



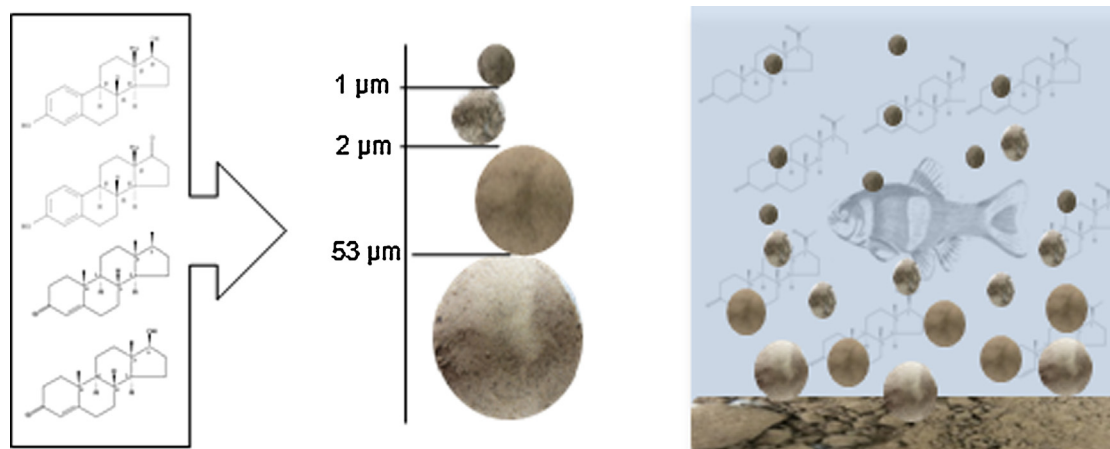
The effect of particle size on sorption of estrogens, androgens and progestagens in aquatic sediment



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GRAPHICAL ABSTRACT



HIGHLIGHTS

- Two sediments were used to evaluate the effects of particle size on steroid sorption.
- Sorption capacity did not increase with decreasing particle size for all steroids.
- Particle interactions affect the distribution of steroids within the whole sediments.
- Preferential sorption to fine particles was observed.

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ABSTRACT

There is growing concern about the biologic effects of steroid hormones in impacted waterways. There is increasing evidence of enhanced transport and biological effects stemming from steroid hormones associated with soils or sediments; however, there are limited studies evaluating how steroid hormone distribution between various particle sizes within whole sediments affects steroid fate. In this study, sorption of 17β -estradiol, estrone, progesterone, and testosterone was evaluated to different size fractions of two natural sediments, a silty loam and a sandy sediment, to determine the steroid sorption capacity to each fraction and distribution within the whole sediment. Sorption isotherms for all steroid hormones fit linear sorption models. Sorption capacity was influenced more by organic carbon content than particle size. Interactions between size fractions were found to affect the distribution of steroids

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within the whole sediments. All four steroids preferentially sorbed to the clay and colloids in the silty loam sediment at the lowest aqueous concentration (1 ng/L) and as aqueous concentration increased, the distribution of sorbed steroid was similar to the distribution by weight of each size fraction within the whole sediment. In the sandy sediment, preferential sorption to fine particles was observed.

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1. Introduction

In addition to being naturally occurring in the environment, large quantities of steroid hormones including 17 β -estradiol (E2), estrone (E1), testosterone (T), and progesterone (Pr) are used annually for both human health and animal production. Studies have shown that these compounds pose risks to fish and other aquatic organisms at extremely low concentrations (\sim 1 ng/l) such as decreased fertility, feminization, and hermaphroditism [1]. In particular, attention has focused on the feminization of fish in surface waters receiving discharges of steroid-containing municipal wastewater effluent [1,2]. E2, E1, T, and Pr are commonly found in wastewater effluents and in sediments and receiving water downstream of municipal wastewater discharges [3,4]. More recently, attention has focused on animal production as a source of steroid hormones to aquatic environments as animals may be given growth promotants in the form of hormones. Wastes contain androgens, estrogens, and progestagens, as well as synthetic hormones such as trenbolone (Tb) are commonly land applied to agricultural fields as a source of fertilizer. Hormone transport from agricultural fields and feedlot surfaces has been documented during irrigation and precipitation events [5–7]. The presence of steroidogenic compounds in surface water and the corresponding endocrine-disrupting effects on aquatic species has been documented adjacent to animal production facilities [8–10]. Due to their hydrophobicity, hormones will predominantly sorb to sediment which act as potential sinks and sources of hormones and exposure to hormones bound to sediment can elicit biologic effects in aquatic organisms, either by acting as a source of free hormone through desorption from the sediments and/or by direct contact between fish and sediment-associated hormone [11]. Zhou et al. [12] found 4–26% of E1 and 15–30% of E2 downstream of a wastewater treatment plant was associated with suspended aquatic colloids. Increasing evidence suggests sediment texture and particle size may play an important role in the persistence [13] and biologic effects [2,14] of hormones in aquatic systems.

