*IOSR Journal of Environmental Science, Toxicology and Food Technology (IOSR-JESTFT)*
*e-ISSN: 2319-2402,p- ISSN: 2319-2399.Volume 12, Issue 9 Ver. II (September. 2018), PP 50-58*
*www.iosrjournals.org*

# Effect of Induced pH Change on Overburden Topsoil Nutrient Availability in Bituminous Area of Ondo State, Nigeria

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***Abstract:*** *In the development of open pit mining scheme for bituminous sand exploitation, one of the primary operations carried out is the stripping operation (the removal and displacement of overburden materials overlying the deposit) including the plant supporting layer of topsoil. This greatly affects the soil chemistry making it unsuitable for vegetative growth. To this effect, this study was carried out to evaluate the effects of induced change in overburden (topsoil) pH on availability of exchangeable bases as indicators for plants nutrients. The study involved collection of topsoil samples from the overburden of bituminous deposit located in Ilubirin - Gbelejuloda axis of Ondo State, Nigeria and leaching with basic (pH 8.5), neutral (pH 7.0) and acidic (pH 4.5) doses of ammonium acetate ($NH_4OAc$). Additionally, the organic matter and nitrogen contents of the samples were also determined. Multivariate statistical analysis of variance (ANOVA) was conducted based on the obtained laboratory data in conjunction with Post Hoc test of multiple comparisons. The statistical analysis (ANOVA) showed that there was a statistically significant difference between each exchangeable cation's concentrations based on pH of the extractant at ($p < 0.05$). Post-hoc test further confirmed that $Na^+$, $K^+$,$Ca^{2+}$ concentrations were significantly different, mostly between pH groups pH 4.5 and pH 8.5 at $p<0.001$; Mg was only slightly different at $P<0.05$. This implies that the exposure of the topsoil to conditions liable to change the soils' pH by mining operations will have a serious impairing effect on the cation exchange functionality of the soil to store and supply plants' essential base metals nutrients.*

***Keywords:*** *Overburden, Topsoil, pH level, Exchangeable cations, Ecosystem*

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Date of Submission: 26-08-2018 Date of acceptance: 11-09-2018

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## I. Introduction

The surface mining of bituminous sand through open pit comes with its attendant environmental challenges. Fundamentally, bituminous sands mining is carried out through surface mining and *in-situ* extraction techniques. When the deposit is located within (0-75) m depth to the ground surface, open pit mining is deployed[1]; but when it is situated beyond 75 m depth, the extraction is done through *in-situ* extraction techniques or enhanced oil recovery. Open pit bituminous sand mining comes with its concomitant environmental footprints affecting the ecosystem [2,3,4] they include deforestation, alteration of landforms, depletion of water quality and quantity and soil degradation. In the open pit mining operation, most material waste generation comes from overburden removal and tailings disposal. One critical disruption to the ecosystem in stripping operation of overburden is the loss of topsoil, rich in organisms and plant nutrients [5,6]. Harold [7] defines soil as the layer(s) of generally loose mineral and/or organic material that are affected by physical, chemical, and/or biological processes at or near the planetary surface and usually hold liquids, gases, and biota and support plants. The Improper handling of the topsoil could result to loss of plants vital nutrients[8].

The sustainable environmental management of a mine aims to minimize the mining impact on the environment. Good reclamation and the ecological restoration of a mined out area is predicated on proper management of the soil which holds numerous restorative properties including plants nutrients cycling [6,9.] The provisions of the Nigerian Minerals and Mining Acts, 2007 stipulate that mine operators conduct environmental impact assessment (EIA) prior to obtaining operating license. The assessment report is submitted as environmental impact statement (EIS) in which environmental management plans (EMP) on how best to minimize impact to the environment is well stated. These plans which include reclamation, restoration and rehabilitation plans are primarily aimed at securing the future use of the land. Hamanaka *et al.*[10] and Tóth *et al.*[11] also stressed the advocacy for the need to protect the environment through proper management of the soil. The bituminous sand deposit in the study area is known to occur in sedimentary terrain that is ecologically active, forested with lush vegetation and thriving majorly with small to medium scale agricultural activities[12]. Mining of the bituminous sand will impact the soil component greatly; hence, it is imperative to undertake discrete management of the overburden material which contains topsoil, the nutrient-rich portion of the overburden profile for effective post mining restoration and rehabilitation programmes. Generally, alkali metals found naturally in soil are essential for proper plant growth. They constitute the primary macro elements and are generally referred to as nutrient base cations ($K^+$, $Na^+$, $Mg^{2+}$, $Ca^{2+}$)[13]. These exchangeable base cations are an indicator of the quality of the soil nutrition. The availability or relative abundance of these base metals in soil for plant uptake is dependent on a number of physicochemical factors including the nature and texture of the mineral matter, the presence and type of the colloidal particles as well as the pH of the soil. The change in soil pH could influence and alter the solubility, mobility and plant availability of these essential nutrients elements in the soil. Soil pH change could be caused by both or either natural or anthropogenic sources. Natural factors that affect soil pH include climate, mineral content, and soil texture [14]. Soils that have a high content of colloidal particles (clay and organic matter) are more resistant to changes in pH due to higher buffering capacity than are sandy soils.

