

## HYDROMETALLURGICAL TREATMENT OF BIOTITE ORE FOR PRODUCTION OF MAGNESIUM HYDROXIDE (Mg(OH)<sub>2</sub>) FOR INDUSTRIAL VALUE ADDITION

Ayinla I. K.<sup>1</sup>., Baba A. A.<sup>1</sup>., Babamale H.F.<sup>1</sup>., Padhy S.K.<sup>3</sup>., Adio O.<sup>2</sup>., Ananfi G. A.<sup>2</sup>, Ilesanmi R.O.<sup>1</sup>., Tripathy B. C.<sup>3</sup>.

<sup>1</sup>Department of Industrial Chemistry, University of Ilorin, P.M.B 1515, Ilorin-240003, Nigeria.

<sup>2</sup>Department of Science Laboratory Technology, Kwara State Polytechnic, Ilorin, Nigeria

<sup>3</sup>Hydro & Electrometallurgy Department, CSIR- Institute of Minerals and Materials Technology, Bhubaneswar – 751013, India

\*Corresponding author's E-mail: ibkuranga@gmail.com, ayinla.ik@unilorin.edu.ng Tel.:+2347065360971

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### ABSTRACT

Magnesium hydroxide is a popular inorganic compound used in wide range of applications such as: flame retardant in polymers, special ceramics, fillers in bleaching agent and a raw material for preparation of magnesium oxide, which were currently imported into Nigeria from foreign countries using hard earned currency. Thus, this current study focused on dissolution kinetics of a biotite ore treated via leaching in sulphuric acid leachant. During leaching, parameters such as leachant concentration, particle size and reaction temperature on the extent of ore dissolution were examined. At optimal leaching conditions (2.5 mol/L H<sub>2</sub>SO<sub>4</sub>, 80°C), 98.23 % of the initial 10 g/L ore reacted within 120 minutes. The shrinking core dissolution model was adopted to evaluate the various dissolution parameters for better understanding of the rate determining step. Experimental results indicated that the reaction rate of the leaching process was controlled by diffusion model, with calculated activation energy (E<sub>a</sub>) of 38.56 kJ/mol. By considering the solubility differences of corresponding elements, a two-step precipitation process was applied to remove Fe and Al and recovery of Mg. Subsequently, magnesium hydroxide was prepared by precipitation from the leach solution with ammonia as the precipitant. X-ray diffraction (XRD) and scanning electron microscope (SEM) analyses showed that the high purity Mg(OH)<sub>2</sub> existed in the form of flower-like spherical aggregation. Furthermore, the final solution of ammonium–potassium chlorate can be used for agricultural compound fertilizer.

Keywords: inorganic, optimal, leaching, ammonium-potassium chlorate, fertilizer

### INTRODUCTION

Biotite is a mineral with aluminosilicate layered which consist of some valuable elements of magnesium and potassium (Zheng *et al.*, 2015) compared with other kinds of mica minerals which are of practical importance in industry. It contains some traces of iron inside and greatly restrained from practical use due to its poor insulation performance. As a result, biotite slice could only be used as cheap fillings of building materials (Liu *et al.*, 2011). Biotite is the darkest members of the mica group of minerals with typically brown to black in color. Its occasionally occurs in a dark green variety (Rominger *et al.*, 1976; Camtakan *et al.*, 2012). The mineral exhibit the perfect single cleavage that characterize the mica group and allows it to be easily separated into thin flexible sheets that transmit light. Their cleavage is a reflection of the minerals crystal structure that consists of aluminium silicate sheets

that are weakly bound together by layers of positive ions (Yu *et al.*, 2004; Ma *et al.*, 2014). This positive ion in biotite is dominantly potassium and iron with only minor magnesium (Xiong *et al.*, 2014; Tran *et al.*, 2013). Because the chemical bonds between the aluminium silicate sheets are much weaker than those within the sheets, both of these mica minerals can be easily separated into thin sheets that are both flexible and elastic. If biotite is bent, they tend to return to their original shape once the applied stress is removed (US Geological Survey, 2014; Jane *et al.*, 2014). Metallic magnesium is found in many other minerals such as muscovite, magnesite, dolomite, brucite, serpentinite (Elsner and Rothon, 1988).

