Occurrence of organo-mineral complexes in relation to clay mineralogy of some Sri Lankan soils

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Abstract: The major products of the interactions of soil minerals with organic components and organisms are organo-mineral complexes. This study was conducted to investigate the naturally occurring organo-mineral complexes and to develop a relationship between extractability of carbon with type and amount of clay minerals present in soils. Representative soil samples of Ultisols of four different locations and an Alfisol were collected. The clay fraction (< 2 μm) was separated and organic substances were extracted and determined from the separated clay fractions sequentially by 0.1M NaOH (C1), HF/HCl (C2), 0.1M NaOH (C3), and the residue (C4). Clay mineralogy of the soils was determined using X-ray diffraction technique. Amounts of C present in stable organo-mineral complexes were 20% to 48% of the total C. Amounts of organic C present in organo-mineral complexes were closely related with the amounts of total C extracted from the soils. Regression analysis showed a significant relationship between clay % and the organic C present in the organo-mineral complexes. Clay minerals of kaolinite, illite and traces of vermiculite were present in Ultisols while kaolinite, illite and smectite were present in Alfisols. Presence of smectite in Alfisol contributed significantly for the retention of C with the organo-mineral complexes. This study showed that the total organic C content, percentage of clay and the presence of smectite were the contributing factors for the amount of organo-mineral complexes present in these soils.

Key words: Alfisols, clay minerals, organo-mineral complexes, sequential extraction, Ultisols.

INTRODUCTION

Minerals, organic matter, and micro-organisms are integral parts of the pedosphere and related environments. Interactions of organic and inorganic components of soils govern mechanisms of mineral weathering reactions, abiotic catalysis of the formation of humic substances, formation of organo-mineral complexes, enzymatic activity, soil structure stability and transformations of metals and organic pollutants in the terrestrial environment.[1] Soils and clays interact with organic compounds to form organo-mineral complexes through a number of different mechanisms. Soil organic matter can be intimately associated with mineral particles of various sizes. The interaction of organic substances with clay has a multitude of consequences that are reflected in the physical, chemical and biological properties of the soil matrix. Formation of organo-mineral complexes in Andosols showed low mineralization activity in organic matter.[2] Organic matter in Vertisols contributes to soil colour by formation of organo-mineral complexes and affects morphological, physico-chemical, biological and biochemical properties.[3] Trace metals in the soil solution partly occur in the organically bound forms.[4] Organic ligands on one hand could enhance the availability of trace elements to plants and on the other hand it could reduce the toxicities through binding.[5] Pesticides of various types are adsorbed by clay as well as organic matter and rendered inactive. The activities of enzymes in soil may be modified through adsorption to clay. Obviously, there are wide gaps in our knowledge of naturally occurring resistant organic compounds, which constitute one of the most abundant natural resources in the terrestrial environment.

Adsorption of organic molecules in soil is influenced by several factors; the most important ones are the type and amount of clay mineral and the amount of soil organic matter. Generally 2:1 type clays can adsorb greater amounts of organic materials than 1:1 types due to large specific and high charge densities in 2:1 type clays. Products resulting from interactions of organic matter with inorganic soil constituents are likely to affect the physical, chemical and biological properties of soils. The extractability of organic matter by an extracting solution depends on the extent to which the organic matter is linked to the soil mineral fraction.[6] The extent to which humus materials are associated with the soil inorganic colloids, and especially the strengths of these associations are important for the design of a dissolution procedure.[7] In his review Hayes[8] has named a variety of single extractants, such as alkali, dilute and concentrated mineral acids, neutral and organic reagents, to separate soil organic matter.

Information on naturally occurring organo-mineral complexes and its applications to soil properties is very scarce for Sri Lankan soils. The nature and stability of organo-mineral complexes in soils determine rate of release of nutrients, heavy metals and toxic pollutants, activities of microorganisms and enzymes and many more reactions taking place in soils. Extraction of organic matter from organo-mineral complexes is difficult and depends on the stability of these complexes. The Red Yellow Podsolic soil (Ultisol) is the model soil group in wet zone and the soils found in Kandy district, which belongs to the agro-ecological zone of mid country wet zone and is reported to have 1 -2 % organic matter content.[9] Due to the accumulation of organic matter, Ultisols found in Nuwara Eliya district (upcountry wet zone) have 4 - 8% of organic matter content.[10] Reddish Brown Earth (Alfisols) is the most widespread great soil group in Sri Lanka and it is the model soil group of the dry zone of Sri Lanka. Decomposition rate of organic matter is very high in Alfisols resulting in very low percentages of organic matter. This study was conducted to evaluate the extractability of C and N from organo-mineral complexes and to develop relationships between amounts of C extracted with the type and amount of clay minerals present in some selected soils of Sri Lanka.

