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Full Length Research Paper

Potassium adsorption characteristics of five different textured soils under enset (*Ensete ventricosom cheesman*) farming systems of Sidama zone, South Ethiopia

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A laboratory study was conducted to determine the potassium (K) adsorption characteristics of five soils under *enset* (*Ensete ventricosom cheesman*) farming system in Sidama. Potassium adsorption isotherms were constructed by equilibrating 5 g soil samples with 5 levels of K (0 to 78mg/L) as KCl in 10 mmoles/L $CaCl_2$ solutions. Suspensions were shaken for half an hour. Adsorption data were fitted to Freundlich, Langmuir and Temkin adsorption equations. On an average, amount of K adsorbed ranged from 29.2 to 67.82% of added K. Significant positive relationship existed among percent K adsorbed, CEC, percent clay and pH. Langmuir equation gave a better fit of equilibrium K adsorption data for sandy clay loam and silty clay loam soils, while Freundlich equation gave a better fit for clay and loam soils. Furthermore, Temkin isotherm gave a better fit of equilibrium K adsorption data for clay loam soil. Sandy clay loam and loam soils had low 1/n values. The n values on the other hand indicated that the sorption is favorable only for these soils. The Freundlich adsorption capacity, k_f were found low as compared to available soil potassium. The negative values of Langmuir isotherm maximum monolayer coverage capacity (a) for clay loam and clay soils agreed with the absence of best fit to the model. Sandy clay loam and silty clay loam which indicated best fit had high monolayer coverage. Value of Temkin isotherm equilibrium binding constant (A*T*) was high for clay loam soil and this was manifested by high coefficient of determination. Low values of constant related to heat of sorption (*B*) were determined for sandy clay loam and loam whilst clay loam, silty clay loam and clay soils indicated high binding energy (*B*).

Key words: Sorption isotherm, Freundlich, Langmuir, Temkin model, monolayer coverage, equilibrium binding constant, heat of sorption.

INTRODUCTION

Enset (*Ensete ventricosum Cheesman*) farming system is a part of sustainable production and enset has been cultivated in Ethiopia since ancient times (Garedew et al., 2017). It is most commonly grown in home-gardens, frequently intercropped with peas or beans, which is suitable to compensate for the low protein level in enset foods (Abebe et al., 2010). Enset is also intercropped with cereal crops (maize), root crop (sweet potato), vegetable (cabbage) and coffee.

Potassium is an essential element for crop production and productivity. It is required for the activation of over 60 enzymes involved in the formation of carbohydrates, translocation of sugars, various enzyme actions, yield, quality parameters, tolerance to certain diseases, mechanisms to overcome abiotic stress, cell permeability and several other functions (Askegaard et al., 2004). Plants require immediately available forms of K such as exchangeable and soluble forms. Availability of K is however affected by physical, chemical and biological processes of soil (International Plant Nutrition Institute, 1998). Soils K availing capacity can be assessed using different techniques. Among these, the study of K adsorbing potential or transforming available K forms into unavailable ones is one of the techniques.

Adsorption is the accumulation of a chemical species at the interface between solution and the solid phase (Sposito, 1989). It affects the mobility and fate of nutrients in the soil. Soils K adsorption potential depends upon the amount and type of clay minerals (Pal et al., 1999). Moreover, equilibrium among the potassium retained by the interlayer sites, surface and edge sites of mineral crystal lattice and potassium in soil solution also affect soils' K adsorption potential. The mobility of soil potassium is affected by the dynamic equilibrium that exists in soil system. This dynamic equilibrium is affected by clay minerals types, pH, soil organic matter (SOM), hydroxide aluminum, soil moisture status, cation exchange capacity (CEC), fertilization and tillage system (Pannu et al., 2003). Among the clay mineral types influencing the dynamic equilibrium, illite and vermiculite were found to have positive associations with quantities of adsorbed K (Goulding, 1987). Due to this, the fate of K fertilizer in soils differs for different soils and the responses of crops to applied K are erratic and unpredictable.

Studies show that adsorption of K increases as the K imbalance is increased by addition of fertilizer and the degree of adsorption varies from soil to soil. Pal et al. (1999) reported the range of percent K adsorbed to be 5 to 67% of added K and also reported the negative effect of high K adsorption capacity of soils on the availability of K to crops. This implies that the rise in percent of K adsorbed by a given soil indicates the soils poor capacity to avail K to crops.

To visualize the relationship between the quantities of K fixed per unit soil weight and the concentration of K in solution, several equations or adsorption isotherms were developed. Among the isotherms, Langmuir and Freundlich equations are mostly employed and described the adsorption phenomena satisfactorily (Boschetti et al., 1998). The Freundlich equation is an empirical equation which corresponds to a model of adsorption where the affinity term decreases exponentially as the amount of adsorption increases. Potassium adsorption was described well often by Freundlich equation over a limited range of concentration (Barrow, 1978). The theory of Langmuir is restricted to cases where only one layer of molecule can be adsorbed on the surface. Therefore, the non-conformity to the Langmuir model suggests the presence of several types of K sorption sites in the soils, each with different selectivity for K (Hannan et al., 2007). The study of K adsorbing potential of soils could help clarify the relationship that exists between K adsorption capacity and other soil properties (Murashkina et al., 2007). It is undertaken by investigating the relation existing between amounts of soil solution K and its amount adsorbed by the soil (sorption isotherm). This provides information on whether any of the applied plant nutrients fix, react or make complex with the soil and helps optimize fertilizer to enhance crop productivity (Hunter, 1980). Thus, K sorption isotherm can be used to evaluate the ability of soils to supply K to plants and to determine interchangeable K in order to understand its dynamics in soil (Yunda et al., 1997). It can also be used to describe the exchange of K from the soils by other ions particularly, Ca (Bedrossian and Singh, 2002).

To date, there has been a general perception that soils of Ethiopia have sufficient amount of potassium. This perception is mainly due to the earlier work done by Murphy (1968) and hence; soil K adsorption studies were limited in Sidama, South Ethiopia. The present study was therefore conducted to assess and describe the K adsorption characteristics of soils under enset farming systems of Sidama, South Ethiopia. The results of this study are expected to illuminate light on the effects of some soil properties on the potassium adsorption characteristics of soils in the study area.

MATERIALS AND METHODS

Study area

The study was conducted in Awassa-Zuriya, Dale and Hula districts of Sidama zone, Southern Ethiopia (Figure 1) in 2016. Sidama administrative zone is located within 5°45'- 6°45'N latitude and 38°39'E longitude, covering a total area of 6,538.17 sq km of which 97.71% is land and 2.29% is covered by water (SZPEDD, 2004). It lies in the area varying from flat land (19.9 to 24.9°C) to highland (15 to 19.9°C) (Sidama Development Corporation, 2000). The regional and zonal capital, Hawassa, which is located in the northern tip of Sidama zone, has a distance of 275 km from Addis Ababa. In the present study, sample districts from the zone were randomly selected because nearly all areas in the zone have good

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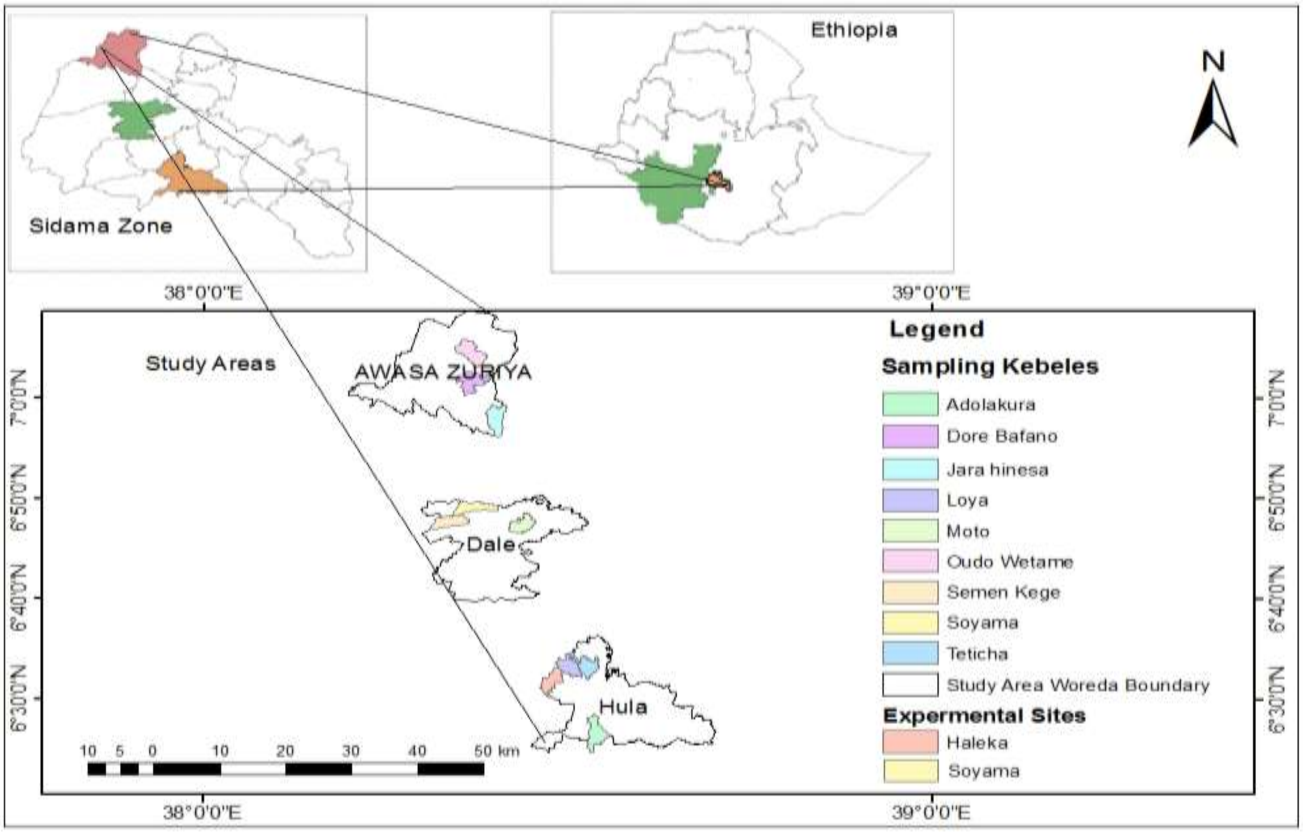

