International Journal of Environment and Pollution Research
Vol.3, No.2, pp.69-83, June 2015
Published by European Centre for Research Training and Development UK (www.eajournals.org)

SULPHUR STATUS OF SELECTED CRUDE OIL POLLUTED AND UNPOLLUTED SOILS IN BAYELSA, NIGER DELTA, NIGERIA

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ABSTRACT: *Soil sulphur status provides valuable information about its bioavailability and potential environmental consequences. Sulphur forms (total, organic, water soluble, adsorbed, organic matter bound and elemental S) of selected crude oil polluted and unpolluted soils in Bayelsa, Niger Delta, Nigeria were evaluated. Experimental design was a 3 x 2 x 2 factorial of location, soil depth and pollution status arranged in a randomized complete block set-up with 3 replications. Also relationship between S forms and selected soil properties was estimated using correlation and regression analyses. Averaged over soil depths and pollution status, soil S varied distinctly (LSD $_{0.05}$) as 1.59, 2.57 and 1.98 (elemental S), 26.25, 35.15 and 25.67 (water soluble S), 6.07, 6.34 and 6.58 (organic matter bound S), 51.71, 58.08 and 53.42 (adsorbed S), 1014.87, 998.90 and 940.13 (organic S) and 1094.92, 1092.14 and 1019.22 mg kg^{-1} (total S) in Agudama-Epie, Elebele and Imiringi respectively. Also interactions of location x soil depths x pollution status yielded best elemental S (3.79 mg kg^{-1}) in surface depth of polluted soil at Elebele, water soluble S (43.34 mg kg^{-1}), organic matter bound S (10.00 mg kg^{-1}) and adsorbed S (68.00 mg kg^{-1}) in the subsurface depths of unpolluted soils at Agudama-Epie, Imiringi, and Elebele respectively. Best organic (1228.06 mg kg^{-1}) and total S (1296.73 mg kg^{-1}) were in the surface depth of unpolluted soils in Agudama-Epie. Soil OM, pH, sand, silt, clay, P, N, Mg, Ca, K, Na and ECEC correlated with S forms. Besides clay which accounted for more than 40% of water soluble and adsorbed S, other S forms were poorly predicted by the soil properties. In general, concentrations of most S forms were higher in unpolluted than polluted soils, probably due to favourable conditions for S transformation and existence of important S sources other than crude oil in the former than the later.*

KEYWORDS: Sulphur, Polluted, unpolluted soils, Bayelsa and Niger Delta

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INTRODUCTION

Sulphur is a secondary nutrient element that is a component of some amino acids especially cystine, cysteine and methionine. It is associated with vitamins and other biomolecules and thus important in the growth and development of all forms of lives (Solomon et al., 2005). It activates many enzyme and coenzyme systems and essential for the characteristic odour and flavor of plant species as onion, mustard and garlic (Havlin et al., 2012). Due to its high reactivity, it often influences cation availability and metal pollutant mobility in soils (Martinez et al., 2002; Hamburg et al., 2003).

Sulphur could be of natural or anthropogenic origin, with natural sources including rock mineral weathering and the decomposition of organic sulphur compounds. Anthropogenic sources include acid rain deposition due to SO_2 emission from the combustion of fossil fuel, environmental disaster involving hydrocarbons, coal combustion, vulcanization of rubber and the over-use of chemical fertilizers and pesticides (Puacz et al., 2001; Sager, 2012). Two forms of soil sulphur include organic and inorganic (Scherer et al., 2012), with the former consisting of more than 90% and the later less than 5% of total soil sulphur (Likens et al., 2002; Schroth et al., 2007; Balik et al., 2009; Kulhanek et al., 2011; Forster et al., 2012). Organic sulphur includes hydroiodic reducible and carbon-bonded S species (Mcbride, 1994). Hydroiodic reducible S consists of ester sulphates (C-O-S) especially sulphated polysaccharides, Cholin sulphates, phenolic sulphates (Edward et al., 1998) and sulphamate species (C-N-S) while carbon-bonded species include the amino acid containing fractions like cysteine and methionine (Forster et al., 2012). Inorganic S consists of sulphate and sulphite S (Yunas et al., 2010; Havlin et al., 2012) with sulphate S including the water soluble and adsorbed S fractions and involves the fraction immediately available for plant and microbial uptake or lost by leaching (Scherer et al., 2012).

Techniques of estimating soil sulphur forms vary, with the most common types including the indirect wet-chemical degradation technique in which S-compounds are reduced to H_2S by hydroiodic acid (HI) (Kowalenko, 1993). Another includes the synchrotron-based S-K edge X – ray absorption near edge structure (XANES) spectroscopy that provides a means of estimating directly the various S species based on the energy required for core electron transitions to bound states or ejection into a continuum (Morra et al., 1997; Xia et al., 1998; Solomon et al., 2003). Its utility has been widely reported in S speciation and quantification in petroleum (Waldo et.al., 1991), marine sediments (Vairavamorthy et al., 1991) and size separates of mineral soils (Pietzel et al., 2003; Solomon et al., 2003). Though very efficient methods, the XANES technique has been reported superior to the HI due to its tendency to overcome some inaccuracies in calculation of S fractions (Pietzel et al., 2003) and the ability to identify intermediate oxidation states of sulphur (Solomon et al., 2005). However, due to the complexity of the Xanes method, use of the HI method has enjoyed the greatest acceptability in most laboratories.

Soil properties especially pH, redox potential, OM, P, N, sand, silt, clay, Ca, Mg, K, Na and ECEC affects soil sulphur forms (Biswas et al., 2003; Isitakhale et al., 2013). Wang et al. (2006) have reported a significant correlation between total organic sulphur with soil nitrogen and organic carbon. Also Increased S concentration in the clay than sandy soil ascribable to greater leaching tendency in the later than former has been reported (Yunas et al., 2010). Equally, high concentration of all S forms in silt and clay than sand fractions have been indicated and attributed to the high adsorption capacity of the fine textured fractions (Shamsuddin and Bhatti, 2001; Solomon et al., 2011).