Many studies revealed that magnesium could be recovered from tailings and waste residues by treating boron mud with magnesia which is a potential magnesium source for synthesis of

Mg(OH)<sub>2</sub> and proposed the formation mechanism of the flower-like Mg(OH)<sub>2</sub> via anti-drop precipitation method (Shen et al., 1997). In another study hydrochloric acid was applied to recovery magnesium from magnesite tailings and MgCl<sub>2</sub> · 6H<sub>2</sub>O with a purity of 91% was extracted by evaporation of leach solution obtained at 40 °C after one hour, 1.0 M acid, solid-to-liquid ratio of 10 g/L (Zhang et al., 2009). Also, Karidakis and co-workers, investigated the recovery of magnesium from nickel laterite soil using sulphuric acid as leachant. Leaching process was followed by precipitation with Ca(OH)<sub>2</sub> and the precipitate was a mixture of Mg(OH)<sub>2</sub> and CaSO<sub>4</sub> · 2H<sub>2</sub>O. While pure magnesium recovery rate of 90–99% was achieved at only 20 °C after 45 minute (Karidakis et al., 2005)

In this current work, an innovative process to recover magnesium from the Akiri, Awe local Government of Nigeria biotite rich microcline is proposed, which is suitable to deal with magnesium-rich muscovite. The aims focused on optimization of various leaching conditions for magnesium recovery from biotite by leaching with sulfuric acid solution and to determine factors affecting its leaching efficiency. Various process parameters like the concentration of sulfuric acid, temperature and leaching particle diameter are investigated in terms of metal leaching efficiency. This is followed by a two-step precipitation process to prepare Mg(OH)<sub>2</sub> and recovery of potassium from the leach solution using ammonia (Zheng et al., 2015; wang et al., 2017). Finally, the purity of Mg(OH)<sub>2</sub> produced was also determined by XRD and SEM methods.

## EXPERIMENTAL

### Ore sample and Instrumentations

The biotite sample was obtained from an ore deposit at Akiri Village, Awe Local Government Area of Nasarawa State, Nigeria. It was crushed, ground, and sieved using the American Society for Testing and Materials (ASTM) standard sieve into three size fractions: -90 + 75, -112 + 90, and -300 + 112 μm. All experiments were performed with -90 + 75 μm size, unless otherwise stated. The elemental analysis of the ore sample was carried out using an X-ray fluorescence (XRF) technique (Philip's model 120454/3). The mineralogical

purity of the ore was examined using a PHILIP PW 1800 X-ray diffractometer (XRD) with Cu K $\alpha$  1 (0.154 nm) radiation, generated at 40 kV and 55 mA. The microstructural characterization was done by scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) respectively. Deionized water and British Drug Houses (BDH) reagent grade hydrochloric acid and ammonia were used to prepare all solutions.

### Equipment and methods

All Leaching experiments were carried out in a 250 mL spherical glass reactor equipped with a mechanical stirrer. Typically, for each run, 100 mL H<sub>2</sub>SO<sub>4</sub> solution of predetermined molarity was charged to the reactor and heated to the required temperature, following the procedures developed by (Baba et al., 2013; Baba and Adekola, 2015; Ayinla et al., 2018; Baba et al., 2015). For every leaching experiment carried out, the solution mixture was freshly prepared by dissolving 10 gL<sup>-1</sup> of the ore in the leachant at 55°C. According to previous experimental results, it was found that the effect of stirring speed could be neglected when it was larger than 360 rpm. All leaching experiments were carried out at the stirring speed of 400 r/min (Jena et al., 2014). The optimal concentration of sulphuric acid, which gave the maximum dissolution (2.5 mol.L<sup>-1</sup>), was subsequently used for optimization of other leaching parameters including temperature and particle size. The activation energy (*E*<sub>a</sub>) and constants were determined from the Arrhenius plots. The fraction of dissolved biotite was calculated from the initial differences in mass of the amount dissolved or undissolved at various intervals up to 120 min, after oven drying at 60°C for 12 h (Baba et al., 2005). The post leaching residual products obtained at 80°C in 2.5 mol.L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> solution was then analyzed by XRD and scanning electron microscopy (SEM) for morphological studies.

### Production of Mg(OH)<sub>2</sub>

Finally, a Mg(OH)<sub>2</sub> precipitate was obtained by adjusting the pH to 11.0, whereas the potassium/ammonium sulfate solution was achieved by filtration using the pressure filtration unit. The precipitate was dried at 90 °C for 1 h to obtain magnesium hydroxide (Mg(OH)<sub>2</sub>) The

concentrations of metal ions ( $Mg^{2+}$ ,  $Al^{3+}$  and  $Fe^{3+}$ ) determined were then used to characterize the precipitation rate as a function of pH. The precipitation rate of the ion was calculated as follows:

$$\text{Precipitation rate} = \left[1 - \frac{C_i}{C_o}\right] \times 100 \quad (1)$$

Where  $C_o$  and  $C_i$  are the concentrations of the

specific element in initial and titrated leachate solution.