METHODS AND MATERIALS

Ultisols from four different locations; i.e., Meepilimana (MP), Sita Eliya (SE), Kiribathkumbura (KK), Peradeniya (PD) and an Alfisol from Mahailluppallama (MI) were collected at two depths, *viz*; 0 – 15 and 15 – 30 cm for the study. Considerations were given to organic matter contents in the undisturbed soils when these locations were selected. The locations MP and SE were from Nuwara Eliya district, KK and PD from Kandy district and MI was from Anuradhapura district. Soil samples were air dried and passed through a 2 mm mesh sieve prior to laboratory analysis.

Soil analysis: Soil pH was measured in 1M KCl solution (1:2.5, soil: solution). Organic C content was determined by digestion with acid dichromate solution and titrating with ferrous ammonium sulfate using Walkley-Black procedure.[11] Clay content of the soils was determined by the Pipette Method.[12] Soil total N was determined by the micro Kjeldhal method.[13]

Separation of clay fraction and extraction of organic substances: Clay fraction needed for extraction of organic substances in naturally occurring organo-mineral complexes was separated by sedimentation without any pretreatments to remove flocculating-cementing agents.

Sequential extraction of organic substances: Organic C and N contents in separated clays were extracted by the sequential extraction procedure described in a previous study.[14] Four extractions were conducted sequentially for one sample of 0.5 g of clay. The first fraction of the organic substances was extracted by treating with 0.1M NaOH (C1). The residue of the clay sample was then treated with HF / HCl solution to dissolve the silicate minerals (C2). The organic substances made available by HF/HCl solution were extracted again using 0.1M NaOH (C3). Finally the organic substances in the residual clay fraction was also determined (C4).

Clay Mineralogical Analysis: Clay fraction of the soils was separated after giving pretreatments to remove cementing-flocculating materials in soils.[15] The soluble salts were removed by washing the sample with distilled water and 30% hydrogen peroxide was used to remove organic matter. Removal of iron oxides was done by adding sodium citrate-bicarbonate-dithionite solution and heating it to 80 $^\circ$C. After saturating the soil sample with 1 M NaCl to facilitate dispersion, clay fraction ($<$ 2 μm) was separated by sedimentation.[16] Clay fractions were saturated with K and Mg by washing with 1M KCl, and mixture of 0.5M magnesium acetate / 0.5M $MgCl_2$, respectively. K and Mg saturated clay samples were mounted onto glass slides and dissolved with acetone. Glycerol solvated samples were obtained by adding few drops of glycerol onto the Mg-saturated clay slide. X-ray diffraction test was carried out by the SHIMADZU XD-7A X-ray diffractometer operated at the potential of 40 kV and the amperage of 30 mA, producing Cu Kα radiation at a wave length of 1.54 Å equipped with Ni filter. The samples were made using a continuous scanning technique between 2° 2θ and 30° 2θ and a scan speed of 0.02° 2θ per min. Crystalline phases were identified using the Powder Diffraction File.[17]

