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## **An Energy Saving Option for Production of pure $\alpha$ – Alumina for Industrial Applications Using Waste Aluminium Dross**

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### **Abstract**

Globally, over four million tonnes of aluminium dross are reportedly produced throughout the world each year, in which about 95% of this material is land-filled. This material represents a major waste from primary and secondary aluminium production that contain about 15 to 70% recoverable metallic aluminium that can be extracted and later used for industrial and engineering purposes. In this study, the production of alumina from aluminium dross via leaching-cum-precipitation route for other industrial purposes was examined. Arrangement of elements, compounds present within the aluminium dross particle size -150 + 70  $\mu\text{m}$  was initially determined by X-ray Fluorescence (XRF), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) to give basis for explaining the dissolution process of aluminium the intermediate material for alumina production. The results obtained showed that 99% of aluminium was leached out using -150+78 mesh ore particle size at a reaction temperature of 60°C for 60 min reaction time with 1.5 M hydrochloric acid. The morphology analysis of the produced alumina at optimal leaching by XRD showed that it contains  $\alpha$ -alumina ( $\alpha\text{-Al}_2\text{O}_3$ ) and hydrated hydroxyl molecule (OH) as the major compound.

**Keyword:** *alumina, aluminium dross, dissolution kinetics, hydrochloric acid, leaching*

### **1. Introduction**

Aluminium dross is one of major waste products emanated during aluminium smelting and refining from bauxite ore of aluminium scraps. This waste material consists of metal, salts oxides, and other non-metallic substances which are basically classified as either black or white (Amer, 2002).

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The blackdross variety consist of high amounts of metallic oxides with low metals and granular in form of sand while the white dross is extremely of high metal content with small amounts of oxides and salts in form of large clumps or blocks (Bhattacharya *et al.*, 2004). Production of aluminium dross originated from the melting of aluminium scrap such as used beverage containers, aluminium siding, castings and treating of the melt with salt flux. The salt flux accumulates on top of the melt and forms dross or skim that contains aluminium and other elements such as magnesium, silicon and others in trace amount (El-katatny *et al.*, 2003). It is obvious that aluminium dross is a by-product of aluminium production where over fourmillion tonnes of white dross and more than a million tonnes of black dross are producedevery year across the globe, and UK alone disposes about 200,000 tonnes of white and black dross (Garret, 1982; Zuck, 1995) A report from India shows that, a rough estimate of 75,000 tons of dross is generated annually and mostly found applicable in making crackers, impure chemicals and low quality refractory bricks or stock piled (Huckabay, 1984).

Typical application of aluminium dross has been earlier reported as landfill. These quantities of dross land filled nowadays are expected to be lower because larger portion of it is being reprocessed to recover metallic aluminium and other compounds of aluminium (Huckabary and Skiatha, 1982). Other engineering utilization of this material can be found in fortification of asphalt to improve stiffness and also improve abrasion resistance in order to control micro-cracking due to its toughness (Ozerkan *et al.*, 2014). More so, the potential use of dross as a filler in concrete products such as, non-aeratedconcrete, concrete bricks and concrete roof tiles has duly investigated (Lavoie and Lachance, 1995; Lavoie *et al.*, 1991). The non-metallic residues produced during processing of aluminium dross is a major source of aluminium oxide found applicable in cement's recipe and the part adjudged to be of insignificant commercial value is declared as process waste as a direct release to the environment as waste and can have adverse impact on the ecological system (Osborne, 1995; Baba *et al.*, 2005).Increase in the global demand of valuable materials and environmental standard enforcement has led to the development of suitable treatment facilities for all industrial wastes, because the sustainable development goal and replacement of primary resources with secondary resources has become very necessary due to scarcity of former. The general overview of literatures showed that many attempts have been made by numerous researchers to utilize aluminium dross by adopting either pyro- or hydrometallurgical methods. The conventional dross oxide treatment consists mainly of grinding the dross, sieving to recover the metal value followed by water leaching to dissolve the salt in water from residue oxide which would later recover back by filtering and

evaporation technique. The residue which contains primarily alumina and other alloying elements is then calcined to obtain high grade alumina (Eslami, 2008). In another process aluminium is mechanically separated from the oxide part and the remaining oxide fines are blended into a product that can be found applicable in steel industries.

