic activation (Herrmann, 2005; Malato et al., 2009). Thus, in our study, the temperature was maintained at 23 ± 2°C.

The photocatalytic degradation of MTBU using the tandem ZnO/$Na_2S_2O_8$ and $TiO_2$/$Na_2S_2O_8$ is presented in **Fig. 4**. In the presence of ZnO/$Na_2S_2O_8$ complete disappearance was achieved after 60 min of illumination. The residual level of MTBU for $TiO_2$/$Na_2S_2O_8$ at the end of the experiment was 0.1 μg/L. Therefore, ZnO appears to have a greater effect on MTBU oxidation than $TiO_2$. This finding agrees with other studies carried out with different pesticides (Fenoll et al., 2011, 2012a, 2012d). The observed differences are almost certainly related to the optical properties, particle diameters and structure of the photocatalyst (Salah et al., 2004). Thus, the main limitation of using of $TiO_2$ as catalyst is due to the fact that it can only take in a small portion of solar spectra in the UV region (Chatterjee et al., 2006) and has high recombination rate of the photoinduced electron-hole pairs at or near its surface (Jiang et al., 2008). In addition, the electron mobility of ZnO in their bulk single-crystal phases is over 2 orders of magnitude higher than that for $TiO_2$, which may in turn minimize recombination rates (Kitsiou et al., 2009). However, the process using ZnO has several drawbacks compared with $TiO_2$ since the pH must be adjusted to precipitate the $Zn^{2+}$ obtained by the photodissolution induced by self-oxidation of ZnO (ZnO + $2h^+ \rightarrow Zn^{2+} + \frac{1}{2}O_2$) and the solution must be neutralized before being released to the environment, as in the case of the photo-Fenton treatment (Fenoll et al., 2012c). Thus, the concentration of $Zn^{2+}$ in water after precipitation was below 0.1 mg/L. Zinc removal is not complete until the metal solid is physically removed from the water, by subsequent sedimentation and filtration. In the case of $TiO_2$ (particle size < 21 nm), the separation of particles from treated water is mainly achieved by microfiltration or by induced accelerated sedimentation and subsequent microfiltration (Fernández-Ibáñez et al., 2003).

The kinetic parameters of these compounds in optimal conditions (ZnO = 200 mg/L, $TiO_2$ = 200 mg/L, $Na_2S_2O_8$ = 200 mg/L, and initial pH = 7.1) were calculated. The apparent rate constants for this compound in the presence of ZnO, $TiO_2$, ZnO/$Na_2S_2O_8$ and $TiO_2$/$Na_2S_2O_8$ were 0.36, 0.09, 1.28 and 0.14 $min^{-1}$, respectively The degradation fitted the exponential decay curve well, following first-order behaviour that was consistent with the Langmuir-Hinshelwood mode with $R^2 > 0.989$:

$$-dC_P/dt = k_{ap}C_p \quad (1)$$

where, $C_p$ (mol/L) is the concentration of the pesticide and $K_{ap}$ ($time^{-1}$) is the apparent first-order rate constant.

The half-lives for this compound in the presence of ZnO, $TiO_2$, ZnO/$Na_2S_2O_8$ and $TiO_2$/$Na_2S_2O_8$ were 1.9, 7.3, 0.5 and 5.0 min, respectively. Finally, metabolism of methabenzthiazuron can be influenced by the competitive pathways of *N*-demethylation and hydroxylation of the side-chain methyl group (Roberts, 1998).

## 3 Conclusions

This study summarizes the effect of using different techniques to minimize MTBU residues in leaching waters. As general rule, the presence and/or addition of organic matter drastically reduced the mobility of this compound in soils with a low organic matter content. The reduction in leaching can be attributed to the increased sorption capability on the amended soils. On the other hand, the results obtained in this work point to the efficacy of using photocatalysis with zinc oxide and titanium dioxide for degrading MTBU in leaching water irradiated by artificial light. The rate constants of the catalyst were in the order: ZnO/$Na_2S_2O_8$ > ZnO > $TiO_2$/$Na_2S_2O_8$ > $TiO_2$. The half-lives for MTBU ranged from 0.5 to 7.3 min. However, the process using ZnO had several drawbacks compared with $TiO_2$ since the pH must be adjusted to precipitate the $Zn^{2+}$ obtained by the photodissolution induced by self-oxidation of ZnO.

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