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Quantification of Humic Substances Fractions in Soils under Different Tree Stands Plantation in Tropical Rainforest, Nigeria

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Abstract

For better understanding of the dynamics of natural organic matter in the tropical forest ecosystem, quantitative assessment of humic substances (humic acid (HA), fulvic acid (FA), and humin) was done for soils underneath six tree stands plantation (secondary re-growth natural forest (SRNF): *Terminalia superba* (TSP), *Pinus caribaea* (PCP), *Gmelina arborea* (GAP), *Tectona grandis* (TGP) and *Theobroma cacao* (TCP) in Nigeria. Extraction of the soil humic substances was done according to the International Humic Substances Society (IHSS) methodology, based on the principle of differential solubility in basic and acidic media. Humin that could be extracted with a 0.1 M NaOH–0.1 M Na₄P₂O₇ solution from the residue after HAs and FAs was determined on weight basis. The highest yield of HAs, 0.31 g C kg⁻¹, was recorded in soil under *Terminalia superba* plantation, while the highest yield of FAs was recorded in soil under *Tectona grandis* plantation. The yield of humin was significantly ($p < 0.05$) higher in the soil under secondary re-growth natural forest compared to those under *Pinus caribaea* and *Tectona grandis* plantations, but not significantly different from the soil under *Terminalia superba* plantation. Further research is needed to provide information on biochemical compounds in the soil organic matter fractions under different tree stands.

Keywords: Humic substances, Soil organic carbon sequestration, Tree stands, Plantation, Polyvinylpyrrolidone, Visking dialysis

Introduction

The emergence of plantations of different tree species in Nigeria forest reserves may limit the ability of existing protected areas to sequester more carbon (C), if the right tree species are not planted to mitigate C emission. Changes in the quality and quantity of plant litter input to the soil environment may be affected by forest management decisions, as is the case of promotion of exotic tree species for reforestation in Nigeria. Increasing concentration of carbon dioxide (CO₂) ($\approx 76\%$) of the total greenhouse gas in the atmosphere has been a critical concern to global community (Lambers *et al.*, 2008; IPCC, 2014) because of the severe impact on climate change. The flow of C from the atmosphere into plant and later into soils through litter fall determines whether a stand is

a C sink or a net source leading to rising of CO₂ in the atmosphere. The input of litter by plants represents the link between the above-ground and below-ground dimensions of terrestrial ecosystems that can influence the functioning of the whole ecosystem (Faboya *et al.*, 2015). Soil C sequestration ability of tree plantation is a function of litter quantity and quality that replenish the soil underneath it stand. The magnitude and rate of soil organic C (SOC) sequestration with afforestation depend on climate, soil type, nutrient management and tree species (Lal and Follet, 2009). Many tropical forest plantations were designed to provide environmental benefit in addition to timber production (Healey and Gara, 2003). Now that protective function is driving the current objective for afforestation and reforestation through the Clean Development Mechanism (CDM) of the Kyoto Protocol. It is very important to note that, to date, available data on the ability of different forest

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stands in sequestration of C is still very few especially in the tropics (Chapin III *et al.*, 2003). The ability of different tree plantations in maintaining natural organic matter is imperative in order to guide future selection of tree species for afforestation aiming to enhance soil C management. This research therefore, intends to advance knowledge in understanding the variation in abundance of humic substances across different tree stands in the tropics.

Materials and Methods

Study Area

This study was carried out in Shasha Forest Reserve in Ile-Ife area of southwestern Nigeria. Shasha Forest Reserve, which was originally created in 1925 (Isichei, 1995) and has a land area of 310,798 km² (Salami *et al.*, 2007), was selected because its long-term land use history is known. The reserve shares a common boundary with Ago Owu, Oluwa, and Omo forest reserves. The area has a fairly high relief with elevation as high as 800 feet above sea level according to the topographic map of the area. It has witnessed excessive logging, conversion to plantations and farm lands. It has a mean annual rainfall of 1,421 mm (Adekunle, 2006), most of which falls during the rainy season from April to October. Shasha Forest Reserve is a fragmented habitat whose landscape has been partitioned as result of plantation development and agricultural intensification into secondary re-growth natural forest (SRNF), plantations of both local and exotic tree species, farm settlement and agricultural land. These physiognomies were separated from each other by forest road, footpath or abandoned grassland. The vegetation of the area is classified as the Guinea-Congolian drier forest types (White, 1983). The five major soil types recognized in this area are; inselberg soils, Hill creep soils, sedimentary non-skeletal soils, drift soils, and alluvial deposits (Hall, 1969). Those soils are derived from old basement complex which is made up of granite metamorphosed sedimentary rock (Hall, 1969) and have been classified into Lixisols and Ultisols (FAO/UNESCO, 1988). The soil in this area is well-drained with a major river (known as Shasha) and its tributaries which provide a good network of rivers and streams that defines dendritic patterns suggest a fairly homogenous rock of uniform resistance which makes up the basement complex.

