



# Retention and loss of water extractable carbon in soils: Effect of clay properties



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## HIGHLIGHTS

- Little is known about which clay properties are important for retention and loss of water extractable C (WEOC).
- Clay with higher native TOC and lower Fe/Al concentrations sorbed less and lost more WEOC.
- Clays with low TOC and high Fe/Al concentrations had a high WEOC retention capacity.
- Retention of WEOC was not related to clay mineralogy, CEC, and exchangeable Ca concentration.

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## ABSTRACT

Clay sorption is important for organic carbon (C) sequestration in soils, but little is known about the effect of different clay properties on organic C sorption and release. To investigate the effect of clay content and properties on sorption, desorption and loss of water extractable organic C (WEOC), two experiments were conducted. In experiment 1, a loamy sand alone (native) or mixed with clay isolated from a surface or subsoil (78 and 96% clay) resulting in 90, 158 and 175 g clay kg<sup>-1</sup> soil. These soil treatments were leached with different WEOC concentrations, and then CO<sub>2</sub> release was measured for 28 days followed by leaching with reverse osmosis water at the end of experiment. The second experiment was conducted to determine WEOC sorption and desorption of clays isolated from the loamy sand (native), surface soil and subsoil. Addition of clays isolated from surface and subsoil to sandy loam increased WEOC sorption and reduced C leaching and cumulative respiration in percentage of total organic C and WEOC added when expressed per g soil and per g clay. Compared to clays isolated from the surface and subsoil, the native clay had higher concentrations of illite and exchangeable Ca<sup>2+</sup>, total organic C and a higher CEC but a lower extractable Fe/Al concentration. This indicates that compared to the clay isolated from the surface and the subsoil, the native clay had fewer potential WEOC binding sites because it had lower Fe/Al content thus lower number of binding sites and the existing binding sites are already occupied native organic matter. The results of this study suggest that in the soils used here, the impact of clay on WEOC sorption and loss is dependent on its indigenous organic carbon and Fe and/or Al concentrations whereas clay mineralogy, CEC, exchangeable Ca<sup>2+</sup> and surface area are less important.

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## 1. Introduction

Dissolved organic carbon (DOC), derived from microbial activity, root exudates, leachate of canopy and leaf litter, accounts for only a small proportion of total soil carbon and is the most mobile and bio-available fraction of soil organic matter (Marschner and Kalbitz, 2003; McDowell, 2003). DOC has an important role in ecosystems because it is an easily accessible carbon source for microbes (Burford and Bremner, 1975; Meyer et al., 1987). The DOC concentration retained within the soil–water system depends on supply and adsorption capacity of the soil (Nodvin et al., 1986). Retention and mobility of DOC are influenced by soil properties such as pH, metal concentrations

(e.g. amorphous Fe and Al oxides) (Kaiser and Guggenberger, 2000; Kaiser et al., 1996), clay and/or soil minerals (Gonzalez and Laird, 2003; Kaiser and Zech, 2000; McDowell, 2003).

Clay can reduce accessibility of the organic matter to soil microbes by sorption and occlusion in aggregates (Nelson et al., 1997). For example, Shi and Marschner (2013) found that subsoil clay addition to sandy soil reduced cumulative respiration and extractable C concentration. Clay-sized particles can bind organic matter via cations and oxides covering the clay surface (Amato and Ladd, 1992; Nelson et al., 1997). Sorption of organic matter to clay is influenced by surface area (Kaiser et al., 1996; Nelson et al., 1997), cation exchange capacity (CEC) (Amato and Ladd, 1992) and clay mineralogy (Benke et al., 1999; Nelson et al., 1997; Ransom et al., 1998). Organic C is preferentially sequestered in smectite-rich sediments compared to clays fraction dominated by chlorite (Ransom et al., 1998). Illite has a lower DOC sorption capacity

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than smectite and kaolinite (Nelson et al., 1997; Ransom et al., 1998) and Benke et al. (1999) showed that C sorption increased in the following order: kaolinite < haematite < goethite. However, organic C sorption also seems to be related to the native organic matter content because there is a negative correlation between DOC retention and organic matter content of subsoil (Jardine et al., 1989).