This present study presents the results of research on the utilisation of waste aluminium dross materials for the production of high grade alumina. At this moment, it is important to note that aluminium dross is widely used as landfill for general building purposes, road beds and railway lines in Nigeria. Thus, it is a worthwhile venture to upgrade its use or producing high grade alumina a material currently imported into Nigeria to meet the demand of many chemical and agro-allied industries. To the best of our knowledge, there is practically limited data in this area of research in Nigeria. Therefore, utilization from this abundant waste deposit for producing high grade alumina would no doubt add to country's economic viability, growth and development. Hence, the aim of this investigation was geared towards producing high purity alumina powder that would be able in diverse engineering applications: including electronics industry, wear resistant parts, corrosion resistant parts, chemical processing, synthetic gems, oil and gas via acidic leaching, precipitation, and calcinations techniques.

## **2. Materials and Methods**

### **2.1 Sample Location and Reagents used**

The aluminium dross used for this study was sourced from Inyishi Industrial Layout, Ikeduru Local Government Area of Imo State, Nigeria. The dross was washed with warm distilled water to remove excess salt and sand. This was oven dried at 60°C for duration of 2 hours. The oven dried dross was later grinded and pulverized into three different particle sizes: 75+63  $\mu\text{m}$ , -63+75  $\mu\text{m}$  and -75+90  $\mu\text{m}$  fractions. All reagents such as HCl and NaOH used were of analytical grade (BDH grades) and deionized water was used to prepare all aqueous solution.

### **2.2 Aluminium Dross Dissolution Process**

Leaching experiments were performed in a 1 L glass reactor equipped with a mechanical stirrer, a reaction temperature control unit and a bulk-cooler. For each experiment, 150 mL of hydrochloric acid solution of pre-determined molarity (1.5 M) and 20 g dross were mixed in the reactor vessel making 200 g/L bulk density. The contents of the reactor were initially heated with mild agitation during heating stage and on attaining the set temperature, the leaching

duration start from this point. After the desired reaction time of 2 hours, the leached slurry was immediately separated by filtration.

### **2.3 Synthesis of Alumina Powder**

The solution obtained after leaching and filtration processes were subjected to iron removal using 2% hydrogen peroxide. The purified solution was later subjected to precipitation to form aluminium hydroxide using 10% solution of aqueous ammonia that was added drop wise into the solution using a burette. The product aluminium hydroxide was calcined at various temperatures range from 200 to 1100 °C in a muffle furnace to observe the phase transformation and to identify the temperature for  $\alpha$  -alumina transformation.

### **2.4 Characterization of prepared Alumina Product**

The physico-chemical analysis of raw aluminium dross was done using standard methods (Ayinla, 2013, Baba et. al, 2010). X-ray Fluorescence (XRF) analytical equipment was used for the dross and alumina product analysis. The X-ray diffraction analysis (XRD) was carried out using a Philips Powder diffractometer Model PW 1830 X'pert system to ascertain the chemical component of the dross and the obtained product.