Description of the Different Stands

The plantations investigated were established between 1990 and 1995 from existing natural forest reserve. Details of 6 soil sampling sites are as follows.

Secondary Re-growth Natural Forest (SRNF; 7° 04' 10.46" N and 4° 34' 26.43" E, 178 m above sea level) has a closed forest canopy and vegetation with the principal layers consisting of trees. Its vegetation was mostly dominated by *Strombosia pustulata* Oliv. of the family Olacaceae, *Musanga cecropioides* R. Br. of the family Moraceae, and *Diospyros dendo* Welw. ex Hiern of the family Ebenaceae. Also there were shrubs, herbs, climbers, and forest litter covered ground surface.

Terminalia superba Plantation (TSP; 7° 02' 13.85" N and 4° 35' 10.17" E, 168 m above sea level) was established in 1992. *Terminalia superba* Engl. ex Diels (Limba) is a local tree species. The plantation has been selectively logged in the past, which has encouraged gaps in the plantation. There were presence of herbs and climbers, and the canopy was fairly closed.

Pinus caribaea Plantation (PCP; 7° 02' 2.20" N and 4° 34' 52.69" E, 186 m above sea level) has not been logged since it was established in 1989. There were high presence of native trees, herbs, and climbers which were co-habiting with the planted tree. The plantation had a closed canopy. Any post planting operation, such as weeding, pruning, or thinning, has not been conducted since it was established.

Gmelina arborea Plantation (GAP; 7° 03' 23.82" N and 4° 35' 50.98" E, 201 m above sea level) was established in 1990-1993 and since then any post planting operation has not been conducted. There were presence of climbers, herbs, shrubs, and indigenous tree species. The forest canopy was fairly close and the section chosen has not previously been logged.

Tectona grandis Plantation (TGP; 7° 02' 34.26" N and 4° 34' 55.60" E, 180 m above sea level) was established between 1990 and 1995. The main objective of establishing *Tectona grandis* plantation was to meet up with the wood shortage as a result of over-exploitation of the natural forests by wood users. The forest canopy was fairly closed, and the soil surface was covered with forest litters. The section chosen has not previously been logged. Any post planting operation was applied since it was established.

Theobroma cacao Plantation (TCP; 7° 02' 51.75" N and 4° 34' 8.95" E, 2179 m above sea level) was

established over 30 years ago. It was made up of *Theobroma cacao*, *Cola nitida*, *Elais guineensis*, and scattered indigenous trees. The canopy can be described to be a closed and shaded type. Weeding and thinning have been conducted every year. No traces of logging activities were observed.

Collection of soil samples and determination of humic substances

Simple random sampling was adopted in collecting triplicate soil samples using stainless soil auger from three quadrats of 5 m x 5 m within 25 m x 25 m plots (two at the edges and one in the middle), from the surface soil layer (0–20 cm). The collected composite soil samples were air-dried and passed through a 2-mm mesh sieve.

Extraction and fractionation of fulvic acids (FAs) and humic acids (HAs) were conducted according to the International Humic Substances Society (IHSS) methodology (Swift, 1996). Soil samples (1 g) were equilibrated to a pH 1–2 with 1 M HCl and 0.1 M HCl was added to provide a final ratio of 10 mL g⁻¹ dry soil sample. The suspension was shaken for 1 h at room temperature and then the supernatant was separated from the residue by decantation after allowing the solution to settle. The supernatant (FA Extract 1) was saved for the isolation of FAs. The soil residue was neutralized (pH 7.0) with 1 M NaOH and 0.1 M NaOH was added to give a final extractant to soil ratio of 10:1 (v/w). The alkaline suspension was shaken intermittently for a minimum of 4 h and allowed to settle overnight at room temperature under N₂ atmosphere. Then the supernatant was collected by centrifugation, acidified to pH = 1.0 with 6 M HCl, and allowed to stand for 13 h before centrifugation for separating HA (precipitate) and FA (supernatant - FA Extract 2) fractions.

