Adsorptive removal of sulfonamide antibiotics in livestock urine using the high-silica zeolite HSZ-385

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APPENDIX

Chemicals

Sulfathiazole, sulfamethizole (Tokyo Kasei, Ltd., Tokyo, Japan), sulfamerazine, sulfamethoxazole, sulfamonomethoxine, sulfadimethoxine (Wako Pure Chemical Industries, Ltd., Osaka, Japan), sulfadimidine (Sigma Aldrich, St Louis, MO, USA). All chemicals used were reagent grade and of high quality. The molecular structures of sulfonamide are summarized in Figure A1. Other reagents, including acetonitrile, sulfuric acid, sodium hydroxide and formic acid, were used without further purification.

Solid phase extraction (SPE)

A 6-mL SPE cartridge with 200 mg sorbent (Oasis HLB, Waters, Milford MA, USA) was conditioned subsequently with 1 mL methanol and 5 mL deionized water. The zeolite powder was removed using a membrane filter. The pH of the urine sample was adjusted to 3. After sample loading, the SPE cartridge was washed with 5 mL deionized water and the analytes eluted with 1 mL of 10% methanol and 4 mL of methanol.

LC/MS/MS analysis

The concentrations of sulfonamides were measured by standard additional method using LC/MS/MS (ACQUITY UPLC-Xevo TQ; Waters) after SPE. LC/MS/MS analyses were carried out using a BEH C18 column (2.1 × 150 mm, Waters) with a linear gradient from 10% acetonitrile in 0.05% formic acid (isocratic for 0.5 min) to 90% (0.5–7.0 min) at a constant flow rate of 0.3 mL/min. The conditions of cone voltage and collision energy MS/MS experiments on the Xevo-TQ instrument were optimized for each sulfonamide.
Other analyses

The NPOC content of porcine urine was quantified. Briefly, urine was diluted 100 times using Millipore water (Millipore, Bedford, MA, USA) and filtered with a membrane filter (DISMICD; pore size, 0.20 μm; ADVANTEC, Ltd). The NPOC content was measured using a Shimadzu Total Organic Carbon analyzer (TOC-5000A; Shimadzu, Kyoto, Japan) based on CO₂ quantification by non-dispersive infrared analysis after high-temperature catalytic combustion.

Concentrations of urea and uric acid were measured using an Automatic Analyzer (Fuji Drichem 3500V; Fuji Photo Film, Tokyo, Japan).

Concentration of ions in porcine urine was measured using ion chromatography (DX-120; Dionex, Sunnyvale, CA, USA). The cations were measured by adopting a CS12A column (4 × 250 mm; Dionex) and 19.8 mM methanesulfonic acid as eluent (flow rate 1 mL min⁻¹). The anions were analyzed by using AS12A anionic column (4 × 200 mm; Dionex) and a mixture of 2.7 mM Na₂CO₃ and 0.3 mM NaHCO₃ (flow rate 1.5 mL min⁻¹).