## RESULTS AND DISCUSSION

### Ore characterization

#### Elemental analysis by XRF

The elemental chemical analysis of the -90+75  $\mu m$  biotite ore fraction by XRF as contained in the ore is summarized in Table 1.

Table 1: Elemental composition of biotite ore by XRF.

compound	MgO	Al <sub>2</sub> O	SiO <sub>2</sub>	TiO <sub>2</sub>	K <sub>2</sub> O	SO <sub>2</sub>	Fe <sub>2</sub> O	CaO	P <sub>2</sub> O <sub>5</sub>	LIO	Total
Composition %	25.04	8.67	53.59	1.31	5.82	0.24	0.94	0.13	0.15	4.23	100

According to the results (Table 1) of the analysis, the biotite crystal contains 25.19% MgO, 5.7% K<sub>2</sub>O, 8.60% Al<sub>2</sub>O and 53.59% SiO<sub>2</sub> as the major element. Other traces elements such as Fe, Ca and P. in minor less than 1%.

### Phase analysis

Powdered X-ray diffraction of the magnesium ores (Figure 1) gave phase composition dominated by biotite ( $MgAl_2(Si_3Al)(OH)_2$ ), microcline ( $KAl(SiO_{11})$ ), kaolinite ( $Al_2Si_2O_5(OH)_4$ ) and quartz ( $SiO_2$ ) which may be a mixture of pyrothite ( $Fe_7S_8$ ).

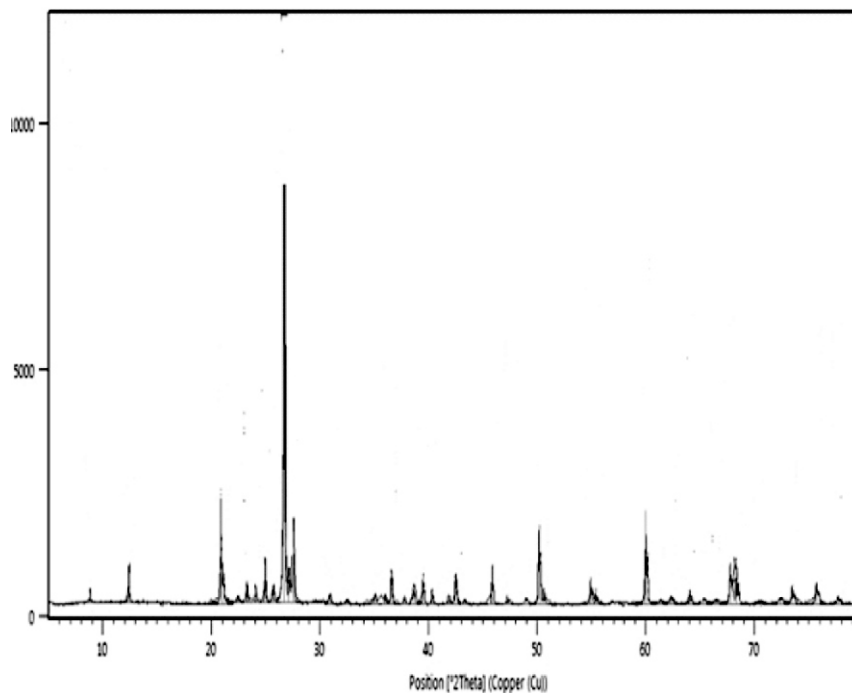


Figure 1: XRD spectrum of raw muscovite ore (a) biotite ( $MgAl_2(Si_3Al)(OH)_2$ ) (b) microcline ( $KAl(SiO_{11})$ ) (c) kaolinite ( $Al_2Si_2O_5(OH)_4$ ) (d) quartz ( $SiO_2$ )

The XRD data also showed the presence of minor abundance (62 wt %) of other associated compounds, including galena (PbS), cassiterite (SnO<sub>2</sub>) and hausmannite (Mn<sub>3</sub>O<sub>4</sub>).

### Petrography and Mineralogy

Microscopic studies of the ore (Figure 2) also showed highly reflecting bright yellow to redish brown biotite as the dominant phase. Other

associated minerals include pleochroic pale yellow  $\alpha$ -quartz (SiO<sub>2</sub>), opaque pyrite (FeS<sub>2</sub>), light and dark gray to opaque lead sulphide galena (PbS). This was corroborated with scanning electron microscopy analytical technique as depicted in Figure 3. It shows a rectangular structure with perfect sets where particles appeared to be ultra-fine and having agglomeration.