The HA fraction was redissolved in a minimum volume of 0.1 M KOH under N₂ atmosphere. Solid KCl was added to attain a concentration of 0.3 M [K⁺] and then centrifuged to remove insoluble materials. The HAs were reprecipitated by adding 6 M HCl with constant stirring to pH = 2.0 and the suspension was allowed to stand for 12–16 h. The HA precipitate collected by centrifugation was suspended in 0.1 M HCl/0.3 M HF solution in a plastic container and shaken overnight at room temperature. Removal of the supernatant by centrifugation and treatment of the precipitate with fresh HCl/HF were repeated 6

times until the ash content of HAs was below 1%. The precipitates was transferred into a Visking dialysis tube by slurring with water, dialyzed against distilled water until the dialysis water gives a negative chloride ion (Cl⁻) test with silver nitrate, freeze dried, and weighed.

The supernatant designated “FA Extract 1” was passed through a column of polyvinylpyrrolidone (PVP; 0.15 mL of resin per gram of initial sample dry weight at a flow rate of 15 bed volumes per h). The PVP column containing sorbed FAs was rinsed with 0.65 column volumes of distilled H₂O. The sorbed FAs were back eluted with 1 column volume of 0.1 M NaOH, followed by 2 to 3 column volumes of distilled H₂O. Immediately the FA solution was acidified with 6 M HCl to pH = 1.0 and concentrated HF was added to a final concentration of 0.3 M HF. Fulvic acids in the supernatant designated “FA Extract 2” were also recovered by similar manners to “FA Extract 1” using a column of PVP (1.0 mL of resin per gram of initial sample dry weight). The final eluates from each FA extract were combined and passed through another column of PVP (equivalent to one-fifth of sample volume). The column was rinsed with 0.65 column volumes of distilled H₂O and back eluted with 1 column volume of 0.1 M NaOH, followed by two column volumes of distilled H₂O. The eluate was passed through H⁺-saturated cation exchange resin (AG-MP-5, Bio-Rad, Richmond, CA) using three times the mole of Na ions in solution. The eluate was freeze dried to recover the H⁺-saturated FAs and weighed.

The soil residue was further washed with distilled water until pH becomes neutral and then shaken with 200 ml of 10% HF for 24 h. The suspension was centrifuged and the supernatant was discarded. The residue was washed with distilled water repeatedly until the pH of the supernatant reached 7. After that, 500 mL of a mixture of 0.1 M NaOH and 0.1 M Na₄P₂O₇ (1:1) was added to the residue and the remaining alkali-soluble and acid-insoluble humus was prepared by similar manners to the preparation of HAs and weighed. This fraction is designated humin in the present study.

Results and Discussion

The concentration of the humic substances across the different tree plantations exhibited wide variation

(Fig. 1). Across the different plantations the yield of humin was the highest among the three fractions. Stevenson (1994) stated that regardless the soil use system there is predominance of humin in the soil in relation to FAs and HAs. Our results agreed to this, although the humin that was recovered in the present study may be only a portion of the whole humin. The yield of humin was also significantly ($p < 0.05$) higher in the secondary re-growth natural forest soils ($0.55 \pm 0.02 \text{ g kg}^{-1}$) compared to the *P. caribaea* soils ($0.36 \pm 0.03 \text{ g kg}^{-1}$) and *T. grandis* soils ($0.44 \pm 0.04 \text{ g kg}^{-1}$) but not significantly different from the soils under TSP ($0.49 \pm 0.05 \text{ g kg}^{-1}$), GAP ($0.46 \pm 0.03 \text{ g kg}^{-1}$), and TCP ($0.47 \pm 0.03 \text{ g kg}^{-1}$). As tree

species encountered within the secondary regrowth natural forest were most diverse, the largest accumulation of humin there might be attributed to larger biomass resulted from higher tree populations and the presence of species that grow faster.

