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Distribution of humic substances in sieved aggregates of soil under contrasting land use

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ABSTRACT

Soil quality indicators that control aggregate stability need to be extensively investigated so as to maintain our soils. Humified carbon (HC), humified acid carbon (HAC), and aggregate-associated fulvic acid carbon (FAC) in forest soils, cocoa plantations, five-year fallow, and five-year continuous cultivated soils were studied. Samples of soil were collected at 0-15 cm topsoil in order to measure the amount of humic materials in both the wet sieved and dry sieved aggregates. Findings revealed a significant input of land use on values of HC, HAC, and FAC occluded in sieved soil aggregates. The HC and FAC were preferentially stored in micro aggregate fractions less than 0.25 mm, while the HAC was greater in macro aggregates 2-1 mm and 1-0.5 mm. Concentration of HC was 18.8 g kg^{-1} in dry sieved and 17.2 g kg^{-1} in wet sieved micro aggregates less than 0.25 mm. The HC increased significantly ($p<0.05$) under a 5-year fallow. The HAC was stored in macro aggregates larger than 1 mm, whereas the HC and FAC fractions were occluded in micro aggregates > 1.0 mm. Continuous cropping decreased MWD of water-stable aggregates by 55%, while bulk density increased by 18%. The correlation coefficient between HC and MWD was significant ($r = 0.811$, $p < 0.01$), revealing the positive role of HC in aggregate stability. This study will help in understanding soil management strategies that will raise the accumulation of HC and HAC in macro-aggregates, thereby protecting the soil mass from structural degradation.

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1. INTRODUCTION

Soil organic carbon regulates the numerous ecological and environmental processes, such as preserving soil fertility, promoting plant development, and determining what happens to environmental contaminants da Silva et al. (2014); (Gerke, 2018). Humic substances (HS) and non-humic substances (NHS) make up humus. winch often referred to as SOM. The NHS is usually characterized as classes of simple or complex substances, whereas, humification is the process by which plant and microbial bodies disintegrate and transform to form a wide range of very acidic substances. Humic substances (HS) are compounds with a high molecular mass that range in color from yellow to black (IHSS, 2019).

Humic substances can be further classified as extractable humic substances (EHS), water-soluble humic substances (WSS), residual humin (RH), and water-floating humic substances (WFS) (Dou et al., 2020). The WFS and WSS, in this case, are SOM, but they are not humus in a strict sense. The primary components of HS are in Equations 1–4.

$$SOM = WFS + WSS + EHS + HC \quad [1]$$
$$Humus = WSS + EHS + HC \quad [2]$$
$$EHS = HAC + FAC \quad [3]$$
$$HS = FAC + HAC + HC \quad [4]$$

HS is the main component of SOM, according to García et al. (2019). It is widely distributed around the world and typically contributes to the preservation of soil, particularly the protection of soil structure from disturbance during cultivation.

The role of humus and its hydrophobic components in the long-term stability of soil aggregates cannot be overemphasized (Hayes & Swift, 2018; Olk et al., 2019). This is because, humus and humic materials can significantly affect soil fertility and form the constituent parts of soil aggregates

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(Guan et al., 2015). Thus, humic substances are important in limiting possible loss of SOC in the soils, through preserving soil aggregates. Guan et al. (2015) state that the main factors affecting the stability of soil aggregates are the iron hydroxides and oxides that makeup HS and their interactions with the organo-mineral. The quantity and quality of humic substances in SOM typically functioned as an adhesive agent that affects the total and labile compounds in the soil.

