Phase and Micro-Chemical Characterization of Water Works Sludge Minerals and their Thermally-Decomposed Products

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Abstract

Environmental Regulations worldwide have created a critical need for a long term approach to sludge management for environmental sustainability. Several sludge disposal concepts adopt the reuse of sludge as a viable alternative to material application and utilization in engineering design. In-depth understanding of the mineralogical compositions of water works sludge and its decomposed products became inevitable for optimal use in engineering applications. In this paper, sludge from Lower Usuma Dam Water Treatment Plant (LUDWTP), Abuja, Nigeria is assessed for reuse potentials in engineering applications. The processing steps considered include gravity thickening, air drying and thermal treatment at temperatures of 105 ^{o}C, 800 ^{o}C and 1000 ^{o}C respectively. The effect of temperature on the microstructure and chemical properties of the resultant sludge ash were investigated using scanning electron microscopy (SEM), energy dispersion spectroscopy (EDS) and x-ray diffraction (XRD) techniques. The results showed the presence of phyllosilicates. The thermally-treated sludge at 105 ^{O}C did not show significant changes in structure while the diffraction patterns at 800 ^{o}C gave a featureless x-ray band of amorphous meta-kaolinite and the formation of gamma-alumina and amorphous silica at 1000 ^{o}C. Toxicity Characteristic Leaching Procedure (TCLP) tests showed that the metal leaching level is within the acceptable National and International environmental limits.

Keywords: *amorphous silica, gamma-alumina, kaolinite, meta-kaolinite, Water works sludge.*

1.0 INTRODUCTION

Water works sludge is an inevitable by-product from the production of potable water. Large quantities of this sludge are generated, thus making its management a big challenge for water utilities.

The objective of sludge treatment is to produce treated sludge suitable for safe discharge into the environment or reuse. The treatment processes of water works sludge involves dewatering through natural, mechanical or electrical methods for the purposes of making further management of this sludge an easier task. The choice of water works sludge treatment method depends on the quantity and quality of sludge and other site-specific conditions (USEPA, 1996; Syed, 2004; Tantawy *et al*., 2012).

The composition and properties of water treatment plant sludge depends on the quality of raw water and the type of treatment chemicals used in the production of potable water. Water Works sludge has traditionally been classified as wastes. They contain different kinds of oxides, such as aluminum oxide, silicon oxide, calcium oxide and iron oxide, and also some heavy metals such as As, Cd, Cr, Cu, Hg, Ni, Pb, Se and Zn. (Wen-Ten *et al*., 2006; USEPA, 1996). Thus, it becomes necessary that these wastes are properly handled in accordance with approved Standards and Regulations which stipulate that quantity of generated waste be reduced, re-used and recycled to minimize pollution (NESREA, 2009).

The disposal methods of water treatment plant sludge include land application, disposal in a sanitary sewer, disposal in surface waters and landfill (Syed, 2004). Space limitations on existing landfills and increasing health and environmental concerns such as groundwater pollution from landfill leachate, odour emission and soil contamination have motivated the investigation of alternative disposal methods (Park and Heo, 2002).

Beneficiation of water works sludge and reusing them in material applications is a win-win method of sludge management for economic and environmental sustainability. Water works sludge contains some clay minerals like kaolinite and quartz (Anyakora *et al*., 2012). Kaolinite is a clay mineral often mixed with other clay minerals such as montmorillonite and illite (Gonzalez *et al*., 2007). At high temperatures kaolinite dehydroxylates to meta-kaolinite, and the meta-kaolinite formed subsequently decomposes to amorphous SiO_2 and other alumina groups such as gamma-alumina (Anwarul *et al*., 2009). Due to the physical and chemical properties of Kaolinite, it has been reported to be used for different industrial applications such as refractories and ceramics (31 %), fiberglass (6 %), cement (6 %), rubber and plastic (3 %) and others (4 %), (Murray, 2002).

Furthermore, the characteristics of water works sludge is similar to those reported in literature as raw materials used to elaborate ceramic products by Odegaard *et al*. (2002) for the preparation of adsorbents and catalysts in Park and Heo, (2002), as soil substitute for land application, elaboration of fire-breaks in woods by Casado-Vela *et al*. *(*2006). Also, Dunster and Petavratzi (2016) in their investigation reported that if only 5 % of the clay raw materials used to make bricks were to be substituted by alternative raw materials, this would create a market for over 400,000 tonnes of waste.

Recently, research has shown that sludge ash has been used as an additive in the production of building and construction materials by Ali and Chang (1994) and in concrete by Monzó *et al*. (1996). As concluded in the work of Monzó *et al*. (1999) sludge containing high sulphate content is found to be compatible with cements with high C_3A content as binder in mortars. The results from investigations reported in the forgoing references suggest that the toxicity leaching potentials of the heavy metals contained in the sludge-blended materials are well contained below environmental acceptable limits (Tantawy *et al*., 2012).