In the Niger Delta and Bayelsa State in particular, large quantities of sulphur is usually discharged into the environment during crude oil spillage. It has been noted that heavy petroleum especially crude oil contains many complex hydrocarbon molecules as well as such impurities as sulphur, nitrogen, polycyclic aromatics and asphaltens, heavy metals and metal salts (Martinez et al., 2002;

Hanni and Mustafa, 2004; Mohammed et al., 2012). A mechanistic understanding of S speciation dynamics in the soil could be useful in estimating their environmental consequences. However, information on speciation, turnover dynamics, and ecological significance of specific S-compounds in tropical and subtropical agro-ecosystems is currently limited (Neptune et al., 1975; Solomon et al., 2001; Moller et al., 2002). The main objectives of the present study were therefore to evaluate the sulphur forms of selected crude oil polluted and unpolluted soils in Bayelsa, Niger delta and correlate with them selected soil properties.

MATERIALS AND METHODS

Study Location

The study locations were Agudama-Epie, lying between Latitudes 4^o 55^1 29^{11} and 4^o 57^1 32^{11} N and Longitudes 6^o 15^1 51^{11} and 6^o 18^1 53^{11} E, Elebele (Latitudes 4^o 51^1 22^{11} and 4^o 54^1 25^{11} N and Longitudes 6^o 20^1 35^{11} and 6^o 25^1 39^{11} E) and Imiringi (Latitudes 4^o 85^1 25^{11} and 4^o 89^1 27^{11} N and Longitudes 6^o 37^1 25^{11} and 6^o 39^1 27^{11} E) in Bayelsa State, Niger Delta, Nigeria. The areas have a mean annual rainfall range of between 2000-4500 mm, mean daily temperature range of $28\text{-}32^{oC}$ and mean relative humidity range of 78-85% (Sutton and Loganathan, 1986). Climax vegetations included cassava for unpolluted and spear grass (Imperata Cylindrica) for polluted sites in Imiringi, Eupathora Odorata for both polluted and unpolluted sites in Agudama Epie and Elebele. Soils of the area are underlain by the Meander Belt deposits (Okezie, 1974) and classified as Typic Troposamment in Agudama-Epie and Oxic Dystropepts in Elebele and Imiringi. Main economic activities of the area include fishing, farming, trading and crude oil exploitation.

Sample Collection and Analyses

Triplicate soil samples were collected from two depths (0-15 and 15-30cm) each of crude oil polluted and unpolluted sites in Agudama-Epie, Elebele and Imiringi, in Bayelsa State and giving a total of 18 samples. The 0-15cm depth represents the surface while the 15-30 cm the subsurface depth. The samples were air dried, sieved using a 2 mm diameter mesh and the fine earth soil samples stored for laboratory analysis. Sub samples of the fine earth soil fractions were then analyzed for particle size after dispersion with calgon using the hydrometer method (Gee and Or, 2002), pH in 1:2.5 soil/solution (water and 1N KCl) ratios (Thomas, 1996), ECEC (Thomas, 1996), available P (Olson and Sommers, 1982) and OM (Nelson and Sommers, 1996). Sulphur forms were determined as follows: Inorganic sulphates (water Soluble and adsorbrd sulphate) were determined by sequential extraction using the methods proposed by Freney et al. (1970). In this, water soluble sulphate was determined by weighing 1 g fine earth soil sample into a centrifuge tube and shaking with 10 ml of distilled water in a reciprocating shaker for 30mins. The suspension was centrifuged for 30 mins at 12000 x g and the supernatant decanted into a test tube without disturbing the soil at the bottom of the centrifuge tube. To the soil residue left in the centrifuge tube was added 0.032 mol/litre NaH_2PO_4 and treated as for the water soluble sulphate above. All the extractions were in duplicates and the extracts further filtered through a 0.45 μm cellulose acetate filter (Macherey & Nagel, Düren, Germany) before measuring. Sulphate concentrations in the extracts were determined by anion chromatography. The NaH_2PO_4 extract represented the adsorbed sulphate. Total S was measured with a CNS analyzer (Eurovector EUROEA, Redavalle, Italy) while organic S was obtained as the difference between total S and the inorganic S

Statistical Analyses

All data generated for the sulphur forms were subjected to analysis of variance (ANOVA) and treatment means separated using least significant difference (LSD) at 5% probability. Correlation and regression analyses were used to determine the relationships between sulphur forms and selected soil properties. All analyses were conducted using Genstat statistical package (Buyse et al., 2004).

RESULTS

Characterization of the Soils Studied

Mean sand, silt and clay ranged from 385-520, 320-505 and 60-160 in unpolluted and 375-508, 318-511 and 100-186 g kg^{-1} in polluted soils respectively (Table 1). Mean soil pH values for Agudama-Epie, Elebele and Imiringi were 5.66, 5.70 and 5.44 in unpolluted and 5.20, 5.56 and 5.50 in polluted soils respectively. In each location, distribution pattern of the pH values for surface and subsurface depths of polluted and unpolluted soils was irregular. Mean organic matter ranged from 15.83-21.85 and 19.46-30.27 g kg^{-1} for polluted and unpolluted soils respectively and with surface better than subsurface depths in the various locations. Amongst locations, organic matter was better in Agudama-Epie for polluted and Imiringi for unpolluted soils. Total N ranged from 0.28-0.61 (mean = 0.45-0.50 g kg^{-1}) and 0.28-0.68 (mean = 0.47-0.54 g kg^{-1}) in polluted and unpolluted soils respectively, with concentration in unpolluted better than polluted soils. Irrespective of pollution status, concentrations were higher in the surface than subsurface depths of all locations. Ranges of available P were 12.43-17.87 (mean = 13.41-16.53 mg kg^{-1}) and 16.30-21.80 (mean = 18.12-21.00 mg kg^{-1}) for polluted and unpolluted soils respectively, with surface higher than the subsurface depths. In both soil depths and pollution status, concentrations were better in Agudama-Epie than the other locations. Mean exchangeable Ca, Mg, Na and K of polluted soils ranged from 8.60-16.74, 2.74-5.74, 0.04-0.05 and unpolluted from 0.02-0.03 cmol$_c$ kg^{-1} and 14.55-19.50, 3.26-4.68, 0.05-0.06 and 0.03-0.03 cmol$_c$ kg^{-1} respectively. Exchangeable Ca followed by Mg dominated the exchangeable cations in the soils. Mean ECEC varied as 22.54, 13.87 and 13.35 in polluted and 25.90, 20.42 and 18.53 cmol$_c$ kg^{-1} in unpolluted soils for Agudama-Epie, Elebele and Imiringi respectively, with Agudama-Epie higher than others. In most locations, ECEC was higher in the surface than the subsurface soil depths.