Figure 1. Location map of Sidama zone in Southern Ethiopia, study districts in Sidama zone and soil sampling kebeles in the study districts.

potential for enset production irrespective of productivity variation due to rainfall and altitude discrepancy. The sites are located between 6°28'15.5" - 7°04'50.3"N latitude and 38°20'7.8" - 38°32'36.5"E longitude.

Soil sampling

Samples were taken from enset farm fields in Sidama, South Ethiopia from the depth of 0 to 50 cm. From samples collected, five samples were chosen based on OECD guideline for the testing of chemicals (2000). The samples represent Awassa-Zuriya district, Jara hinesa and Oudo Wetame; the Hula district, Teticha and Adola kura and the Dale district, Moto dorsiso villages. The soil samples then were air-dried and ground to pass through 2 mm sieve before use.

Soil analysis

The pH was determined by 1:2.5 soil-water suspension by a glass electrode, EC was determined by conductivity meter at 25°C (Jackson, 1967) and CEC was determined by the 1 N NH_4OAc extraction method (Lu, 1999). The exchangeable potassium was determined using Mehlich III extractant (Mehlich, 1984). Particle size distribution was determined by the sedimentation procedure using hydrometer method after dispersing the soil with sodium hexametaphosphate (Bouyoucos, 1951). Organic carbon was determined by the wet oxidation method of Walkley-Black (1934).

Potassium adsorption experiment procedure

Soils were analyzed by the quantity/intensity procedure based on the method outlined in Beckett (1964). A stock solution of potassium chloride, 10 mmole/L KCl and calcium chloride, 10 mmole/L $CaCl_2$ were prepared separately. From these separate solutions, graded concentrations of potassium (0.0, 0.2, 0.4, 1.0 and 2.0 mmole/L in 10 mmole/L $CaCl_2$) were prepared and used for equilibration study. From each of the soil samples, duplicates of 5 g soils were weighed and quantitatively transferred into each of the five plastic tubes. Then, 50 ml of the graded concentration solutions was added to the correspondingly labeled plastic tubes. The suspensions were then shaken with a horizontal flask shaker for half an hour (180 rpm and 25 ± 1°C) and allowed to equilibrate overnight. After equilibration, suspensions were filtered using Whatman No. 42 filter paper, and the equilibrium solutions were analyzed for potassium by Flame Photometer. Adsorption isotherms were constructed based on the method described by Rowell (1994). The amount of K adsorbed was obtained by subtracting the amount determined in supernatant solution from the initial amount of K added as in the following.

$$\text{Concentration of Adsorbed K} = CKi - CKf \qquad (1)$$

Where CK_i = initial concentration of added K and CK_f = concentration of K in supernatant solution percent K adsorbed (%) = (concentrations of K adsorbed × 100)/concentrations of added K.
The K adsorption data were fitted into the following adsorption isotherm equations:

Table 1. Physico-chemical properties of selected soils of *enset* farming system in Sidama zone, South Ethiopia.

District	Kebele	Textural class	pH	EC (dS/m)	Organic carbon (%)	Clay (%)	Silt (%)	Sand (%)	CEC (cmol (+) kg^{-1})	Exch. K (cmol$_c$/kg)
Awassa-Z	Jara hinesa	Clay loam	7.3	0.17	3.3	39	40	21	33.91	2.9
Hula	Teticha	Sandy clay loam	4.9	0.14	1.23	23	26	51	21.2	0.34
Awassa-Z	Oudo Wetame	Silty clay loam	7.5	0.15	1.92	39	45	16	36.9	1.75
Dale	Moto dorsiso	Clay	7.1	0.12	3.11	42	39	19	31.64	1.05
Hula	Adola kura	Loam	4.8	0.14	4.12	22	49	29	25.98	0.37

Awassa-Z = Awassa-Zuriya, Exch. = Exchangeable, Kebele = Farmers' association.

Freundlich Adsorption equation:

$$\text{Log}\,(x/m) = \text{Log}\,kf + 1/n\ \text{Log}\,C \qquad (2)$$

Where x/m is the mass of adsorbed K per unit mass of soil mg/kg, C is the equilibrium K concentrations of solutions (mg/L), k$_f$ and 1/n are constants obtained from the intercept and slope respectively.

Langmuir adsorption equation

$$1/(x/m) = 1/a + 1/akLC \qquad (3)$$

Where C is the equilibrium solution K concentration (mg/L), x/m is the mass of K adsorbed per unit mass of soil (mg/kg), k$_L$ is a constant related to bonding energy of K to the soil, and 'a' is the soil's maximum monolayer coverage capacity during K adsorption. The values of 'a' and 'k$_L$' are obtained from the intercept (a) and the slope (b), respectively.

The Temkin equation:

$$x/m = B\ln AT + B\ln C \qquad (4)$$

where, x/m is the mass of K adsorbed per unit mass of soil (mg/kg), *C* is equilibrium solution K concentration (mg/L), 'A$_T$' is Tempkin isotherm equilibrium binding constant and '*B*' is constant related to heat of sorption. The values of 'A$_T$' and '*B*' are obtained from the intercept and the slope, respectively. For all isotherms, graphing and mathematical computations were undertaken using Excel sheet facilities.

Statistical analysis

Data analyses were performed with the statistical analysis system (SAS Institute, 2012). Correlation analysis was used to determine the relationship with percent K adsorbed, CEC, clay and pH.

RESULTS AND DISCUSSION

Soil physico-chemical properties

Power of hydrogen (pH) of soils ranged from 4.8 to 7.5 (Table 1). According to EthioSIS (2014), the Awassa- Zuriya district, Jara hinesa kebele clay loam and the Dale district clay samples showed neutral reaction. The Hula district Teticha kebele sandy clay loam and Adola kura kebele loam samples indicated strongly acidic reaction (pH<5.5) (EthioSIS, 2014). On the other hand, Awassa-Zuriya district Oudo Wetame kebele silty clay loam had high pH (7.5) as compared to the rest soils. The soil OC contents ranged from 1.23 to 4.12%. According to the rating by Landon (2014), very low value was determined for Awassa-Zuriya district, while the Hula and Dale district soils had low values. Soil OC influences the physical, chemical and biological properties of soils such as structure, water retention, nutrient contents and retention as well as soil microbiological activities. The CEC (cmol (+) kg^{-1}) of the samples ranged between 25.98 and 36.9. Based on Landon (2014), the range intervenes in the high rate (25 to 40) of the CEC. According to EhioSIS (2014), the Hula district Teticha and Adola kura kebele soils had low levels of exchangeable K due to low percent clay and pH, while high levels of exchangeable K were determined in Awassa-Zuriya district soils. The Dale district soil had optimum level of soil K (Table 1).

The particle size distribution of the soils ranged from 22 to 42%, 26 to 45% and 16 to 51 for clay, silt and sand, respectively. Electrical conductivity of the studied soils ranged between 0 and 2 dS/m and it has negligible salinity effect on crop plants according to the rating by Rhoades and Loveday (1990).

