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Organic matter contained in soil aggregates from a tropical chronosequence: correction for sand and light fraction

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ABSTRACT

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A cultivation sequence of Ultisols (sandy loam) from the Amazon Basin of Peru was physically fractionated using wet sieving followed by heavy liquid separation and total organic C and N analysis. Soils were sampled to an equivalent plow depth (22.5 cm) in a cultivation chronosequence containing the following treatments: an old growth forest (75 plus years), a 13-year fallowed forest, a 3-year continuous cultivation corn–soybean rotation following a 10-year forest fallow and a 16-year intensively managed corn field. The soil was air dried and re-wetted (misted) to field capacity before shaking for 10 min and wet sieving into five size classes: <0.02, 0.02–0.05, 0.05–0.212, 0.212–1.0 and >1.0 mm. The weight of soil, total organic C and N, and percent sand was determined on each size class. Subsamples of each size class were gently stirred in sodium metatungstate (specific gravity = 1.8), left overnight and the floating material removed by aspiration. The removed material was filtered through pre-ashed glass fiber filters and analyzed for total organic C and N.

The sand content of size classes ranged from 45 to 95%. Light fraction C comprised 24, 28, 14, 5 and 2% of the total C of the size classes ranging from the largest to smallest particle sizes. Total C concentrations were highest in the largest and smallest particle size classes. Within any size class, all treatments had similar C concentration except for the <0.02-mm particles where the 75-year forest had lower concentrations. When corrected for sand content (i.e., expressed on a silt plus clay basis) and with light fraction C subtracted, the organic C content was, with few exceptions, not different among size classes or treatments. However, when all treatments within a size class were averaged, the 0.020–0.053-mm class had a higher concentration of C.

We applied methods that allowed correction of soil organic matter in aggregate size classes and suggest the importance of such corrections when interpreting aggregate organic matter data. Trends in organic matter concentrations were quite different when fractions were corrected for light fraction and sand content compared to when they were not corrected.

INTRODUCTION

As litter is processed it becomes increasingly associated with the mineral portion of the soil. For the purpose of studying soil organic matter, it is often

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useful to distinguish mineral-associated from free particulate organic matter (Turchenek and Oades, 1979; Sollins et al., 1984). Most free soil organic matter is usually undecomposed debris that floats in heavy liquids and is referred to as light fraction. Light fraction has been variously defined as material floating at densities ranging from 1.0 to 1.8 g cm^{-3} (Elliott and Cambardella, 1991). When studying soil organic matter associated with soil aggregates, this distinction becomes especially important because undecomposed particles may be associated with a particular size class of aggregates as a result of their size and are not actually contained within aggregates. However, these particles of litter contribute to the organic matter content of the aggregates if the litter particles are not separated from the aggregates before chemical analysis. Without disruption of aggregates before analysis, intra-aggregate particulate organic matter is considered to be part of the organic matter associated with aggregates. The light fraction contributes proportionately more C than N compared to soil organic matter and may affect the measured C/N of the fractions. If light fraction material constitutes a significant proportion of the total organic matter included with a size class, but not held within aggregates, information on the organic matter concentration in the fraction can be misleading.

The sand content of aggregate size fractions may also vary (Elliott, 1986; Dormaar, 1983). Many aggregate size classes have the same size range as sand (0.05–2.0 mm). Similar to the light fraction, sand may be contained in a particular size class, but not within aggregates. Since sand cannot contain organic matter the organic matter content of aggregate fractions may be diluted by the sand and make it difficult to compare organic matter contents of different aggregate size classes. This problem is compounded where the sand is well sorted because it may dominate a particular size class. However, some sand is coated with organic matter and sand may contribute to the dynamics of soil organic matter in this manner.

To make an appropriate comparison of mineral-associated soil organic matter concentrations in different size classes of aggregates, we suggest that it is necessary to correct for the differing amounts of sand and undecomposed particulate material in the different size classes of aggregates. What follows is a description of a method we used to make these corrections for Amazonian Ultisols and how the corrections influenced our interpretation of the results.