Since most of the processing methods of these sludge-materials involve thermal treatment, it becomes necessary to investigate the behavior of water treatment plant sludge at elevated temperatures in order to optimize its applicability for engineering applications. There exist little research on the micro-chemical characterization of Lower Usuma Dam Water Treatment Plant (LUDWTP) but none on its thermally decomposed products, thus this work is aimed to investigate the behavior of thermally processed sludge from LUDWTP so as to have an in-depth understanding of the mineralogical compositions of its decomposed products for optimal applicability in the production of engineering materials.

2.0 MATERIALS AND METHOD

The raw material used in this study was sludge from the Lower Usuma Dam Water Treatment Plant (LUDWTP), Abuja, Nigeria. Dehydration and de-hydroxylation of the dry sludge was carried out by thermal treatment at different temperatures of 105 ^oC, 800 ^oC, 1000 ^oC to loosen the alumina components (Hosseini et al., 2011). Study of the micro-chemical changes was carried out using scanning electron microscopy (SEM) and Energy Dispersion Spectroscopy (EDS) while the identification of changes in the crystal structure was done using x-ray diffraction (XRD) in accordance with the manual prescribed by Anyakora *et al*. (2012). The Toxicity Characteristics Leaching Procedure

(TCLP) test was performed in accordance with USEPA (1988) method to determine leach ability potentials of the heavy metals.

2.1 Processing of Sludge

Sludge sample was collected from the de-sludging chamber of the clarifier of the LUDWTP. The sludge was subsequently dewatered using gravitational thickening and air drying methods in accordance with the manual prescribed by Anyakora *et al*. (2012).

2.2 Thermal Treatment of Sludge

50 g of three different samples were placed in a crucible and subsequently were thermally treated in a laboratory furnace at different temperatures (105 ^oC, 800 ^oC, 1000 ^oC) with heating program at 5 $^oC.min^{-1}$ from the room temperature to the working temperature and was maintained at this temperature for 2 hours (Gonzalez *et al*., 2007). When the required temperature was attained, the samples were immediately placed in a desiccator to cool. The weights of the samples before and after the thermal treatment were measured to determine the weight loss on ignition (LOI) during calcination process before analysis using XRD, SEM, EDS, and toxicity characteristic leaching procedure (TCLP) Technique.

2.3 Characterization of Sludge

2.3.1 Determination of Mineralogical Composition of Sludge

The phase identification of the dry and thermally treated sludge samples were performed in accordance with the manual prescribed by Anyakora *et al*. (2012) using XRD machine PW3050/60-XPERT-PRO MPD type x-ray diffractometer (XRD) at Shestco, Gwagwalada, Abuja, Nigeria. Study of phase analysis was performed under K α-ray of Cu from 3^o to 75^o 2θ pipe pressure 40 KV, pipe flow 30 mA, with a scanning step of 0.06^o at scanning speed $4°min^{-1}$. The diffraction data were detected by automated detector X-celarator. The samples were then analyzed to determine the mineralogical components and phase analysis (Anyakora, 2013).

2.3.2 Determination of Microstructure and Chemical characterization of Sludge

The Sludge samples were quantitatively analyzed using EVO/MA 10 Scanning electron microscope (SEM) and Energy dispersion spectroscopy (EDS) at Shestco, Gwagwalada, Abuja, Nigeria. The test pieces, measuring approximately 15 x 15 mm^2 were mounted on sample holder already prepared with conductive (carbon) adhesive tapes to hold samples. The sample holder, with the held sample specimens was subsequently loaded into the SEM specimen chamber, and operated on variable pressure mode, with the vacuum created to the level needed to propagate electron beam. As the indicator showed 'Blue', the microscope was subsequently directed on each of the samples for magnification. Each sample was magnified and viewed at separately. Scanning electron microscopes are equipped with a cathode and magnetic lenses to create and focus a beam of electrons, and have been equipped with elemental analysis capabilities (EDS). A detector was used to convert X-ray energy into voltage signals; this information was sent to a pulse processor, which measured the signals and passed them onto an analyzer for data display and analysis.