Humic substances (HS) in aggregates and their storage can offer a diagnostic link between aggregate stability and SOM (Gerke, 2018; Olk et al., 2019). Several discussions have been made in recent times concerning the necessity of HS's participation in aggregate formation and the role it contributes to the quality and stability of soil aggregates (Kleber & Lehmann, 2019). Certain fractions of humic substances, such as water-soluble humic substances (WSS) and water-floating humic substances (WFS), were found to be susceptible to slaking by water and land use types (Dou et al., 2020). Whereas, a positive correlation between fulvic acid carbon (FAC) and macro aggregates >0.25 mm were reported (Lehmann & Kleber, 2015; Olk et al., 2019). They came to the conclusion that the sole component of humus that helped build water-stable aggregates was the FAC percentage. However, there are knowledge gaps about how soil aggregates respond to slaking in water and about the distribution of humic materials in cultivated and forested land uses. As a result, this research will resolve some debates and increase our understanding of how HS functions within an aggregate hierarchy. This study was to quantify the humic carbon fractions associated with aggregates under cropping and forested soils, using both wet and dry sieved samples.

2. MATERIALS AND METHODS

2.1. Study area and soil sample collection

This study examined data from four different land use types in the Southern ecological zone of Nigeria. The USDA Soil Taxonomy classifies the soil texture as sandy clay loam. The area is located on 5° 20' and 5° 30'N, 7° 28' and 7° 42'E. The rainfall in the area is typical of a tropical rainforest climate (NiMet, 2019). The maximum annual temperature is 31°C. For this study, four different land use types were selected: (1) a 100-hectare secondary forest that is mostly covered with shrubs like *Ficus exasparata* and *Alchornea cordifolia;* (2) a 100-hectare 5-year fallow plot (5-year fallow); (3) a 120 ha cocoa plantation; and (4) an 85 ha 5-year cropping area that was planted to cassava continuously for five years. Five transects, or blocks, were created from each land use area according to differences in physiographic positions. For a total of 40 bulk and 40 undisturbed samples, five replicates of bulk and undisturbed soil samples were taken at 0–15 cm along each transect. After being labeled, the soil samples were brought to the University of Port Harcourt Teaching and Research laboratory for analyzes for water-stable aggregates, humic compounds, and other soil characteristics.

2.2 Soil bulk density, pH, and particle size distribution measurements

The Blake and Hartge (1986) method was used to determine the bulk density using the core soil samples. The pH of the soil was measured using a Bechman zeromatic pH meter in a ratio of 1:2.5 in distilled water. The particle-size distribution was obtained using the modified hydrometer method, and the dispersion was accomplished using sodium hydroxide.

2.3. Aggregate stability, saturation hydraulic conductivity, and organic carbon calculations

Using Kemper and Rosenau (1986) approach, aggregate stability was calculated based on the mean weight diameter (MWD) of water-stable aggregates. After sand fractions were eliminated, the MWD was computed using Equation 5.

$$MWD = \sum_{i-1}^{n} X1\, Wi \quad [5]$$

where, For each given size class of aggregates separated by wet sieving, Xi = mean diameter, and Wi = weight of aggregates in that size class as a function of sample's total dry weight. Using Equation 6, water stable aggregates (WSA) were determined.

$$WSA = (\frac{MR}{MT} \times 100) \quad [6]$$

where MR = mass of resistant aggregate (g) and MT = total mass of wet sieved soil (g).

The constant head method (Reynolds et al., 2002) was used in determining saturated hydraulic conductivity. The result was computed by rearranging Darcy's equation for constant head conditions as Equation 7.

$$A = \frac{VL}{AT\Delta H} \quad [7]$$

where, V is the volume of water collected (cm^3), L the height of soil core (cm), A the area of core (cm^2), T is the time (h) and ΔH is change in hydraulic head (cm). The wet combustion method was used to determine organic carbon (Nelson & Sommers, 1996).

2.4. Measurement of humic substances in wet-and dry-sieved aggregate sizes

To measure water stable aggregates, wet-sieving method (Nimmo & Perkins, 2002) was employed. Soil aggregates measuring 4.75 mm were placed in the uppermost sieves with varying diameters of 2.0, 1.0, 0.5, and 0.25 mm, as part of the process. A mechanical agitator was used to vertically oscillate soil samples in water twenty times after they had been pre-soaked for ten minutes. Prior to being weighed, the stable aggregates on each sieve were oven-dried for 24 hours at 50 °C. The resulting aggregates were used to determine the humic compounds.