MATERIALS AND METHODS

Sampling

Soils were sampled from a sandy loam Ultisol in the Amazon Basin of Peru near Yurimaguas. The four treatments consisted of a 75-year or older forest, a 13-year fallow forest, a 3-year continuously cropped corn–soybean field fol-

lowing a 10-year forest fallow and a 16-year continuously cropped corn–soybean field. Three replicate 7-cm diameter intact cores were taken and kept intact during shipping. All treatments were sampled to the same depth (22.5 cm) which was the plow depth of the most deeply plowed treatment (16-year corn–soybean field). It is crucial for studies of this type (comparison of organic matter contents of different agricultural management treatments) that the depth of the most deeply plowed treatment be used as the basis for sampling the other treatments because of the soil mixing and dilution of surface with subsoils in plowed treatments.

In most undisturbed natural soils, organic matter contents decrease with depth. Upon plowing, the lower organic matter concentration of the deeper soil is mixed with the organic matter-rich surface soils. Therefore, the depth of plowing will determine the initial concentration of organic matter in the plow layer. If one were to sample the top 5 cm of a field newly plowed out of forest and compare the organic matter concentration to that of the top 5 cm of an adjacent unplowed forest, the forest values would almost certainly be higher, not because of losses due to cultivation, but simply because of dilution of topsoil with subsoil.

Physical fractionation

The physical fractionation scheme we used to separate soil organic fractions is shown in Fig. 1. The samples were shipped in a field moist state, although some drying occurred en route. Samples were carefully hand crumbled to pass an 8-mm sieve. Large roots and litter fragments were removed at this time. After air drying, samples were mixed and subsamples taken for the determination of total organic matter C and N. One hundred-gram subsamples were taken from each of the three field replicate cores per treatment and placed in aluminum pans. The pans were placed in a mist chamber (see Kemper and Rosenau, 1986, for details of misting), slowly wetted to field capacity and held under refrigeration overnight.

Estimates of field capacity to be used for mist wetting were determined by filling a 50-ml graduated cylinder with soil to the 50-ml mark with gentle tapping. Cylinders were weighed with and without soil to determine bulk density. The field capacity was roughly estimated based on the soil texture and one-half of the required water to bring soil to field capacity was very slowly added to the surface of the soil in the cylinder. The cylinders were covered and the water allowed to equilibrate for 24 h. By definition, the soil wetted after this period of time is at field capacity. The depth of the wetting front was determined and the volumetric water content calculated. This was converted to a gravimetric water content based upon the amount of soil held in the 50-ml volume (bulk density).