2.3.3 TCLP test of sludge

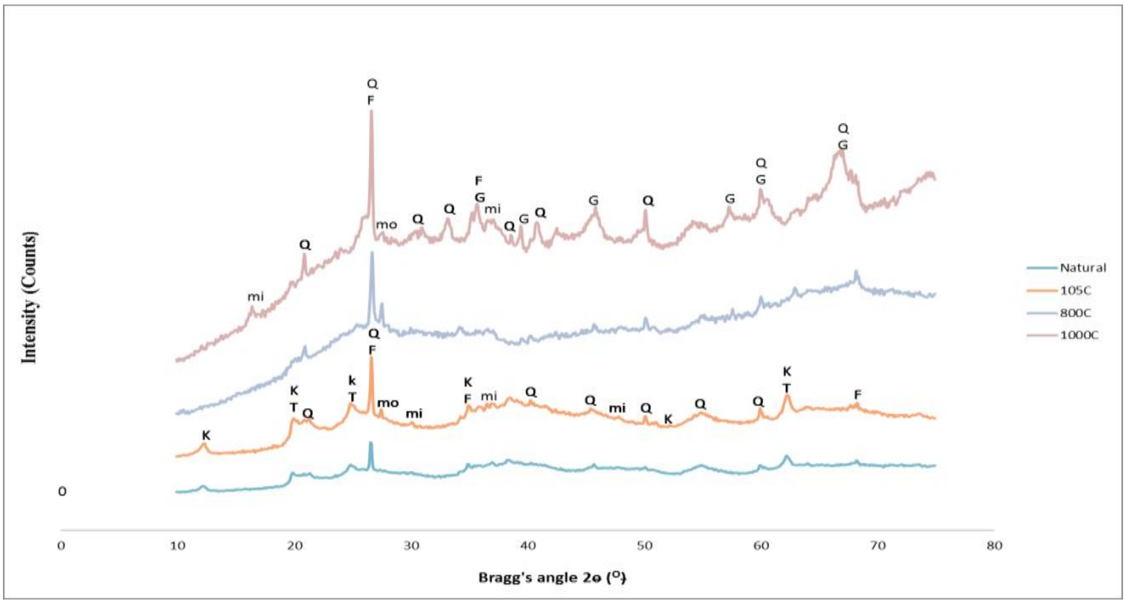
The test of toxicity characteristic leaching procedure was performed on the sludge samples to investigate the leach ability of heavy metals using USEPA (1988) Method.

3.0 RESULTS AND DISCUSSION

3.1 Mineralogical Composition of Sludge using X-Ray Diffractometer (XRD) analysis

Figure 1 illustrates the XRD patterns of natural and the thermally treated sludge at different temperatures of 105 ^{o}C, 800 ^{o}C, and 1000 ^{o}C respectively, in which it can be observed that the processed sludge is an amorphous material whose composition is of both organic and inorganic origin. There is evidence of some minerals like kaolinite, quartz, and iron oxide, montmorillonite, ferric oxide, gamma alumina, mica and talc as identified by their prominent peaks (Chen, 1977 and Mineralogy, 2013).

As shown in **Figure 1**, it can be observed that for the natural sludge, peaks corresponding to the kaolinite and quartz phases were pronounced. It is also evident that the crystallinity of sludge is low as can be seen by the background bulge which is bigger and the intensity of diffraction peak is relatively low in whole (Anyakora *et al*., 2012).



K: Kaolinite; Q: Quartz; M: Montmorillonite; F; Ferric oxide; G; Gamma alumina; Mi: Mica; T: Talc

Figure 1: XRD patterns of residue from LUDWTP sludge processed by natural drying, and thermal treatment at 105^{o}C, 800^{o}C and 1000^{o}C / 2hrs.

The result of XRD measurements of the thermally treated sludge at 105 ^{o}C did not show significant changes as it maintained its structure, thus indicating that only free water was removed. While at higher temperatures the diffraction patterns became amorphous at 800^{o}C and 1000 ^{o}C respectively. The result in **Figure 1** also showed that at 800 ^{o}C, the characteristic peaks of kaolinite observed in the natural sludge (Mineralogy, 2013) disappear giving a featureless band of X-ray amorphous meta-kaolin, which was further transformed at 1000 ^{o}C to gamma alumina and amorphous silica as indicated by their prominent peaks (Chen, 1977).

The observed traces of montmorillonite at higher temperatures (800 ^{o}C and 1000 ^{o}C) evidenced by their prominent peaks indicate that LUDWTP sludge undergoes a series of phase transformations upon thermal treatment similar to the behaviour of some kaolinite clays. Kaolin-clays undergo endothermic dehydroxylation, which begins at 550 °C – 600 °C to produce disordered meta-kaolin, but continuous hydroxyl loss is observed up to 900 °C. This can be attributed to the gradual oxolation of the meta-kaolin (Tay *et al*., 1991). **Table 1** shows the percentage weight loss (L.O.I) of LUDWTP sludge at various temperatures.