## **3. Result and Discussion**

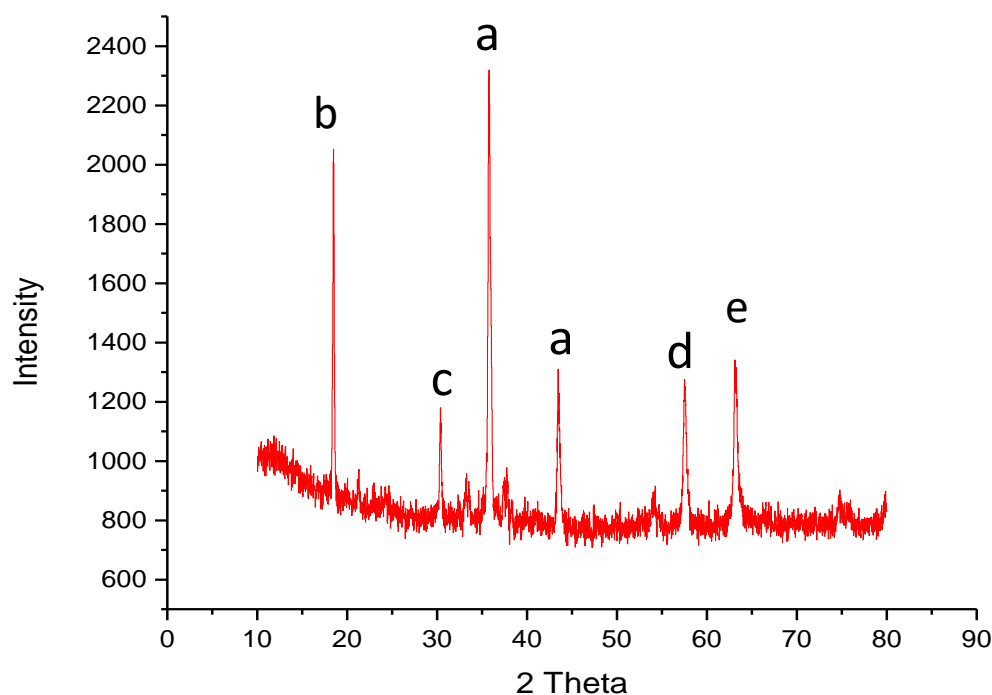
### **3.1 Physico-chemical analysis**

The results of the physico-chemical analyses performed on the pulverized Al-dross are summarized as follows: (i) The moisture content (MC) of the dross is  $3.41 \pm 0.01\%$ . This value enables us to find out the state of dryness of the ore and to make appropriate corrections in the analytical data (Tenorio and Espinosa, 2005); (ii) the loss of mass on ignition (LMOI) is  $0.98 \pm 0.011\%$ . This LMOI may account for possible presence of organic/decayed matters present in the ore under study (Tenorio and Espinosa, 2005). (iii) The pH of the aluminium dross-water suspension was 8.24. This value indicates the surface morphology of the dross – water solution to be slightly basic.

### **3.2 Spectroscopic results of used Al-dross**

The semi-quantitative chemical analysis of aluminium dross used for the production of alumina by EDXRF showed that  $\text{Al}_2\text{O}_3$  (52.86%), CaO (1.87%),  $\text{SiO}_2$  (10.9%),  $\text{Na}_2\text{O}$  (7.56%),  $\text{Fe}_2\text{O}_3$

(3.8%) and Al (20.77%) constituted the major elemental species. Other compounds detected in trace amount ( $\leq 1\%$ ) are MgO (0.14%),  $\text{SO}_3$  (0.57%),  $\text{Mn}_2\text{O}$  (0.02%) and  $\text{K}_2\text{O}$  (0.23%). The X-ray diffraction results are presented in Figure 1.



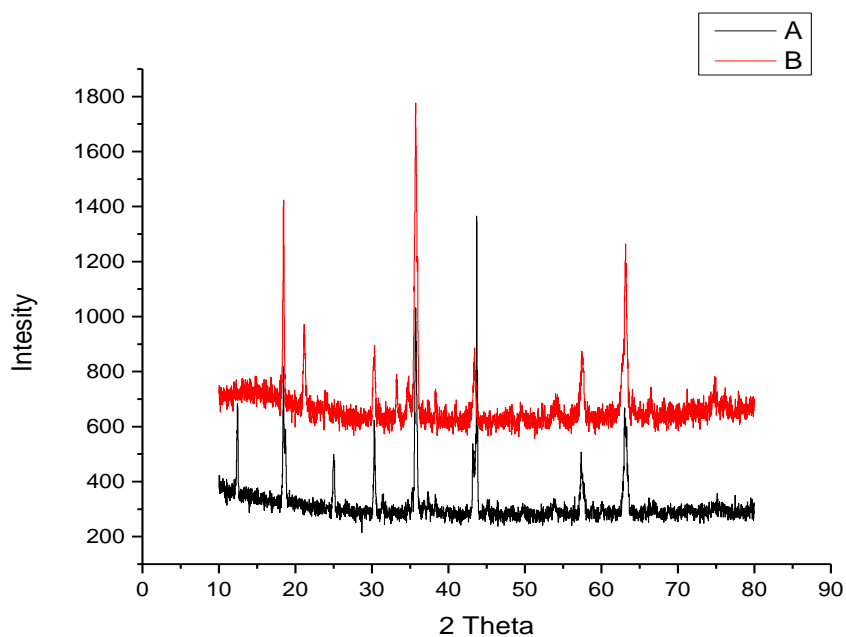
**Figure 1:** XRD spectral of raw aluminium Dross: (a) aluminium oxide (b)  $\alpha$ -quartz (c) zinc traces (d) iron traces (e) salt of chloride

