



## Evaluation of potentially labile soil organic carbon and nitrogen fractionation procedures

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### ABSTRACT

Particulate organic matter (POM) and light fraction (LF) organic matter are potentially labile (active) fractions of soil organic matter (SOM) that have been shown to be indicators of short-term changes in soil management practices (e.g. tillage, manure and fertilizer applications, and crop rotation). These two fractions consist mainly of partially decomposed plant residues, microbial residues, seeds, and spores forming organo-mineral complexes with soil mineral particles; however, they cannot be used as synonyms because of their different chemical composition and structure. Particulate-OM is recovered by size-based procedures while LF is generally recovered in two distinct fractions [free-LF (FLF) and occluded-LF (OLF)] using density-based solutions in conjunction with soil-aggregate disruption. Solutions used in these density-based separations have most commonly varied in density from 1.6 to 2.0 g cm<sup>-3</sup>. Sodium iodide (NaI) and sodium polytungstate (SPT) are the chemicals most often used to prepare the density solutions in LF recovery but comparisons of the effectiveness of two solutions have not been conducted. The objectives of this research were: (1) compare the efficiency of similar density solutions of NaI and SPT in recovering FLF; and (2) compare POM, FLF, and OLF as possible sensitive indices of short-term soil changes due to tillage management. Soil samples were collected at 0–15 cm depth from a cropping system experiment conducted on a silt loam Ultisol. Plots selected for sampling had received either reduced till (RT) or no-till (NT), and cropping was continuous corn silage for a period of 3 years prior to sampling. Solutions of NaI and SPT at densities of 1.6 and 1.8 g cm<sup>-3</sup> were used to recover FLF, and OLF was recovered with SPT solution at a density of 2.0 g cm<sup>-3</sup> from the soil pellet remaining after FLF recovery with SPT 1.6 g cm<sup>-3</sup>. The average total soil organic carbon (SOC) content of these samples was of 12.7 g kg<sup>-1</sup>, and carbon-POM (C-POM), carbon-FLF (C-FLF), and carbon-OLF (C-OLF) represented 22.4, 5.5, and 5.2% of it, respectively. In general, C-FLF and nitrogen-FLF (N-FLF) contents recovered did not differ significantly between chemical solutions (NaI or SPT) adjusted to the same density (1.6 or 1.8 g cm<sup>-3</sup>). Increasing the density within a specific solution (NaI or SPT) resulted in significantly higher C-FLF and N-FLF recovery. For instance, C-FLF recovery averaged 637 and 954 mg kg<sup>-1</sup> at 1.6 and 1.8 g cm<sup>-3</sup>, respectively. For both chemicals increasing density from 1.6 to 1.8 g cm<sup>-3</sup> reduced the variability in recovering C-FLF and N-FLF with coefficient of variation values decreasing from a range of 14.9–19.1% for densities of 1.6 g cm<sup>-3</sup> to 6.7–10.4% when densities increased to 1.8 g cm<sup>-3</sup>. In the present work, POM and OLF were more sensitive than FLF to changes in tillage management, with significantly greater amounts of the sensitive fractions in RT samples. A better sensitivity of FLF would be expected if treatments dealing with residue input (e.g. crop rotation and cover crop) were evaluated.

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### 1. Introduction

Soil organic matter (SOM) consists of functional pools differing in their turnover rate. Depending on the authors, the pools are

termed as labile or active, slow or intermediate, and recalcitrant, passive, stable, or inert (von Lütow et al., 2007; Wander, 2004). Typically, soil fractions belonging to the labile pool present turnover rates of days to a few years, while for fractions in intermediate and passive pools turnover rates range from a few years to centuries (Stevenson, 1994; Wander, 2004). Soil fractions consisting largely of labile SOM are recognized as potential indicators of soil quality changes in the short-term due to variation in management practices (e.g. tillage, fertilizer and manure applications, and crop

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rotation). Particulate organic matter (POM) and light fraction (LF) are important soil fractions that, depending on the system evaluated (e.g. soil type and quality of residue input), to a large extent consist of labile or active SOM, and are potentially sensitive indicators to changes in soil management practices (Marriott and Wander, 2006b; Sharifi et al., 2008; Yoo and Wander, 2008). Assuming that sorption is an important mechanism in SOM stabilization (formation of organo-mineral complexes), soil fractions within the sand fraction are allocated to the labile pool and soil fractions in silt and clay fractions to the intermediate and passive pools (von Lützow et al., 2007). These fractions consist mainly of partially decomposed plant residues, microbial residues, seeds, and spores forming organo-mineral complexes with soil mineral particles (Baisden et al., 2002; Gregorich et al., 2006). Despite of their similarity, POM and LF contain different proportions of carbohydrates and other aliphatic compounds, which precludes their use as synonyms (Gregorich et al., 2006).

The POM and LF fractions have been associated with changes in the mineralizable organic nitrogen (N) pool in soils. Sharifi et al. (2008) found that carbon-POM (C-POM) was one of the most sensitive indices to changes in the intermediate mineralizable N pool due to tillage for loam, silt loam, and clay loam soils in Canada. In addition, C-POM and nitrogen-POM (N-POM) had significant but low relationships with potentially mineralizable N ( $N_0$ ) for soils classified as Spodosols, Mollisols, Alfisols, Inceptisols, and Entisols coming from Canada and USA (Sharifi et al., 2007). This suggested varied suitability of these fractions for assessing  $N_0$  from SOM and that the variability is associated with soil, management, and climate characteristics.