Table 1: Percentage weight loss (L.O.I) of LUDWTP Sludge at various temperatures

Component (%)	Normal feed	105°C	800°C	1000°C
L.O.I	22.6	20.78	1.36	0.04

From **Table 1**, it can be seen that the weight loss, which occur at 105 °C is due to the elimination of moisture and absorbed water. While, weight loss which occur at 800 °C and 1000 °C is due to emission of volatile organic and non-volatile organic matter, and elimination of structural water of the clay mineral and decomposition of the carbonaceous matter present in the water treatment plant sludge (Karayildirim *et al*., 2006).

3.1.2 Chemical and Microstructural characterisation of LUDWTP Sludge (SEM/EDS)

Figures 2a, 3a, 4a, 5a show the SEM micrographs of residues of LUDWTP sludge while, **Figures 2b, 3b, 4b, 5b** show the corresponding EDS spectrum of the residues processed by natural drying method and at 105 °C, 800 °C and 1000 °C respectively. **Figures 2a and 2b** show the SEM micrograph of residue of LUDWTP sludge and the corresponding EDS spectrum of the residue processed by natural drying method respectively.

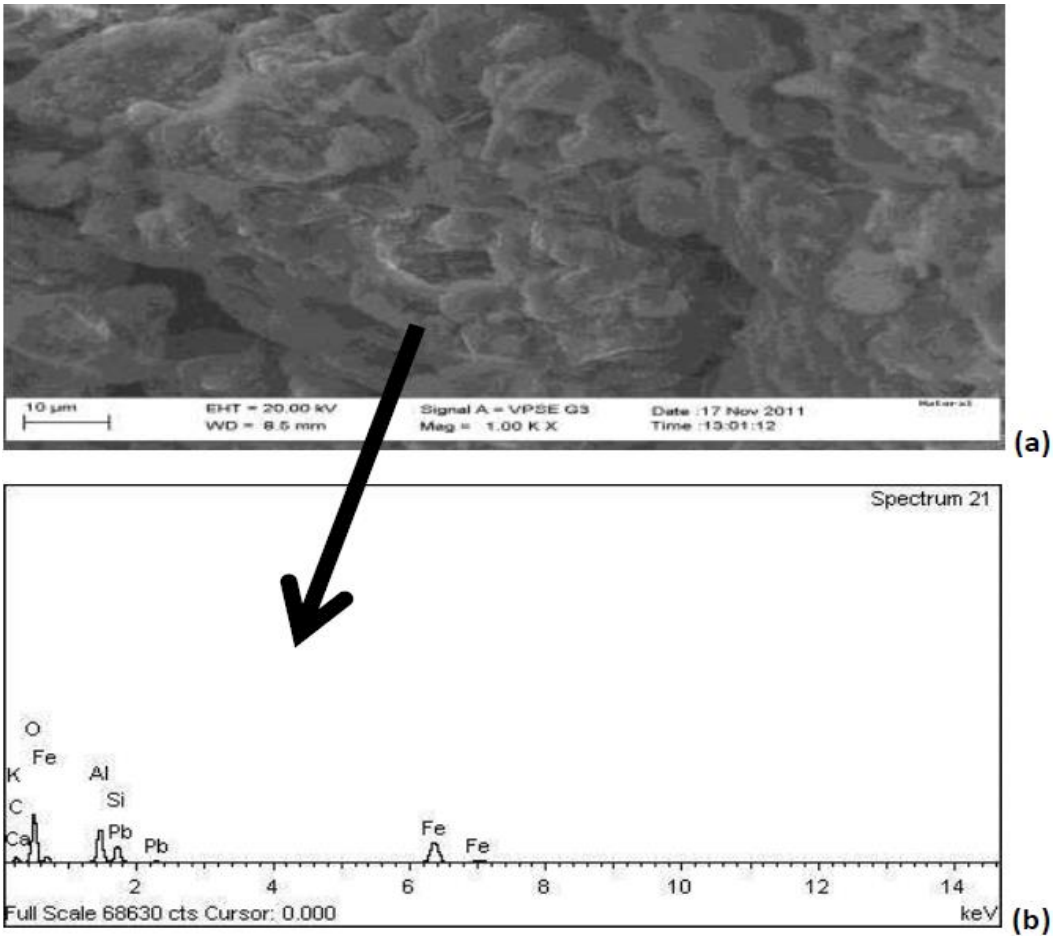


Figure 2: (a) SEM Micrograph and (b) a corresponding EDS spectrum of residue of LUDWTP sludge processed by natural drying method

From **Figure 2a**, the SEM analysis revealed that the residue has irregular shape and contained lots of pores on the surface, which could be attributed to the strong adsorption capacity of organic and heavy metals in the raw water by the coagulants. The grey regions are the bonding phases while the dark areas are the pores. The microstructure of sludge comprised of flakes of fine kaolinite clay particles (Anwarul *et al*., (2009). The presence of cracks is noted as a result of drying stress encountered during the drying process (Vieira *et al*., 2008a).