The result shows that the aluminium dross consists primarily of aluminium oxide, traces of elements like Zn, Fe, salts of chlorides such as NaCl or KCl and  $\alpha$ -quartz ( $\text{SiO}_2$ ).

### 3.3 Synthesized alumina analyses

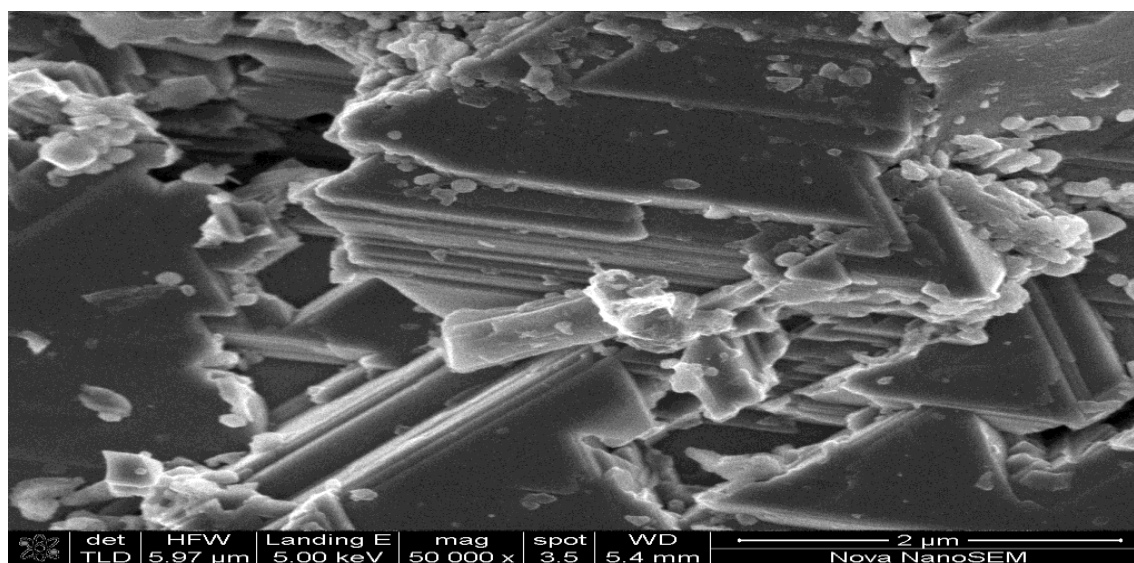
#### 3.3.1 Alumina morphology by XRD and SEM/EDS result

The XRD spectra of both precipitated aluminium hydroxide and the resulted product after calcinations is depicted in Figure 2.



**Figure 2:** XRD spectra of synthesised product: (A) aluminium hydroxide (B)  $\alpha$ -alumina

The XRD study of the product aluminium hydroxide carried out at different temperatures showed different changes of phases. At 800°C amorphous material was shown, but later change to  $g$ -alumina at 900°C and finally to  $\alpha$ -alumina at 1000°C. A similar observation was also observed in other studies (Tenorio and Espinosa, 2005; Unger and Backmann, 1982; Das *et al.*, 2007) where aluminium hydroxide was precipitated from purealuminium sulphate liquor and transformed to  $g$ -alumina at 900°C after calcinations. The XRD result was corroborated with scanning electron microscopy (SEM) analytical technique as depicted in Figure 3.