Gregorich et al. (2006) defined LF as the organic fraction recovered by density fractionation and POM as the organic fraction recovered by size fractionation alone, or by the combination of size and density fractionation procedures. In this paper, Gregorich et al.'s (2006) nomenclature will be used. In the size-based procedure, the soil sample is dispersed in a sodium hexametaphosphate [(NaPO<sub>3</sub>)<sub>6</sub>] solution, and the organic material retained on a 53  $\mu\text{m}$  sieve is operationally defined as POM (Cambardella and Elliott, 1992; Gregorich and Beare, 2008).

The density-based procedure recovers the LF using solutions adjusted to different densities, with a common range between 1.6 and 2.0  $\text{g cm}^{-3}$  (Gregorich et al., 2006; Wander, 2004). The LF is generally recovered in two distinct pools as either free-LF (FLF) or occluded-LF (OLF) in a two-step fractionation. In the first step the soil sample is gently shaken with a density solution and the supernatant is filtered to obtain FLF. In the second step the remaining pellet from the first step is shaken with more energy with a density solution at the same density used in the previous step, or greater, and the supernatant is filtered to obtain OLF. The greater energy applied in the second step is designed to break stable soil-aggregates releasing OLF. The FLF organic matter exists outside of soil aggregates, or is associated with weak aggregates, while OLF organic matter is associated with stable aggregates (Bird et al., 2008). The FLF is considered a more labile fraction of SOM than OLF because it is more closely associated with plants (chemical composition and structure) and exists external to the aggregate; however, OLF is generally considered decomposable when it is released from the aggregate, making the degree of protection to be aggregate-turnover dependent (Gulde et al., 2008).

Sodium iodide (NaI) and sodium polytungstate (SPT) [Na<sub>6</sub>(H<sub>2</sub>W<sub>12</sub>O<sub>40</sub>)] are the most commonly used chemicals to prepare solutions employed to recover FLF and OLF. Sodium polytungstate has become the most used chemical for certain research groups due to lower toxicity and capacity to achieve densities up to 3.1  $\text{g cm}^{-3}$  (Shang and Tiessen, 2001; Six et al., 1999). Sodium iodide is still used for density fractionation in many studies, but human

health issues through chronic exposure (iodism) is a concern, and the highest density achievable with NaI is approximately 1.85  $\text{g cm}^{-3}$  at 25 °C (MSDS, 2009). These two issues are distinct disadvantages for using NaI. However, NaI is significantly less expensive than SPT. These density solutions have been employed in various procedures with both being employed to recover FLF and OLF. In one method the pellet left in the bottle after FLF recovery is resuspended with the same density solution and higher disaggregation energy is used to disperse soil aggregates (Kölbl and Kögel-Knabner, 2004; Sohi et al., 2001). This sequential recovery of FLF and OLF has also been achieved by the combination of higher disaggregation energy and using the same solution at a higher density to recover OLF after FLF recovery (Bird et al., 2002; Gulde et al., 2008; Marriott and Wander, 2006a; Wander and Yang, 2000).

The use of determined density solution for recovering LF organic matter seems to be based on: (1) cost, since NaI is less expensive than SPT; (2) human health concerns, since SPT has lower toxicity than NaI; and (3) the range of densities necessary for specific experiments since these chemicals have different maximum achievable densities. Apparently there has not much concern if a certain chemical-density solution is more efficient than another in extracting LF. Data have been compared across studies for these chemical-density solutions, when adjusted to the same density, which assumes they have the same efficiency in recovering LF. Such an assumption ignores possible solution pH effect on organic substances solubility, different buffering potentials of different density solutions as well as numerous other factors associated with SOM reactions. Data are not available to show that the NaI and SPT solutions at the same density do recover similar amounts of LF SOM.

The objectives of this research were: 1) compare the efficiency of NaI and SPT density solutions when adjusted to equal densities in recovering FLF organic matter; and 2) compare POM, FLF, and OLF organic fractions as possible sensitive indices of short-term changes in soil organic carbon (SOC) and soil organic nitrogen (SON) in response to tillage management.

## 2. Materials and methods

Soil samples were collected in July 2008 from a cropping system experiment in the Valley region of Virginia initiated in the spring of 2006 on an Ultisol developed from limestone parent material. The experiment was a split-split-plot design with crop rotation as whole-plot treatment factor, tillage as sub-plot treatment factor, cover crop management as sub-sub-plot treatment factor. The experiment had three replications (blocks). Each plot was 9 m by 37 m. Crop rotation had three levels (continuous corn, corn/alfalfa, and corn/soybean/corn), tillage had two levels (reduced till (RT) and no-till (NT)), and cover crop management had three levels (remaining on the field after killed, removed from the field, and no cover crop planted). The RT management consisted of one pass disc harrow and one pass of field cultivator before planting at approximately 10 cm depth. In this study, only soil samples from plots in RT and NT with continuous corn and with no cover crop planted were sampled, resulting in a total of six plots sampled. Thus, only the effect of tillage management on C and N fractions was evaluated. Fifteen soil cores (3 cm diameter soil probe) were collected at 0–15 cm depth from each selected plot and mixed to form a composite sample. Soil samples were transferred to the lab within 8 h of collection and rapidly air-dried.

### 2.1. Objective 1: comparisons of NaI and SPT density solutions for recovery of FLF

The FLF was recovered from each composite sample in triplicate using NaI density solutions adjusted to 1.6 and 1.8  $\text{g cm}^{-3}$ , and by

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