Similarly, in **Figure 2b**, it can be observed that the constituent sample elements of the EDS analysis indicated that aluminium, silicon, and oxygen with varied peaks correspond closely to the kaolinite stoichiometry (Gonzalez *et al*., 2007). This is in alliance with the elements present in the crystalline phases as observed by the XRD (**Figure 1**). Also in the **Figure 2b**. It was observed that all the peaks correspond to the element present in the phase except the carbon peak, which is a normal contaminating agent in the SEM machine as a result of the conductive carbon adhesive tape used to hold the sample in the sample holder.

Figures 3a shows the SEM micrograph of residue of LUDWTP sludge and **Figures 3b** is the corresponding EDS spectrum of the residues processed at 105 ^oC.

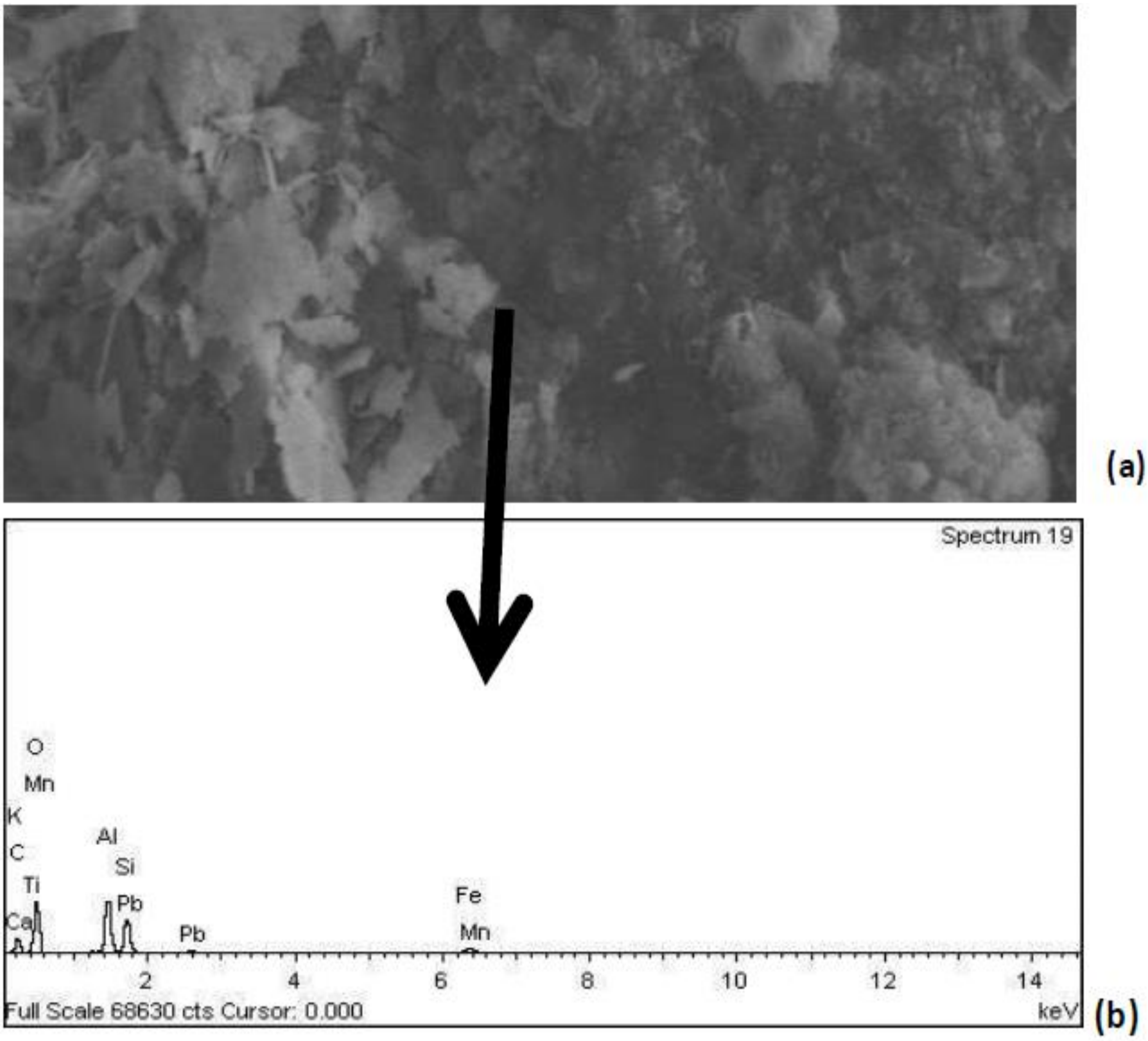


Figure 3: (a) SEM Micrograph and (b) a corresponding EDS spectrum of residue of LUDWTP sludge thermally treated at 105 ^oC/2 hours