Soil Sulphur

Total, organic, adsorbed, organic matter bonded, water soluble and elemental sulphur contents of the soils are presented in Table 2. Total sulphur was distinctly (LSD $_{0.05}$) higher in Agudama-Epie relative to Imiringi but not Elebele, with Imiringi distinctly lower than Elebele. Values included 1094.92, 1092.14 and 1019.22 mg kg^{-1}for Agudama-Epie, Elebele and Imiringi respectively. Averaged over pollution status and locations, concentrations were significantly (LSD $_{0.05}$) higher in the surface (1082.28) than the subsurface (1055.24 mg kg^{-1}) soil depths. Unpolluted (1071.72) was specifically higher than polluted (1065.80 mg kg^{-1}) soils, averaged over soil depths and locations. Amongst locations and soil depths, total S was significantly higher in the surface than subsurface depths for Agudama-Epie and Elebele, with the reverse being the case for Imiringi. Averaged over soil depths, concentrations were distinctly (LSD 0.05) better in polluted than unpolluted soils for Elebele and Imiringi and with the reverse being the pattern for Agudama-Epie. Total S content of surface and subsurface depths of various locations were 1037.83 and 1093.76 and 1126.72 and 1016.72mg kg^{-1} for polluted and unpolluted soils respectively. This indicated that total S was higher in the surface depth of unpolluted and the subsurface depth of polluted soils. In all locations, best total S was in the surface depth of unpolluted soils of Agudama-Epie.

Organic sulphur constituted about 91-95% of total S and ranged from 940.13-1014.87 (mean = 984.63 mg kg^{-1}), being distinctly higher in Agudama-Epie than others (Table 2). Averaged over locations and pollution status, concentrations were significantly (LSD $_{0.05}$) better in the surface (1005.12) than the subsurface (964.15 mg kg-1) depths.

Table 1. Physical and Chemical Properties of Crude oil polluted and unpolluted soils Studied

Site Status	Soil depth	Sand	Silt	Clay	pH		OM	N	P	Ca	Mg	Na	K	ECEC
	cm	g kg^{-1}			H_20	1N KCl	g kg^{-1}		mg kg^{-1}	Cmol kg^{-1}				
Imiringi														
Non Polluted	0-15	580	350	70	5.56	4.72	40.59	0.60	18.60	17.10	1.67	0.05	0.03	19.20
	15-30	290	660	50	5.31	4.63	19.95	0.48	17.64	12.00	4.84	0.04	0.02	17.86
	Mean	435	505	60	5.44	4.68	30.27	0.54	18.12	14.55	3.26	0.05	0.03	18.53
Polluted	0-15	340	560	90	5.13	4.46	19.95	0.52	14.30	5.20	2.67	0.05	0.03	9.45
	15-30	410	410	180	5.87	4.93	19.95	0.48	17.64	12.00	4.84	0.04	0.02	17.25
	Mean	375	485	135	5.50	4.70	19.95	0.50	15.97	8.60	3.76	0.05	0.03	13.35
Elebele														
Non Polluted	0-15	530	320	150	5.65	4.56	27.56	0.64	16.30	14.80	2.00	0.04	0.02	17.41
	15-30	510	320	170	5.74	4.88	11.35	0.29	20.30	18.30	4.98	0.05	0.04	23.42
	Mean	520	320	160	5.70	4.72	19.46	0.47	18.30	16.55	3.49	0.05	0.03	20.42
Polluted	0-15	519	316	187	5.58	4.43	21.52	0.61	14.38	12.45	3.21	0.04	0.02	15.72
	15-30	497	319	184	5.54	4.56	10.14	0.28	12.43	9.68	2.27	0.05	0.02	12.02
	Mean	508	318	186	5.56	4.50	15.83	0.45	13.41	11.07	2.74	0.05	0.02	13.87
Agudama-Epie														
Non Polluted	0-15	470	480	50	5.70	4.13	27.56	0.68	21.80	20.00	4.34	0.07	0.03	27.35
	15-30	300	490	210	5.61	4.72	12.04	0.34	20.20	19.00	5.01	0.05	0.03	24.44
	Mean	385	485	130	5.66	4.43	19.80	0.51	21.00	19.50	4.68	0.06	0.03	25.90
Polluted	0-15	430	481	89	5.34	4.71	32.14	0.58	17.87	18.28	6.34	0.03	0.03	24.68
	15-30	348	541	111	5.06	4.26	11.56	0.42	15.19	15.19	5.14	0.05	0.02	20.40
	Mean	389	511	100	5.20	4.49	21.85	0.50	16.53	16.74	5.74	0.04	0.03	22.54

Table 2. Sulphur Forms of Polluted and Unpolluted Surface and Sub soils of Agudama-Epie, Elebele and Imiringi

Locations	Sulphur Forms (mg kg^{-1})					
	Elemental S	Water Soluble S	Adsorbed S	OM bound S	Organic S	Total S
Ag1a	1.61	16.33	44.00	6.00	873.04	941.71
Ag2a	1.89	29.00	43.84	4.33	1040.00	1112.84
Mean	**1.75**	**22.67**	**43.92**	**5.17**	**956.52**	**1027.27**
Ag1b	1.67	16.34	52.34	8.33	1228.06	1296.73
Ag2b	1.17	43.34	66.67	5.60	918.38	1028.39
Mean	**1.42**	**29.84**	**59.50**	**6.97**	**1073.22**	**1162.56**
El1a	3.79	37.60	60.00	5.67	1070.79	1168.39
El2a	2.81	39.34	48.16	6.00	1002.56	1090.06
Mean	**3.30**	**38.47**	**54.08**	**5.83**	**1036.67**	**1129.23**
EL1b	1.84	31.34	56.17	7.34	1015.89	1103.39
EL2b	1.84	32.34	68.00	6.34	906.38	1006.72
Mean	**1.84**	**31.84**	**62.09**	**6.84**	**961.13**	**1055.06**
IM1a	1.11	8.67	56.00	4.00	938.71	1003.38
IM2a	2.20	33.00	56.67	7.00	988.72	1078.39
Mean	**1.66**	**20.83**	**56.34**	**5.50**	**963.71**	**1040.88**
IM1b	3.26	34.34	41.50	5.33	904.21	980.05
IM2b	1.33	26.67	59.50	10.00	928.88	1015.05
Mean	**2.30**	**30.50**	**50.50**	**7.67**	**916.55**	**997.55**
LSD$_{0.05}$: Fact A =	0.08	0.33	1.19	0.72	3.76	2.83
Fact B	0.05	0.71	0.85	0.37	2.44	2.18
Fact C	0.05	0.50	0.61	0.70	2.12	2.15
Fact A x B	0.08	0.88	1.35	0.74	4.11	3.34
Fact A x C	0.08	0.65	1.23	1.00	3.99	3.39
Fact B x C	0.06	0.80	0.96	0.76	2.96	2.81
Fact A x B x C	0.11	1.14	1.61	1.35	5.17	4.71