2.5. Fulvic acid carbon (FAC), humified carbon (HC), and humic acid carbon (HAC) measurements

To Calculate the humic fractions in the soil, the Mostafa et al. (2021) method was used to determine the humified carbon (HC), humic acid carbon (HAC), and fulvic acid carbon (FAC). The amount of humified carbon (HC) was measured by wet oxidation procedure. Humified acid carbon (HAC) was determined using 10 ml of the extract after acidifying it with H_2So_4 to a pH of 2. The difference between the HC and HAC was used to calculate the amount of fulvic acid carbon (FAC).

2.6. Statistical analyses

SAS Institute software was used to conduct statistical analyses (SAS Institute, 2016). Variations in soil characteristics were tested using the parametric statistical method of ANOVA. LSD was used to differentiate the means. The associations between HS and several soil characteristics were examined using the correlation coefficient.

3. RESULTS

3.1. Textural class, bulk density, and soil pH

After five years of continuous cultivation, the soil texture ranged between sandy clay loam and sandy loam, with a percent sand content of 709 to 548 g kg^{-1}. Silt and clay content ranged between 202 g kg^{-1} in Forested soil and 115 g kg^{-1} in 5-year year fallow. During a five-year period of continuous cropping, clay content in forested soil was 250 g kg^{-1}, which was considerably ($p<0.05$) greater (Table 1). The soil's textural class remained unchanged despite the high sand content promoted by 5-year cropping' and the Forested the high clay in forested soil. pH was generally moderately acidic in Forested soil (Table 1) at 6.2. Five years cropping had a bulk density value of 1.51 g cm^{-3}, and Forested soils had the lowest value of 1.28 g cm^{-3}.

3.2 Water stability of aggregates, organic carbon, and hydraulic conductivity

In forested soil, saturated hydraulic conductivity (Ksat) was rapid (32.5 cm h^{-1}), and moderately (21.3 cm h^{-1}) in 5-year fallow soil. The 5-year cropping had led to very slow saturated hydraulic conductivity (Table 2). In contrast to forested soil, 5-year cropping reduced water-stable aggregates by 55% and SOC by 217%. However, 5-year fallow raised the MWD of water stable aggregates by 65%, compared to 5-year cropping, which decreased water–stable aggregates. Forested, cocoa plantation, 5-year fallow, and 5-year cropping soils had mean weight diameters (MWD) of 1.69, 1.25, 0.81, and 0.76 mm, respectively. The 5-year fallow increased SOC by 123% and MWD by 65% as compared to 5-year continuous cropping. SOC was generally in the range of Forested > Cocoa plantation > 5-year cropping > 5-year fallow (Table 2). Soil organic carbon (SOC) was significantly higher (26.3 g kg^{-1}) in Forested soil ($p<0.05$), whereas, the soil five-year cropping had the lowest value, (8.3 g kg^{-1}).

3.3. Humic substances in stable aggregates

Results in Figures 1a and 1b showed that both the dry-sieved and wet-sieved aggregates and the whole soils had significantly different amounts of humic compounds in water stable aggregates ($p<0.05$) in forested soil. The percentage of humified carbon (HC) in forested soil was mostly stored in micro-aggregate fractions less than 0.25 mm. On the other hand, the dry-sieved macro-aggregates 2-1 mm and 1-0.5 mm in the Forested soils had a significantly ($p<0.05$) greater humic acid carbon (HAC) concentration. Wet-sieved forest soils generally had greater levels of HC for all aggregate sizes in the 5-year fallow soils. In the sieved aggregates HC was significantly occluded in both the macro and micro-aggregates, followed with HAC (Fig. 2a and 2b). In Figures 3a and 3b, the HC and FAC were significantly stored in macro-aggregates greater than 2 mm and micro-aggregates less than 0.25 mm, indicating that Cocoa plantation helped in retaining the humic substance in the soil. In all the soils, the order of occlusion of humic substances in water-stable aggregates was cocoa plantation > 5-year cropping > forested > 5-year fallow. In Figures 4a and 4b, 5-year continuous cultivation decreased HC in micro-aggregates, and increased FAC in similar aggregates compared to the Forested soil (Fig. 4). Thus, continuous cultivation can accelerate depletion of HC in soils in micro-aggregates, whereas, the traditional 5-year fallow increased the HC and decrease HAC and FAC in the soil.