From **Figure 3a**, it was observed that moisture loss caused interior particle separation, which signifies shrinkage. The processed specimen showed reduced porosity with increase in temperature and this attributes to increased density and mechanical strength as well as the formation of cracks resulting in the difference in shrinkage as noted by Callister (1999). It was observed that **Figure 3b** showed no significant difference from that in **Figure 2b**. This could be attributed to the fact that during thermal treatment at 105 ^{o}C, only moisture was removed. This is in line with the earlier result from XRD (**Figure** 1).

Figure 4a, shows the SEM micrographs of residues of LUDWTP sludge and **Figure 4b** is the corresponding EDS spectrum of the residues processed at 800 ^{o}C. **Figure 4(a)** shows the microstructure of the residue processed at 800 ^{o}C in the course of which dehydroxylation takes place giving an amorphous more reactive product (meta-kaolinite).Thus, it could be inferred that the LUDWTP sludge has pozzolanic potentials. Also in **Figure 4(b**), it was observed that the relationship between the atomic concentrations of the elements in the residue depicts the stoichiometry of the meta-kaolinite phase (Anwarul, 2009).

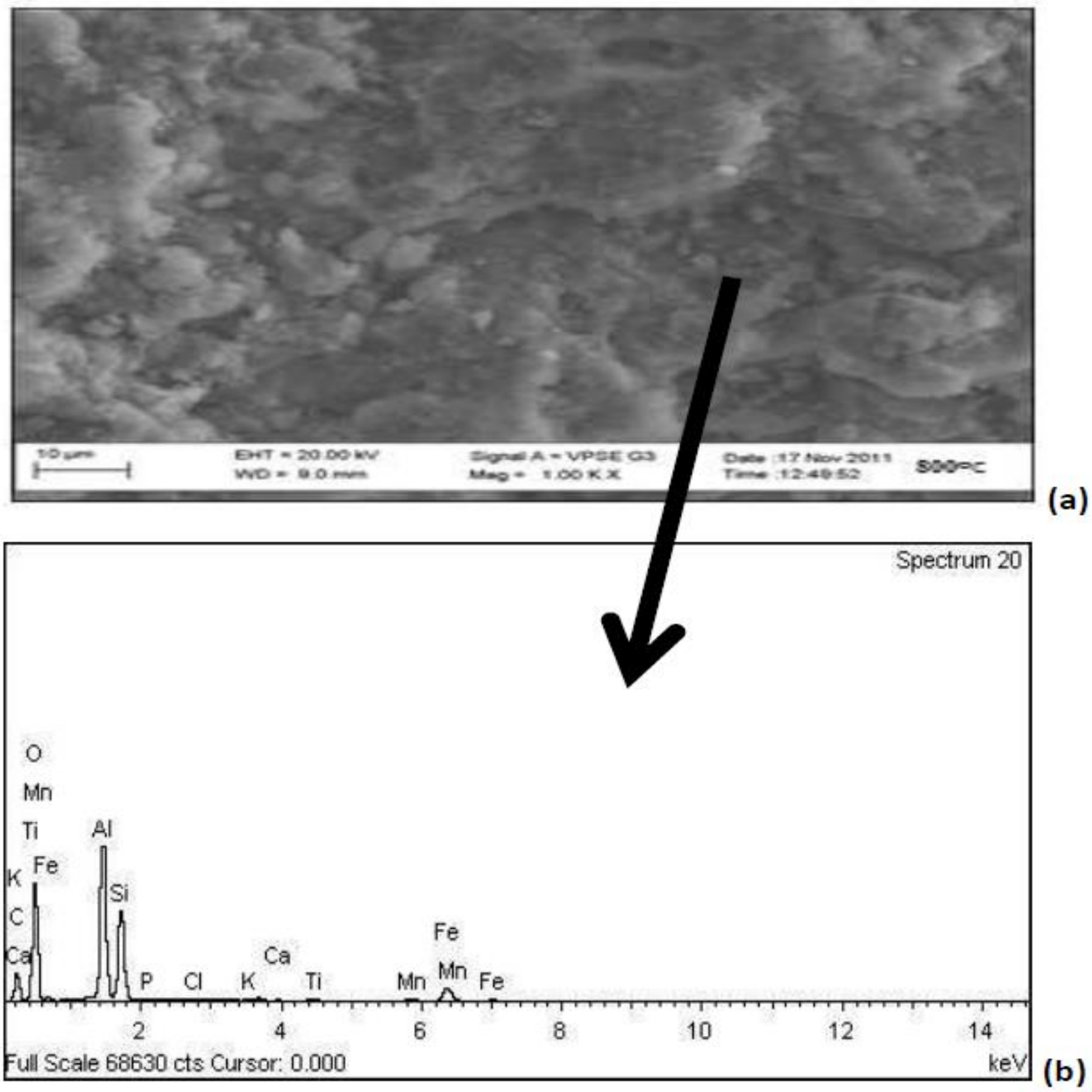


Figure 4: (a) SEM Micrograph and (b) a corresponding EDS spectrum of residue of LUDWTP sludge thermally treated at 800 ^{o}C/2hours

The presence of meta-kaolinite was not observed in the natural sludge nor the residue processed at 105 ^{o}C. This could be attributed to the transformation of kaolinite to meta-kaolinite at 800 ^{o}C, as also observed by the XRD prominent peaks in **Figure 1**. However, this transformation can be confirmed through further experimental investigations using new equipment/ technique.

Figure 5(a) shows the microstructure of the residue processed at 1000 ^{o}C in the course of which the meta-kaolinite was further transformed to gamma-alumina and amorphous silica as revealed by their particle sizes (Hosseni *et al*., 2011). The stability of the gamma-alumina was improved in the presence of alkali-earth ions (Na, K, Mg, Ca) and Silica, phosphorus or in their combinations, Belver *et al*., (2008) and Liu *et al*., (2007) and Zhou *et al*., (2007).

From **Figure 5(b),** it was observed that the EDS spectrum corresponding to the gamma-alumina and amorphous silica were almost consistent with previous studies (Anwarul *et al*., 2009).