Ag = Agudama-Epie, El = Elebele, IM = Imiringi, 1 = 0-15cm soil depth, 2 = 15-30cm soil depth, a = polluted, b = unpolluted and OM = Organic matter

Polluted soils (985.64 mg kg^{-1}) were none distinctly (LSD $_{0.05}$) higher than the unpolluted (983.63 mg kg^{-1}), averaged over locations and soil depths. Concentrations of organic S varied as1050.55 and 979.15, 1043.54 and 954.47 and 921.46 and 958.80 mg kg^{-1} for surface and subsurface depths of Agugama-Epie, Elebele and Imiringi respectively. Concentrations of the surface depths of Agudama-Epie and Elebele were seriously better than the subsurface while the reverse was the case for Imiringi. Averaged over soil depths, concentrations of polluted and unpolluted soils were 956.52 and 1073.22, 1036.67 and 961.13 and 963.71 and 916.71 mg kg^{-1} Agudama-Epie, Elebele and Imiringi respectively, with polluted soils of Elebele and Imiringi significantly better than unpolluted and reverse being the situation for Agudama-Epie. Highest organic S was in the unpolluted surface depth of soils of Agudama-Epie.

Averaged over soil depth and pollution status adsorbed S varied as 51.71, 58.08 and 53.42 mg kg^{-}

^{1} equivalent to 4.72, 5.32 and 5.24% of total S in Agudama-Epie, Elebele and Imiringi respectively. Concentrations of surface and subsurface depths were 51.67 and 57.14 mg kg^{-1} respectively averaged over locations and pollution status while that for polluted and unpolluted soils were 51.45 and 57.36 mg kg^{-1} respectively averaged over soil depth and locations. This showed that subsurface unpolluted soils were better than surface polluted surface soils. In Agudama-Epie, Elebele and Imiringi mean concentrations of surface and subsurface depths were 48.17 and 55.25, 58.09 and 58.08 and 48.75 and 58.09 mg kg^{-1} respectively and that for polluted and unpolluted soils were 43.92 and 59.50, 54.08 and 62.09 and 56.34 and 50.50 mg kg^{-1} respectively. This showed that adsorbed S was better in the subsurface than surface depths of the locations exception being Elebele and that besides Imiringi, concentrations were higher in unpolluted than polluted soils. Amongst locations best adsorbed S was in the unpolluted subsurface depth of Elebele (68.00 mg kg^{-1}) and the least polluted surface depth of Imiringi (41.50 mg kg^{-1}).

Sulphur bonded to soil organic matter varied as 6.07, 6.34 and 6.58 mg kg^{-1} equivalent to 11.74, 10.92 and 12.32% of adsorbed S or 0.55, 0.58 and 0.65% of total S in Agudama-Epie, Elebele and Imiringi respectively, averaged over soil depth and pollution status (Table 2). Mean concentrations of surface and subsurface depths were 6.11 and 6.55 mg kg^{-1} respectively and polluted and unpolluted 5.50 and 7.16 mg kg^{-1} respectively. In Agudama-Epie, Elebele and Imiringi concentrations varied as 7.17 and 4.97, 6.50 and 6.17 and 4.67 and 8.50 mg kg^{-1} for surface and subsurface depths respectively and 5.17 and 6.97, 5.83 and 6.84 and 5.50 and 7.67 mg kg^{-1} for polluted and unpolluted soils respectively. This signified that besides Imiringi with better subsurface content, surface depth was better in Agudama-Epie and Elebele. In the soils, best concentration was in unpolluted subsurface depth (10.00 mg kg^{-1}) and the least in polluted surface depth (4.00 mg kg^{-1}) of Imiringi.

Mean water soluble S ranged from 25.67-35.15 (mean = 29.02 mg kg^{-1}) with Elebele (35.15 mg kg^{-1}) significantly (LSD $_{0.05}$) higher than Agudama- Epie (26.25 mg kg^{-1}) and Imiringi (25.67 mg kg^{-1}) while Agudama-Epie did not differ significantly with Imiringi (Table 2). Averaged over locations and pollution status, concentrations were distinctly lower in the surface (24.10 mg kg^{-1}) than the subsurface (35.95 mg kg^{-1}) depths while averaged over location and soil depth it was prominently lower in polluted (27.32 mg kg^{-1}) than unpolluted (30.73 mg kg^{-1}) soils. In Agudama-Epie, Elebele and Imiringi, mean water soluble S varied as 16.34, 34.47 and 21.50 mg kg^{-1} in the surface and 36.17, 35.84 and 29.84 mg kg^{-1} in the subsurface depths respectively, with subsurface better than surface soil depths in all locations. Interactions of locations x soil depth x pollution status, yielded best water soluble S in unpolluted subsurface depth of Agudama-Epie (43.34 mg kg^{-1}) and the least in the polluted surface depth of Imiringi (8.67 mg kg^{-1}).

Elemental sulphur was the least of all sulphur forms and decreased in the order Elebele > Imiringi > Agudama-Epie. Averaged over locations and pollution status, mean concentration was significantly (LSD $_{0.05}$) higher in the surface (2.21 mg kg^{-1}) than subsurface (1.87 mg kg^{-1}) depths while averaged over locations and soil depths, polluted (2.24 mg kg^{-1}) was better than unpolluted (1.85 mg kg^{-1}) soils. Variation in elemental S for Agudama-Epie, Elebele and Imiringi included 1.64 and 1.53, 2.81 and 2.32 and 2.19 and 1.77 mg kg^{-1} in the surface and subsurface depths respectively and 1.75 and 1.42, 3.30 and 1.84 and 1.66 and 2.30 mg kg^{-1} for polluted and unpolluted soils respectively. This showed that besides unpolluted subsurface soils of Imiringi, polluted surface soils had higher elemental S. Best elemental S was in surface polluted soils of Elebele (3.79 mg kg^{-1}) while the least was in the unpolluted subsurface soils of Imiringi (1.17 mg kg^{-1}).