Soils potassium adsorption characteristics

Potassium adsorption isotherms of the soils studied are presented in Figure 2. The soils showed noticeable variation in their K adsorption

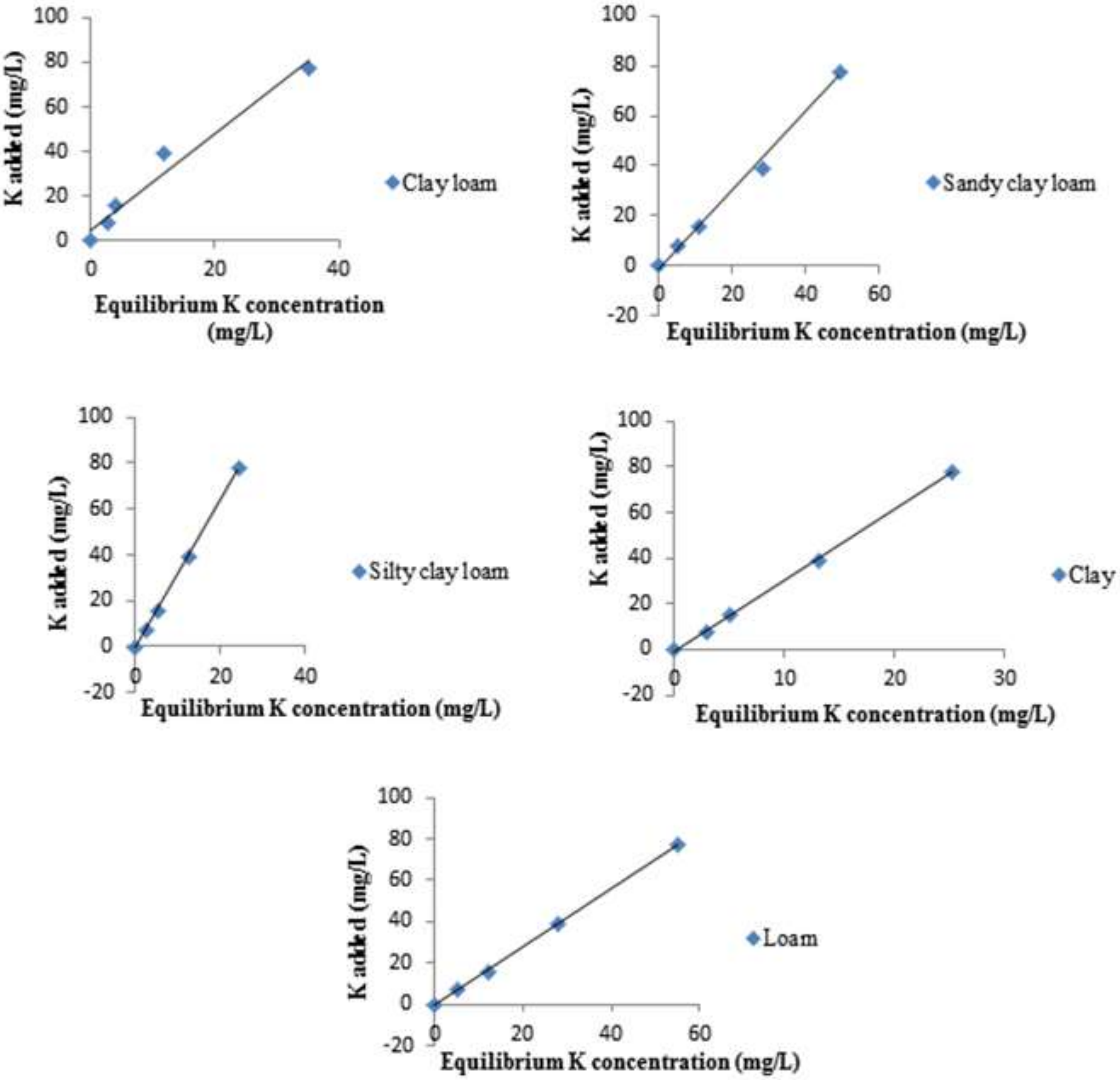

Figure 2. Potassium adsorption behavior of enset farming system soils of Sidama zone, South Ethiopia.

characteristics.

The percent of K adsorbed was not uniformly increased with increasing K concentrations (Figure 3). For the initial added concentration of K (7.8 mg/L), K adsorbed varied from 35.89 to 69.3% and for the highest added concentration of K (78 mg/L), it varied from 29.5 to 68.9%. The silty clay loam, sandy clay loam and loam soils indicated decreased adsorption after initially adding K and following this, slightly increasing trend in K adsorption was observed. The clay soil increased K adsorption for all added K levels. Apart from this, the clay loam soil increased adsorption for the first three K levels and decreased adsorption upon K levels increase. The equilibrium K concentration ranged from 2.4 to 5 mg/L for 7.8 mg/L of added K and from 24.2 to 55 for 78 mg/L of added K. Here, the lowest equilibrium K was detected for Awassa-Zuriya district Jara hinesa silty clay loam soil while the Hula district Adola kura loam soil had the

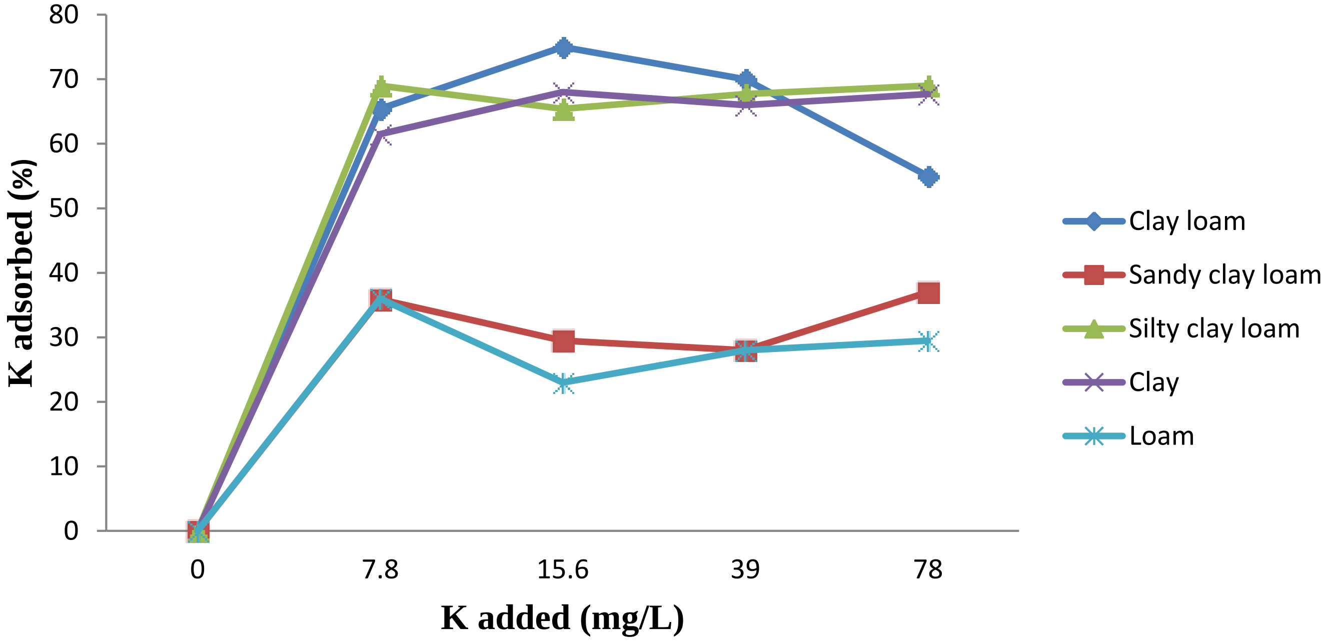

Figure 3. Percent of K adsorbed as a function of different K concentration levels of enset farming system soils in Sidama zone, South Ethiopia.

highest Equilibrium K. The high K in equilibrium solution for the Hula district Adola kura kebele loam soil could be due to relatively low clay percent (22) (Bangro et al., 2012) and strongly acidic pH (4.8) (Table 1). The linear relationship between added and equilibrium K showed the direct proportionality that existed (Figure 2). Furthermore, the soils indicated maximum adsorption behavior up to 53.8 mg/kg (silty clay loam for 78 mg/L added K) and the adsorption of K was found to increase with the amount of added K irrespective of soil types and texture.

Soils varied in percent of adsorbed K for different concentrations of added K. On average, highest adsorption (67.82%) was recorded for silty clay loam Awassa-Zuriya district Oudo Wetame kebele soil. High K adsorption potential of this soil is due to high silt fraction (45%), clay fraction (39%), CEC (36.9) and pH (7.5) of the soil. Following this, Awassa-Zuriya district Jara hinesa clay loam soil adsorbed 66.38% of added K. This could be due to high pH (7.3) and percent silt (40), percent clay (39) and CEC (33.91). Further, the clay soil adsorbed 65.89% of added K and it might be attributed to high clay proportion (42%) and CEC (31.64). Bangro et al. (2012) also found similar correlation that soils with high clay and CEC values fixed more K as compared to soils with low clay and CEC values. These soils may contain dominant amount of illite (hydrous mica) type of clay minerals since such clay types increase K adsorption capacity of soils (Mengel and Busch, 1982). Lastly, the Hula district Teticha kebele sandy clay loam soil adsorbed 32.69% of added K and followed by Adola kura kebele loam soil which adsorbed 29.2% of added K. Relatively low percentages of K was adsorbed by these soils when compared with the first three is likely due to low clay percentages and strongly acidic (4.9) and pH (4.8) values, respectively (Table 1). From the results, it could be concluded that adsorption was mainly governed by CEC (Jafari and Baghernejad, 2007) clay content (Zhang et al., 2009) and pH (Loannou et al., 1994) of the soils. In general, the soils were found to adsorb high proportion of added K than letting it stay in equilibrium solution manifesting their high K fixation capacity.