RESULTS AND DISCUSSION

The soils collected from Meepilimana and Sita Eliya showed the lowest pH values and the highest organic C % among the tested soils (Table 1). These two soils also showed higher clay % than the other three soils. The pH values of the Kiribathkumbura and Peradeniya soils were in the range of 4.2 to 5.1 and the Mahailluppallama soil which belongs to the order Alfisols showed a pH close to 6.0. Similar pH values and clay % were observed at the two depths in each soil. Organic C and N percentages showed higher values for the surface soils than sub-surface soils. The organic matter content of all mineral soils decreases rapidly from the surface to lower depths.[18]
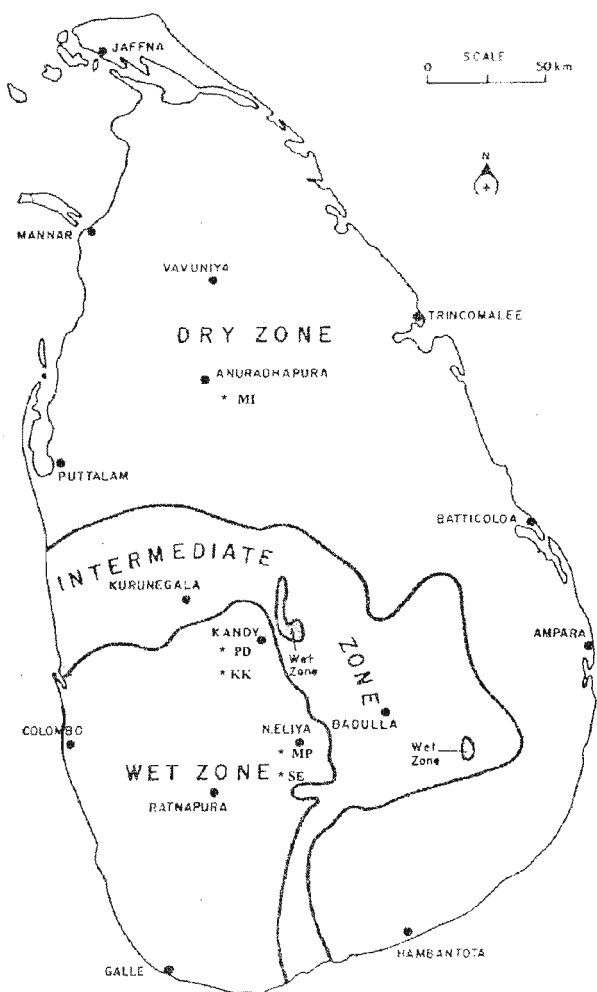
Figure 1: Site map indicating the locations of the samples collected (MP = Meepilimana, MI = Mahailluppallama, SE = Sita Eliya, KK = Kiribathkumbura, PD = Peradeniya)

Results of organic C and N analyses in different soil extractions are presented in Table 2. Physical fractionation according to size and density of soil particles emphasizes the importance of interactions between organic and inorganic soil components in the turnover of organic matter. It allows the separation of free and occluded uncomplexed organic matter and of primary and secondary organo-mineral complexes.[19] The organic C and N amounts extracted by 0.1M NaOH indicate the loosely bound organic substances in soils. It was reported that 60% - 72% of total C was extracted in 0.1M NaOH.[20] The amounts of organic C extracted in 0.1M NaOH from the soils tested ranged between 54% - 80% of the total C (Table 2). The repeated shaking of samples with HF / HCl treatment removes the silicate minerals and facilitates the extraction of organic substances by NaOH solution. Results indicated that 3% to 10% of the organic C was removed with the silicate minerals by HF / HCl solution (Table 2). Amounts of

organic C extracted after removal of silicate minerals were in the range of 7% - 25% of the total C. However the results of the residue fraction indicate that 6% - 30% of the total C remained with the clay fraction. Presence of organic substances in the clay fraction of soils even after the treatment of HF/ HCl solution indicates the presence of highly-stable organo-mineral complexes in these soils. Radiocarbon dating has shown that some of the organic matter in soil are extremely stable.[21] Amounts of organic C extracted by different solvents of the sequential extraction procedure indicated the presence of organo-mineral complexes with varying stabilities in clay fraction of the soils. Amounts of C extracted from clay fractions using C2, C3 and C4 treatments indicate the presence of stable organo-mineral complexes and accounted for 20% to 48% of the total C. Organic substances are retained in soils as insoluble polymeric complexes of humic and fulvic acids, polymeric complexes of humic and fulvic acid bound together by di- and tri-valent cations, substances held on clay mineral surfaces and substances held within the interlayers of expanding-type clay minerals.[22] Amounts of organic N extracted using different solvents followed a similar trend as with organic C (Table 2). As in organic C, the removal of silicate minerals by HF / HCl solution increased the amount of N extracted. Results of the sum of N in C2, C3 and C4 treatments point to the role of organo-mineral complexes in interfering with the extraction of soil N. Amounts of N in this combined fraction ranges from 41% to 65% of the total N in soils. Evidence obtained by extraction of a large number of soils with various reagents suggests that soil clays may protect organic N components from extraction.[23, 24] Table 2 also indicates the C/N values calculated from C and N presented in different extractions of the soils at two depths. The results showed that C/N value for C1 treatments was higher than the (C2 + C3) or C4 treatments and the C/N values for C1 ranged from 12.1 to 3.3. Higher C/N values were observed in surface soils than in the subsurface soils. This may be due to the higher organic matter content in surface soils than in the sub-surface soils (Table 1). Brady[25] suggested that generally, C:N ratio of sub soils is narrower than the corresponding surface soils. The narrower C: N ratios obtained for the C2 + C3 or C4 than C1 (Table 2) indicate the involvement of N for stabilizing the organo-mineral complexes. Apart from that presence of NH_4^+-N is also responsible for narrower C:N ratios.[26]