Sulphur forms correlated with some soil properties (Table 3). For instance, total S correlated significantly ($P < 0.05$) with exchangeable Ca ($r = -0.58$) and ECEC ($r = -0.52$) but not sand ($r = -0.30$), silt ($r = 0.16$), clay ($r = 0.14$), OM ($r = --0.34$), P ($r = -0.30$), N ($r = -0.03$), pH ($r = -0.23$), Mg ($r = -0.24$), K ($r = -0.06$) and Na ($r = 0.06$). A regression equation showed that besides Ca, other soil properties (clay, ECEC, pH, OM and P) accounted for less than 30% of total sulphur (Table 4). Also, correlation between organic S with ECEC ($r = -0.48$) and Ca (-0.55) were significant ($P < 0.05$) while that with sand ($r = -0.33$), silt ($r = 0.24$), clay ($r = 0.02$), OM ($r = -0.26$), P ($r = -0.24$), N ($r = 0.04$), pH ($r = -0.31$), Mg ($r = -0.23$), K ($r = 0.003$) and Na ($r = -0.01$) were not. Besides ECEC which predicted about 23% of organic S, other properties (clay, OM, P and pH) accounted for less than 2%. Equally, adsorbed S correlated distinctly ($P < 0.05$) with silt ($r = -0.43$), clay ($r = 0.69$), OM ($r = -0.55$), pH ($r = 0.48$) and K ($r = -0.48$) but not with sand ($r = 0.10$), P ($r = -0.38$), N ($r = -0.26$), ECEC ($r = -0.36$), Ca ($r = -0.35$), Mg ($r = -0.29$) and Na ($r = 0.20$). Clay, OM and pH accounted for about 48, 30 and 23% of soil adsorbed S respectively while less than 20% was accounted by soil Ca, ECEC, Mg and P. Furthermore, water soluble S distinctly ($P < 0.05$) related with silt ($r = -0.40$), clay ($r = 0.68$), OM ($r = 0.44$), N ($r = -0.47$), ECEC ($r = 0.57$) and Na ($r = -0.66$) and not with sand ($r = 0.08$), P ($r = -0.23$), pH ($r = 0.32$), Ca ($r = -0.06$), Mg ($r = 0.23$) and K ($r = -0.20$). Water soluble S was accounted by about 46 and 23% of Clay and ECEC respectively while less than 20% was due to others (Ca, pH, P and OM). Besides Na ($r = -0.48$), other soil properties (sand, silt, clay, OM, P, N, ECEC, pH, Ca, Mg and K) were not significantly correlated with elemental sulphur. Less than 10% of elemental S was due to the soil properties (clay, Ca, ECEC, pH and P). Finally, S bonded to organic correlated seriously with silt ($r = 0.42$), clay ($r = 0.44$), P ($r = -0.57$), ECEC ($r = -0.51$) and Ca ($r = -0.48$) but not with sand ($r = -0.15$), OM ($r = -0.33$), N ($r = -0.26$), pH ($r = -0.38$), Mg ($r = -0.19$), K ($r = 0.05$) and Na ($r = -0.21$). Sulphur bonded to organic matter was accounted by about 26 and 32% of ECEC and P respectively but less than 5% of the other properties (clay, OM and pH).

DISCUSSION

Silt and sand dominated the soil fractions and ascribable to the nature of the parent materials. It has been reported that soils of the area are underlain by Coastal Plain Sands, Meander belt deposits, Sombreiro-Warri Deltaic deposits, Mangrove Swamps and Beach River Ridges (Sutton and Lonagathan, 1986; Youdeowei and Nwakwoala, 2011). The high silt content could be due to alluvial deposition of fine soil particles. Soil pH was low indicating high acidity, probably due to high base leaching occasioned by intense rainfall (Sutton and Lonagathan,

Table 3. Simple Correlation between S Forms and Selected Soil Properties

Soil Properties	Sulphur Forms Total S	Organic S	Adsorbed S	H_2O Soluble S	Elemental S	Organic bound S
Sand	-0.30	-0.33	0.10	0.08	0.27	-0.15
Silt	0.16	0.24	-0.43	-0.40	-0.31	0.42
Clay	0.14	0.02	0.69	0.68	0.19	0.44
OM	-0.34	-0.26	-0.55	-0.44	0.02	-0.33
P	-0.30	-0.24	-0.38	-0.23	0.01	-0.57
N	-0.03	0.04	-0.26	-0.47	-0.05	-0.26
ECEC	-0.52	-0.48	-0.36	0.57	0.20	-0.51
pH	-0.23	-0.31	0.48	0.32	-0.01	-0.38
Ca	-0.58	-0.55	-0.35	-0.06	0.29	-0.48
Mg	-0.24	-0.23	-0.29	0.23	0.08	-0.19
K	-0.06	0.00	-0.48	-0.20	0.19	0.05
Na	-0.06	-0.01	0.20	-0.66	-0.48	-0.21

Table 4. Linear Regression of S Forms with Soil Properties

Parameters	Regression Equation	r^2
Organic S vs. clay	Y = 984.4 + 0.03 Clay	0.004
vs. ECEC	Y = 1138.9 - 7.93 ECEC	0.23
vs. OM	Y = 1048.9 - 2.80 OM	0.07
vs. P	Y = 1122 - 7.71 P	0.06
vs. pH	Y = 1749 - 137 pH	0.10
Adsorbed S vs. clay	Y = 40.94 + 0.10 Clay	0.48
vs. Ca	Y = 63.47 -0.65 Ca	0.13
vs. ECEC	Y = 64.77 - 0.56 ECEC	0.13
vs. Mg	Y = 60.71 - 1.71 Mg	0.09
vs. OM	Y = 66.11 - 0.55 OM	0.30
vs. P	Y = 73.9 - 1.14 P	0.14
vs. pH	Y = 19.47 pH - 53.6	0.23
Water soluble S vs. clay	Y = 13.99 + 0.12 Clay	0.46
vs. Ca	Y = 30.97 - 0.13 Ca	0.04
vs. ECEC	Y = 32.59 - 0.18 ECEC	0.32
vs. OM	Y = 40.47 - 0.52 OM	0.19
vs. P	Y = 42.8 - 0.79 P	0.05
vs. pH	Y = 15.30 pH - 55.3	0.10
Organic matter bound S vs. clay	Y = 4.91 + 0.01 Clay	0.19
vs. ECEC	Y = 8.25 - 0.11 ECEC	0.26
vs. OM	Y = 7.14 - 0.05 OM	0.11