To facilitate a moderate degree of disruption, the misted soils were gently

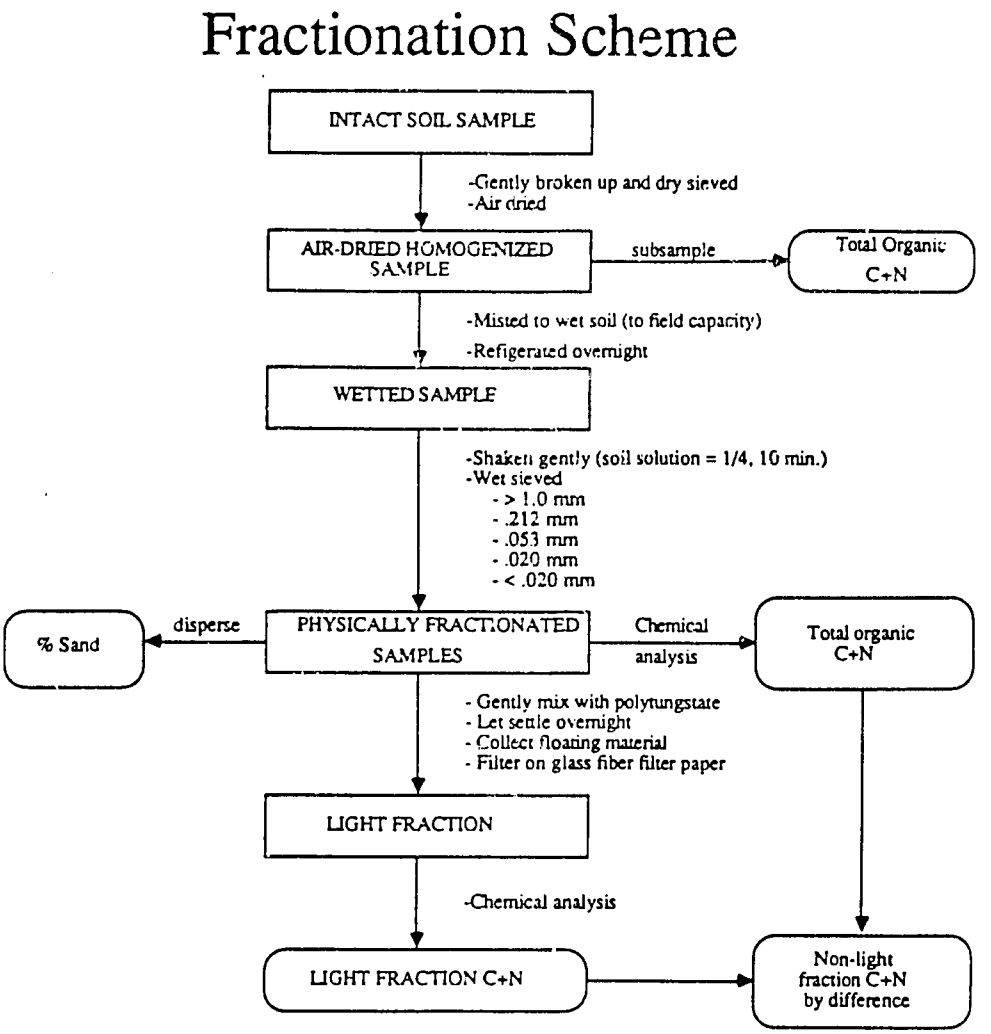


Fig. 1. This fractionation scheme was used to obtain soil organic C and N concentrations in soil aggregate fractions obtained from Peruvian Ultisols sampled from a cultivation chronosequence in the Amazon River basin. The scheme allowed for correction of variable amounts of light fraction and sand in the different aggregate size classes which, we feel, was necessary to obtain meaningful data.

shaken at a speed of 100 cycles min^{-1} on a reciprocal shaker with a soil solution ratio of 4:1 for 10 min before wet sieving by hand (see Elliott and Cambardella, 1991, for sieving method). Soils were separated into five size classes: > 1.0, 0.212–1.0, 0.053–0.212, 0.020–0.053 and < 0.02 mm. During the sieving process, large pieces of floating organic debris were removed from each sieve with a polypropylene sponge. Sieve contents were transferred to aluminum pans and dried in a forced air oven at 60°C.

The sand content of the physically fractionated samples was determined by dispersing 5 g of the fraction in 20 ml of sodium hexametaphosphate and sieving through a 0.053-mm screen for all size classes except for the 0.020–0.053-mm size where a 0.020-mm screen was used. Total organic C was determined on each fraction by the method of Snyder and Trofymow (1984) and total organic N by Kjeldahl digestion (Nelson and Sommers, 1980) and autoanalysis.