Potassium adsorption study from graphical analysis of Freundlich, Langmuir and Temkin equations plots

The degree of accuracy of the sorption isotherms varied from soil to soil. The coefficient of determination (R^2) values in Table 2 indicated that Langmuir equation gave a better fit (Figure 5) of equilibrium K adsorption data for silty clay loam soil. This is due to the homogeneity of sorption sites in the soil that allows only complete monolayer adsorption of solutes. On the other hand, the coefficient of determination (R^2) value indicated that Freundlich equation gave a better fit (Figure 4) of equilibrium K adsorption data for clay soil. This implied that clay soil had unlimited adsorption sites having heterogeneous surfaces (Hutson and Yang, 2000).

Lastly, Temkin isotherm gave a better fit (Figure 6) of

Table 2. Langmuir, Freundlich and Temkin Isotherm constants for the adsorption of potassium on the soils of Sidama zone, South Ethiopia.

Soils	Langmuir Isotherm			Freundlich Isotherm				Temkin Isotherm		
	a	k_L	R^2	1/n	N	k_f	R^2	A*T*	*B*	R^2
Clay loam	-1000	-0.002	0.93	1.04	0.965	0.610	0.91	0.743	12.7	0.97
Sandy clay loam	66.7	0.008	0.97	0.82	1.213	1.205	0.96	0.481	6.26	0.68
Silty clay loam	333	0.007	0.99	1.19	0.837	0.760	0.98	0.646	15.7	0.84
Clay	-143	-0.011	0.97	1.22	0.823	0.851	0.99	0.608	15.6	0.85
Loam	32.3	0.017	0.88	0.76	1.312	1.191	0.95	0.495	5.10	0.74
Mean	-142.2	0.0038	0.95	1.00	1.03	0.92	0.96	0.595	11.1	0.82
SD	508.85	0.0107	0.05	0.21	0.22	0.27	0.03	0.109	5.08	0.11

Table 3. Cross-correlation among percent potassium adsorbed and some soil properties.

	pH	CEC	Clay	Percent potassium adsorbed
pH	1			
CEC	0.9354*	1		
Clay	0.9749**	0.8682*	1	
Percent potassium adsorbed	0.9968***	0.9081*	0.9878**	1

*Significant at $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$.

equilibrium K adsorption data for clay loam soil.

Since Freundlich isotherm assumes low energy of adsorption, its constants ‘k_f’ and ‘1/n’ (Table 2) may be taken as a measure of the extent of adsorption and rate of adsorption or energy of adsorption (Voudrias et al., 2002). According to Kenyanya et al. (2013), the constant 1/n (Eq. 2) also represents the buffering capacities of soils. In the present study, it ranged from 0.76 to 1.22 kg/mg with a mean of 1.00±0.21 kg/mg. In the range, sandy clay loam and loam soils had low 1/n values due to high sand proportion. The values of 1/n also indicate a heterogeneity parameter where smaller 1/n values reveal greater heterogeneity (Dada et al., 2012). Accordingly, sandy clay loam and loam soils had high heterogeneity (Table 2). The n values indicate whether the sorption is favorable or not (Goldberg, 2005). For favorable sorption processes, it lies between one and ten (Goldberg, 2005). From the data, only sandy clay loam and loam had the n values that lie between one and ten, indicating that the sorption is favorable only for these soils since they assume more than a single layer of adsorbed molecules.

The adsorption capacity, k_f (Eq 2) represents the amount of potassium held on non-specific sites that is ready to be released for uptake by plants during cropping season (Kenyanya et al., 2013). It ranged from 0.61 to 1.205 mg/kg and had a mean of 0.92±0.27 mg/kg. These values were low as compared to available soil potassium (Table 1), suggesting that part of exchangeable potassium is held on exchange sites by high bonding energy.

For Langmuir isotherm, it is assumed that the adsorption sites have equal affinities for molecules of adsorbate. Therefore, the presence of adsorbed molecules at one site will not affect adsorption of molecules at an adjacent site (Dada et al., 2012). In the present report, the maximum monolayer coverage capacity (a) from Langmuir isotherm model ranged between -1000 and 333 mg/kg. In the range, the monolayer coverage capacity of clay loam and clay soils was found to be below zero. These negative intercepts suggest that the adsorption behavior of the tested systems does not follow the assumption of the Langmuir approach. These soils also had no best fit to coefficient of determination (R^2) of Langmuir model. On the other hand, soils possessing best fit to Langmuir model (sandy clay loam and silty clay loam) indicated high monolayer coverage. The Langmuir isotherm constant (k_L) ranged from -0.011 to 0.017 L/mg. From the Temkin plot shown in Figure 6, values of Temkin isotherm equilibrium binding constant, A*T* (L/kg) ranged between 0.481 and 0.743. In this range, the clay loam soil had high binding constant and high coefficient of determination. Furthermore, low values of constant related to heat of sorption, *B* (J/mol) indicate a weak interaction between adsorbent and adsorbate supporting a mechanism of ion exchange (Dada et al., 2012). In the soils studied, the sandy clay loam and loam had low values of *B*, while clay loam, silty clay loam and clay soils had comparatively high values of *B.* High repression existed due to high clay proportion in latter soils which could hamper the

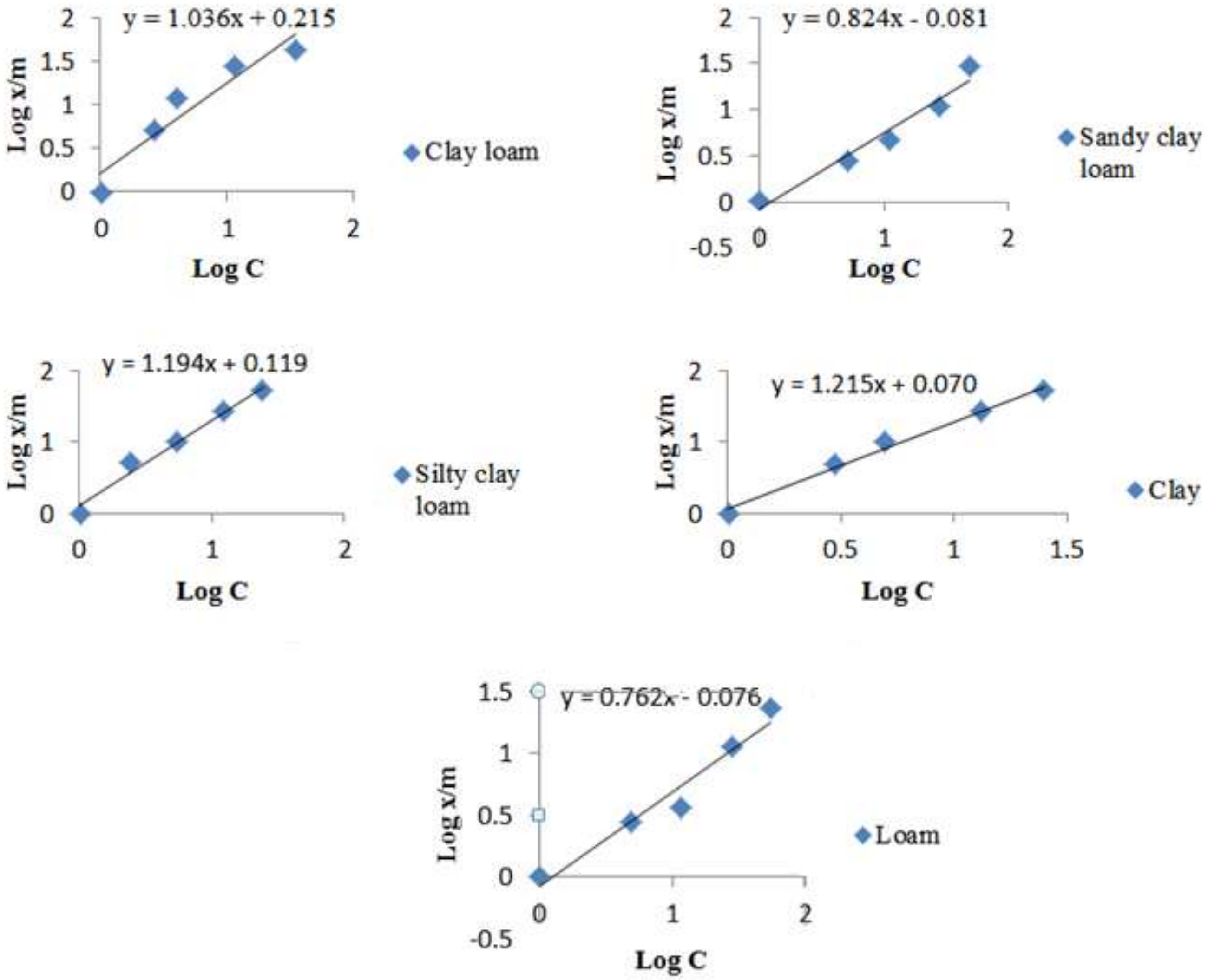

Figure 4. Freundlich adsorption isotherms for enset farming system soils of Sidama zone, South Ethiopia.

movement of K into soil solution where the uptake of nutrients takes place.