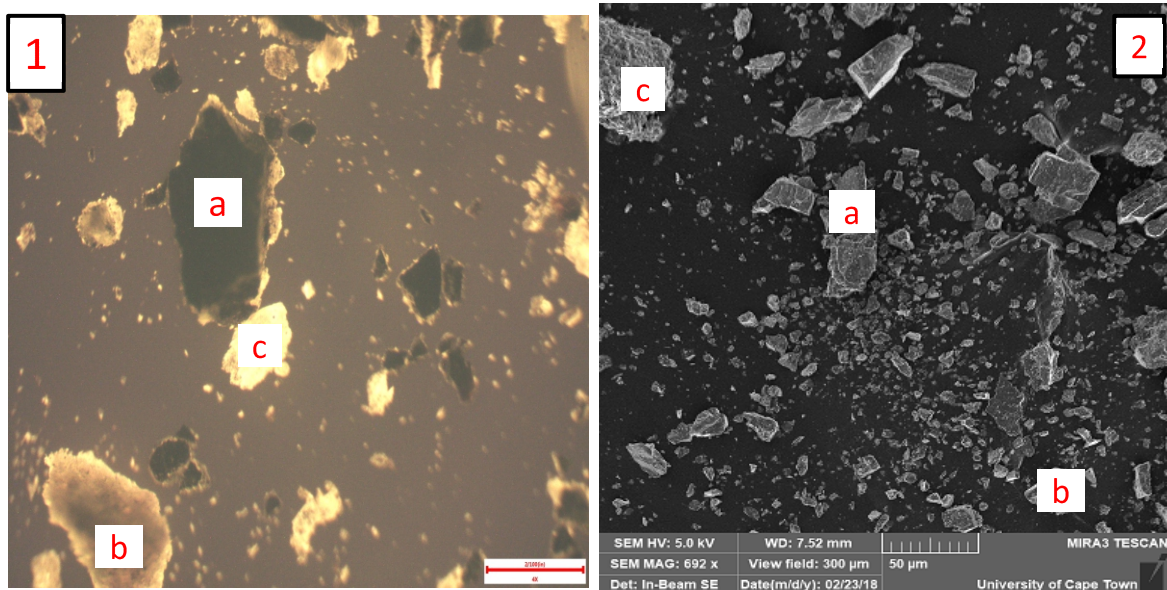


Figure 3: Photomicrograph (1) and SEM micrograph (2) of raw biotite ore: (a) biotite (b)  $\alpha$ -quartz (c) pyrite

### Condition Leaching Tests Influence of leaching time

Figure 3 shows the effect of the reaction time on

the leaching efficiency of Mg at sulfuric acid concentration of 2.5 mol/L, 55 °C, and sulfuric acid-to-biotite ratio of 4 mL/g.

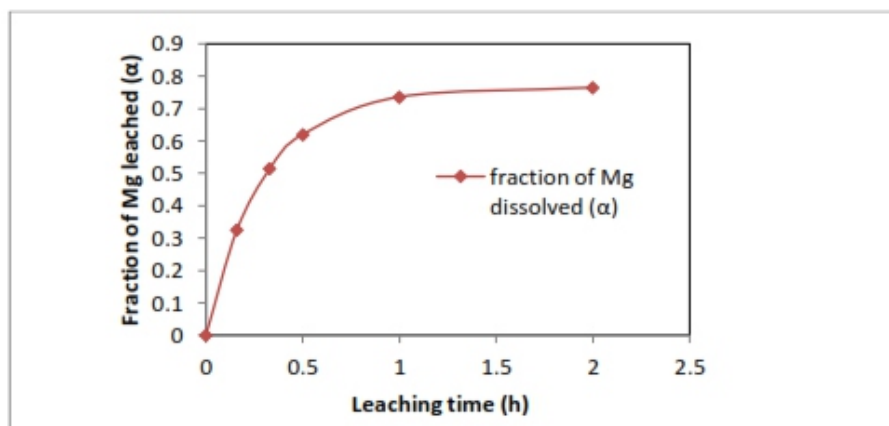


Figure 3: Effect of leaching time on Mg extraction



The leaching efficiency of Mg into solution increases with adding leaching time. When the leaching time is 1 h, the leaching efficiency of Mg is less than 65%. While extending time to 1.5 h, the leaching efficiency of Mg extends up to 71%, and more than 80% of Mg was leached out after 2 h. Also, Figure 3 illustrates that the dissolution rates of Mg are very close and reach an apparent steady-state within 2h.

### Influence of agitation

The significant influence of agitation on the Mg extraction from the biotite ore was carried out in the range of 145-650 rpm at 55°C. The result

obtained shows that the stirring speed had a significant effect on the magnesium dissolution up to about 350 rpm. Above 400 rpm, agitation had no further effect on the dissolution rate. Therefore, the stirring speed of 400 rpm was maintained for subsequent study. This was considered to be appropriate to eliminate the effect of this variable.