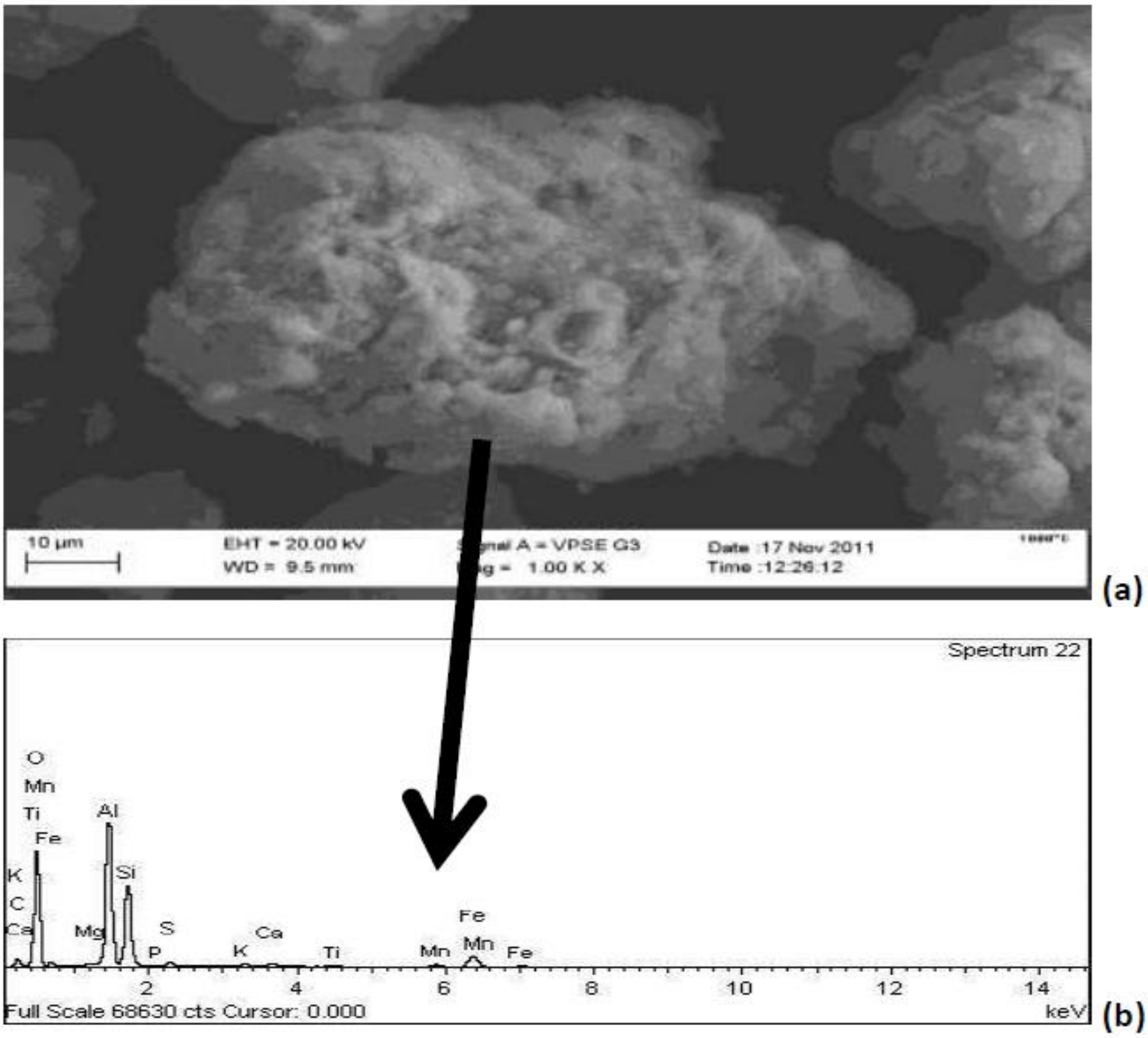


Figure 5: (a) SEM Micrograph and (b) EDS spectrum of residue of LUDWTP sludge thermally treated at 1000 ^{o}C/2hours

Gamma alumina is used as catalyst and catalyst support because of its higher specific surface area and thermal stability. Thus LUDWTP sludge could find useful application in the production of gamma-alumina.

Observed in **Figures 2 to 5**, is the high content of alumina in the LUDWTP sludge. This could be attributed to the presence of aluminium hydroxide (Alum) as the coagulant and the iron oxide, giving it a distinct rust hue. These properties are often desired in brick products as fluxing agent and colorant respectively (Dunster and Petavratzi, 2016). The results (**Figures 2 to 5**) additionally showed that porosity reduced with increase in temperature as a result of increased bonding between the particles and chemical change occurring within the entire matrix. The improved bonding generally resulted in increased strength (Callister, 1999).

3.1.3 TCLP Test Results

Table 2 shows the TCLP test results of the residues from LUDWTP sludge processed by natural drying, and thermal treatment at 105 ^oC, 800 ^oC and 1000 ^oC per 2hrs, respectively.

Table 2: TCLP test result of residues of LUDWTP sludge

Metals	Natural Sludge	105 ^oC	800 ^oC	1000 ^oC	TCLP Nigeria/USEPA
Cd	0.009	0.006	0.007	0.007	1
Cr(total)	0.193	0.277	0.278	0.202	5
Cu	0.000	0.008	0.006	0.043	NL
Hg	<0.001	<0.001	<0.001	<0.001	0.2
Pb	<0.001	<0.001	0.027	0.005	5
Zn	0.034	0.042	0.053	0.100	NL
Fe	0.844	0.877	1.643	3.432	NL
Mn	2.007	2.181	1.068	2.538	NL
Ni	0.062	0.051	0.13	0.070	NL
Al	20.649	21.369	24.750	26.844	NL