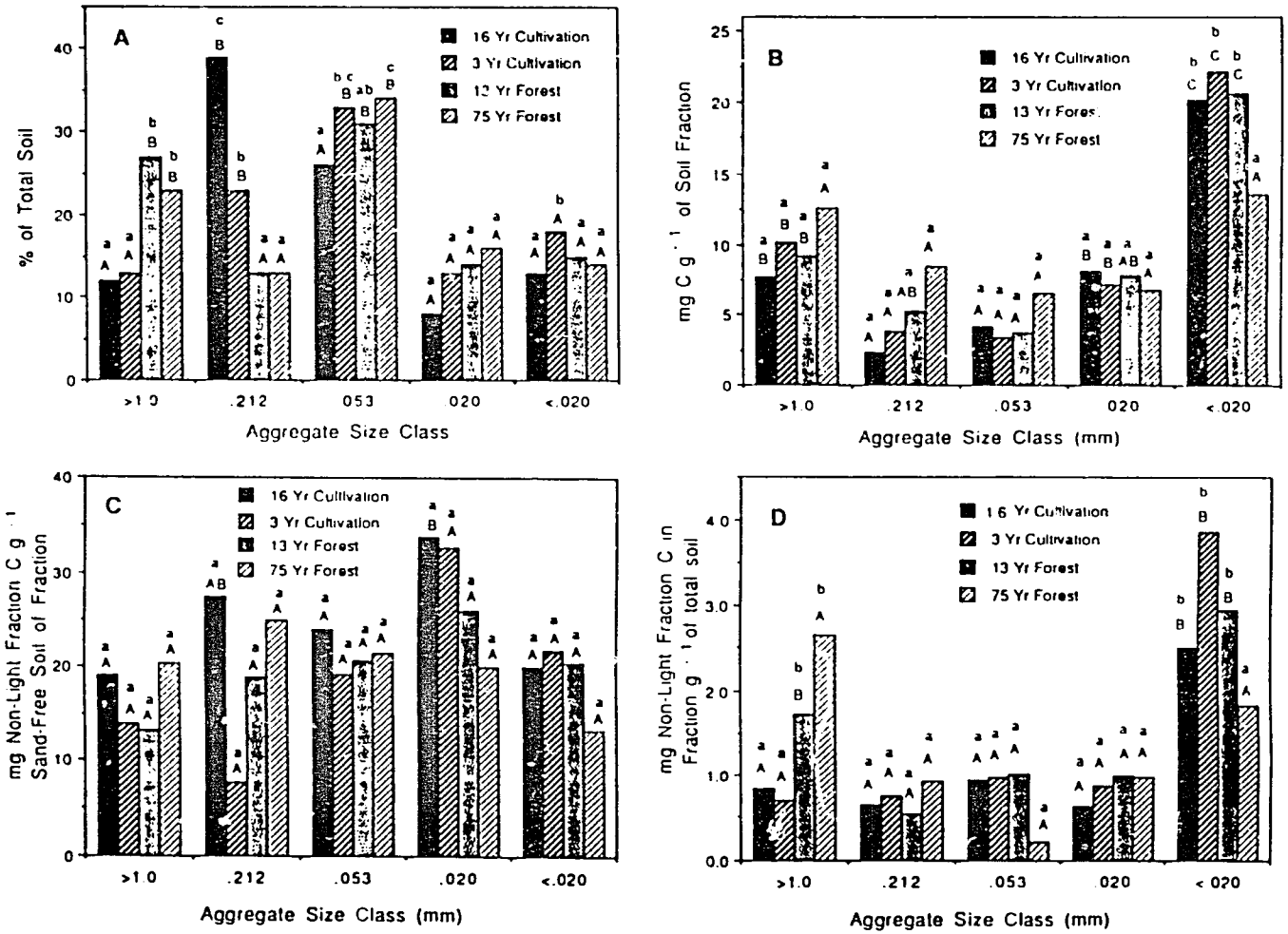
Light fraction C and N in the soil fractions was determined using sodium metatungstate, as suggested by Oades (1988). This compound has several advantages over other heavy liquids. A wide range of densities can be obtained (>3.0 g cm^{-3}), it is inert and non-toxic (as far as has been determined), and it does not interfere with the C analysis method of Snyder and Trofymow (1984) as halogen salts do. It also does not become oxidized with time or use, does not appear to accumulate soluble organic compounds (at least for the soils we have tried) and it can be recovered ($\sim 80\%$) and reused.