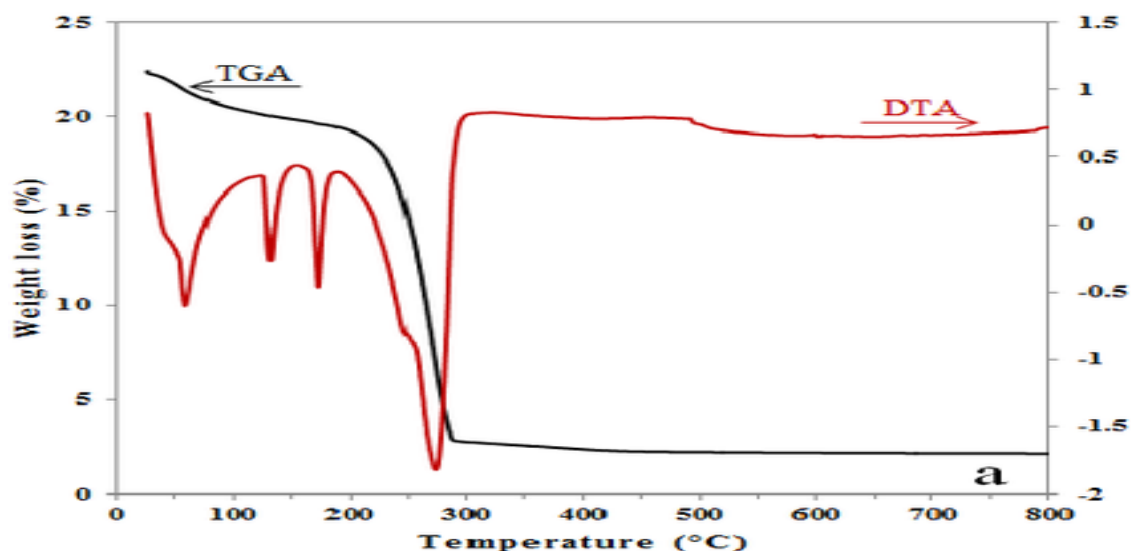


**Figure 3:** Scanning electron micrograph of  $\alpha$ - alumina.

The SEM image shows a rectangular plate structure with perfect arrangement where particles appeared to be ultrafine and having agglomeration with pores.

### 3.3.2 TGA Results

The thermogravimetric analysis result of the product synthesis is depicted in Figure 4.