Although clay content cannot be altered, organic matter content can be altered by management practices. The disruption of soil occasioned by vegetal cover removal impacts its pH levels. Sandy soils commonly have a low content of organic matter, resulting in a low buffering capacity and a high rate of water percolation and infiltration. Thus, they are susceptible to acidification[14]. A study by [15] noted that areas of forestland tend to be more acidic than areas of grassland. Anthropogenic activities such as land use and management also impact on soil pH. Conversion of land with forest or grass to another land use with less vegetal cover can result in drastic changes in pH over time. These changes result from a broad range of activities including, loss of organic matter primarily concentrated in the topmost soil layer, removal of soil minerals, and erosion of the exposed surface layer. Increased soil acidicity has been attributed to accelerated depletion of essential base cations through mobilization and subsequent leaching from the reach of plants root[16]. The question of will extreme pH (alkaline-acidity) induced level potentially due to anthropogenicalally (mining) disturbed surface soil affect the release or hold back of essential base plant nutrients in the study area is the focus in the study.

## II. Materials and Methods

### Description of the Study Area

The study area is located within latitudes 6° 38' 17.45" N and 6° 39' 6.69" N; and longitudes 4° 49' 48.27" E and 4° 53' 22.62" E respectively along the bituminous deposits belt in Irele and Odigbo Local Government Areas of Ondo State. This area falls within the rain forest belt in the South western part of Nigeria and generally belongs to the sedimentary terrain of the Benin (Dahomey Basin). The sediments are said to be stratified according to depositional era. Evidences from lithologic and microfossil examinations of the strata (source and reservoir rock) revealed that the sediments were deposited in varying sedimentary environments varying from continental at the base through brackish and shallow marine in the middle to full marine towards the top. The bitumen bearing zone is made up of sandy and shaley layers occurring mostly in the middle transitional level. It is said to have proven reserve of 42.74 billion barrels of untapped bituminous deposit[12]. The bitumen zone is overlain by a profile of overburden materials varying in thickness from less than 5 m in the North of the study area to approximately 60 m in the Southern part. At the topmost part of this overburden profile is a layer of biophysico-chemically active topsoil material which plays a key role in ecosystem regulation and sustenance.

### Sample Collection

Field sampling was carried out using sampling tools including soil hand auger, plastic bags, GPS equipment, labeling tape, marker, cutlass and hand shovel. Random sampling of the soil was carried out within the study area to reduce sampling bias. Plant liters were first removed from the ground surface (where applicable) before sample collection. Ten locations were sampled and five sub-samples were obtained from each location in a quadrangular manner within 0-20 cm soil depth at approximately 10 m from each other using a combination of soil auger and hand shovel. Each location sub-samples were thoroughly mixed and composited to 1 kg sample size. The composites were put in clean polythene bags and transferred to the laboratory for analysis.

### Determination of Exchangeable cations in Soil

The exchangeable cationic or alkali metals ($Ca^{2+}$, $Mg^{2+}$, $K^+$ ,$Na^+$) in the composite samples were determined in accordance with Rengasamy and Churchman[17] procedure. Two and half grammes (2.5 g) of each air-dried (< 2 mm) composite soil sample was weighed into a 125 ml Erlenmeyer flask and 25 ml of 1 M neutral ammonium acetate (1 M $NH_4OAc$) of pH 7.0 was added. The sample was placed in a rotor shaker for 2 hours. Thereafter, the solution was filtered and the filtrate was analyzed by atomic absorption spectrometry (AAS) with Bulk Scientific VGP 210 model. Sodium, potassium, magnesium and calcium standards were aspirated into the

AAS and the emissions recorded and plotted against concentrations. Soil extracts were also aspirated and emission of sodium, potassium, magnesium and calcium recorded and concentration evaluated from the graph. pH of extracting agent was adjusted to pH of 4.5 and pH of 8.5 respectively and the procedure was repeated to determine the exchangeable bases in acidic and basic media.