The results showed that Cd, Cr, Hg, Pb, leached to a very low concentration below the permissible limits. Other leached metals from sludge such as Al, Ni, Mn, Cu, Zn and Fe were of insignificant concern as there were no limits for them at the time of this report. Thus, it is likely that under the recommendations of the minimum requirements NESREA (2009) and USEPA (1996), the use of sludge for material applications in engineering design would not pose any environmental effects for sustainable development.

4.0 CONCLUSION

The phase and micro-chemical characteristics of LUDWTP sludge minerals and their thermally decomposed products investigated in this work using XRD, SEM and EDS techniques appear to be adequate. The phase transformation temperatures determined for the LUDWTP sludge minerals were found consistent with previous studies.

The XRD results showed that kaolinite and quartz were the major phases present in the LUDWTP sludge with ferric oxide and trace amounts of mica and talc including montmorillonite, metakaolinite and gamma alumina at higher temperatures. Also, the result of XRD measurements of the thermally treated sludge at 105 ^oC did not show significant changes, while at higher temperatures the diffraction patterns became amorphous at 800 ^oC and 1000 ^oC respectively.

The microstructure of the LUDWTP sludge comprised flake-like kaolinite agglomerates which were transformed to metakaolinite at 800 ^oC, gamma alumina and amorphous silica at 1000 ^oC in agreement with some previous studies. The EDS analysis showed that alumina and silica are the major constituent elements with high alumina content.

Thermal treatment of LUDWTP sludge affects the chemical and microstructural composition of the sludge. Thus, appropriate control of the working conditions became necessary as to optimise the applicability of sludge in engineering design and material applications for sustainable development. Meta-kaolinite (pozzolanic additive), gamma alumina and amorphous silica may be obtained by thermal treatment of LUDWTP sludge.

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