



Sorption of sulfadiazine on Brazilian soils

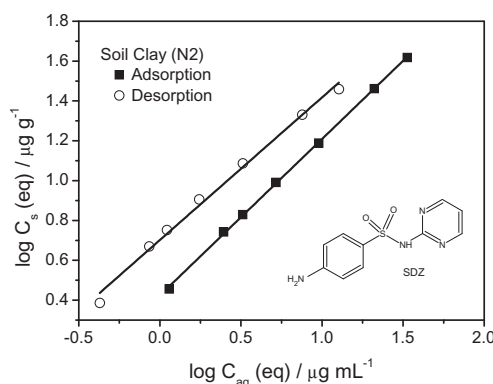
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HIGHLIGHTS

- ▶ The sorption of sulfadiazine (SDZ) was studied in four tropical soil–water systems.
- ▶ The low values of the K_F indicate that SDZ is highly mobile in the evaluated soils.
- ▶ The desorption values were higher than those obtained for adsorption of SDZ.
- ▶ A small positive hysteresis was observed with all soils under study.
- ▶ The main mechanism of the sorption of SDZ is partitioning to organic matter.

GRAPHICAL ABSTRACT



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ABSTRACT

Antimicrobials, among them sulfonamides are widely used in veterinary medicine and can contaminate the environment. The degree to which antimicrobials adsorb onto soil particles varies widely, as does the mobility of these drugs. Sulfadiazine (SDZ) was used to study the adsorption–desorption in Brazilian soil–water systems, using batch equilibrium experiments. Sorption of SDZ was carried out using four types of soils. Adsorption and desorption data were well fitted with Freundlich isotherms in log form ($r > 0.999$) and ($0.984 < r < 0.999$), respectively. An adsorption–desorption hysteresis phenomenon was apparent in all soils ranging from 0.517 to 0.827. The experimental results indicate that the Freundlich sorption coefficient (K_F) values for SDZ ranged from 0.45 to $2.6 \mu\text{g}^{1-1/n} (\text{cm}^3)^{1/n} \text{g}^{-1}$.

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1. Introduction

Sulfonamide antimicrobials comprise a class of synthetic sulfanilamide derivatives which are widely used in human and veterinary medicine. Sulfadiazine (SDZ) belongs to the class of sulfonamides and is – in addition to its use in human medicine – one of the most widely used antibacterial veterinary drugs (Thiele-Bruhn, 2003). Sulfonamides are quickly eliminated from the organism, mainly as the parent compounds or as their acetyl- or hydroxyl-conjugated

metabolites (Boxall et al., 2002; Lamshöft et al., 2007). Therefore, sulfonamides may reach the environment through grazing livestock or spreading of manure on agricultural soils. A further pathway into the environment is the direct use of drugs in aquacultures. Once in the environment, they may sorb onto the soil and sediment, be transported to ground or surface waters, or be degraded. In addition, antimicrobials can provoke the formation of resistant microorganisms (Hirsch et al., 1999; Boxall et al., 2003).

The fate of sulfonamides, including SDZ, in soils, ground waters or surface waters is dependent on the processes of sorption, degradation and leaching. These processes are, in turn, governed by physico-chemical properties, such as structure, size, shape, solubility,

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hydrophobicity, and speciation, and soil properties, such as organic matter content, texture, mineralogy, clay content, pH, ionic strength, and cation exchange capacity, apart from local weather conditions (Sarmah et al., 2006).

For a reliable assessment of sulfonamide mobility and bioavailability, the identification of factors affecting sorption to soils and sediments is essential. Thiele-Bruhn et al. suggested that for sulfonamide sorption to soils, not only is the organic matter content important to be considered, but also the presence of clay minerals and pedogenic oxides (Thiele-Bruhn et al., 2004). In sorption experiments, the addition of manure to soil samples resulted in decreasing sorption of sulfonamides compared to soil without manure. Studies have suggested that sulfachloropyridazine is relatively weakly sorbed in soils and slurry amended soils and is, thus, quite mobile in soil (Boxall et al., 2002). Research indicates that sulfonamides are sorbed on soil particles with distribution constants (K_D) ranging from 0.9 to 3.5 L kg⁻¹ (Boxall et al., 2002; Thiele-Bruhn, 2003). Other results reported by Thiele-Bruhn and Aust suggests lower sorption of sulfanilamide, sulfadimidine, sulfadiazine, sulfadimethoxine and sulfapyridine in pure soil systems than in pig slurry and pig slurry amended soils (K_D ranging from 0.3 to 2.0 L kg⁻¹) (Thiele-Bruhn and Aust, 2004).

Almost all data reported in the literature about antimicrobial sorption in soils were obtained through studies carried out in temperate regions. Different environmental conditions found in the tropics, such as increased rainfall and temperature, predominance of low-activity clays, a lower percentage of organic matter in soils and lower pH will contribute to different behaviors of these antimicrobials in Brazilian soils and need to be evaluated. The aim of this study was to investigate the fate of sulfonamides in four types of Brazilian soils, using sulfadiazine as a model compound. For this purpose batch sorption kinetic experiments were carried out.