### Influence of H<sub>2</sub>SO<sub>4</sub> concentration

The influence of H<sub>2</sub>SO<sub>4</sub> concentration on the extent of magnesium extraction was studied in the acid concentration range 0.5-3.0 M (Figure 4).

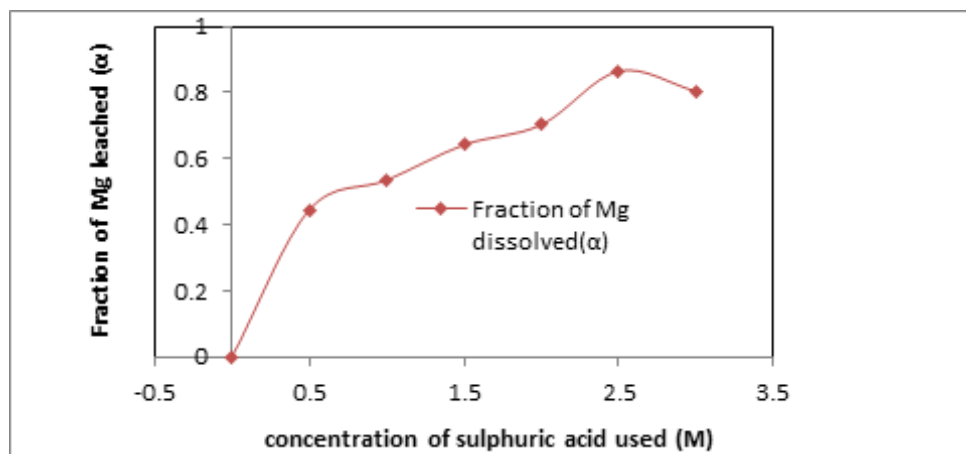


Figure 4: Effect of H<sub>2</sub>SO<sub>4</sub> concentration on Mg extraction (55°C, 0.09 nm particle size).

It can be seen that the acid concentration has a significant impact on the leaching efficiency of whose leaching efficiency was raised from 45% to 85% when the acid concentration of H<sub>2</sub>SO<sub>4</sub> was varied from 0.5 mol/L to 2.5 mol/L respectively. The increase of magnesium leaching efficiency while increasing the acid concentration is due to acid concentration effect on increasing the H<sup>+</sup> activity that results in further dissolution of biotite (Harvie et al., 1984). The leaching efficiency reached the maximum value at the acid concentration of 2.5 mol/L. The reason for this

may be due to the biotite structure collapsing at this concentration or Mg<sup>2+</sup> species block H<sup>+</sup> diffusion. Similar behaviour was also observed in other studies (Özdemir and Çetişli, 2005; Özdemir et al., 2009).

### Effect of leaching temperature

The effect of reaction temperatures on the leaching efficiency of Mg at sulfuric acid concentration of 2.5mol/L, time of 2 h and sulfuric acid-to-biotite ratio of 4 mL/g is presented in Figure 5.

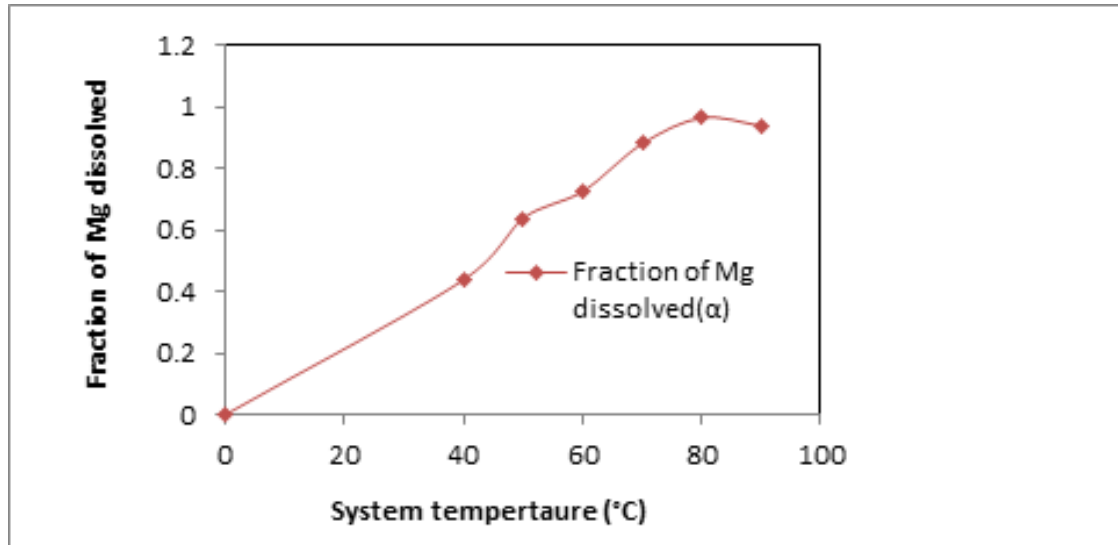


Figure 5: Effect of system temperature on Mg extraction ( $\text{H}_2\text{SO}_4 = 2.5 \text{ M}$ ,  $0.09 \text{ nm}$  particle size, Leaching duration = 2h)