The amounts of organic C present in C1, C2, C3 and C4 extractions (total C) and organic C present in C2, C3 and C4 extractions (C present in organo-mineral complexes) of the clay fractions of 0 -15 cm depth is shown (Figure 2). Total organic C contents indicated that soils collected from Meepilimana and Sita Eliya
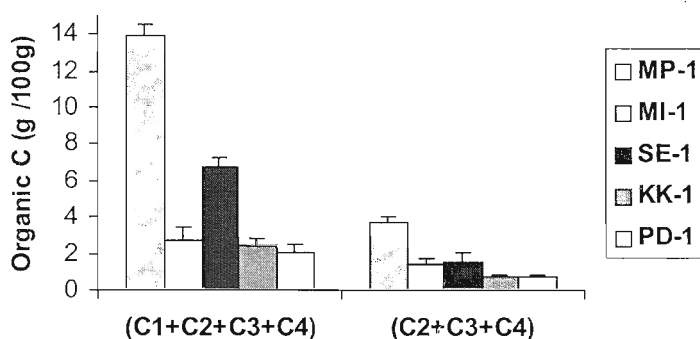
Figure 2: Amounts of total organic C (C1+C2+C3+C4) and organic C present in organo-mineral complexes (C2+C3+C4) of the soils. (MP-1= Meepilimana depth 1, MI-1= Mahailluppallama depth 1, SE-1= Sita Eliya depth 1, KK-1= Kiribathkumbura depth 1, PD-1= Peradeniya depth 1). Bars represent standard errors of the mean.

Table 1: Some important physical and chemical properties of the experimental soils.

Location	Depth (cm)	Agro-ecological zone	Sample	pH(1M KCl)	Carbon (%)	Nitrogen (%)	Clay (%)
Meepilimana	0 – 15	WU3	MP-1	3.8	8.24	0.84	43.2
	15 – 30		MP-2	3.9	4.12	0.46	36.3
Mahailluppallama	0 – 15	DL1	MI-1	6.0	1.52	0.32	30.5
	15 – 30		MI-2	5.9	0.96	0.18	28.3
Sita Eliya	0 – 15	WU3	SE-1	3.9	2.48	0.32	40.3
	15 – 30		SE-2	3.8	1.52	0.20	37.2
Kiribathkumbura	0 – 15	WM2	KK-1	4.4	1.20	0.18	32.4
	15 – 30		KK-2	4.2	1.23	0.14	29.6
Peradeniya	0 – 15	WM2	PD-1	5.1	1.81	0.20	30.5
	15 – 30		PD-2	4.5	0.96	0.18	28.5

Table 2: Amounts of organic carbon (C) and nitrogen (N) extracted by different solutions from the clay fraction of the soils.