Soil from the fraction (0.75 g) was gently stirred (one stroke with a rubber policeman to minimize disruption) with 15 ml of 1.8 g cm^{-3} sodium metatungstate and left to settle overnight. The floating material was removed by aspiration. The heavy liquid containing the light fraction was filtered through pre-ashed (525°C) glass fiber filters and digested for C or N with the above described methods, separate samples being floated for C and N analyses. Depending upon the amount of light fraction retained on the filter, it was cut up into several pieces (up to five), each of which was digested separately. In these Peruvian Ultisols, there were size fractions in some samples that were composed of up to 50% light fraction organic C.

The amount of C or N contained in the light fraction per gram of soil fraction was subtracted from the total soil C or N to calculate the non-light fraction C and N in a gram of soil fraction. Likewise, sand contents were used to correct soils to a sand-free (silt plus clay) basis. These calculations were used to determine the non-light fraction sand-free soil organic C and N in each fraction. Stated in another way, these calculations allowed us to determine the silt-plus-clay-associated soil organic matter.

We did not measure the weight of light fraction material held on the filter because it was contaminated with the heavy liquid. Therefore, we were not able to determine the actual C or N concentration of the light fraction material (although we were able to calculate its C/N ratio). We were not able to devise a way to determine the amount of heavy liquid contaminating the light fraction material. However, our main interest was in determining the proportion of total soil C or N that was in the light fraction, which we were able to do with the above method.

One-way analyses of variance were run, either comparing size classes within each treatment or across treatments within a size class. A two-way analysis of


A
% of Total Soil
16 Yr Cultivation
3 Yr Cultivation
12 Yr Forest
75 Yr Forest
>1.0
.212
.053
.020
<.020
Aggregate Size Class
B
mg C g⁻¹ of Soil Fraction
16 Yr Cultivation
3 Yr Cultivation
13 Yr Forest
75 Yr Forest
Aggregate Size Class (mm)
C
mg Non-Light Fraction C g⁻¹ Sand-Free Soil of Fraction
16 Yr Cultivation
3 Yr Cultivation
13 Yr Forest
75 Yr Forest
Aggregate Size Class (mm)
D
mg Non-Light Fraction C in Fraction g⁻¹ of total soil
16 Yr Cultivation
3 Yr Cultivation
13 Yr Forest
75 Yr Forest
Aggregate Size Class (mm)

variance was not run with treatments by sizes because we were not interested in determining if different size classes of different treatments were different from each other. The Newman–Keuls multiple comparison test was used to determine significant differences among means at $P \leq 0.05$.

RESULTS AND DISCUSSION

The concentration of organic C was 7.6, 8.4, 7.8 and 10.2 mg C g^{-1} soil for the 16-year cropped, 3-year cropped, 13-year forest and old growth forest, respectively. However, when the light fraction C was subtracted from the total organic C concentrations the values were 5.5, 6.8, 7.2 and 8.5 mg C g^{-1} soil; a more predictable pattern considering the cultivation chronosequence. Similarly, non-light fraction (mineral-associated) N concentrations were 466, 679, 719 and 772 $\mu g\ g^{-1}$ soil for the above chronosequence.