**Determination of pH of Soil** The pH values of the soil samples which indicate the degree of its acidity or alkalinity were determined in accordance to ASTM D 4972 [18] using electronic pH meter calibrated with buffer solutions of pH 7.0 and pH 4.0 respectively.

**Determination of Soil Organic Carbon (SOC) and Organic Matter (SOM)**The soil organic carbon and organic matter contents are co-indicators of soil richness which indicate the ability of the soil to release essential nutrient (nutrient store house). The Walkley- Black method as described by Schumacher [19] was used in determining the SOC and SOM.

**Determination of Soil Total Nitrogen**The total nitrogen was determined using Kjeldahl method through a three- phase process of digestion of organic material to convert nitrogen to $HNO_3$; distillation of ammonia into absorbing surface and volumetric analysis of ammonia derived from digestion according to Food and Agricultural Organization [20] procedure.

**Statistical analysis**

The data obtained from the laboratory tests were analyzed using Statistical Package for Social Sciences SPSS version 20 (IBM SPSS 20). A One Way Analysis of Variance (One way ANOVA) was conducted complemented with Post Hoc test to determine the measure of significance in the variances of the means of the soil base cations.

## III. Results and Discussion

The biochemical results of soil analysis in the study are shown in Tables (1 and 2). Table 1 presents the natural field status of the pH, Organic Matter and percentage Nitrogen of the composite samples. The pH of the mixed samples reflects the acid nature of the entirely soil of the study area. This phenomenon is typical of forest soils which dominates more than three-quarters of the study area. The organic matter contents of the samples are low ranging from (2.2- 5.43) % (Table 2). FAO [21] describes several functions of organic matter to include cation holding capacity on its exchange site. Considering the mean low organic matter content (3.116 %) of the soils in Table 2, and the sandy loam texture of the soils in the study area, there is a likelihood of accelerated leaching of these nutrients base metals if the tops soil is unduly exposed without proper management.

**Table 1:** The pH, Organic Matter and Total Nitrogen of Soil Composites samples

| Sample Codes | pH | % Organic Matter | % T. Nitrogen | pH Classification according to (USDA) |
|---|---|---|---|---|
| AA | 5.70 | 2.61 | 0.39 | Moderately acid |
| AB | 5.80 | 2.78 | 0.38 | Moderately acid |
| AC | 5.40 | 2.59 | 0.36 | Strongly acid |
| G1 | 4.20 | 3.37 | 0.37 | Extremely acid |
| G2 | 4.30 | 3.44 | 0.41 | Extremely acid |
| G3 | 5.10 | 3.40 | 0.39 | Strongly acid |
| GR | 5.90 | 5.43 | 0.47 | Moderately acid |
| IA | 6.00 | 2.20 | 0.42 | Moderately acid |
| IB1 | 6.40 | 2.82 | 0.33 | Slightly acid |
| IB2 | 6.10 | 2.52 | 0.37 | Slightly acid |

**Table 2:** Descriptive Statistics of all the Samples pH, Organic Matter and Nitrogen Contents

| Statistics | pH | % Organic Matter | % Nitrogen |
|---|---|---|---|
| Minimum | 4.2 | 2.2 | 0.33 |
| Maximum | 6.4 | 5.43 | 0.47 |
| Mean | 5.49 | 3.116 | 0.389 |
| Standard deviation | 0.746 | 0.9148 | 0.0381 |

Soil with extremely low pH tend to leach away essential metal nutrients, however, results in Table 1 alone could not be sufficiently used to conclude the effect pH had on the dissolution and release of the soil essential exchangeable metals. Figs. 3, 4 and 5 show the concentrations of the base cations under three different pH of extracting agent (pH 7.0, pH 4.5, pH 8.5) are shown.