As expected, the leaching efficiency of Mg increases with raising temperatures and the leaching efficiency of Mg was up to 95.8% at 80 °C. However, the temperatures continue to rise is to the formation of jarosite because of the presence of iron, which could lead to a poor leaching efficiency of (Wang et al., 2005; Baron and Palmer, 2002). In addition, Al may also incorporate into the leached residue as a result of precipitation of alunite and therefore causes a fallen in the purity of silica gel (Liu and Papangelakis, 2005). Thus, the optimized condition of the leaching temperature is fixed for 80 °C. The activation energy was calculated from Arrhenius equation:

$$k = Ae^{-E_a/RT} \quad (2)$$

where,

$k$  = reaction rate constant,  $A$  = frequency factor,  $E_a$  = activation energy,  $R$  = Gas constant,  $T$  = absolute temperature.

By plotting natural logarithm of reaction rate constant versus inverse of temperature, the activation energy was calculated to be 38.56 kJ/mol. From result of the obtained activated energy it can be conclude that the dissolution process followed diffusion control kinetic model.

### Effect of particle sizes

Figure 6 illustrates the effect of the reaction time on the leaching efficiency of Mg at sulfuric acid concentration of 2.5 mol/L, 90 °C, and sulfuric acid-to-biotite ratio of 4 mL/g. The leaching efficiency of Mg into solution increases with decrease in particle diameter of the ore. When the leaching time is 1 h, the leaching efficiency of Mg is less than 73%. While extending time to 1.5 h, the leaching efficiency of Mg reached up to 85%, and more than 98.23% of Mg can be leached out after 2 h.

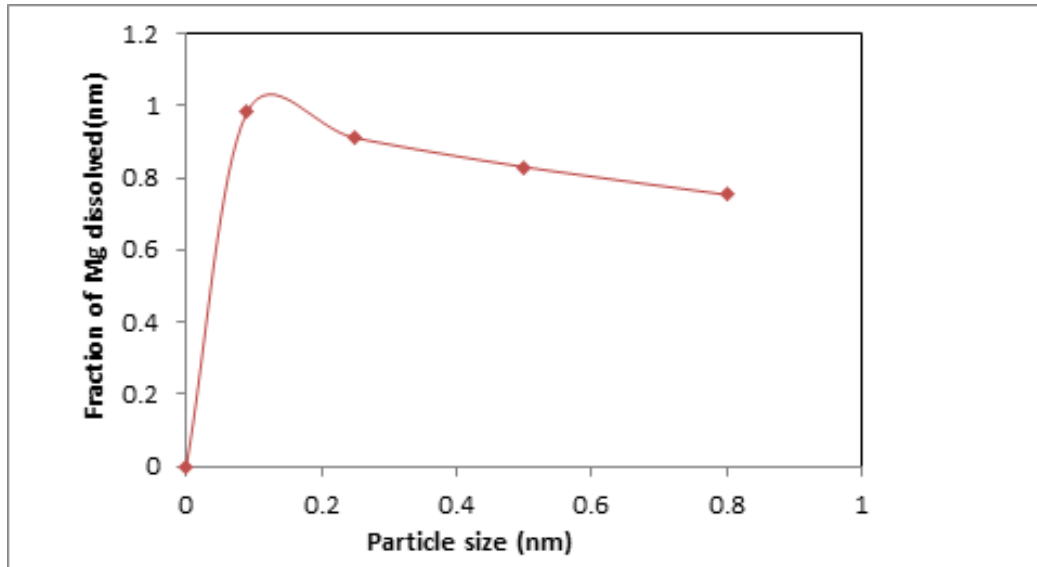


Figure 6: Effect of particle on Mg extraction (80°C, H<sub>2</sub>SO<sub>4</sub> concentration = 2.5 M, Leaching time = 2 h)

### Effect of pH on precipitation of Mg from leach liquor

Figure 7 presents the precipitation rate of Mg<sup>2+</sup>, Fe<sup>3+</sup>, where Fe<sup>3+</sup> has been oxidized using hydrogen peroxide and Al<sup>3+</sup> from leach solutions considering pH (Figure 7). The chemical reactions

for the available metal ions in the leached liquor with ammonia (NH<sub>3</sub>) solution are as follows:

