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A Modified Method of Weight Loss on Ignition to Evaluate Soil Organic Matter Fractions

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Abstract: Chemical fractionation and quantification of Soil Organic Matter (SOM) pools involve laborious and time-consuming processes, which are also not very accurate. Therefore an attempt was made here to quantify the Fulvic Fraction (FF), Humic Fraction (HF) and soil litter which are known to be very important in nutrient availability in tropical ecosystems, by using weight Loss on Ignition (LOI) method. Cambisols in Sri Lanka was used to extract the humic substances and soil litter. The result indicated that the soil litter combusted at temperatures 170-190°C. Peak weight losses of the FF occurred between 200-250°C. The weight losses of the HF occurred at 500°C. The study clearly showed that soil litter combusted at temperatures <190°C whereas FF and HF showed weight loss signals at temperatures >190°C. Therefore the LOI can be used as a quick and simple technique for the determination of soil litter, in particular and further the FF and HF, compared to conventional extraction methods. This will be very much useful in comparative studies where there are large numbers of samples to be analysed.

Key words: Soil organic matter, humic fraction, fulvic fraction, soil litter, weight loss on ignition

INTRODUCTION

There are several methods for physical and chemical fractionations and quantifications of the SOM pools. Among these, density fractionation (Christensen, 1992) and wet chemical oxidation (Baker, 1976) have been reported to be common methods used in the tropics. Humic substances are the most widespread natural organic materials occurring in soils (Allard, 2006). Fulvic and humic acids generally represent the important fraction of humic substances and display very complex chemical structures (McDonnell *et al.*, 2001).

There are number of methods for the extraction of FF and HF from soil. The method developed by the International Humic Substance Society (IHSS) is an acceptable method for the extraction of humic substances from soils. However, this contains certain errors (Swift, 1996). Technically, fractions separated in this way contain significant amounts of impurities specifically unbound polysaccharides etc. Therefore, extraction and quantification of FF and HF is a difficult task.

Separation and quantification of soil litter is also a laborious and time-consuming process (Smcker *et al.*, 1982). Litter separation in high clay content soils involve overnight soaking in 10% sodium hexametaphosphate to disperse the soil particles (Smcker` *et al.*, 1982). Soil litter quantification in a large number of samples is a difficult task. Therefore, an attempt was made here to quantify the SOM fractions (FF and HF) and soil litter by using the LOI method.

The LOI measures weight loss of the SOM fractions due to oxidation upon heating. In this procedure, the SOM is oxidized from moderate (150°C) to high (550°C) temperatures, with the weight loss being proportional to the amount of SOM in the sample (Konen *et al.*, 2002). The LOI analysis thus allows establishing weight loss signals for quantifying the SOM. Soils with different quantities

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of SOM fractions produce weight loss signals of different intensities (Siewert, 2004). The variations among these profiles allow the quantitative assessment of the SOM fractions.

Recent studies by several authors indicated that the LOI is an accurate method for estimating soil organic carbon and hence the SOM. The LOI method is much simpler, requires less labour and allows the carbon content of an entire sample to be measured without grinding, mixing, or sub-sampling (Kaspar *et al.*, 2000). Advantages of this method include the large sample numbers that can be run simultaneously and accurately (Konen *et al.*, 2002). Kaspar *et al.* (2000) were able to evaluate the spatial variability of soil carbon accurately by the LOI method in an agricultural field having very large variations and detection was found to be extremely difficult by other methods.

The LOI method was used in above studies to determine the total SOM. However in the present study an attempt was made to use the LOI method to quantify separate SOM fractions. Thus, it was hypothesized here that the LOI can be used to quantify separate organic matter fractions and soil litter.

MATERIALS AND METHODS

Soils were sampled in January 2005 from an undisturbed forest at 700 m above sea level belonging to tropical wet evergreen type in Sri Lanka (5°54' N - 9°52' N latitudes and 79° 39' E - 81° 53' E longitudes). Five composite soil samples (Cambisols) were collected at the depth of 30 cm and used for the extraction of humic substances and soil litter.

Extraction of Humic Substances

The soil was extracted for FF and HF using International Humic Substances Society method (IHSS) (Swift, 1996). The soil sample was shaken for 1 h with 0.1 M HCl (solution to soil ratio 10 L kg^{-1}) to eliminate carbonates and alkaline-earth metals. The supernatant was separated by decanting (FF extract 1). The residue was neutralized using a solution of 0.1M NaOH (final extractant to soil ratio 10 L kg^{-1}). The suspension was then shaken for 4 h and allowed to stand for 12 h. After centrifugation and decanting, humic substances were precipitated from the supernatant by acidification with 6M HCl to pH 1. Then humic (precipitate) and fulvic (supernatant: FF extract 2) fractions were separated by centrifugation. Several purification steps were carried out with 0.1M HCl and 0.3M hydrofluoric acid to reduce the amounts of inorganic impurities of the precipitate, which was then taken as the HF (Swift, 1996). The FF extract 1 and 2 were combined. The extract was then dialyzed against spectrapor membrane (1000 Dalton) to remove salts and finally freeze dried to obtain the FF.

Extraction of Soil Litter

Soil was also extracted to obtain the soil litter using the method described by Smucker *et al.* (1982). The soil sample was agitated in water to separate soil particles and soil litter particles. Organic matter in suspension was carefully decanted onto a 0.25 mm sieve. Soil litter materials collected on the sieve were collected and washed with distilled water. Then it was oven dried at 85°C.