2. Materials and methods

2.1. Soil samples

Sorption studies of sulfadiazine on four different characteristic soils from the state São Paulo, Brazil, named N1 (sandy), N2 (clay), S1 (sandy-clay) and S2 (clay) were carried out.

Soils were collected in 2005 from different locations and transferred to lysimeters (1 × 1 × 2 m) located in the experimental area of the Brazilian Agricultural Research Corporation (Embrapa), Jaguariúna, SP, Brazil.

The origins of the soils were:

N1 – sandy, city of Santa Rita de Passa Quatro, SP (21°42'18,12"S and 47°28'04,82"W, altitude 773 m) (pasture).

N2 – clay, city of Sertãozinho, SP (21°05'20,44"S and 47°48'10,73"W, altitude 538 m) (sugar cane plantation).

S1 – sandy-clay, city of Jagariúna, SP (22°43'14,92"S and 47°01'14,20"W, altitude 617 m) (citrus plantation).

S2 – clay, city of Jagariúna, SP (21°42'59,50"S and 47°01'00,05"W, altitude 609 m) (covered with *Brachiaria*).

The soils were collected from each lysimeter in July 2010, air-dried, sieved to a particle size ≤ 2 mm, and stored in plastic bags at room temperature until use. The physical and chemical characteristics of each soil are presented in Table 1.

2.2. Reagents and chemicals

All solvents used were HPLC grade and all reagents were at least analytical grade. Calcium chloride was supplied by Nuclear (Brazil).

Table 1

Physical and chemical properties of the selected soils.

Property	Soil			
	N1	N2	S1	S2
pH (in 0.01 mol L ⁻¹ CaCl ₂)	5.0	4.9	4.1	4.4
Depth of soil (cm)	0–20	0–20	0–20	0–20
Organic material (% w/w)	1.53	2.88	2.48	3.23
Organic carbon (% w/w)	0.89	1.67	1.44	1.87
Texture (%): sand	91.1	14.9	52.9	43.5
Silt (0.053–0.002 mm)	1.8	30.2	10.5	7.0
Clay (<0.002 mm)	6.2	54.6	36.2	49.2
Cation exchange capacity (mmol _c kg ⁻¹)	19.3	52.7	51.9	66.0

Sulfadiazine (SDZ; benzenesulfonamide, 4-amino-N-2-pyrimidyl; CAS Registry No. 68-35-9; 99%) was purchased from Sigma-Aldrich, Belgium. Molecular structure and some physico-chemical properties of SDZ are shown in Table 2. Throughout the study, water was obtained from a Milli-Q purification system (Millipore, USA). A standard stock solution (750 µg mL⁻¹) of SDZ was prepared by dissolving 75 mg of SDZ in 100 mL warm water. Working solutions of SDZ were prepared by appropriate dilution of the standard stock solution with 0.01 mol L⁻¹ CaCl₂.

2.3. HPLC analyses

Chromatographic analysis were carried on an Agilent HPLC Series 1200 system (Agilent, USA) equipped with a G1311A quaternary pump, a G1315D photodiode array detector (PAD), a G1316A column oven and G1329A auto sampler. Chromatographic separation of SDZ was achieved using a XBridge™ reversed-phase column (RP-18, 150 × 4.6 mm, 3.5 µm particle size) from Waters (Ireland) at 40 °C. Methanol with 0.1% (v/v) formic acid added and water with 0.1% (v/v) formic acid added (10:90 v/v) was the mobile phase. Injection volume was 20 µL and the flow rate was 0.7 mL min⁻¹. Quantitation of SDZ was performed at 266 nm. The retention time for SDZ was 8.6 min.

2.4. HPLC method validation

The method was *in house* validated and the following parameters were evaluated: linear range, linearity, selectivity, matrix effect, intra- and inter-day precision, limit of detection and limit of quantitation. The linearity and linear range were established through calibration obtained, by triplicate analyses, of five concentrations levels (0.05; 0.1; 2.0; 6.0 and 10.0 µg mL⁻¹) of SDZ in 0.01 mol L⁻¹ CaCl₂.

The matrix effect was evaluated for all soils under study through fortification (10.0 µg g⁻¹ SDZ) of the supernatant obtained after soil samples were equilibrated (48 h, at room temperature) with CaCl₂, centrifuged and filtered with 0.22 µm membrane filters. The fortified extract was analyzed by HPLC-PAD and the area obtained compared with the area of a SDZ solution at the same concentration level in 0.01 mol L⁻¹ CaCl₂. All analyzes were carried out in triplicate.

The selectivity of the method was evaluated by the analyses of the chromatograms obtained with equilibrated 0.01 mol L⁻¹ CaCl₂ blank soil sample solutions and with SDZ in 0.01 mol L⁻¹ CaCl₂. The purity of SDZ using the spectral data generated by the photodiode array detector was verified by the full spectrum at the upslope-, the apex- and the downslope in order to ensure that no coeluting impurities or compounds of the soil matrix contributed to peak response.

The intra-day precision of the method was evaluated through the analyses of the CaCl₂ extracts obtained of each soil fortified with 10.0 µg g⁻¹ SDZ, after an apparent equilibrium time of 48 h.

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