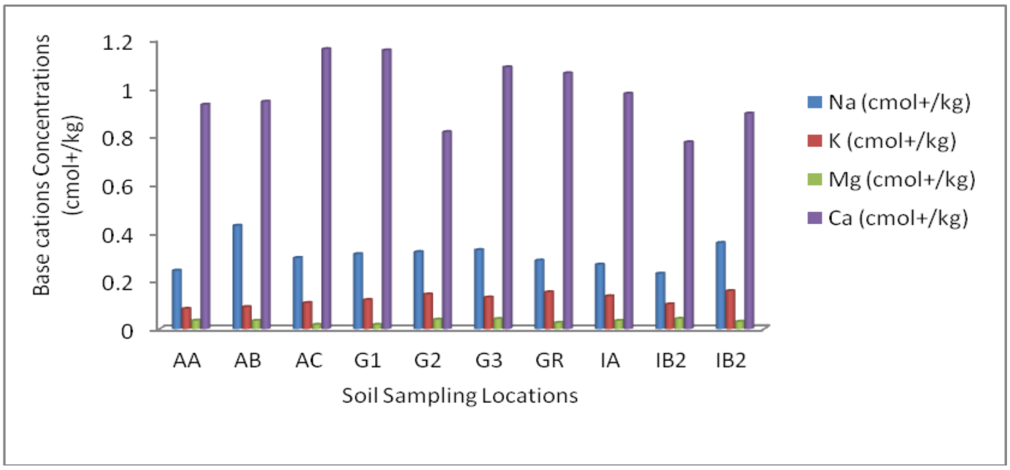


**Fig. 1:** Exchangeable Cations extracted with Ammonium Acetate (pH of 7.0)

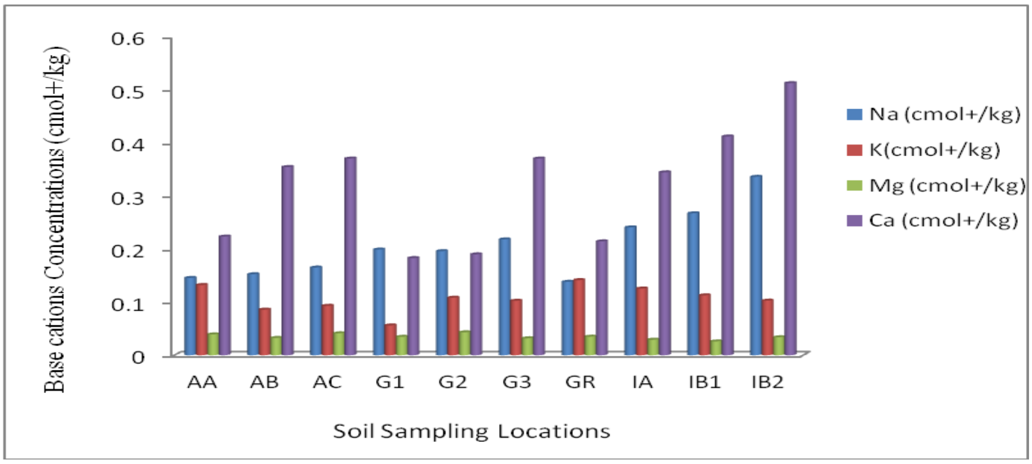


**Fig. 2:** Exchangeable Cations extracted with Ammonium Acetate (pH of 4.5)

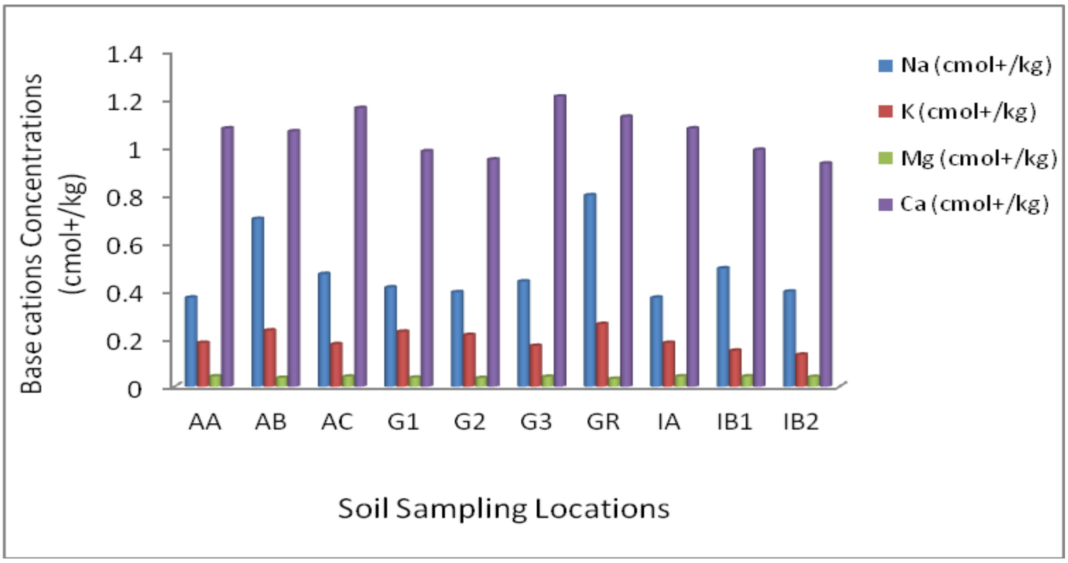


**Fig. 3:** Exchangeable Cations extracted with Ammonium Acetate (pH of 8.5)

Figs. (4-6) show the relative susceptibility of three ($Mg^{2+}$, $k^{+}$, $Ca^{2+}$) of the four base cations examined in this study to pH levels of the extractant. Calcium appeared to be readily (soluble) in the acidic medium (pH 4.5) compared with other base metals. This is closely followed by potassium Figs. (4 and 5). Magnesium on the other hand showed slight response to the change in pH of the extracting agent (Fig. 6). The observation tends to show the susceptibility of each base metal to leaching by the varying degree of pH of the extracting agent in the order Ca> K > Mg.