Different from humin, the yield of HAs was the highest in the *T. superba* soils ($0.31 \pm 0.01 \text{ g kg}^{-1}$), followed by the *G. arborea* soil ($0.26 \pm 0.06 \text{ g kg}^{-1}$). The lowest yield of HAs was recorded by the *T. grandis* soils ($0.11 \pm 0.02 \text{ g kg}^{-1}$). The difference in the quality of the inputted litter materials might have been responsible for the variation, although details are unknown. The yield of FAs tended to be higher in the *T. grandis* soils ($0.18 \pm 0.06 \text{ g kg}^{-1}$) while lower in the *T. cacao* plantation soils ($0.12 \pm 0.01 \text{ g kg}^{-1}$) compared to the other plantations. However, there were no significant differences in the yield of FAs across the different physiognomies. This result suggests that the FA content in forest soil is stable and less affected by the variation in tree species.

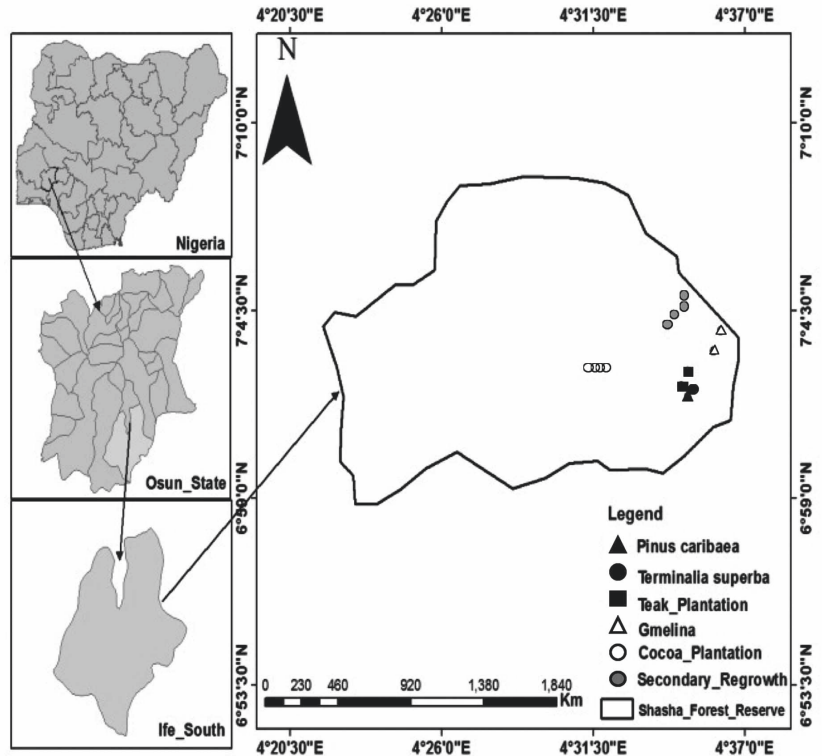


Figure 1. Maps of the study area in Ife South Local Government Area, Osun State, Nigeria with soil sampling sites.

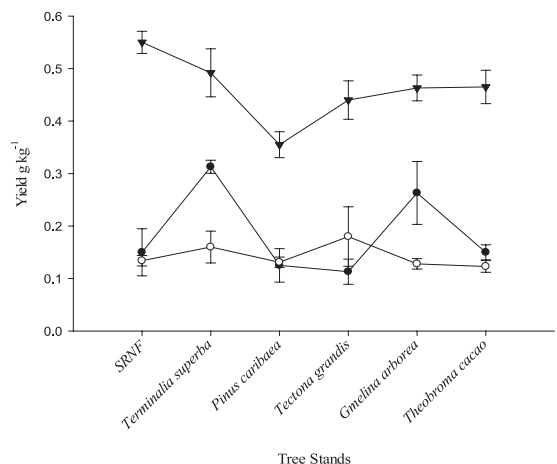


Figure 2. Yields of humic acids, fulvic acids, and humin in Nigerian soils under different tree stands. ●, Humic acid content. ○, Fulvic acid content. ▼, Humin content. Vertical bars indicate standard errors. SRNF, Secondary re-growth natural forest.

Conclusion

Considering the afore analysis, it could be concluded that the development of plantations from Shasha forest reserve has not only transformed the structural composition of the forest reserve but also impacted on the natural organic matter composition.

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