3.4. Relationship between humic substances and aggregate stability

A strong positive correlation ($r = 0.811$, $p<0.01$) accounted for around 80% of the relationship between humified carbon (HC) and MWD in Table 3. HAC and MWD showed a non-significant positive association ($r = 0.573$, $p > 0.05$), indicating that HAC had minimal effect on soil aggregation. There was a significant correlation between FAC and MWD in almost 74% of the correlations ($r = 0.741$, $p<0.05$).

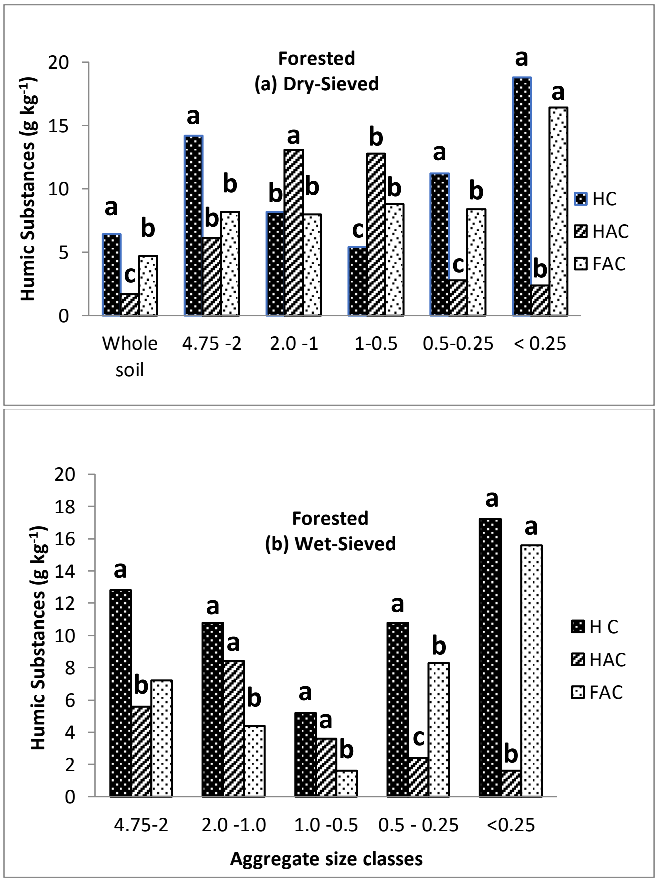

Figure 1a and b. Distribution of humic substances in dry- and wet-sieved stable aggregates of forested soils. Means followed by the same letter within column were not significantly different at $p < 0.05$. HC- humified carbon, HAC- humified acid carbon, FAC- fulvic acid carbon

4. DISCUSSION

In the five years of continuous cropping, the soil's bulk density increased significantly in tandem with an increase in sand content. This suggests that the soil had a 25% increase in bulk density along with a large increase in sand content.