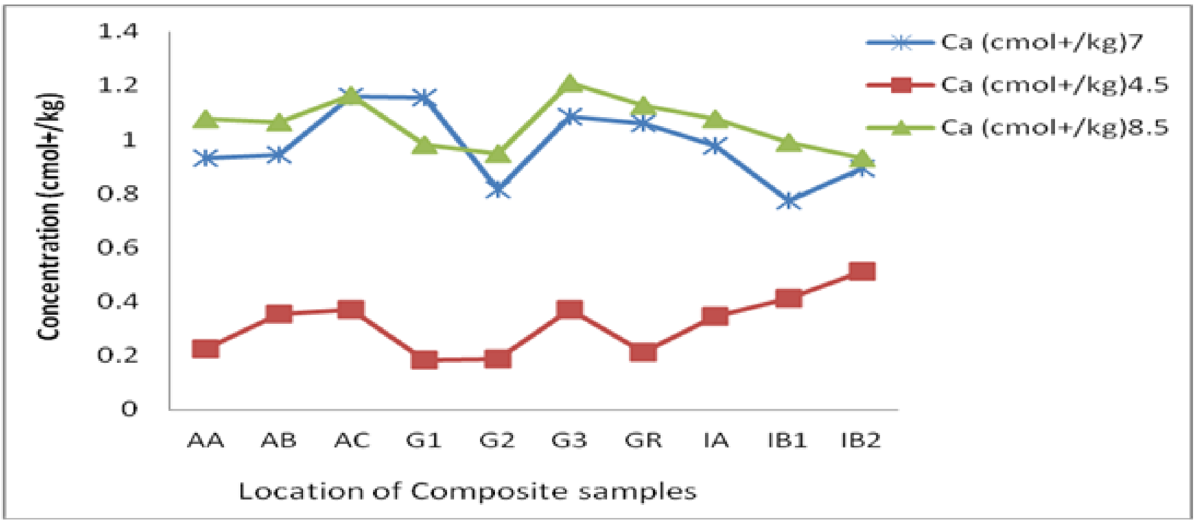


**Fig. 4:** Exchangeable Calcium with different pH of the Extraction Agent

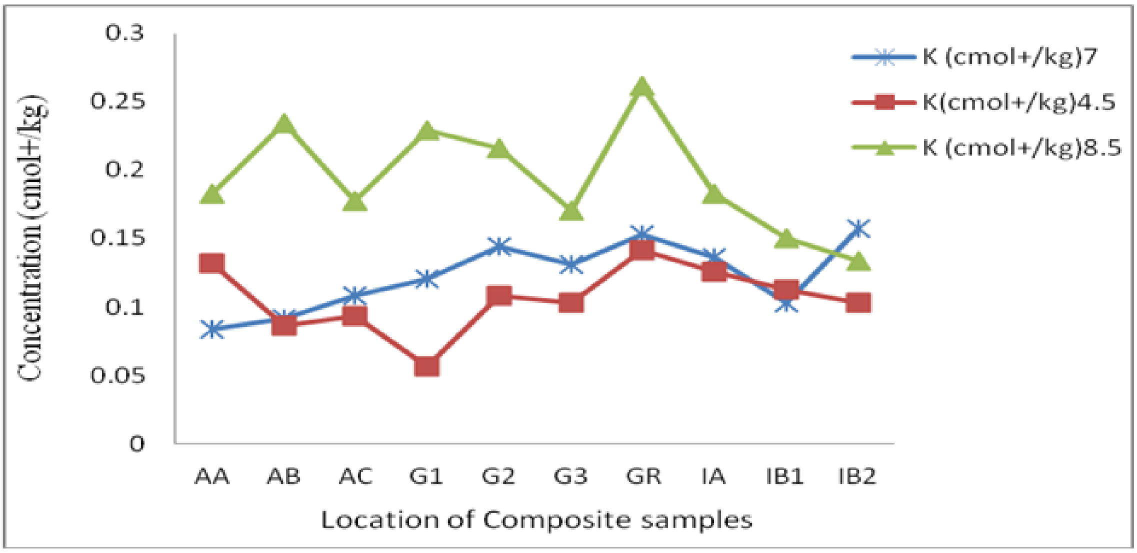


**Fig. 5:** Exchangeable Potassium with different pH of the Extracting Agent

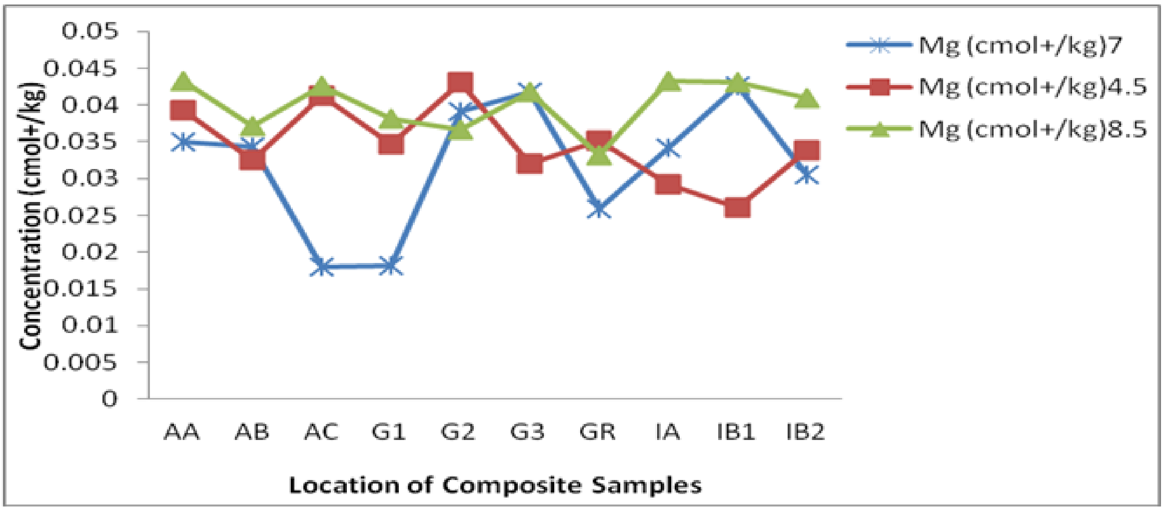


**Fig. 6:** Exchangeable Magnesium with different pH of the Extracting Agent

Table 3 shows that there is a statistical significant difference between each set of groups of the essential nutrient base cations relative to pH of extracting agent at P< 0.5. However, results from the ANOVA could not identify which of the soil groups were statistically significantly different from each other in terms of response to pH of the extractant, hence, a further test to resolve the ambiguity was done using Tukey's HSD Post Hoc Test. The test as shown in Table 4 indicates the multiple comparisons of each group of the analytes (base cations) with one another. It showed that the ionic concentrations of the nutrient base cations in pH 4.5 medium were statistically significantly different at p < 0.5 from those of neutral pH of 7.0 and pH of 8.5 respectively.