The effects of soil particle size on contaminant distribution has been evaluated experimentally and in the field for a variety of contaminants including trace metals [15] and other hydrophobic organic compounds including octylphenol, nonylphenols, bisphenol a (BPA), polychlorinated biphenyls, dibutylphthalate, polycyclic aromatic hydrocarbons, and various perfluorinated compounds [16–18]. However, there have been a limited number of studies evaluating how steroid hormones distribute among various particle size fractions in aquatic sediment. Existing studies focus on interactions between estrogens and colloids [12,19] or dissolved organic matter [20] or selected soil/sediment particle size fractions [2,21] or mineral constituents [22] and do not look at distribution among fractions within a whole soil or sediment. Sun et al. [23] systematically evaluated the sorptive behavior of a synthetic estrogen, 17 α -ethinyl estradiol (EE2), and two other endocrine active compounds, BPA and phenanthrene, in soil and sediment particle size fractions and determined sorption of all three compounds was strongly correlated with the organic carbon (OC) content of the soil. Recently, Yong et al. [24] evaluated sorption behavior of T in five fractions from an agricultural soil. Results show T has a higher affinity for smaller particle size fractions which is likely due to both increased surface area and OC content in the smaller parti-

cle size fractions. While, studies like these provide valuable insight into the sorptive behavior of soils, each of these studies used soils or sediment fractionated prior to sorption testing, which may not represent how steroids would naturally distribute between particle sizes within whole sediments.

This study focuses on evaluating the sorptive behavior of four steroid hormones to different size fractions from two natural sediments. The objective was to evaluate not only sorption capacity of particle size fractions within natural aquatic sediments, but also to evaluate preferential distribution of steroid hormones between various particle sizes within whole sediment. To do this, three types of sorption experiments were utilized; whole sediment sorption, sorption capacity where sediment was fractionated prior to equilibration with hormones, and preferential sorption where whole sediment was equilibrated with hormone prior to rapid fractionation. The results will improve the understanding of steroid hormone distribution and bioavailability in aquatic systems.

2. Materials and methods

2.1. Sediment collection and preparation

Two natural aquatic sediments were chosen for use in this study to represent differences in physical characteristics; a silty loam (16% sand, 60% silt, 24% clay) was collected from Plum Creek in Seward, Nebraska, U.S.A and a sand (92% sand, 6% silt, 2% clay) was collected from the Elkhorn River near Winslow, NE, U.S.A. Although, these sediments represent differences in physical characteristics, it should be noted that they do not represent the full range of physical characteristics observed in natural sediments. However, sediments from these systems have been used frequently in laboratory and field studies either directly [11,14] or in close proximity to the collection sites used in this study [8–10]. Sediments were air dried before use. Properties of each sediment are given in Table 1. Additional information about mineralogy can be found in Supporting information (Table S1).

Each sediment was categorized according to particle size ranges as defined by USDA [26]. For sorption capacity experiments, sediment was fractionated using methods detailed in [26] utilizing both sieve and deposition methods. Briefly, sand was removed from the whole sediments using a wet sieve technique and was rinsed to remove any fine particles or debris that may have adhered to the sand particles. A repeated sedimentation procedure was used to isolate the remaining silt, clay, and colloid fractions. Approximately, 100–200 g of the remaining sediment was placed in a 1 L beaker with water and sonicated to separate particles. This was allowed to sit undisturbed until particles became stratified. It was possible to estimate settling time needed for specific particles based on standard particle settling velocity equations and verifying the size of the particles in suspension using a zetasizer (NanoS90). The suspension containing the desired particle sizes was removed using a pipette and allowed to air dry before use. Due to a limited amount of fines in the sandy sediment, it was decided to assess the fines as a single fraction. 5 fractions consisting of sand ($>53 \mu\text{m}$), large silt (53–15 μm), small silt (15–2 μm), clay (2–1 μm), and colloids ($<1 \mu\text{m}$) were evaluated in the silty loam sediment and 3 fractions consisting of sand ($>106 \mu\text{m}$), very fine sand (106–53 μm),

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