| Soil	0.1M NaOH			HF/HCl + 0.1M NaOH			Residue		
	C (g/100g)	N (g/100g)	C/N	C (g/100g)	N (g/100g)	C/N	C (g/100g)	N (g/100g)	C/N
MP-1	10.19	0.90	12.1	2.80	0.52	5.4	0.90	0.28	3.2
MP-2	2.74	0.57	6.1	0.90	0.28	3.2	0.38	0.15	2.5
MI-1	1.31	0.21	7.4	0.75	0.23	3.3	0.69	0.15	4.6
MI-2	0.73	0.26	3.3	0.53	0.23	2.4	0.20	0.05	3.8
SE-1	5.15	0.56	12.7	0.66	0.20	3.4	0.89	0.28	3.2
SE-2	1.93	0.36	5.8	0.76	0.18	4.3	0.18	0.08	2.3
KK-1	1.58	0.16	7.8	0.37	0.18	2.1	0.39	0.06	6.8
KK-2	1.30	0.17	7.1	0.23	0.16	1.4	0.27	0.07	3.8
PD-1	1.31	0.20	6.2	0.37	0.17	2.2	0.42	0.07	6.0
PD-2	1.17	0.17	6.0	0.15	0.18	0.8	0.26	0.05	5.4
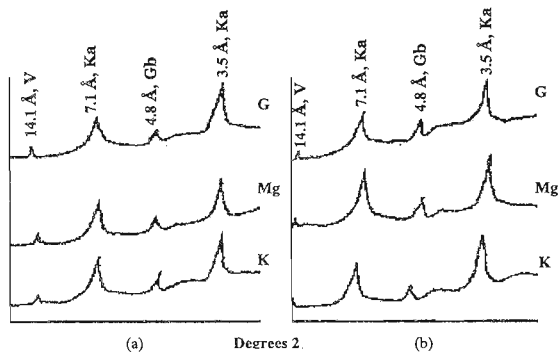
Figure 3: X-ray diffractograms of the clay fraction of Meepilimana soils {(a). 0 – 15 cm depth and (b). 15 – 30 cm depth}. Ka = Kaolin, V = Vermiculite, Gb = Gibbsite, K= K-saturated, Mg= Mg-saturated, G= glycerol-solvated.
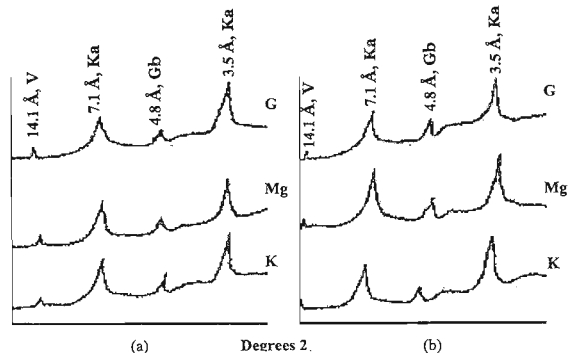

Figure 5: X-ray diffractograms of the clay fraction of Sita Eliya soil {(a). 0 – 15 cm depth and (b). 15 – 30 cm depth}. Ka = Kaolinite, V= Vermiculite, Gb = Gibbsite, K= K-saturated, Mg= Mg saturated, G= glycerol-solvated.
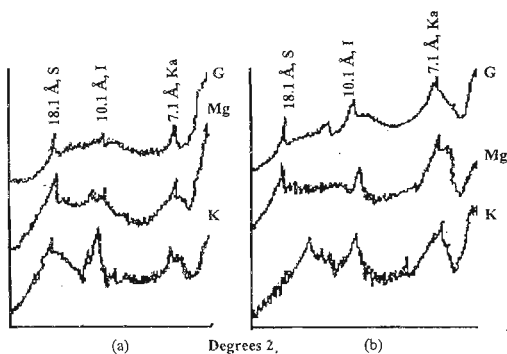

Figure 4: X-ray diffractograms of the clay fraction of Mahailluppallama soil {(a). 0 – 15 cm depth and (b). 15 – 30 cm depth}. Ka = Kaolinite, S = Smectite, I = Illite, K= K-saturated, Mg= Mg-saturated, G= glycerol-solvated.
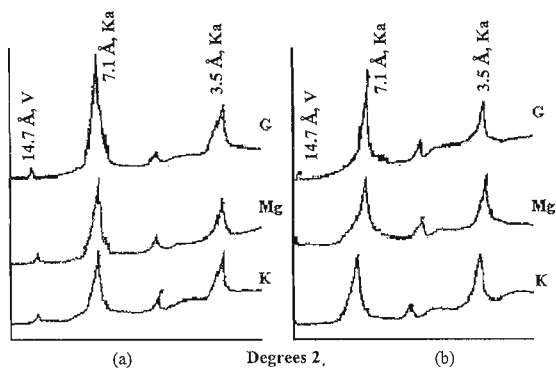