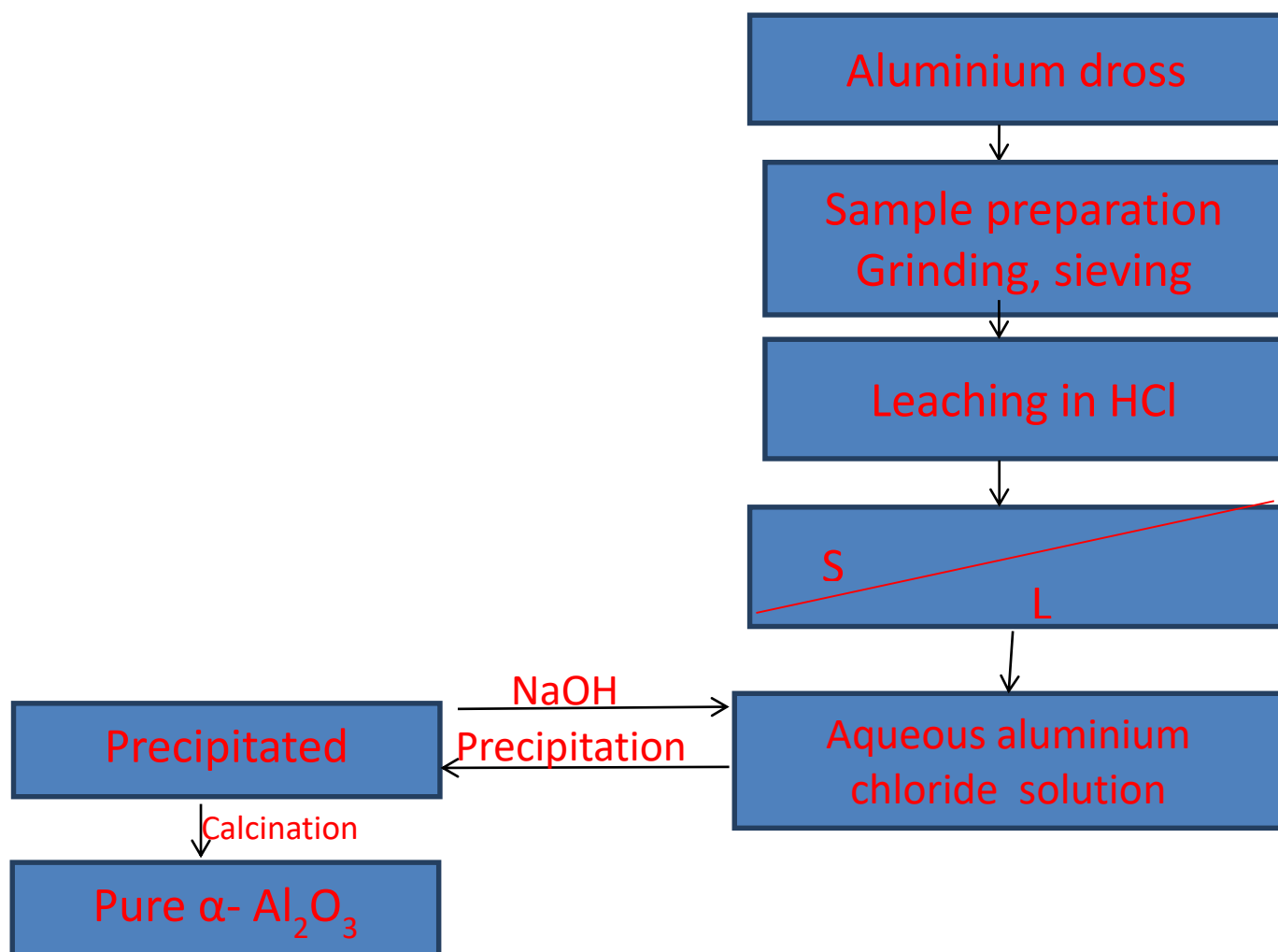


**Figure 4:** TGA diagram of calcinations process of aluminium hydroxide  $\text{Al}(\text{OH})_3$  showing the degrading form of the precipitated

TGA result of the precipitated aluminium hydroxide indicated two distinctive high rates of sample disintegration at 250°C and 900°C. The first zone disintegration was due to dehydration or dehydroxylation while that of second zone was due to desulphurization reaction (Goodboy and Downing, 1990). These aforementioned effects result lead to increase in the porosity of the material produced, a factor which makes  $\alpha$ - alumina a good industrial adsorbent.

### 3.4 Processing routes

Fig. 5 is the proposed synthesis flow sheet for aluminium dross processing to recover aluminium from waste aluminium dross and processing of leach liquor for producing value added  $\alpha$  – alumina product.



**Figure 5:** Schematic illustration of the processing route of  $\alpha$ -  $\text{Al}_2\text{O}_3$  production.

#### 4. Conclusion

The process was developed to treat waste aluminium dross following leaching cum- co-precipitation process. The established treatment option was used to optimize the recovery of pure alpha alumina. The recovery optimization was 96.9 % using 1.5 M HCl at 60°C and -75+90  $\mu\text{m}$  particles diameter. The aluminium ion containing leached solution was further treated with aqueous ammonia to obtain amorphous aluminium hydroxide. This aluminium hydroxide was calcinated at 900°C to obtain  $\alpha$ - $\text{Al}_2\text{O}_3$ , as ascertained by XRD technique, which is a high valued activated alumina and can be used as an adsorbent or for catalytic purpose.