Strong and significant positive correlation occurred between percent potassium adsorbed and soil properties such as pH, clay content and CEC of soils (Table 3 and Figure 7). The results are in line with the findings of Loannou et al. (1994) who reported positive association between pH and potassium adsorption as a result of formation of new sites where competition between H^+ and K^+ for these sites decreased due to high pH. Furthermore, the results coincide with the findings of Zhang et al. (2009) who found similar correlation that soils containing high clay content and CEC fixed more K as compared to those with low clay content and CEC. The amount and type of clay minerals affect the distributions of K between exchange and solution phases.

Study on relationships occurring between adsorbed K and soil solution K may help in formulating precise fertilizer recommendations based on the adsorption capacity of the soils (Samadi, 2003).

CONCLUSION AND RECOMMENDATIONS

Although, apparent variations were observed in adsorption behaviors; all soils adsorbed the added K to high degree. This indicated the high potential of studied soils to decrease crop availability of applied K. This indicates that crops in the sampling districts were in K deficient condition and hence K recommendation should be one of the soil management choices.

A better fit of equilibrium K adsorption data to Langmuir, Freundlich and Temkin isotherms was different for different textured soils. This helps in describing the

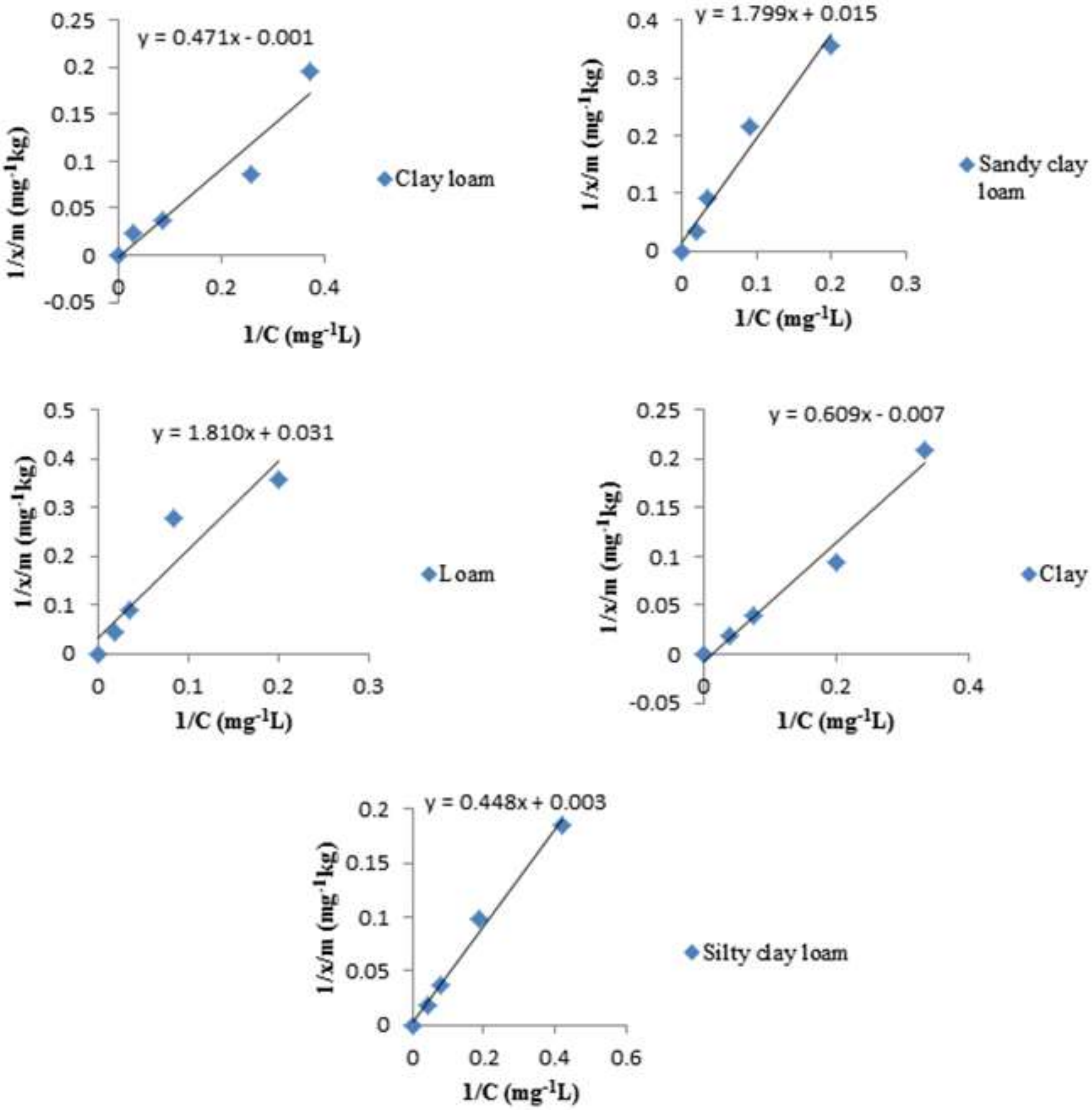

Figure 5. Langmuir adsorption isotherms for enset farming system soils of Sidama zone, South Ethiopia.

adsorption behaviors of soils based on their textures. Thus, the present research findings indicated the significance of the isotherms to assess soils K adsorption behaviors. The low 1/n values of Hula district Teticha kebele sandy clay loam and Adola kura kebele loam soils indicate low K buffering capacity and high heterogeneity of the soils. In addition to this, low values of the Freundlich adsorption capacity, k_f, as compared to available soil potassium (Table 1 and 2) for Awassa-Zuriya district clay loam and silty clay loam and for Dale district clay soils suggested the binding of part of exchangeable potassium on exchange sites by high energy. These manifested the limitedness of K availability to crops in the studied areas. Hence, it can be concluded that the studied districts would require K fertilization to replenish the soil solution K. The weak interaction between adsorbent and adsorbate indicated by constant related to binding energy (*B*) for Hula district sandy clay

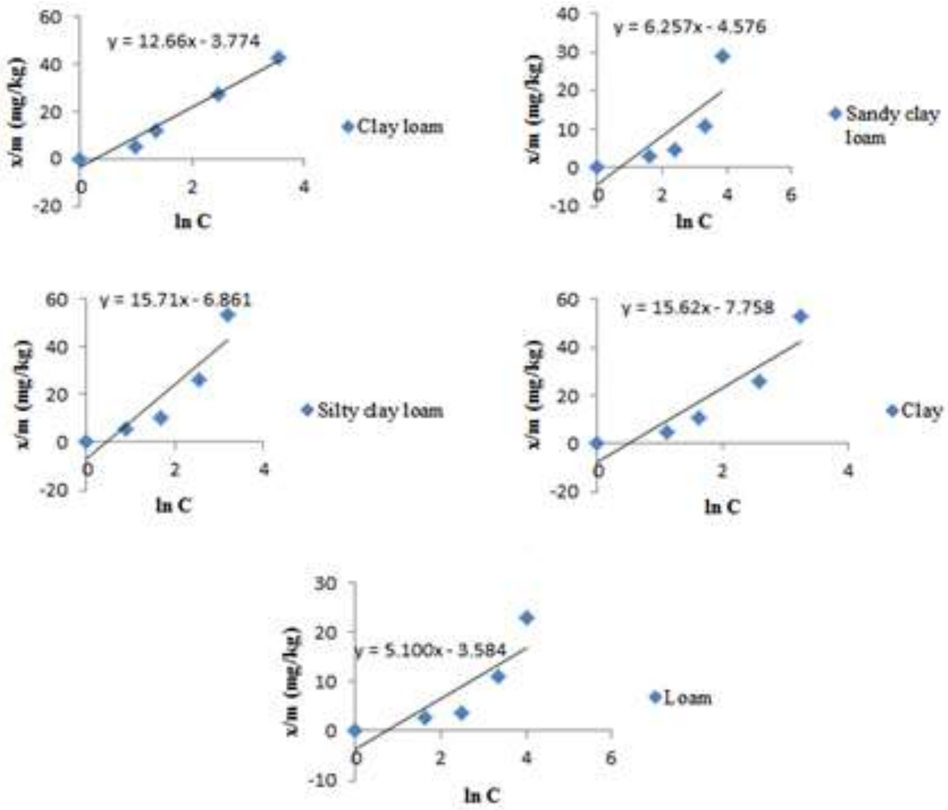

Figure 6. Temkin adsorption isotherms for enset farming system soils of Sidama zone, South Ethiopia.

