



# Sorption of testosterone on partially-dispersed soil particles of different size fractions: Methodology and implications



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## ABSTRACT

Sorption of hormones to soil particles of different size fractions (DSFs) has been studied to understand their fate and transport (F/T) in soils. Conventional studies fractionated the soil particles into DSFs by using the high speed stirring method and/or adding surfactants to fully disperse the bulk soil. However, the natural processes (e.g., soil erosion, irrigation) often are relatively mild, and many soil particles may be still in the aggregate form. In this study, a method was developed for conducting the sorption test of a representative hormone (i.e., testosterone) to bulk soils first and then analyzing the results against DSFs. Results indicated the particle size distribution (PSD) of the two representative soils tested with partially-dispersed and fully-dispersed methods was significantly different due to the attachment of clay particles on sand and silt. Testosterone was sorbed mainly by the dominant aggregates even though they might have relatively lower sorption affinity than that of clays. However, the small particles (<2000 nm), even with ~5% mass of the bulk soil, contributed more than 30% of sorbed testosterone in the “whole” soils. The partially-dispersed soil particles of DSFs should be used to understand the transport of hormone in runoff, because using the fully-dispersed soil particles will overestimate while the whole soil method will underestimate the transport potential. With the methodology developed in this study, the sorption tests will not compromise soil's original properties (e.g., aggregates) or the competition (e.g., sorption) among soil particles, and the contribution of DSFs (particularly the partially-dispersed aggregates) to the sorption of the “whole” soil can be determined.

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## 1. Introduction

The increasing natural and synthetic steroidal hormones found in environments worldwide have caused wide public concern because of their potential of adverse effects on aquatic lives and human beings at very low concentrations (Kolpin et al., 2002; Orlando et al., 2004; Martinovic et al., 2007; Young et al., 2014). Currently, one of the critical research areas is to investigate the fate and transport (F/T) of hormones in the soil environment (Das et al., 2004; Salvia et al., 2014), with sorption properties of hormones to soils of different textures and organic contents being a major research focus. Soils with finer texture and higher organic content were reported to have a higher sorption affinity (Lee et al., 2003; Casey et al., 2004; Ma, 2009).

Soils contain a wide range of particles with different size

fractions (DSFs). These particles have different mobility during rainfall events (Mouri et al., 2014). Over 80% of the total soil particles found in storm water runoff were less than 20  $\mu\text{m}$  (Randall, 1982; Furumai et al., 2002). It has been reported that the sorption and desorption properties of pesticides (e.g., atrazine and diuron) associated with soils exhibited significant particle-size dependence (Wang and Keller, 2008). A slight change in clay content in the sediment can greatly change the sediment's potential for transporting chemicals sorbed on the solid phase (Foster et al., 1985). Therefore, it is necessary to determine the sorption–desorption properties of hormones associated with soil particles of DSFs. Qi et al. (2014) used wet-sieve-washing and repeated-sedimentation methods to fractionate soils into five soil particles of DSFs and used each of the DSFs for conducting sorption-desorption tests of testosterone. They found that the sorption and desorption properties of soil particles of DSFs associated with hormones are very different; clay particles have relatively high sorption capacity, mainly due to their high specific surface area. These previous studies fractionated the soil particles into DSFs by using the high

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speed stirring method and/or adding surfactants (ASTM, 2004), and then conducted the sorption tests with soil particles of each size fraction independently (Wang and Keller, 2008; Qi et al., 2014). These methods can be used to determine the ultimate texture of the soil with respect to certain chemical or physical properties and the sorption-desorption behavior of these soil particles of DSFs.

One concern is that these sorption tests may compromise the soil's original properties (e.g., aggregates, attached organic matter) and the competition among soil particles of DSFs. It is well-known that the fundamental difference between fully-dispersed and partially-dispersed soil particles is very significant (Fig. 1). A soil aggregate is defined as a group of primary soil particles that cohere to each other more strongly than to other surrounding particles (Le Bissonnais, 1996). Many soil aggregates are stable under the forces of erosion (Young, 1980). In nature, the dispersion processes (e.g., irrigation, soil erosion during storm events) often are relatively mild, and these soil particles of DSFs are mixed together in the form of aggregates (i.e., partially-dispersed soil particles). Therefore, one should conduct the sorption tests with the 'bulk' soil first, and then fractionate the soil into particles (including soil colloids) of DSFs using partially-dispersed methods (e.g., no chemical, mild stirring). However, the existing knowledge gap is: *after the sorption test with the bulk soil, how to fractionate the soil particles of DSFs and then evaluate their contributions to sorption of hormones?*

The goal of this study is to fill this knowledge gap. The specific objectives of this study were to: 1) develop the procedures to conduct sorption test with bulk soil first, and then fractionate soil particles into DSFs under series sedimentation and centrifugation conditions; 2) compare the properties [e.g., total organic carbon (TOC), particle size distribution (PSD)] of partially-dispersed and fully-dispersed soil particles of DSFs; and 3) investigate the sorption distribution of testosterone among partially-dispersed soil particles of DSFs in the bulk soils.