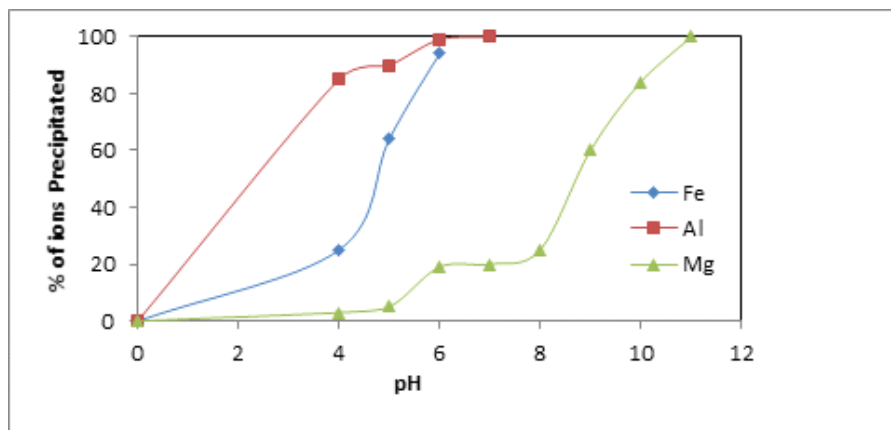
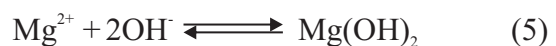
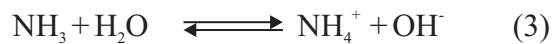


Figure 7: Precipitation trend of major metal ions from the leached liquor.

Most of Fe<sup>3+</sup> and Al<sup>3+</sup> precipitate when the pH value is 4 whereas about 3% of Mg<sup>2+</sup> precipitates. The precipitation rate of Mg<sup>2+</sup> begins to increase as the pH value increases from 8.0 to 11.0. Therefore, according to the different precipitation rate between Mg<sup>2+</sup> and the other two metal ions, a two-step precipitation process with the neutralizing agent was adopted. Fe and Al removal increased by adding the pH, whereas the magnesium loss from aqueous solution increased distinctly at pH 8.0. Consequently, the optimum

pH value at the first step for Fe, Al ions and Mg ion separation is about 8.0, at which the majority of Fe<sup>3+</sup>, Al<sup>3+</sup> have been precipitated as Fe(OH)<sub>3</sub> and Al(OH)<sub>3</sub> mixture which could be used to prepare ferric oxide, aluminium hydroxide separately, while near 3% of magnesium is in the solid residual. The recovery of Mg in the second step of precipitation reached 99.9% Mg at pH 11.0. This was affirmed by the XRD pattern, which gave Mg(OH)<sub>2</sub> [86-0438] as the only major peak detected (Figure 8).

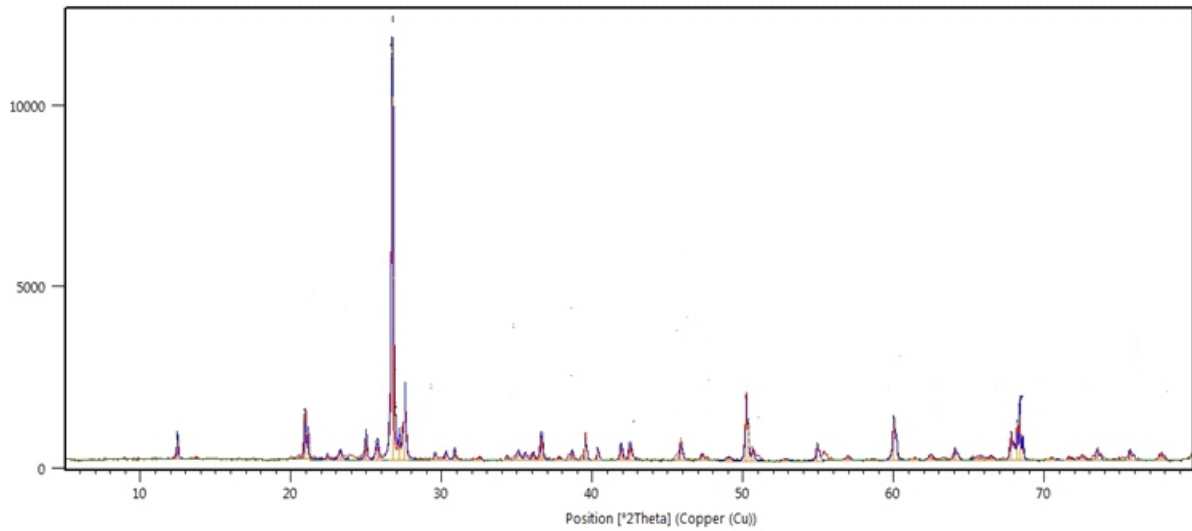


Figure 8: XRD pattern of precipitated  $\text{Mg}(\text{OH})_2$  product.