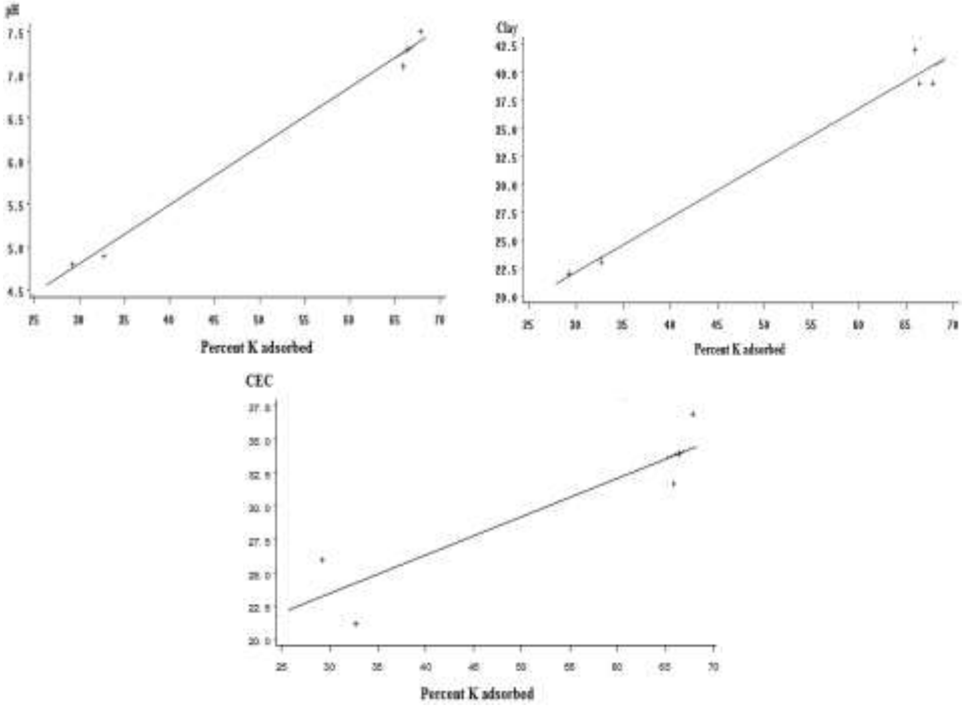

Figure 7. Correlation plots of percent potassium adsorbed with the pH (0.997**), clay (0.988*) and CEC (0.908*), respectively (**significant at P<0.001, *Significant at P<0.05).

loam and loam soils implies the exposedness of K to be lost via leaching. However, potassium availability in these soils is relatively better as compared to those soils with high values of *B* such as clay loam, silty clay loam and clay. Over all, Awassa-Zuriya and Dale district soils require K application since K flow is favored to the direction where its fixation is boosted due to high binding potential. The Hula district also requires K fertilization since less binding potential may favor leaching of K.

Significant positive correlation existed among percent K adsorbed, CEC, percent clay and pH which is convincing since the rise in contents of these soil properties creates room for large number of negative sites which adsorb and or fix K from soil solution.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

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Comparison of bulk density methods in determining soil organic carbon storage under different land use types

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The different methods used for determining soil bulk density make the result of soil organic carbon (SOC) estimation vary. The study was conducted on five land use types in Gergera watershed, Tigray, Ethiopia. This study investigates two methods (Core and Excavation methods) of soil bulk density and their relative consequence on SOC; it also evaluates which method is better for estimating SOC stock. Both methods were undertaken in top 0 to 30 cm soil depth. For comparison of bulk density methods, statistical package for social science (SPSS) version 23 was used. The result revealed that using core method, SOC was 59.30, 74.70, 64.18, 45.35 and 54.61 Mg/ha in pasture land, homestead agroforestry, crop land and woodlot respectively. Whereas, land use types were scored 56.40, 69.08, 62.20, 43.86 and 52.83 Mg/ha in exclosure, pasture land, homestead agroforestry, crop land and woodlot respectively using excavation method of bulk density determination. Although SOC stock exhibits statistically significant difference among land use types in the bulk density methods, the statistical effect was not because of bulk density methods but because of other variables in the land use types. SOC of bulk density results and mean SOC difference in each land use types were calculated using core and excavation methods. In conclusion, soil excavation method of bulk density determinations used for SOC estimation is the lower standard error. Furthermore, this work provides new insights into improving the bulk density methods and assists in the accurate estimation of soil carbon stock management.

Key words: Bulk density methods, land use types, soil organic carbon stock.

INTRODUCTION

Currently, soil carbon storage is generally expressed as a mass of carbon per unit area as recommended by the Intergovernmental Panel on Climate Change (IPCC). In measurements of bulk density, care should be taken to avoid any loss of soil from the cores (Pearson et al., 2007). Bulk density is one of the most important parameters used to calculate soil organic carbon (SOC) storage (Xu et al., 2016). To obtain an accurate

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estimation of SOC stocks in the mineral soil or organic soil three types of variables must be measured: soil depth, soil bulk density, and concentrations of organic carbon within the sample (Pearson et al., 2007).

The main reason for high uncertainty of soil organic carbon stock results from the difference in bulk density methods (Dawson and Smith, 2007; Xu et al., 2015). It is described as the weight of dry soil for a given volume. It is used to measure soil compaction. Several methods can be used to determine bulk density of soils although the most commonly practiced methods are core method and excavation method.

From Dane and Topp (2002), obtaining an unbiased measurement of soil bulk density is difficult, and different methods of measurement for soil bulk density yield different results. Therefore, selecting best estimator of bulk density is important for soil carbon stock estimation. Accurate SOC estimation results from improved sampling methods. Recent concern over the effects of small changes in soil organic carbon has been encouraged to evaluate the accuracy of methods for quantifying soil carbon (VandenBygaart and Angers, 2006).

Additionally, different soil conditions, sampling equipment, or techniques can cause unintentional biases in depth measurements and the method as well (Wuest, 2009). Different methods of bulk density result in different SOC storage under different land use types. According to the study by Petrokofsky et al. (2012), soil carbon represents the largest carbon pool of terrestrial ecosystems, and has been globally estimated to have the largest potentials to sequester carbon. However, there are numerous challenges of soil organic carbon estimation. There is need to monitor small incremental changes in soil carbon content relative to large carbon pools, long-time periods to accrue the full carbon benefits, high local variability of soil carbon content, and relatively costly soil carbon measurement procedures. Moreover, bulk density is one of the most important parameters used to calculate SOC storage (Wiesmeier et al., 2012).

Palta et al. (1969) reported several methods (core method, mercury displacement method, kerosene saturation method, kerosene displacement using water as impregnating liquid, coating the soil clods with molten wax of 65 and 100^{o}C, coating the soil clods with collodion, coating the soil clods with rubber solution of varying dilutions and excavation method) that have been used for determining bulk density**.**

However, the most common used methods are core and excavation methods in and around the study area. As a result, there is a need to select unbiased and best method of bulk density from the common methods to have an accurate information of soil organic carbon stock measurement in the different land use types.

According to Sakin (2012), very strong relationship was determined between soil organic carbon stock and soil textural groups. Moreover, few studies have focused on the effects of bulk density methods on soil organic carbon stock and the accuracy of the different methods worldwide. Best estimator method of bulk density gives good estimation on carbon stock. Therefore, the study quantitatively evaluates the SOC stock estimated from different methods of bulk density.

MATERIALS AND METHODS

Study area

The study area, Gergera watershed, is situated in Atsbi-Wonberta District, Tigray regional state, Northern Ethiopia (Figure 1). It lies in the eastern part of Tigray, about 65 km North East of Mekelle, the regional capital of city of Tigray Regional State. Geographically, it is located between 39°30' to 39°45' E and 13°30' to 13°45' N. The total size of the watershed (study area) is an area of 2302 ha. Mean elevation of the watershed ranged from 2141 to 2859 meter above sea level, with minimum (2076) in crop land and maximum (2859) in exclosure.

Farming system of the study area was dominantly subsistent involved on mixed crop-livestock production (Figure 2). Farmers of the study area do not integrate crop residues into the soil because of fuel wood and animal feed constraints, respectively. The study area experiences highly soil erosion and degradation through flooding and runoff from nearby exclosure, which is one of the main land degradation factors in the watershed. Livestock raring embraces cattle, sheep, goat and donkey. The main crops growing in the farming system were wheat (*Triticum aestivum*), maize (*Zea mays*) teff {*Eragrostis tef*), millet (*Eleusine coracana*), Faba bean (*Viciafaba*) and sorghum (*Sorghum bicolor*) with rotation year by year.

The soil type classification by its coverage in the study area is clay loam, loam, sand, sandy loam, silt and silt loam with total area of 25.8, 41.23, 741.1, 108, 1322.7 and 63.2 ha, respectively. The percentage was 1.08, 2.2, 32.19, 4.69, 57.45 and 2.74%, respectively. Some challenges in the study were climate change impacts like unseasonal heavy rainfall, rainfall interruption and others. The study area is faced with soil nutrient depletion due to continuous plowing of the cultivated lands. Continuous year by year application of chemical fertilizer may affect the soil structure and texture, not only on cultivated land but also on the other land use types; while repeated drought and weather fluctuation may damage the watershed.

In the study area, governmental and non-governmental organizations have been working in the watershed to improve the livelihood standard of the community through climate smart agriculture.