## 2. Materials and methods

Figs. 2 and 3 shows the major procedures developed in this study. Details are as follows.

### 2.1. Selected hormones and soils

Testosterone was selected as the representative hormone because 1) it is a prototype for other synthetic androgenic hormones (Kanayama and Pope, 2012); 2) little is known about the F/T of androgenic hormones (Casey et al., 2004); and 3) it was used previously (Qi et al., 2014; Qi and Zhang, 2015a; Ma et al., 2015). In the present study, sorption tests were conducted with testosterone

at environmentally relevant low concentration levels (20–150 ng/L) (Finlay-Moore et al., 2000).

Two representative soils were selected to evaluate the effects of soil properties on methodology establishment and sorption properties of hormones; they were HAL (Haskell Agriculture Laboratory, Concord, NE, USA) soil and Iowa Black (IA, USA). These two soils were selected because they were sampled from the sites where the concentrated animal feeding operations (CAFOs) were nearby, and they were used in the previous studies (Ma et al., 2015; Qi et al., 2014). The HAL soil (Nora silty clay loam, fine-silty, mixed, mesic Udic Haplustolls) contains 0.95% total organic carbon (TOC). The total Fe, Cu, Ca, Mg are 21, 4.7, 2131, 318 mg/kg, respectively. The cation exchange capacity (CEC) of HAL soil is 13.9 meq/100 g. The Iowa Black (Dickinson sandy loam soil, coarse-loamy, mixed, superactive, mesic Typic Hapludolls) contains 0.68% TOC. The CEC of Iowa Black is 13.8 meq/100 g. No residual testosterone was detected in the soil samples (Ma et al., 2015). After air drying and sieving with a 2-mm sieve to remove larger particles, the soils (called original soils) were ready for use.

### 2.2. Chemicals and solution

<sup>14</sup>C-labeled testosterone came from the American Radio Chemicals Society (St. Louis, MO, USA). Low activity stock solution (= 1 µg/L, directly used in the experiment) was made by adding 1 µL of high activity (= 50 µCi) stock solution of <sup>14</sup>C-labeled testosterone to 10 mL deionized (DI) water (electrical resistivity = 18.2 MΩ cm). Unlabeled testosterone stock solution (= 10 µg/mL) was made by dissolving 0.001 g testosterone into 100 mL methanol, and preserved at 4 °C in a brown glass container under the dark condition. The concentration of methanol was less than 1% by volume. Under this concentration, no effect of methanol on sorption of soils was observed by other researchers (Wauchope and Koskinen, 1983; Fan et al., 2007). CaCl<sub>2</sub> (96% purity) and NaN<sub>3</sub> (99% purity) were purchased from Fisher Scientific Inc.

### 2.3. Fully-dispersed and partially-dispersed particle size distribution of original soils

In this study, six diameter size ranges were studied for partially-dispersed soil particles (Table 1): sand-sized aggregates (SDA = primary sand + large aggregates) (2–0.075 mm), silt-sized aggregates (STA = primary silt + small aggregates) (0.075–0.002 mm), clay 1 (2–0.9 µm), Colloids#1 (0.9–0.6 µm), #2 (0.6–0.4 µm), and #3 (0.4–0.2 µm). For fully-dispersed ones, corresponding size ranges of primary particles were investigated by Qi et al. (2014).

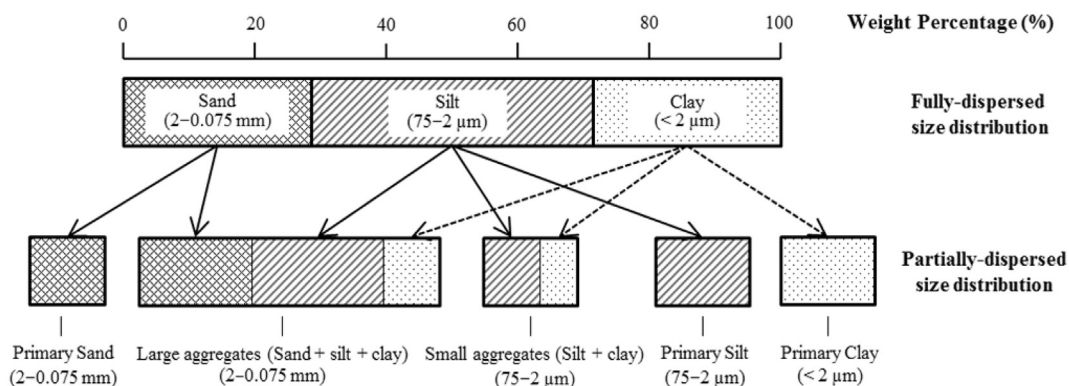


Fig. 1. Relationship between the fully-dispersed and partially-dispersed soil particle size distribution (Foster et al., 1985; Di Stefano et al., 2002).

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