**Table 3:** One Way Analysis of Variance of the Soil Base Cations

| | | | | Sum of Squares | df | Mean Square | F | Sig. |
|---|---|---|---|---|---|---|---|---|
| Na | Between Groups | (Combined) | | .400 | 2 | .200 | 20.863 | .000 |
| | | Linear Term | Contrast | .364 | 1 | .364 | 37.958 | .000 |
| | | | Deviation | .036 | 1 | .036 | 3.768 | .063 |
| K | Between Groups | (Combined) | | .044 | 2 | .022 | 22.640 | .000 |
| | | Linear Term | Contrast | .034 | 1 | .034 | 35.383 | .000 |
| | | | Deviation | .010 | 1 | .010 | 9.897 | .004 |
| Mg | Between Groups | (Combined) | | .000 | 2 | .000 | 4.184 | .026 |
| | | Linear Term | Contrast | .000 | 1 | .000 | 2.369 | .135 |
| | | | Deviation | .000 | 1 | .000 | 5.998 | .021 |
| Ca | Between | (Combined) | | 3.302 | 2 | 1.651 | 128.088 | .000 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Groups | Linear Term | Contrast | 3.042 | 1 | 3.042 | 236.022 | .000 |
| | | | Deviation | .260 | 1 | .260 | 20.155 | .000 |

However, there were no significant differences for $Na^{+}$, $K^{+}$ and $Mg^{2+}$ concentrations at pH 7.0 and pH 4.5 and for $Ca^{2+}$ at pH (7.0 and 8.5).

**Table 4:** Post Hoc Test of Multiple Comparisons (Tukey's HSD)

| Dependent Variable | | (I) pH | (J) pH | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | Lower Bound | Upper Bound |
| Na | | 4.50 | 7.00 | -0.100087 | 0.043784 | 0.075 | -0.20865 | 0.00847 |
| | | | 8.50 | **-0.279130**$^{*}$ | 0.043784 | 0.000 | -0.38769 | -0.17057 |
| | | 7.00 | 4.50 | 0.100087 | 0.043784 | 0.075 | -0.00847 | 0.20865 |
| | | | 8.50 | **-0.179043**$^{**}$ | 0.043784 | 0.001 | -0.28760 | -0.07048 |
| | | 8.50 | 4.50 | **0.279130**$^{**}$ | 0.043784 | 0.000 | 0.17057 | 0.38769 |
| | | | 7.00 | **0.179043**$^{**}$ | 0.043784 | 0.001 | 0.07048 | 0.28760 |
| K | | 4.50 | 7.00 | -0.016744 | 0.013863 | 0.459 | -0.05112 | 0.01763 |
| | | | 8.50 | -0.**087846**$^{**}$ | 0.013863 | 0.000 | -0.12222 | -0.05347 |
| | | 7.00 | 4.50 | 0.016744 | 0.013863 | 0.459 | -0.01763 | 0.05112 |
| | | | 8.50 | **-0.071103**$^{**}$ | 0.013863 | 0.000 | -0.10547 | -0.03673 |
| | | 8.50 | 4.50 | **0.087846**$^{**}$ | 0.013863 | 0.000 | 0.05347 | 0.12222 |
| | | | 7.00 | **0.071103**$^{**}$ | 0.013863 | 0.000 | 0.03673 | 0.10547 |
| Mg | | 4.50 | 7.00 | 0.002742 | 0.002820 | 0.600 | -0.00425 | 0.00973 |
| | | | 8.50 | -0.005283 | 0.002820 | 0.166 | -0.01228 | 0.00171 |
| | | 7.00 | 4.50 | -0.002742 | 0.002820 | 0.600 | -0.00973 | 0.00425 |
| | | | 8.50 | -0.**008025**$^{*}$ | 0.002820 | 0.022 | -0.01502 | -0.00103 |
| | | 8.50 | 4.50 | 0.005283 | 0.002820 | 0.166 | -0.00171 | 0.01228 |
| | | | 7.00 | **0.008025**$^{*}$ | 0.002820 | 0.022 | 0.00103 | 0.01502 |
| Ca | | 4.50 | 7.00 | **-0.661600**$^{**}$ | 0.050772 | 0.000 | -0.78749 | -0.53571 |
| | | | 8.50 | **-0.739450**$^{**}$ | 0.050772 | 0.000 | -0.86534 | -0.61356 |
| | | 7.00 | 4.50 | **0.661600**$^{**}$ | 0.050772 | 0.000 | 0.53571 | 0.78749 |
| | | | 8.50 | -0.077850 | 0.050772 | 0.292 | -0.20374 | 0.04804 |
| | | 8.50 | 4.50 | 0.**739450**$^{**}$ | 0.050772 | 0.000 | 0.61356 | 0.86534 |
| | | | 7.00 | 0.077850 | 0.050772 | 0.292 | -0.04804 | 0.20374 |

$^{**}$ The mean difference is significant at the $p < 0.001$ level; $^{*}$ at $p < 0.05$