Soil sampling methods

The time of soil sampling started from 12 October to 20 November, 2015. Soil samples were collected from exclosure, pasture land, homestead agroforestry, crop land and woodlot. Soil samples were taken from every sampling plot measured 20 × 20 m at each corner and center (Bajigo et al., 2015). A total of 75 representative soil samples were collected from soil depth (0 to 30 cm) (IPCC, 2006) on each land use types; 15 plots from the 75 experimental plots were taken out and mixed together in order to have approximately 1 kg composite sample. Soil samples for chemical analysis were air dried under shade, ground using pestle and mortar and sieved to pass through 2 mm to obtain fine fractions (Yitaferu et al., 2013). Undisturbed soil samples were also collected from the same plots of the land use types using core method and disturbed excavated

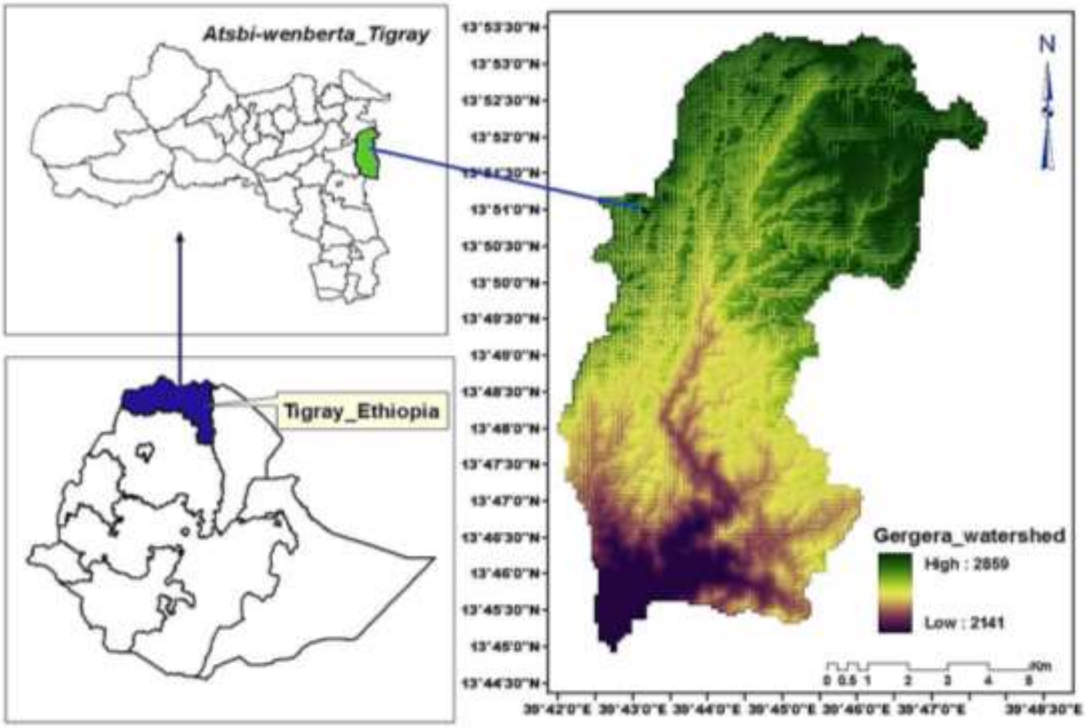

Figure 1. Gergera watershed map.

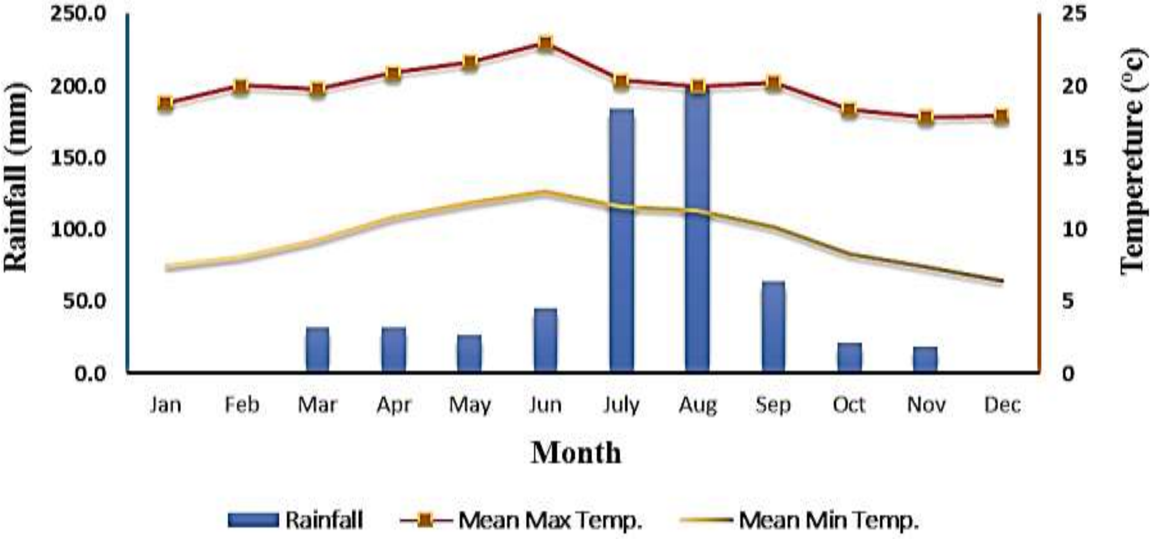

Figure 2. climate data of the study area **(**Tigray regional meteorological agency, 2015)

method for determination of bulk density from each 75 soil samples.

Core method

Five land use types were selected in Gergera watershed, Tigray, Ethiopia for soil bulk density measurement. Using core method, soil samples were collected using a core sampler made from 5 cm height and 5 cm diameter metal cylinder pressed into the soil. The cutting edge was sharpened without disturbing the height of core. The cylinder is removed, extracting a sample of known volume which is 98.12 cm^3. The moist sample weight is recorded. The sample is then dried in an oven and weighed. A total of 75 plots of undisturbed soil sample were collected from five land use types, 15 plots each (exclosure, homestead agroforestry, pasture land, crop land and woodlot) were taken from the land use types using core

Figure 3. Remove excess soil from the bottom of cylinder with knife.

sampler for determination of bulk density. While taking cores for measurements of bulk density, great care was taken to avoid any loss of soil from the cores. The weights of dry mass of soil sample were determined after oven dried at 105°C for 24 h (Hunde, 2015; Kuyah and Rosenstock, 2015), till no further changes in weight occurred.

Procedures

(1) Carefully dig up to 0 to 30 cm then drive cylindrical core ring (5cm*5cm) horizontally in the middle of 0 to 30 cm that is, 12.5 to 17.5cm.
(2) Weigh the empty cylindrical core sampler.
(3) Remove excess soil from the bottom of cylinder core sampler with knife as shown in Figure 3 and if not full, an independent measurement must be made to the volume of core sampler so as to measure accurate volume of soil.
(4) Close and pack the soil fill core sampler, label further sampled soil by known weight of the core sampler and record its total fresh weight.
(5) The packed soil fill core sampler was transported to the soil laboratory, oven dried at 105°C for 24 h (Hunde, 2015; Kuyah and Rosenstock, 2015), till no further changes in weight occur.
(6) Place a metal on each soil fill core sampler and carefully place in a plastic bag.
(7) Weigh the dry and undisturbed soil sampled by core sampler.
(8) Then bulk density is calculated by dividing the dry mass to the volume core sampler.

Excavation method

The excavation method entails digging a pit that is wide enough to collect the soil to the depth desired (Pearson et al., 2007) which is at least 0-30 cm in five land use types. A hand shovel can be used to collect material. It is important to collect material from the entire depth to avoid biasing the sample. Uniform rings were used to sample the sides of the pit for bulk density. Bulk density is determined on both the total soil and fine fraction (<2 mm). The fine fraction bulk density is critical when converting to carbon balance studies in soils with high coarse fragment content, since usually only the fine soil fraction is analyzed for carbon. The main disadvantage of the excavation method is more labor-intensive than simple coring. This method requires one to sieve out the course fragments greater than 2 mm in size, retain and weigh the weights recorded and deducted from the volume of the core.

Procedures

(1) Record empty core weights (CW).
(2) Prepare flat surface and dig a deep hole at required depth in sampling pit
(3) Press or drive core sampler into soil horizontally
(4) Take soil sample using known height and diameter core sampler; in this case take six times (Figure 4) in each five cm of 0-30 cm and place all sampled soil in a plastic bag.
(5) At the laboratory, remove the soil from the plastic bags and air dried the soil then sieve the soil (breaking up the soil clumps only) to <2 mm fraction.
(6) If sample contains rock fragments > 2 mm, dry and weigh the rock fragments that are retained on the sieve (Figure 5). Record weight of rock fragments (RF). Determine density of rock fragments (PD).
(7) Oven-dry the <2 mm soil at 105°C for 24 h.
(8) Weigh the <2 mm fraction of soil
(9) Measure and record cylinder volume (CV)
(10) Determine the oven-dry weight of the sample and calculate bulk density

Data analysis

Bulk density by core sampling was determined using Lichter and Costello (1994) (Equation 1). Whereas, Excavation method of bulk density determination was analyzed using Pearson *et al*., 2007 (Eq.2). For the soil organic carbon stock estimation (Eq.3) Pearson et al. (2007) was also used.

$$\mathrm{BD} = \frac{\text{Oven dry weight of soil}}{\text{Volume of core sampler}} \quad (1)$$

Where; BD = Soil bulk density (g/cm3)

$$\mathrm{BD} = \frac{\mathrm{ODW}}{\mathrm{CV} - \left(\frac{\mathrm{CF}}{\mathrm{PD}}\right)} \quad (2)$$