Extracted SOM fractions (FF, HF) and soil litter were subjected to sequential thermal oxidation from 105 to 600°C using a muffle furnace (Gallenkamp-box furnace F SL-340-0160). Equivalent volumes (approximately 2.5 g) of those fractions were placed in 15 mL crucibles. Samples were oven dried at 105°C overnight, cooled in desiccators and weighed. Weight loss of the fractions at temperature steps of 50°C for 4 h was recorded. This allowed for identifying critical ranges of temperatures oxidizing the SOM fractions. The SOM fractions were quantified in terms of the LOI as follows.

$$\text{LOI} (\text{g} 100 \text{ g}^{-1}) = \frac{(\text{Oven dry soil weight at } 105°C - \text{Soil weight after ignition})}{\text{Oven dry soil weight at } 105°C} \times 100$$

Samples of the soil which was used to extract SOM fractions were also subjected to sequential thermal oxidation from 150 to 550°C.
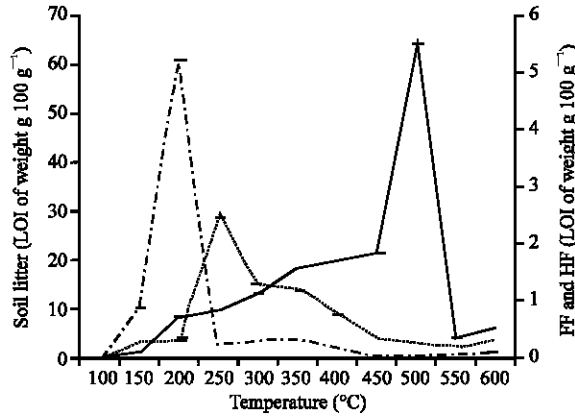
Fig. 1: Loss-on-Ignition (LOI) of weight of Fulvic Fraction (FF) (.........), Humic Fraction (HF) (——) and the soil litter (—·—·—) extracted from the soil, from 105 to 550 °C
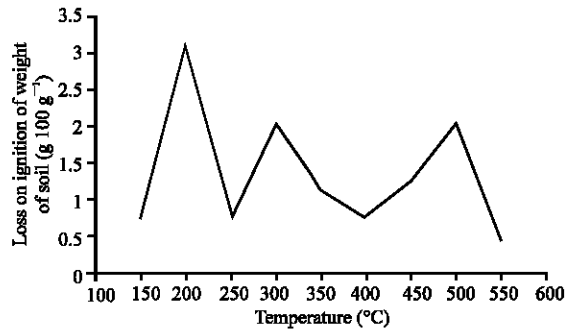

Fig. 2: Loss-on-Ignition (LOI) of weight of the soil used, from 150 to 550°C

RESULTS

The peak weight loss signal for the soil litter occurred between 150 and 200°C (Fig. 1). Two main reactions occurred between 200-500°C. The peak weight loss signal for the FF occurred in between 200-250°C (Fig. 1). The peak weight loss signal for the HF occurred at 500°C.

Figure 2 shows the LOI of weight of soil from 150 to 550°C. Three main peaks were observed at temperature ranges 150-200, 200-250 and at 500°C. These peaks coincided with the peaks of the Fig. 1.

DISCUSSION

Recent studies have used the LOI of weight of soil fractions as well as the whole soil for a wide range of applications and they have determined the SOM accurately (e.g., Cambardella *et al.*, 2001; Konen *et al.*, 2002; DeLapp and LeBoeuf, 2004; Bellamy *et al.*, 2005). During the LOI, a particular range of temperatures oxidizes a certain group of organic compounds depending on their activation energies of oxidation. Therefore, the groups of compounds with different compositions produce weight loss peaks at different temperatures. Thus, quantification of the compounds that are combusted according to their activation energies is more accurate than that using chemical extraction methods.

The weight loss signal between 170-190°C was due to the loss of soil litter with combustion (Morilzumi, 2003). The signal between 200-250°C of the FF in this study could be attributed to the degradation and decarboxylation reactions of fulvic acid, because those reactions of humic substances are known to occur in the range 200-350°C (Flaig *et al.*, 1975; Morilzumi, 2003). The main peak associated with HF at 500°C could be due to the oxidation of humic acid in the HF. The HF oxidizes at a higher temperature than FF, as the former contains many aromatic structures, which need high temperatures to oxidize (Flaig *et al.*, 1975). In addition to the main peak of each fraction, moderate to minor weight losses occurred in a range of temperatures. Because, each acid is a heterogeneous mixture of organic substances having a wide range of molecular weights (Hayes and Swift, 1990).

Comparison of Fig. 2 with Fig. 1 reveals that the highest peak occurred in the soil between 150-200°C is attributed to the loss of soil litter. Two main peaks observed in soil between 200-500°C corresponded to FF and HF, as also reported by Dell' Abate *et al.* (2003) for tropical soils.

The soil litter represents partially decomposed, fragmented and soil incorporated litter materials. They fall between active and slow pools of the SOM (Brady and Weil, 1999), more stable than other active SOM forms (i.e., fresh plant residues) and serve as an important fraction for long-term supply of nutrients (Wander *et al.*, 1994). The LOI was found to be a simple and accurate method for quantification of the soil litter.

It is generally considered that the soil litter and other organic matter fractions as a whole, as the SOM. The study clearly showed that the soil litter combusted at temperatures ranging from 170-190°C. Other SOM fractions showed weight loss signals at temperatures >190°C. Therefore the LOI can be used as a quick and simple technique for the determination of soil litter, in particular and further the FF and HF, compared to laborious and time-consuming extraction methods. However, the possible errors involved in this method are that in addition to the oxidation of SOM, increasingly higher temperatures can drive off structural water from clays and other inorganic constituents, decompose carbonates and hydrated salts and oxidize Fe^{2+}, although carbonates remain stable at temperatures <500°C (Schulte and Hopkins, 1996). This method should be evaluated for other soil types in further studies.

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