The previous studies mentioned above focused on the effect of clay particle size, type and content on C retention (Amato and Ladd, 1992; Gonzalez and Laird, 2003; Nelson et al., 1997), but little is known about the link between clay properties and leaching, sorption and desorption of water extractable organic C (WEOC). The aim of this study was to determine if addition of clays isolated from two different soils to a loamy sand affects leaching and sorption of WEOC derived from compost and how this is related to clay properties.

## 2. Materials and methods

### 2.1. Soils

A loamy sand (0–15 cm) was collected from Monarto, South Australia (35°6'S, 139°37'E). The region is semi-arid with a Mediterranean climate. Surface soil (0–15 cm, 51% sand, 15% silt and 34% clay) was collected in Brinkley, South Australia (35°2'S, 139°2'E) and a subsoil (30–50 cm, 18% sand, 20% silt and 62% clay) from the Agricultural Reserve, La Trobe University, Bundoora, Victoria, Australia (37°72'S, 145°05'E). All soils were air-dried and sieved to <2 mm (Table 1).

### 2.2. Clay isolation

To isolate the clay fraction, the soils were dispersed in reverse osmosis water at 1:5 soil: water ratio and shaken for 24 h. Then the suspension was transferred to a bucket and left to settle for 16 h. The <2 µm clay fraction was collected by siphoning off the top 22 cm of the soil suspension. The settling time (16 h) and the height of the soil suspension (22 cm) were selected based on Stokes' law (Jackson, 2005). Then, the isolated clay was dried at 50 °C (Table 1).

**Table 1**

Physical, chemical and mineralogical properties of loamy sand and clays isolated from the loamy sand (native), surface and subsoil clay soils (n = 3).

Properties	Loamy sand	Native clay	Surface clay	Subsoil clay	LSD (P ≤ 0.05)
Particle size (%)					
Sand	84	0	0	0	
Silt	7	4	22	4	
Clay	9	96	78	96	
Electrical conductivity <sub>1:5</sub> (µS cm <sup>-1</sup> )	59	1355	336	363	
pH	7.3	7.4	8.7	5.7	
Water holding capacity (g kg <sup>-1</sup> )	50.1	615.0	685.4	651.5	
Total organic C (g kg <sup>-1</sup> )	2.0 <sup>a</sup>	30.5 <sup>c</sup>	6.3 <sup>b</sup>	7.7 <sup>b</sup>	1.3
Water soluble organic C (mg kg <sup>-1</sup> )	19 <sup>a</sup>	205 <sup>c</sup>	75 <sup>ab</sup>	94 <sup>b</sup>	49
Cation exchange capacity (cmol <sub>c</sub> kg <sup>-1</sup> )	na	25.1 <sup>b</sup>	15.0 <sup>a</sup>	12.9 <sup>a</sup>	5.7
Water extractable Fe (mg kg <sup>-1</sup> )	na	5.6 <sup>a</sup>	33.5 <sup>c</sup>	24.6 <sup>b</sup>	3.0
Water extractable Al (mg kg <sup>-1</sup> )	na	8.4 <sup>a</sup>	37.4 <sup>c</sup>	33.2 <sup>b</sup>	6.0
Surface area (m <sup>2</sup> g <sup>-1</sup> )	na	315	168	322	ns
Total organic C: Surface area (µg m <sup>-2</sup> )	na	97 <sup>b</sup>	38 <sup>a</sup>	24 <sup>a</sup>	37
Exchangeable cation (cmol <sub>c</sub> kg <sup>-1</sup> )					
Ca	na	13.5 <sup>b</sup>	8.1 <sup>a</sup>	4.9 <sup>a</sup>	3.0
Mg	na	4.1	3.2	4.3	ns
Na	na	1.3 <sup>c</sup>	0.6 <sup>a</sup>	0.9 <sup>b</sup>	0.2
K	na	7.4 <sup>b</sup>	3.6 <sup>a</sup>	3.8 <sup>a</sup>	1.9
Mineralogy (% wt) of <2 µm clay fraction <sup>1</sup>					
Illite		45–50	15–20	20–25	
Kaolinite		25–30	7–12	35–40	
Smectite		<5	5–10	2–5	

<sup>1</sup> Dominant minerals. na is not applicable. LSD is least significant difference at P ≤ 0.05. ns is not significant. Within rows, means followed by the same letter are not significantly different (P > 0.05).