## IV. Discussion

From the results, it was observed that soil reaction had significant influence on the concentrations of the base cations (which are primary indicators of plants nutrients). The result indicated that exchangeable sodium was stable on the exchange site of the tested soil in slightly acid medium but with varied solubility between extreme acidic and alkaline range, same as exchangeable potassium at a significance of 1% as indicated in Table 4. However, according to the analysis, the exchangeable magnesium showed significant soil solution concentration difference occurred within the 'alkaline' neutral – alkaline extraction implying that Mg maintained its relative stability in the slightly acidic soil medium. The exchangeable calcium which comparatively is more prevalent in all analyzed soils Figs (1-3) has dissolution dynamics within the soil natural or background pH level and the extreme lower pH region. There is also a high statistical significance between the low and high alkaline pH. It is instructive to note that during excavation and processing of bituminous sand, a number of environmental disturbing mechanisms are involved which tend to affect the physicochemical properties of the soil. Included in the array of such disturbances are vegetation removal and soil displacement as well as process wastes discharged in form of tailings. Each of these activities has its potential environmental footprints one of which is soil degradation occasioned by extreme lowering or elevation of pH levels of the soil particularly the vital topsoil. The process of surface excavation of the bituminous sands could encourage the buildup of surface soil acidity when stripped soil are left bare and exposed by leaching of exchangeable metals from the soil surface down the soil profile. In addition, processing of synthetic bitumen from bituminous sands could result in emissions of acidic oxides such as sulfur and nitrogen dioxides into the atmosphere. These oxides react with atmospheric water according to equations (1-4) to forms acid rain that eventually precipitate on the ground leading to soil acidification [22].

Sulfur dioxide emitted from the bitumen processing plants could react with water to form sulfurous acid as indicted in eqt 1

$$SO_{2(g)} + H_2O_{(l)} \rightarrow H_2SO_{3(aq)} \tag{1}$$

The reaction between sulfurous acid and oxygen is then catalyzed by substances in the atmosphere to form sulfuric acid in eqt 2

$$2H_2SO_{3(aq)} + O_{2(g)} \rightarrow 2H_2SO_{4(aq)} \tag{2}$$

Likewise nitrogen dioxide from natural bitumen processing plant emissions reacts with water to form a mixture of nitric acid and nitrous acid as indicated in equation 3.

$$2NO_{2(g)} + H_2O_{(l)} \rightarrow HNO_{3(aq)} + HNO_{2(aq)} \tag{3}$$

While substances in the troposphere (upper atmosphere) then catalyzes the reaction between nitrous acid and oxygen to produce more nitric acids eqt 4.

$$2HNO_{2(aq)} + O_{2(g)} \rightarrow 2HNO_{3(aq)} \tag{4}$$

Due to high solubility of these acids in water, they combine with atmospheric water to precipitate as acid rain, thus increasing soil acidity and other similar effects.

On the other hand, derived wastes (tailings) from Clarke's hydrothermal (hot water) separation process of the sands –bitumen phases could have elevated levels of pH due to stripping agents used in liberating the bitumen from the sand matrix. Xiang et al, [23] claim that basic hot water process technology presently in use could provide better recovery or bitumen stripping from bituminous sands ore, if the pH of process water is raised to as high as pH 11. Alkaline waste fluids spillage from hot water separation could be a source of environmental contaminants particularly to water and soil if preventive spill management strategy is not put in place. As a results surface soil and subsoil could be at risk of being contamination. Based on the study, either very low or high pH values of the soil environment could hamper the release or and sorption of the plant nutrition cations.

## V. Conclusion

The study concludes that the availability of base metal nutrients to plants in the topsoil layer of the overburden could be greatly influenced by actions liable to expose the earth material to acidifying conditions such as stripping of overburden materials above the bitumen deposit. Consequently, soil acidification by exposure to natural or chemical agents during open pit mining of the bitumen deposit could render the soil sterile by depletion of essential macro nutrients particularly the base cations. The aftermath of this phenomenon is that the reclamation and restoration of the mined out area back to a healthy and flourishing ecosystem form becomes extremely difficult to achieve. This is due to the fact that most of these nutrient metals bond together with the exchange sites of soil colloidal particles (organic matter and clay contents) in the soil upper profile and are readily dislodge by chemical agents. Hence, it is important to cautious management of the overburden topsoil in order to preserve it from being exposed to acidifying medium during the course of the open pit mining operation.

**Conflict of Interest**

The authors declare that there are no conflicting interests in the funding or any other matter regarding data collection, preparation and publication of this paper.

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