Figure 6: X-ray diffractograms of the clay fraction of Kiribathkumbura soil {(a). 0 – 15 cm depth and (b). 15 – 30 cm depth}. Ka = Kaolin, V = Vermiculite, K= K-saturated, Mg=Mg-saturated, G= glycerol-solvated.
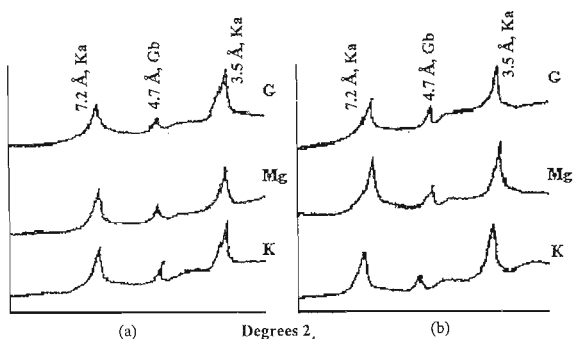

Figure 7: X-ray diffractograms of the clay fraction of Peradeniya soil {(a). 0 – 15 cm depth and (b). 15 – 30 cm depth}. Ka = Kaolin, Gb = Gibbsite, K= K-saturated, Mg= Mg-saturated, G= glycerol-solvated.

were significantly different from each other and showed significantly higher organic C amounts than the other three soils. Meepilimana soils showed significantly higher amounts of organic C present in organo-mineral complexes than in the other soils. This could be explained by the presence of the highest total C amounts and the highest clay % in this soil. The soils collected from Mahailluppallama, and Sita Eliya, have significantly higher organic C amounts with the organo-mineral complexes than those from Kiribathkumbura and Peradeniya (Figure 2). The amount of clay % or the amount of total organic C present in these soils could not explain this observation.

Under similar climatic conditions, soil texture is known to be an important factor in the determination of organic C content in soils. It was stated that higher

the clay content the lower will be the decomposition rates of organic matter. In the present study, regression analysis showed a significant relationship ($r^2 = 0.66$) between clay % and the organic C present in the organo-mineral complexes. This revealed that amount of clay plays a significant role in retaining stable organic C contents in soils.

X-ray diffractograms of Meepilimana soil are shown (Figure 3). A peak at 7.1 Å in K-saturated, Mg-saturated and Mg-Glycerol solvated sample signifies the presence of kaolinite. A peak near 14Å in the Mg-saturate, Glycerol solvated and K-saturated samples indicate the presence of vermiculite. Presence of gibbsite was indicated by the peak at 4.8 Å in the MP X-ray diffraction pattern. X-ray diffraction patterns of 0-15 cm depth and 15-30 cm depth of Mahailluppallama soil are shown (Figure 4). A peak near 14 Å in the Mg-saturate sample and shift of the 14 Å peak to 18 Å following solvation with glycerol indicate the presence of smectite (JCPDF No. 13-135) and the presence of illite was indicated by the peak at 10.0 Å (JCPDF No. 43-685). Smectite, illite and kaolinite are the dominant clay minerals present in MI soils. X-ray diffraction pattern similar to Meepilimana soil was observed in Sita Eliya soil (Figure 5) and the dominant clay minerals present were vermiculite and kaolinite. The X-ray diffraction pattern of Kiribathkumbura soil indicated the presence of kaolinite, vermiculite and gibbsite (Figure 6). Peradeniya soil indicated the presence of clay minerals of kaolin and gibbsite type (Figure 7). Clay mineralogical difference observed between the two soil orders used, i.e., Alfisol (MI) and Ultisols (MP, SE, KK and PD) was the presence of smectite in the Alfisols. Amount of total organic C present in the MI soil was significantly lower than the SE soils but the amounts of organic C present with the organo-mineral complexes in MI and SE soils were comparable (Figure 2). This indicated the contribution of expanding type clay minerals on the stabilization of organic C in MI soils. Many workers[28, 29] have reported adsorption of organic poly-anions in the interlayer spaces of expanding clays.

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