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## References

- Amer, A. M. (2002): Extracting aluminum from dross tailings. *J. Metals*, **54**, 72–75.
- Ayinla, I. K. (2013): Dissolution Kinetics and Solvent Extraction of Copper from Chalcopyrite Ore in Acidic *Media* [Thesis], Department of Chemistry, University of Ilorin, Ilorin, Nigeria, 2012, p. 165.
- Baba, A. A., Adekola, F. A. and Faseki, M.N. (2005): A Study of the Kinetics of the dissolution of a Nigerian Tantalite ore in Hydrochloric acid, *Ife Journal of Science*, **7(2)**, 221-227.
- Baba, A. A., Adekola F.A., and Bale R.B. (2010): Development of a combined pyro- and hydrometallurgical route to treat spent zinc-carbon batteries. *J. Hazardous materials*, **171 (1-3)**, 838-844
- Bhattacharya, I. N., Gochhayat, P. K., Mukherjee, P. S., Paul, S., Mitra, P. K. (2004):, Thermal decomposition of precipitated low bulk density basic aluminium sulfate. *Mater. Chem. Phys.* **88**, 32-39.
- Das, B. R., Dash, B., Tripathy, B. C., Bhattacharya, I. N. (2007): Production of alumina from waste aluminium dross. *Minerals Engineering*, **20**, 252-258
- El-Katatny, E. A., Halany, S. A., Mohamed, M. A., Zaki, M. I. (2003): Surface composition, charge and texture of active alumina powders recovered from aluminum dross tailings chemical waste. *PowderTechnoogy*. **132**, 137–144.
- Eslami, H. (2008): Synthesis and characterization of HAP Nanocrystals via chemicalprecipitation technique, *IJPS*, **4(2)**, 127-134.
- Garret, L.W. (1982): Process for the Production of Sulfates. *US Patent No.4,337,228*.
- Goodboy, K. P., Downing, J. C. (1990): In: Hart, L.D. (Ed.), Production, Process, Properties and Applications of Activated and Catalytic Aluminas. *The American Ceramic Society Inc.*, Westerville, Ohio, p. 93.
- Huckabay, J. A. (1984): Method for Treatment of Aluminum Dross Oxides.*US Patent No. 4,434,142*.
- Lavoie, S., Dube, C., Dube, G. (1991): The Alcan plasma dross treatmentprocess, light metals. In: Elwin Rooy, (Ed.), *TMS Annual Meeting, New Orleans, Louisiana*, 981–985.
- Lavoie, S., Lachance, J. (1995), Waste minimization charges up recycling of spent lead-acid batteries: In: Queneau, P. B., Peterson, R. (Eds.), Proceeding of 3rd International Symposium on Recycling of Metalsand Engineered Materials. *The Minerals, Metals and Materials Society, TMS*, p. 791- 796
- Osborne, W. B. (1995), evaluation of aluminium content of pressed dross:: In: Queneam, P. B., Peterson, R.D. (Eds.), Proceeding of 3rd International Symposium on Recycling of Metalsand Engineered Materials. *The Minerals, Metals and MaterialsSociety, TMS*, p. 947- 954.

- Ozerkan N. G., Maki O. L., Anayeh M. W., Tangen S. and Abdullah A. M. (2014): The effect of aluminium dross on mechanical and corrosion properties of concrete, *Int. J. Innov. Res. Sci. Eng. Technol.* 3(3), 9912–9922.
- Tenorio, J. A. S. and Espinosa, D. C. R. (2002): Effect of salt/oxide interaction on the process of aluminum recycling. *J. Light Metals* 2, 89–93.
- Unger, T. W., Beckmann, M. (1992): Salt slag processing for recycling, light metals. In: Cutshall, E.R., (Ed.), *TMS Annual Meeting, San Diego, California*, 1159–1162.
- Zuck, O. A. (1995): Thermal conductivity of aluminium salt composite: In: Queneau, P.B., Peterson, R. (Eds.), *Proceeding of 3rd International Symposium on Recycling of Metals and Engineered Materials. The Minerals, Metals and Materials Society, T Das MS*, 925 – 930.