The scanning electron micrograph of the synthesized  $\text{Mg}(\text{OH})_2$  also support the purity quality ascertained by XRD as depicted in Figure 9.

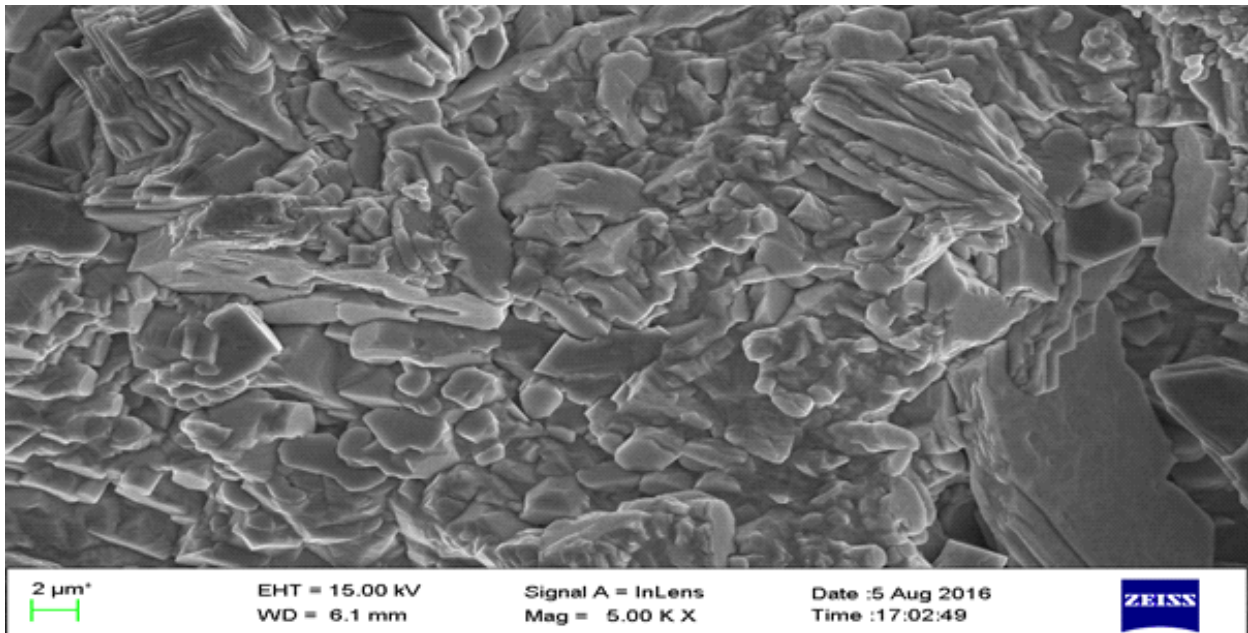


Figure 9: SEM image of synthesized magnesium hydroxide ( $\text{Mg}(\text{OH})_2$ )

Figure 9 appeared to give dense with specific morphological structure quite different from original biotite powder. Thus, there exist some powder particles of irregular shape covering the dense surface. This component is significant to a powder of high potential for filler in bleaching agent and other adsorbents.

## CONCLUSIONS

The ore mineralogical analysis reveals that biotite, kaolinite and quartz are the major phase. The

sulphuric acid leaching is a feasible route for this ore for selective dissolution of magnesium. The magnesium leaching rate is strongly influence by  $\text{H}_2\text{SO}_4$  concentration, temperature and biotite ore particle sizes. Under standard leaching condition of 2.5 mol/L  $\text{H}_2\text{SO}_4$ , 80 °C, and +90-78 µm particle size, it is possible to achieve 85% magnesium recovery in 2 h. however, under higher  $\text{H}_2\text{SO}_4$  concentration and lower particle size above 95% magnesium extraction is feasible. Magnesium was recovered from leached liquor as pure



magnesium hydroxide ( $\text{Mg}(\text{OH})_2$ ) [86-0438] by precipitation with ammonia solution and oven drying at 90 °C for 120 minutes. Lastly, the final solution of ammonium–potassium sulphate that obtained as filtrate can further be used for production of fertilizer, find applicable in agriculture.

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**Conflict of Interest:** The authors want to declare here that there is no conflict of interest

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