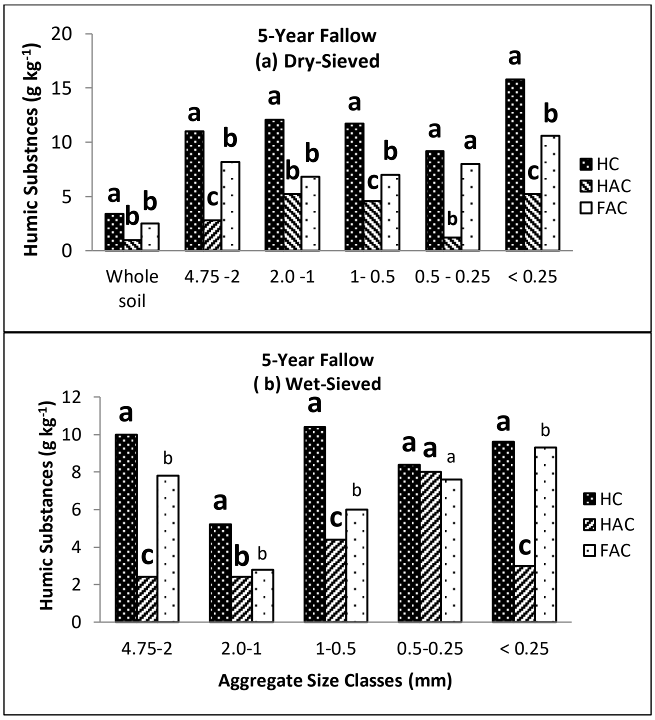

Figure 2a and b. Distribution of humic substances in dry- and wet-sieved stable aggregates in 5-Year fallow soils. Means followed by the same letter within column were not significantly different at $p < 0.05$. HC- humified carbon, HAC- humified acid carbon, FAC- fulvic acid carbon

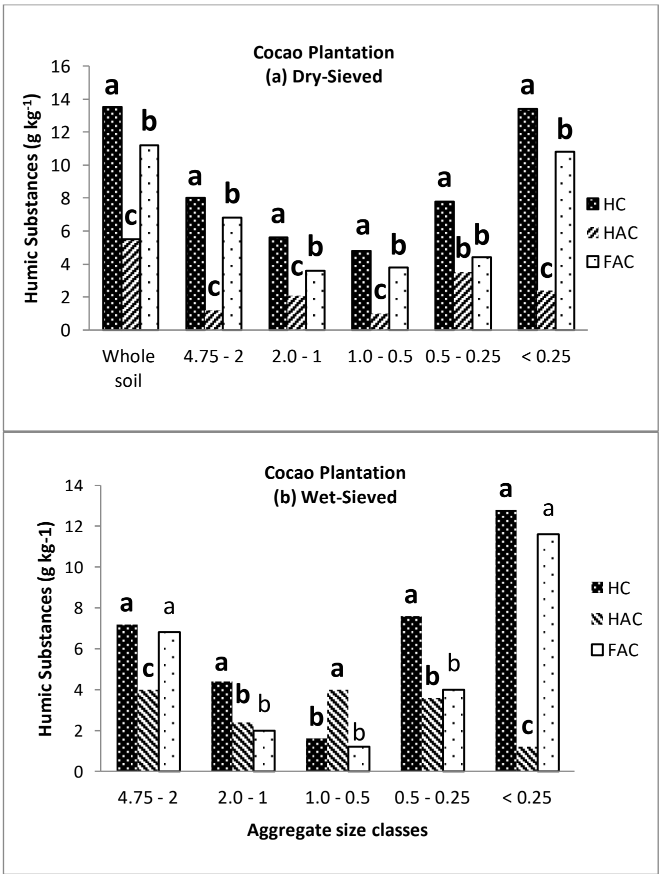

Figure 3a and b. Distribution of humic substances in dry- and wet-sieved stable aggregates in Cocao Plantation soils. Means followed by the same letter within column is not significantly different at $p < 0.05$. HC- humified carbon, HAC- humified acid carbon, FAC- fulvic acid carbon