There was more soil in the largest aggregate size class for the forested treatments than for the cultivated treatments with the reverse being true for the second largest aggregate size class (Fig. 2a). In general, ~80% of the soil was in size classes >0.053 mm. The sand content of size classes ranged from 45 to 95%. Carbon concentrations were highest in the largest and smallest particle size classes (Fig. 2b). Within any size class, all treatments had similar C concentrations, except for the <0.02-mm particles where the 75-year forest had lower concentrations (Fig. 2b). Light fraction C comprised 24, 28, 14, 5 and 2% (averaging across treatments) of the total C of the size classes ranging from the largest to smallest particle sizes. Photomicrographs of the filter paper containing the light fraction showed that a high proportion, perhaps half, of the light fraction material was charcoal, probably the product of previous slash and burn agriculture. There were no significant differences among treatments for the light fraction. When corrected for sand content and with light fraction C subtracted, the organic C content was, with few exceptions, not different among size classes or treatments and averaged ~20 mg non-light fraction C g^{-1} sand-free soil across all fractions (Fig. 2c). However, when all treatments within a size class were averaged, the 0.020–0.053-mm size class

Fig. 2. The amount of soil in each size class is shown in Frame A, the concentration of total organic C in Frame B, the non-light fraction C per gram of sand-free soil (silt-plus-clay-associated C) in Frame C and the milligrams of non-light fraction C held in each size fraction per gram of original unfractionated soil in Frame D. Lower case letters above the bars allow the statistical comparison of treatments within a size class and upper case letters allow comparison within a treatment across size classes; different letters denote significant differences between means at $P \leq 0.05$. Correction of aggregate size class organic matter concentrations for sand and light fraction reveals that the clay- and silt-associated organic matter concentrations do not vary much among treatments or size classes (Frame C). The largest proportion of soil C resides in the smallest particle size class in the two cultivated soils, in the largest and smallest size classes in the 13-year forest, and is equally distributed in all size classes in the old forest (Frame D).

had a significantly ($P \leq 0.07$) higher concentration of silt-plus-clay-associated organic matter.

Another perspective from which to view these data is to consider the total amount of light fraction-free C contained in each size class that was contained originally in a gram of the unfractionated soil. These values were obtained by multiplying the light fraction-free C concentrations by the amount of soil in each size class. When the amount of this C in each size class is summed it equals the total amount of non-light fraction C originally contained in the soil. Although this calculation includes the summated errors made for each of the determinations, it still shows some statistically significant ($P \leq 0.05$) trends (Fig. 2d). The highest proportion of C resides in the smallest size class, except for the forest treatments where there is close to an equal amount in the largest size class (Fig. 2d). Less of the total mineral-associated C resides in the largest size class of aggregates in the cultivated than forested treatments.

Soil organic N showed similar trends as C, except for uncorrected total organic values. This is because the light fraction was considerably enriched in C relative to N. When corrected for light fraction C and N, organic C and N behaved similarly. Soil C/N (corrected for light fraction material) ranged from 10 to 14 and there were essentially no significant differences among treatments or sizes. Light fraction C/N ranged from 15 to 60 with no apparent trends.

CONCLUSIONS

We draw the following tentative conclusions based upon the physical fractionation and chemical analysis obtained from the Ultisol cultivation chronosequence. Contrary to what we see for temperate soils developed under grasslands, where macroaggregates have higher organic matter concentrations than microaggregates, the only difference in the concentration of clay- and silt-associated organic matter of different size classes of soil was in the 0.020–0.053-mm size. This size class of organic matter could be the remains of archaic microbial communities causing local microsites of organic C enrichment. Upon cultivation, there are shifts in the size distribution of aggregates to smaller sizes, but the organic matter concentration remains constant across most size classes. With reversion to forest, the organic matter seems to shift from the smallest silt and clay size fraction to the largest (> 1.0 mm) aggregates. The quality (C/N) of the soil organic matter also did not differ across size classes, as it does in most temperate soils. However, there have been few other instances where light fraction corrections have been made on organic matter contents of particle size classes. It was necessary to correct for sand and light fraction contents of the aggregate size fractions to obtain meaningful data.

Information on the mineralization potential of the different aggregate size

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classes obtained from the different treatments would be useful as another determinant of soil organic matter quality. We do not know if charcoal behaves differently from the rest of the light fraction material, but suspect that it has a much longer residence time in the soil and may adsorb P and soluble organic matter.

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