	vs. P	Y = 10.34 - 0.24 P	0.32
	vs. pH	Y = 18.4 - 2.22 pH	0.14
Elemental S	vs. clay	Y = 1.73 + 0.003 Clay	0.04
	vs. Ca	Y = 1.32 + 0.05 Ca	0.08
	vs. ECEC	Y = 1.53 + 0.03 ECEC	0.04
	vs. OM	Y = 2.04 + 0.002 OM	0.004
	vs. P	Y = 2.04 + 0.003 P	0.001
	vs. pH	Y = 2.38 - 0.05 pH	0.001
Total S	vs. clay	Y = 1042.3 + 0.23 Clay	0.02
	vs. Ca	Y = 1242.2 - 11.77 Ca	0.34
	vs. ECEC	Y = 1237 - 8.68 ECEC	0.27
	vs. OM	Y = 1152 - 3.68 OM	0.12
	vs. P	Y = 1237 - 9.52 P	0.09
	vs. pH	Y = 1642 - 103 pH	0.05

1986).Organic matter content was moderate to high ascribable to high sediment deposition of organic materials. Available phosphorus was also moderate and related to the organic matter content since the fertility of tropical soils are related to their organic matter contents (Hassan et al., 2005). Available Ca dominated the soil CEC probably due to the high vegetal concentration of the sediments and probable accumulation of calcium pectate in plant leaves. Nutrient content and fertility of unpolluted was better than polluted soils attributable to the deleterious influence of crude oil on soil properties (Uzoho et al., 2004).

Total S range of 1019.22-1094.92 mg kg^{-1} for the soils are high when compared with ranges of 75-241 mg kg^{-1} for soils of the free state province of South Africa (Solomon et al., 2005), 19-300 mg kg^{-1} for most South African soils (Du Toit and Du Preez, 1995), 44-281 mg kg^{-1} for Ghanaian soils (Acquaye and Kang, 1987) and 59-398 mgkg^{-1} for Brazilian soils (Neptune et al., 1975). However, they are consistent with values of 353-1231 mg kg^{-1} for surface soils of Puerto Rico (Stanko-Golden and Fizgerald, 1991), 520-1041 mg kg^{-1} for Ethiopian soils (Solomon et al., 2001), 149-802 mg kg^{-1} for Thailand soils (Muller et al., 2002) and 250-2500 kg ha^{-1} for most top arable soils (Scherer, 2012). Differences in total soil S could be due to changes in soil organic matter resulting from variations in vegetation type, soil type, sampling depth, climate (Solomon et al., 2005), land use and anthropogenic activities. In the various locations, variations with soil depth and pollution status could be ascribe to differences in the level of crude oil pollution, anthropogenic infusions from fertilizer, pesticides and acid rain deposition and climate. Also the high concentration in unpolluted than polluted soils could be due to the existence of sources other than crude oil pollution.

Soil organic S constituted more than 90% of the total S as have been reported by others (Solomon et al., 2001, 2003, 2005). Organic S differed between surface and subsurface depths and polluted and unpolluted soils of the locations (Agudama-Epie, Elebele and Imiringi) due to differences in the land use types, organic matter input, climate (Forster et al., 2012; Havlin et al., 2012) and microbial activities (Puacz et al., 2001).

Adsorbed and water soluble S or oxidized inorganic S fraction of the soils were less than 5% of total S. Similar observation have been reported by others (Kertsez and Mirleau 2004; Solomon et al., 2005; Kulhanek et al., 2011; Scherer et al., 2012). It has been indicated that SO_4-S occur in small concentration in soil solution with the amount depending on the balance between plant S uptake, fertilizer input, mineralization and immobilization ((Balik, et al., 2009). According to Solomon et al. (2005), the small concentrations of inorganic SO_4-S in the soils suggests that most of the mineralized SO_4–S could have been directly re-metabolized by the soil microflora, taken up by plants or leached to a greater soil depth by percolating water. Adsorbed SO_4^{-2} varied amongst locations, soil depths and pollution status c ascribable to differences in the amount and type of clays, hydrous oxides, OM, pH, solution SO_4^{-2} and competing anions (Havlin et al., 2012). Water soluble S was higher in the subsurface than surface depths of unpolluted soils of most locations attributable to high leaching by percolating water. It has been reported that water soluble S constitutes the fraction mostly available in soil solution and most easily lost through leaching, plant and microbial uptake (Havlin et al., 2012). Low concentration in the subsurface depth of most polluted soils could be associated with the poor infiltration rate due to structural breakdown by crude oil pollution (Uzoho et al., 2004).

Organic matter bound S was very low probably due to poor S adsorption by the soil organic matter. Elemental S constitutes the reduced inorganic S fraction (Geraldi, 2006) that is transitory and negligible under aerobic or oxic soil conditions (Saggar et al., 1998; Solomon et al., 2005). Its low concentration in the soils confirms the oxic and well aerated or drainage condition of the soils.

Influence of soil properties on various S forms have been reported and included increased total soil S with clay and a decrease with sand content (Solomon et al., 2001; Yunas et al., 2010; Scherer et al., 2012). Also significant correlation has been reported between water soluble, adsorbed and organic S with fine soil particle size fractions (Wang et al., 2006; Scherer et al., 2012). Soil organic carbon and total nitrogen have been noted to be highly and significantly correlated with total S, organic S but not inorganic S (Solomon et al., 2005). Forster et al. (2012) reported a significant correlation between soil pH and adsorbed S. Furthermore, significant relationship between total S with OC, N, P, Ca, ECEC and Silt but not pH, Mg, K, clay and sand and inorganic S with P but not with pH, OC, N, Ca, Mg, K, H, ECEC, clay, silt and sand have been reported for some soils of Edo state, Nigeria (Isitekhale et al., 2013).