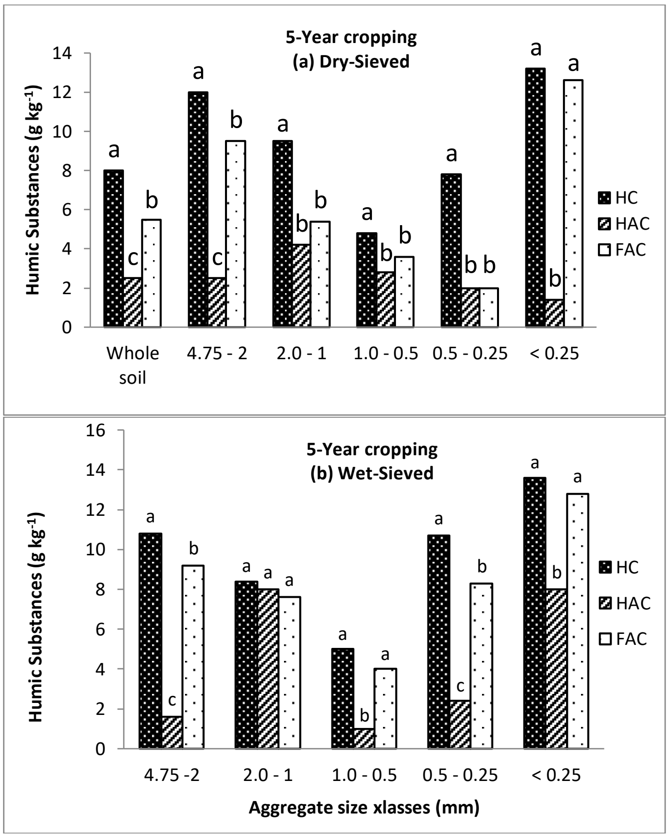

Figure 4 a and b. Distribution of humic substances in dry- and wet-sieved stable aggregates in 5-year cropping soils. Means followed by the same letter within column is not significantly different at $p < 0.05$. HC- humified carbon, HAC- humified acid carbon, FAC- fulvic acid carbon

Numerous writers have connected increased bulk density in continuous cropping soils to the loss of fine-particle fractions and rise in sand content (Udom & Ogunwole, 2015; Udom et al., 2018). The low aggregate stability in continuous cropping soil may have been attributable to notable loss SOM. Similar conclusions were drawn from Udom and Ogunwole (2015), indicating that poor stability of aggregates in soil was caused by continuous cropping. Similar claims were also made in research by Udom et al. (2022), who claimed that the enhanced sand contents and high bulk density of the soil were caused by continuous farming of the coastal plain sand soils. It was previously stated by Stewart et al. (2015) that after five years of continuous farming, soil pH was often expected to be low. It was therefore not surprising that the pH of the soil after five years of farming was only 5.2 instead of the pH of 6.5 and 6.2 discovered in the soil after five years of fallow and forest, respectively. Frequent loss of OM from continuous cropping and the potential removal of basic cations during crop harvesting could have contributed to the low pH of this soil. Baveye and Wander (2019) also documented this development in continuously cultivated soils in their investigations on the biochemistry of soil organic matter after cultivation-induced soil disturbances. These results support the idea that accumulation of SOM affects how macro-aggregates form. Therefore, regarding the improvement in MWD in 5-year fallow and forested soils, these findings further supported the idea that organic litter falls added SOM, which

Table 1. Soil texture, pH, and bulk density of the soils under the different land uses

Land use	Sand ($g\ kg^{-1}$)	Silt ($g\ kg^{-1}$)	Clay ($g\ kg^{-1}$)	Texture	pH	BD ($g\ cm^{-3}$)
Forested	548c	202a	250a	SCL	6.2a	1.28c
5-year Fallow	699b	115b	195b	SCL	6.5a	1.34b
Cocoa plantation	593c	131b	276a	SCL	5.8b	1.49a
5-year Cropping	709a	182a	109c	SL	5.2b	1.51a

Remarks: Means followed by the same letter in each column for each parameter were not significantly different at $p < 0.05$. BD – bulk density, SCL – sandy clay loam, SL – sandy loam

Table 2. Saturated hydraulic conductivity, aggregate stability and total organic carbon

Land use	Ksat ($cm\ h^{-1}$)	MWD (mm)	TOC ($g\ kg^{-1}$)	Permeability class
Forested	32.5a	1.69a	26.3a	Rapid
5-year Fallow	21.3b	1.25b	18.5ab	Moderately rapid
Cocoa plantation	8.2c	0.81c	10.5b	Very slow
5-year Cropping	6.1c	0.76c	8.3c	Very slow

Remarks: Means followed by the same letter in each column for each parameter were not significantly different at $p < 0.05$. Ksat- saturated hydraulic conductivity, MWD – mean weight diameter, TOC – total organic carbon

in turn increased water stable aggregates in the soil.

