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High-resolution mass spectrometry insights into pharmaceutical partitioning and transformation in aquaculture water-sediment microcosms

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Introduction

Aquaculture is a rapidly expanding food production sector, accompanied by increasing use of pharmaceuticals and antibiotics that can accumulate in aquatic environments. Contaminants of emerging concern (CECs) released from fish farms may persist in water-sediment systems, where sediments act as sinks or secondary sources. Understanding compound-specific fate processes is essential for environmental risk assessment, yet comparative data addressing the relative roles of biotic and abiotic mechanisms under realistic conditions remain limited. In this study, HRMS-based suspect screening is applied to investigate the fate of three pharmaceuticals with contrasting properties, paracetamol (APAP), sulfamethoxazole (SMX) and ciprofloxacin (CIP), in reconstructed aquaculture microcosms from temperate and tropical regions.

Methods

Water-sediment microcosms were reconstructed using samples from six active aquaculture sites in Italy and Thailand, covering freshwater and brackish ponds with different physicochemical characteristics. Experiments were conducted under sterilized and non-sterilized conditions to distinguish between abiotic and biotic processes. Target compounds were spiked into the water phase and monitored over time. Sequential extraction protocol was applied to assess contaminant partitioning into dissolved, exchangeable, and strongly bound sediment fractions. Extracts were analyzed using a Thermo Scientific Orbitrap IQ-X Tribrid mass spectrometer with a heated ESI source. Data were acquired in positive and negative mode using dd-MS^2 with HCD fragmentation. LC-HRMS/MS supported transformation products identification through a suspect screening approach. Temporal trends and mass balances were evaluated to elucidate dominant removal mechanisms.

Preliminary data

Results revealed markedly different fate pathways among the investigated pharmaceuticals. APAP was rapidly removed from the aqueous phase under non-sterilized conditions, with >80% loss within 7 days, while remaining stable in sterilized systems. Minimal accumulation in sediment fractions indicated that biodegradation was the dominant removal mechanism rather than partitioning. HRMS suspect screening confirmed the formation of multiple APAP transformation products, including oxidized and dimeric species, supporting active biotransformation.

SMX showed moderate persistence, remaining largely dissolved across all investigated sites, with only 20-40% attenuation over 21 days. Limited sediment sorption was observed, and removal was strongly site dependent, suggesting a key role of local physicochemical conditions and microbial activity. LC-HRMS/MS revealed the formation of several SMX transformation products, including oxidized and acetylated derivatives, indicating slow and incomplete biotic transformation.

In contrast, CIP exhibited a fundamentally different behavior. It disappeared from the aqueous phase within 24 h at almost all sites under both sterilized and non-sterilized conditions. Sequential extraction showed negligible recovery from bioaccessible sediment fractions, indicating rapid transfer to strongly bound, non-extractable pools. The absence of HRMS-detectable metabolites in these experimental conditions, combined

with rapid aqueous depletion, suggests that CIP removal was governed by strong and largely irreversible sediment sorption rather than degradation.
Overall, the combined use of concentration trends, sequential extraction, and HRMS suspect screening enabled the discrimination between biodegradation-driven dissipation, partial transformation with persistence, and sorption-dominated removal. This compound-specific hierarchy of fate processes underscores the importance of integrating high-resolution mass spectrometry with matrix-resolved approaches to correctly interpret pharmaceutical behavior in complex aquaculture water–sediment systems.

Novel aspect

The study establishes a compound-specific fate hierarchy in aquaculture sediments, supported by transformation product evidence.