CONCLUSION

Soil properties varied depending on pollution status and land use types with most soils silty, acidic, high in nutrient levels and with Ca dominating the soil CEC. Averaged over pollution status and soil depths, soil S deceased in the order; Agudama-Epie > Elebele > Imiringi (total and organic S), Elebele > Imiringi > Agudama-Epie (Adsorbed and Elemental S), Imiringi > Elebele > Agudama-Epie (organic matter bound S) and Elebele > Agudama-Epie > Imiringi (water soluble S). Total soil sulphur was dominated by the organic fraction while elemental S was the least. Apart from crude oil, other sources accounted for the soil S. Several soil properties especially ECEC, N, P, Ca, Mg, K, Na, OM, pH, sand, silt and clay influenced S forms but with none predicting up to 50% of the forms. Due to the toxicity of most sulphur compounds and association of S with the mobility of most heavy metals, periodic evaluation of soil S is necessary for efficient environmental control. There is need for more research into an easy and simpler method of estimating soil S fractions.

REFERENCES

Acquaye, D.F. and Kang, B.T. 1987. Sulfur status and forms in some surface soils of Ghana. Soil Science, 144, 43–52.

Balik, J, M. Kulhanek, J. Černy,J. Szakova, D. Pavlikova and P. Čermak, 2009. Differences in soil sulfur fractions due to limitation of atmospheric deposition. Plant Soil Environment 55: 344–352.

Biswas. H, R.K. Rattan, S.P. Datta and A.K. Singh 2003. Adsorption and Translocation of Sulfur in some Tropical Acid Soils. Journal of Plant Nutrition and Soil Science, 166: 519-524

Buyse, W, R.Stern and R.Coe 2004. Genstat Discovery Edition for everyday use. ICRAF Nairobi, Kenya 114pp.

Du Toit, M.C. and C.C. Du Preez 1995. Effect of cultivation period on the decline of total sulphur in selected South African soils. South African Journal of Plant and Soil, 12, 128–131.

Edwards P.J. 1998. Sulfur Cycling, Retention, and Mobility in Soils: A Review. USDA, General Technical Report NE-250, Delaware.

Förster, S, G. Welp G and H.W.Scherer 2012. Sulfur specification in bulk soil as influenced by long-term application of mineral and organic fertilizers. Plant Soil Environment 58(7): 316-321.

Freney, J.R., Melville, G.E. & Williams, C.H. 1970. Extraction, chemical nature, and properties of soil organic sulphur. Journal of the Science of Food and Agriculture, 20, 440–445.

Gerardi, M.H. 2006. Wastewater bacteria. Wiley, Hoboken, NJ.Hamburg, S. P., R. D. Yanai, M. A. Arthur, J. D. Blum, and T. G. Siccama 2003. Biotic control of calcium cycling in northern hardwood forests: Acid rain and aging forests, Ecosystems, 6(4): 399– 406. Gee, G.W and D. Or. 2002. Particle size distribution. In Dane, J/H and G.C.Topp (eds). Methods of soil analysis. Part 4. Physical and mineralogical methods. Soil Science Society of America book series. No.5 ASAS and SSA. Madison, Wisconsin. p 255-293.

Hamburg, S. P, R. D. Yanai, M. A. Arthur, J. D. Blum, and T. G. Siccama 2003. Biotic control of calcium cycling in northern hardwood forests:Acid rain and aging forests, Ecosystems, 6(4), 399– 406.

Hanni, V.D and H.D.Mustafa 2004. Innovative refining technology. Crude oil quality improvement (COQI). Real innovators group, Chemical Engineering Division, Santaeruze, Mumbai, India. Pp 330-335.

Hassan, A.M, B.R.Singh and M. Alkali 2005. Prifile distribution of sesquioxides in a granitic soil in Bauchi,Nigeria. Proceedings of the 29th Conference of the Soil Science Society of Nigeria pp 93-97.

Havlin, J.L, J.D.Beaton, S.L.Tisdale and W.L.Nelson 2012. Sulphur, Calcium and Magnessium. In Soil Fertility and Fertilizers: An introduction to nutrient management 7th ed. PHI Private limited, New Delhi-110001. 513p.

Isitekhale, H.H.E, S.I.Aboh and E.S. Oseghale 2013. Sulphur Status of Some Soils In Edo State, Nigeria. International Journal of Scientific and Technology Reseaerch 2(5): 91-95

Kertesz M.A and P. Mirleau 2004. The role of soil microbes in plant sulphur nutrition. Journal of Experimental Botany, 55*:* 1939–1945.

Kowalenko, C.G. 1993. Extraction of available sulfur. In: Soil Sampling and Methods of Analysis(ed. M.R. Carter), pp. 65–74. Lewis Publishers, Boca Raton, FL.

Kulhánek, M, J. Černý, J. Balík, F. Vašák and O. Sedlář 2011. Changes in different sulfur fractions after long term fertilizing. In: Proceedings of the International Conference Soil, Plant and Food Interactions, 6–8 September, Brno, 215–223.

Likens,G. E, C. T. Driscoll, D. C. Buso, M. J. Mitchell, G. M. Lovett, S.W. Bailey, T. G. Siccama, W. A. Reiners, and C. Alewell 2002. The biogeochemistry of sulfur at HubbardBrook, Biogeochemistry, 60(3): 235–316.

Martinez, C. E, M. B. McBride, M. T. Kandianis, J. M. Duxbury, S. J. Yoon and W. F. Bleam 2002. Zinc-sulfur and cadmium-sulfur association in metalliferous pleats. Evidence from spectroscopy, distribution coefficients, and phytoavailability, Environment, Science and Technology 36 (17):3683–3689.

McBride, M. B. 1994. Environmental Chemistry of Soils, 406 pp., Oxford University Press, New York.

Mohammed, A.D, A.G.Isah, M.Umaru, S. Ahmed and Y.N.Abdullahi 2012. Comparative study on sulphur reduction from heavy petroleum-solvent extraction and microwave irtadiation approach. International Journal of Energy and Environment 3(6): 949-960.

Moller, A, K. Kaiser, N. Kanchanakool, C. Anecksamphant, W. Jirasuktaveekul, A. Maglinao 2002. Sulfur forms in bulksoils and alkaline soil extracts of tropical mountain ecosystems in northern Thailand. Australian Journal of Soil Research, 40, 161–175.