Table 3. Correlation coefficient (R) between mean weight diameter (MWD) and HC, HAC, and FAC (N=20)

Independent	Dependent	R
HC ($g\ kg^{-1}$)	MWD (mm)	0.811**
HAC($g\ kg^{-1}$)	MWD (mm)	0.57
FAC($g\ kg^{-1}$)	MWD (mm)	0.741*

Remarks: *significant at $p < 0.05$. **significant at $p<0.01$, MWD – mean weight diameter, HC- humified carbon, HAC- humic acid-carbon, FAC- fulvic acid carbon

This outcome does not contradict other research, including that of da Silva et al. (2014), Six and Paustian (2014), and Kobierski et al. (2018), who also found an exponential increase in the MWD and soil structural characteristics like macro-porosity and water holding capacity of the soil. The important role of organic matter in preventing soil aggregates from slaking in water and their contribution to the aggregation process was the major contribution in this study. The present investigation has confirmed that humified carbon (HC) and fulvic humic carbon (FAC) fractions exhibited preferential storage in micro aggregates smaller than 0.25 mm in sieved aggregates. Conversely, the humic acid carbon (HAC) fraction was preferentially stored in macro aggregates. This conclusion is however, in variance of the previous report that a larger proportion of HC and FAC were found in macro aggregates <1 mm of dry-sieved soil samples and in water stable aggregates (WSA) >2 mm (Devine et al., 2014; Tobiašová et al., 2018; Wells, 2019). The fact that the quantity of HC and FAC stored in aggregate size classes was determined by the amount of HC and FAC present in the soil provided more evidence in support of their theories.

The findings from the study supported the claim that the amount of humic compounds preserved in macro aggregates and micro aggregates was mostly determined by land use. The earlier studies by Wei et al. (2020) found a significant correlation among aggregates-associated humic substances in forested soils. The contribution of fallow systems to accumulation of SOM in soils can be further explained by noticeably higher concentration of HC and FAC in macro aggregates, particularly for 5-year fallow and forest. The findings of this study further justified the significant impact that land use has on the distribution and quantity of humic compounds in aggregate hierarchy (Carrizo et al., 2015; Wei et al., 2020).

A significant positive association was found between the quantities of humic substances protected in water stable aggregates. This suggests that HC and FAC have a major impact on the stability of aggregates. This assertion is consistent with recent research (Gerke, 2018; Olk et al., 2019), which discovered that HAC and FAC fractions functioned as cementing agents in the arrangement of soil aggregates. Additionally, the positive association between FAC and macro-aggregates larger than 0.25 mm supports the generally accepted theory that FAC contributed to the production of macro-aggregates (Dou et al., 2020; Lehmann & Kleber, 2015).

5. CONCLUSIONS

This study revealed that 5-year cropping of the soils caused loss of humified carbon (HC) and fulvic acid carbon (FAC) fractions in macro-aggregates which help in the protection of soil aggregates against slaking by water. The HC was more sensitive to continuous cropping, with negative impacts on macro-aggregate formation. On the other hand, 5-year fallow increased the accumulation of HC in both larger soil aggregates. The HC and FAC were related to the resistance of soil macro-aggregate against slaking. Continuous cropping reduced the mean size of water-stable aggregates and amount SOC by 55% and 217%, respectively, while bulk density increased by 18%. Therefore, the five years of fallow land improved the concentration of HC and HAC fractions, which is crucial for promoting soil aggregation. The mean weight diameter and HC and FAC had a positive correlation, indicating that the two variables can be used to predict how land use will affect distributions of humic fractions in different soil aggregates

Declaration of Competing Interest

The authors declare that no competing financial or personal interests may appear to influence the work reported in this paper.

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