### 2.3. Water extractable organic carbon

Water extractable organic matter was extracted from fine grade garden compost by shaking 1 g of compost with 6 ml reverse osmosis water overnight at 4 °C. The suspension was centrifuged at 14758 ×g for 10 min and filtered (#42 Whatman™) and then stored at –20 °C until further use. We use the term water-extractable organic carbon (WEOC) because some of the C in the water extract may be colloidal, not dissolved. The organic C concentration of the extract was 1023 mg l<sup>-1</sup>. For experiments, the WEOC stock was thawed at room temperature and then diluted before being immediately added to the soils or isolated clays. The WEOC concentration in all extracts was measured as described below.

### 2.4. Experimental design

This study included two experiments. An experiment in which WEOC was added to soil cores filled with loamy sand alone or loamy sand with isolated clays. WEOC in the leachate and soil respiration were measured. In the second experiment, sorption of WEOC to isolated clays was determined in a batch sorption experiment.

#### 2.4.1. WEOC leaching experiment

There were three soil treatments: unamended loamy sand (native) or amended with the clay isolated from the surface soil (surface) or the subsoil (subsoil) at a rate of 98.1 g kg soil<sup>-1</sup> to increase the clay content from 9 to 18% assuming that the isolated fraction contained only clay. However, particle size analysis of these fractions showed that the clay content was 78% and 96% for the surface and subsoil clay, respectively (Table 1). Therefore the actual clay content of treatments was 90, 158 and 175 g clay kg<sup>-1</sup> soil for the unamended loamy sand (native) and soil amended with clays isolated from surface and subsoil, respectively. Reverse osmosis water was added to reach 60% of water holding capacity. This water content was chosen because it had been shown to result in maximal soil respiration in soils with 10–20% clay (Setia et al., 2011a). The moist soils were pre-incubated for two weeks at 25 °C to reactivate the microbes. Throughout the pre-incubation and the subsequent incubation period, water was added on mass basis to maintain 60% of water holding capacity.

The pre-incubated soils (equivalent to 30 g dry soil) were filled to polyvinyl cores (PVC, 3.7 cm width and 5.0 cm height) with a nylon mesh base (0.75 µm, Australian Filter Specialist) and packed to a bulk density of 1.22 g cm<sup>-3</sup>. The mesh was covered with filter paper (#42 Whatman™) to minimize loss of clay-sized particles during leaching. The cores were placed vertically in plastic funnels with the mesh facing down. 30 ml each of the six water extractable organic C (WEOC<sub>add</sub>) concentrations was added to the cores (0, 252, 494, 712, 865 and 1023 mg C l<sup>-1</sup> or per unit soil weight 0, 252, 494, 712, 865 and 1023 mg C kg soil<sup>-1</sup>) which were then kept overnight at 4 °C to collect the leachate (with three replicates per WEOC treatment and soil). This range was chosen because a preliminary experiment showed that the maximum WEOC sorption of the loamy sand was about 800 mg C kg<sup>-1</sup> soil. The leachate was collected and analysed for WEOC (WEOC<sub>leachate</sub>) as described below. The volume of the leachate was 22 ml for all treatments.

**2.4.1.1. Calculations.** The total organic C (TOC<sub>total</sub>) of the soil treatments is the sum of indigenous TOC of the soils or clays and the different concentrations of WEOC added (Eq. (1)): (WEOC<sub>add</sub>).

$$\text{TOC}_{\text{total}} = \text{TOC} + \sum_{i=0}^n \text{WEOC}_{\text{add}} \quad (1)$$

where, i–n are the different added WEOC concentrations (g C kg<sup>-1</sup>).

The amount of WEOC leached (WEOC<sub>leach</sub>) from WEOC added was calculated by the difference between WEOC in the leachate after addition of the compost extracts (WEOC<sub>com</sub>) (252, 494, 712, 865 and

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