Morra, M. J, S. E. Fendorf and P. D. Brown 1997. Speciation of sulfur in humic and fulvic acids using X-ray absorption near-edge structure (XANES) spectroscopy, Geochim. Cosmochim. Acta, 61(3): 683–688.

Nelson DW and LE Sommers 1996.Total carbon, organic carbon and organic matter, in Methods of Soil Analysis. Part 3. Chemical Methods, Vol. 5 of Soil Science Society of America Book Series,ed. by Sparks DL. Soil Science Society of America/American Society of Agronomy, Madison, WI, pp. 96–101.

Neptune, A.M.L, M.A.Tabatabai and J.J. Hanway 1975. Sulfur fractions and carbon–nitrogen–phosphorus–sulfur relationship in some Brazilian and Iowa soils. Soil Science Society of America Proceedings 39: 51–55.

Olson, S.R and L.E. Sommers 1982. Phosphorus. Pp 403-430. In Page et al.(eds). Methods of soil analysis. Part 2, 2nd ed. American Society of Agronomy and Soil Science Society of America. Madison, Wisconson.

Okezie, C.N 1974. Geological map of Nigeria. Federal Surveys, Lagos, Nigeria.

Prietzel J, J. Thieme, U. Neuhäusler, J. Susini and I. Kögel-Knabner 2003. Speciation of sulphur in soils and soil particles by X-ray spectromicroscopy. European Journal of Soil Science 54**:** 423-433.

Puacz, W. W. Szahun, J. Siepak and T. Sobczyński 2001. Determination of Selected Sulphur Speciation Forms in Fresh Water Lakes and Bottom Sediments. Polish Journal of Environmental Studies10 (5): 365-370

Saggar, S, M.J. Hedley and S. Phimsarn 1998. Dynamics of sulfur transformations in grazed pastures. p. 45–94. In D.G. Maynard ed. Sulfur in the environment. Marcel Dekker, New York.

Sager, M 2012. Levels of Sulfur as an Essential Nutrient Element in the Soil-Crop-Food System in Austria. Agriculture 2: 1-11

Scherer, H.W, G. Welp and S. Förster 2012. Sulfur fractions in particle-size separates as influenced by long-term application of mineral and organic fertilizers. Plant Soil Environment, 58 (5): 242–248

Schroth, A.W, C. B.Benjamin , G.Margaret, M. James, M. Kaste, M.J. Mitchel and A.J. Friedland 2007. Sulfur species behavior in soil organic matter during decomposition. Journal of GeophysicalResearch 112:1-10.

Shamsuddin, and T.A. Bhatti. 2001. Physical properties of soil. p.115. *In:* Soil Science. E. Basher and R. Buntal (eds.). National Book Foundation, Islamabad.

Solomon, D, J. Lehmann, M. Tekalign, F. Fritzsche and W. Zech 2001. Sulfur fractions in particle-size separates of the sub-humid Ethiopian highlands as influenced by land use changes, Geoderma, 102(1 – 2):41– 59.

Solomon, D, J. Lehmann and C. E. Martinez 2003. Sulfur K-edge XANES spectroscopy as a tool for understanding sulfur dynamics in soil organic matter, Soil Science Society America Journal. 67(6): 1721–1731.

Solomon, D, J. Lehmann, I. Lobe, C.E.Martinez, S.Tvitnes, C.C.Du Preez and W.Amelun 2005. Sulphur speciation and biogeochemical cycling in long-term arable cropping of subtropical soils: evidence from wet-chemical reduction and SK-edge XANES spectroscopy. European Journal of Soil Science. 56: 621–634

Solomon, D, J. Lehmann, K. K. de Zarruk, J. Dathe, J. Kinyangi, B. Liang, and S. Machado 2011. Speciation and Long- and Short-term Molecular-level Dynamics of Soil Organic Sulfur Studied by X-ray Absorption Near-Edge Structure Spectroscopy. Journal of Environmental Quality 40 (3):704–718

Stanko-Golden, K.M. and J.W. Fitzgerald 1991. Sulfur transformation and pool size in tropical forest soils. Soil Biology and Biochemistry 23:1053–1058.

Sutton,P.M and P. Loganathan 1986. Characterization and Classification of representative upland soils of Rivers State, Nigeria. Nigerian Journal of Soil Science 6: 14-34.

Thomas,G.W.1996. Soil pH and soil acidity, In D.L.Sparks, A.L.Page, P.A.Helmke,R.H.Loeppert, P.N.Soltanpour,M.A.Tabatabai,C.T.Johnson and M.E.Summer (eds.). Methods of soil analysis. Part 3. Chemical Methods. Soil Science Society of America, Inc,and American Society of Agronomy, Maidson, WI.USA, PP 475-490.

Uzoho, B.U, N.N.Oti and E.U.Onweremadu 2004. Effect of crude oil pollution on maize growth and soil properties in Ihiagwa, Imo State,Nigeria. International Journal of Agriculture & Rural Development 5: 91-100.

Vairavamurthy, A., Manowitz, B., Luther, G.W. III & Jeon, Y. 1993. Oxidation state of sulfur in thiosulfate and implications for anaerobic energy metabolism. Geochimica et Cosmochimica Acta, 57,1619–1623.

Waldo, G. S., R. M. K. Carlson, J. M. Moldowan, K. E. Peters, and J. E.Pennerhahn 1991. Sulfur speciation in heavy petroleums: Information from X-ray absorption near-edge structure, Geochim. Cosmochim. Acta,55 (3): 801–814.

Wang, J, D. Solomon, X. Zhang, J. Lehmann, and W. Amelung 2006. Organic sulfur forms in soils of the Great Plains of North America. Geoderma 133:160–172.

Xia, K., F. Weesner, W. F. Bleam, P. R. Bloom, U. L. Skyllberg, and P. A. Helmke 1998.

XANES studies of oxidation states of sulfur in aquatic and soil humic substances. Soil Science Society of America Journal. 62 (5): 1240– 1246.

Youdeowei, P.O and H.O.Nwankwoala 2011. Studies on sub-soil characteristics of sand deposits in some parts of Bayelsa State, Eastern Niger Delta, Nigeria. Journal of Soil Science and Environmental Management 2(2): 34-38.

Yunas,M. M. Yousaf, R. Khalid and S. Nawaz 2010. Effect of sulfur application on soil sulfur (SO_4-S) status in different textured soils of Pothwar. Soil & Environment 29(1): 7-10.