Where:
BD: bulk density of the < 2mm fraction (g/cm^3), ODW: oven dry mass of fine fraction < 2mm in gram, CV: Core volume (cm3), CF: Mass of the coarse fragment (> 2mm) in gram, PD: Density of rock fragment (g/cm3) or particle density given as 2.65g/cm3

$$SOC\ \left(\frac{\mathrm{Mg}}{\mathrm{ha}}\right) = \text{Soil bulk density} \left(\frac{\mathrm{g}}{\mathrm{cm3}}\right) \text{x soil depth (cm) x \%oc) x 100} \quad (3)$$

Where;
%OC: is percentage organic carbon concentration expressed as decimal fraction, SOC: soil organic carbon stock

RESULTS AND DISCUSSION

Soil organic carbon stock

The main reason for higher SOC stock in pasture land could be the higher organic matter content and manure. Livestock graze on rotational basis, and dung of grazing animals leaves excretion which contributes to increased organic matter. In agreement with this study, Neill et al (1997) reported that soil carbon stock values were better in pastures than in the original forests. The low soil organic carbon content of the crop land could be attributed to continuous cultivation practice and removal of crop residue; decreased fallow due to shortage of land was reported by Ahukaemere et al. (2015). Other study conducted in Southern Tigray Ethiopia by Corral–Nunez et al. (2014), shows declining level of soil organic carbon in crop land soils under current agricultural practices due to the removal and free graze of crop residues after harvesting crop plants and use of manure as an energy source for cooking. Despite the reason for the statistical difference in soil organic carbon stock in the land use types, bulk density methods that cause change on the result of soil organic carbon stock in the land use types are discussed as follows.

Core method

Soil bulk density ranges among land use types between 1.26 and 1.46 g cm^{-3}. The highest is in the exclosure and the lowest in pasture land. Statistically, soil bulk density shows significant difference among land use types ($P < 0.05$). In bulk density using core method of analysis there was statistically significant difference among land use types at $P = 0.05$. Exclosure (1.46±0.05) and crop land (1.42±0.03) have higher significant difference over pasture land (1.26±0.06) ($P = 0.004$ and $P = 0.02$ respectively). No significant different was found among exclosure, crop land, pasture land and woodlot (1.34±0.05) with homestead agroforestry (1.36±0.04). According to Hazelton and Murphy (2007), the rating of bulk density is < 1.0, 1.0 to 1.3, 1.3 to 1.6, 1.6 to 1.9 and >1.9 for very low, low, moderate, high and very high respectively. Therefore, the soil bulk density of the whole watershed was moderate. The result of soil organic carbon with their standard error using core method revealed that 59.30±3.95 Mg/ha, 74.70±9.95 Mg/ha, 64.18±6.42 Mg/ha, 45.35±3.30 Mg/ha and 54.61±6.76 Mg/ha, in exclosure, pasture land, homestead agroforestry, crop land, woodlot respectively. Statistically, soil organic carbon reveals significant difference among land use types ($P < 0.05$). Pasture land was significantly higher compared to exclosure, crop land and woodlot with their probability value of 0.036, 0.000 and 0.007 respectively. Similarly, homestead agroforestry was significantly higher compared to crop land in soil organic carbon stock.

Excavation method

Bulk density using excavated method of analysis, there were no statistically significant differences between land use types ($P < 0.05$). However, the mean difference (mean ± standard error) in between was 1.41±0.06, 1.21±0.06, 1.29±0.04, 1.35±0.03 and 1.27±0.05 for exclosure, pasture land, homestead agroforestry, crop land and woodlot respectively (Table 1). According to Hazelton and Murphy (2007), the rating of soil bulk density of the whole watershed was moderate. Even though there was no significant different in coarse fragment of the land use types, exclosure has higher mean of 43.89 gram compared to pasture land (27.10 gram), homestead agroforestry (24.41 g), crop land (26.24 g) and woodlot (25.49 g) (Table 1)

Soil organic carbon stock estimation using excavated method of bulk density

Using excavated method of bulk density calculation there were significant differences in soil organic carbon stock between land use types ($P = 0.002$). Using excavated method, the organic carbon content varied from 43.86 Mg/ha to 69.08 Mg/ha. The result of this study indicated that pasture land (69.08 Mg/ha) has significantly higher amount of soil organic carbon over crop land (43.86 Mg/ha) ($P=0.001$) and woodlot (52.83 Mg/ha) ($P=0.025$); on the other hand, homestead agroforestry land use type (62.20 Mg/ha) reveals significant difference over crop land (43.86 Mg/ha) ($P=0.012$); whereas there was no significant difference detected between exclosure (56.4

Table 1. Coarse fragment and bulk density mean comparison in different land use types.

Land use types	Variable		
	N	Coarse fragment (g)	Bulk density (g/cm^3)
Exclosure	15	43.89±9.29	1.41±0.06
Pasture land	15	27.10±5.10	1.21±0.06
H. agroforestry	15	24.41±4.38	1.29±0.04
Crop land	15	26.24±4.88	1.35±0.03
Woodlot	15	25.49±4.45	1.27±0.05
Total	75	29.43±2.71	1.30±0.02
P- value (0.05)		NS	NS

Where NS: no significant difference, on the column part of coarse fragment and bulk density; before the plus and minus represent the mean in each land use type and after is for standard error.

Mg/ha) and all the land use types; also pasture land and homestead agroforestry, between homestead agroforestry and woodlot and between woodlot and crop land.

Comparison of soil organic carbon stock results of the two bulk density methods

Bulk density methods are not only used for analyzing the estimation error of soil organic carbon stock, but also used for analyzing soil carbon concentration in laboratory. According to Venkanna et al. (2014), a relationship was established between Walkey–Black carbon and soil organic carbon stock estimated through dry combustion method using CN analyzer; and it was found that Walkley–Black carbon could recover 90% of soil organic carbon for semiarid tropical soils. Although there were no statistically significant differences among the two methods of bulk density there were mean difference between the methods (Table 2). Bulk density values established by excavation method were significantly lower compared to core sampling values (Lichter and Costello, 1994). The volume excavation method requires one to use simple and inexpensive tools, useful for sampling soils of various conditions; but this technique requires greater care in sampling technique, which increases the time required for sampling. Core sampling is a simple, fast and common technique, but is not suitable for sampling rocky, dry or wet soils (Lichter and Costello, 1994), Core sampling is the most common technique for measuring bulk density in agricultural soils. It is difficult to determine the specific reasons for the difference in bulk density values generated by the two methods. The result revealed that, there was a difference between bulk density methods (core and excavation methods) and their effects on soil organic carbon stock result. Average bulk densities measured by excavation method were 4.9, 7.5, 3.1, 3.3 and 3.3% lower than those obtained by core sampling method in exclosure, pasture land, homestead agroforestry, crop land and woodlot respectively (Table 2). The average percentage difference in all land use types was 4.4 g cm^{-3}. It is important to note that although the difference in bulk density values produced by the two methods is relatively small (3.1 to 7.5%), the implications of this difference in terms of soil organic carbon stock may be substantial. Variation of bulk density values within a method was generally low and differences between replicates at each site were not significantly different.

CONCLUSION AND RECOMMENDATIONS

Even though bulk density is the main parameter for calculating soil organic carbon stock, it is also one of the sources of uncertainty in estimating soil organic carbon stock. From the two types of bulk density methods, excavation method provides more accurate estimate of soil organic carbon stock. The core sampling method and excavation method are both useful methods for estimating bulk density of soil. Since excavation method of bulk density generates soil organic carbon values of 3.1 to 7.5% less than the core sampling method, adjustments need to be made when comparing results between these two methods of bulk density. For recommendation it is necessary to know whether the mass of the soil should include the total soil or just <2 mm fraction. Total bulk density which is total mass per total volume would be most useful to know the constitute of total mass of materials in soil. However, for the chemical analysis fine fraction (<2 mm) would tell the soil carbon stock. Therefore, excavation method of analysis is accurate to explain soil organic carbon stock in a soil. And core method is not such an explanatory like excavation method as far as we include coarse fragments when taking sample. This indicates each method has its advantages and disadvantages which need to be considered to select the most appropriate technique for a particular situation.

Figure 4. Take six times in each five cm of 0 to 30 cm, and place all sampled soil in a plastic bag.

Figure 5. Rock fragments > 2 mm.

Table 2. Average bulk density (g cm^{-3}) and SOC (Mg/ha) values for core sampling and excavation sampling methods.

Land use types	No. of observation	Core method		Excavated method		Difference in SOC (Mg/ha)
		BD (g cm^{-3})	SOC (Mg/ha)	BD (g cm^{-3})	SOC (Mg/ha)	
Exclosure	15	1.46±0.05	59.30	1.41±0.06	56.4	2.9
Pasture land	15	1.26±0.06	74.70	1.21±0.06	69.08	5.62
Homestead agroforestry	15	1.36±0.04	64.18	1.29±0.04	62.20	1.98
Crop land	15	1.42±0.03	45.35	1.35±0.03	43.86	1.49
Woodlot	15	1.34±0.05	54.61	1.27±0.05	52.83	1.78

BD; Bulk density.

CONFLICT OF INTERESTS

The author declare that there is no conflicts of interest.

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