International Journal of Research Publication and Reviews

Journal homepage: www.ijrpr.com ISSN 2582-7421

Quantitative Leaching of Awo Mica Ore in Acetic Acid Solutions

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DOI: https://doi.org/10.55248/gengpi.2022.3.10.36

ABSTRACT

Mica is mined and processed throughout the world and used as functional filler in many applications, such as in joint compound, electrical insulation, and plastics. The single most important feature of mica is high aspect ratio, the ratio of width to thickness. High aspect ratio mica filler is desirable in specialty applications, such as in automotive plastics, electrical industry. Considering these applications, Awo mica was quantitatively leached using acetic acid with focus on the determination of elemental composition of the ore using XRF technique, the determination of mineralogical purity of the ore using XRD, SEM and EDS techniques, the determination of the dissolution rate using acetic acid so as to predict the optimum conditions for the beneficiation of the ore for industrial applications and lastly, to investigate the potential for developing flake muscovite deposits in Southwestern Nigeria.

Various parameters like effect of leachant concentration, temperature and the particle size have been investigated. Increase in leachant concentration and temperature brings about increase in dissolution rate and decrease in particle size increases the dissolution rate. The calculated reaction order and Activation energy (Ea) was 0.9437 and 21 kJ/mol respectively. The shrinking core model employed showed diffusion-controlled mechanism as the rate determining step for the dissolution process.

Detailed ore characterization, for example the XRF for the elemental analysis shows that SiO_2 is the most abundant compound (58.30%). It also contains other major compound like Al_20_3 (18.70%) with the other detected compounds found to occur in minor to trace levels (< 1%). The residual product was quantitatively characterized by XRD and SEM instrumentations.

Keyword: High aspect ratio, Ore characterization, quantitative leaching, leachant, Activation energy, shrinking corel, XRD, SEM.

1. INTRODUCTION

Humans have been using mica for many years. Mica was first mined in India about 4,000 years ago, where it was used primarily in medicines, and some Hindu physicians still incorporate biotite mica into medicines today. Early civilizations also used mica for decorations, as windows and as surfaces on which to draw or paint. Maya temples were decorated with mica pigments, which were incorporated into the stucco to make it sparkle in the sun. In North America, ancient inhabitants adorned gravesites and burial mounds with animal figures made of mica.

The word “mica” comes from the Latin word *micare*, meaning to shine or flash, an allusion to mica's ability to reflect and refract light [11]. Mica belongs to a family of minerals known as phyllosilicates, has a monoclinic structure with unit structure of one octahedral sheet sandwiching between two tetrahedral sheets [14]. It is a generic name for a group of complex hydrous potassium-aluminum silicate minerals that differ somewhat in chemical composition; examples are biotite, lepidolite, muscovite, phlogopite, and vermiculite. Muscovite is the commonest of all and whenever the word mica is used it is understood to mean muscovite. Other names of mica include: Cat-gold, Cat-silver, Glimmer, Glist, Katen-silber, Katzen-silber, Katzengold, Or des chats, Rhomboidal Mica [7].

Mica represents 37 phyllosilicate minerals that have a layered or platy texture. These mica minerals all have a crystalline structure that forms layers that can be split into thin sheets, a physical property called perfect basal cleavage [17]. It also possesses some unique characteristics like: an average dielectric constant, excellent mechanical strength, outstanding electrical properties, high surface leakage resistance, low moisture absorption, high heat resistance, low bulk density, ability to dissipate heat.

Mica is a natural mineral resource found in abundance. It is one of the most abundant clayey raw materials [18]. Statistics showed that the world leader in mica production is China with over 30% market share, second place is held by USA with 27.3% of world output. The return of investment on the export of muscovite mica sheets is estimated to be 10 -15% per transaction.

The general formula for minerals of the mica group is $XY_{2\text{-}3}Z_4O_{10}$ $[OH, F]_2$. With X=K, Na, Ba, Ca, Cs, (H_3O), (NH_4); Y=AL, Mg. Fe^{2+}, Li, Cr, Mn, V, Zn; and Z=Si, Al, Fe^{3+}, Be, Ti.

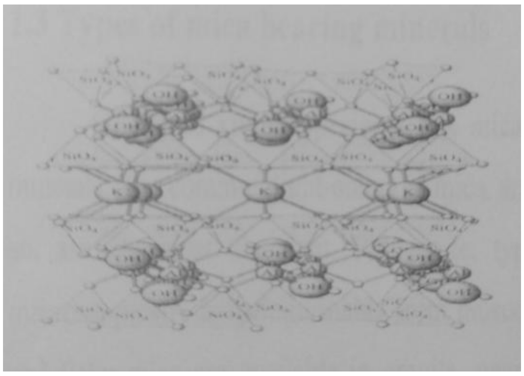


Fig. 1. The structure of muscovite mica [6]

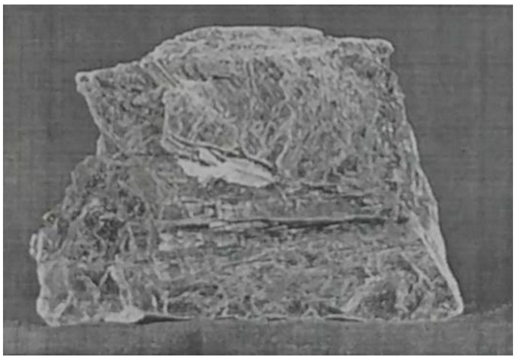


Fig. 2. Sample of mica: [10]

1.1 Properties of mica

Generally, they can withstand temperatures up to 700 °C, they have perfect basal cleavage and capable of splitting into thin laminae and can split into flexible and transparent films. The mineralogy of the mica structure consists of two silica tetrahedral sheets with a central edge-sharing octahedral sheet forming a sandwich.

1.1.1 Physical properties

The physical properties of Mica are summarized in the table below.

Table 1: Physical Properties of Muscovite Mica [6]

Chemistry	$KAI_2(AISi_3JO_{10})(F, OH)_2$; potassium aluminum silicate hydroxide fluoride
Class	Silicates
Subclass	Phyllosilicates
Group	Micas
Colour	Colourless, white, yellow, silver, green, or brown
Hardness	2.5 to 3.5
Avg. Specific Gravity	2.77 to 2.88
Lustre	Vitreous to pearly
Transparency	Transparent to translucent
Cleavage	Perfect in one direction (0011; sheets or flakes with elastic quality
Fracture	Uneven; rarely observed due to perfect cleavage
Streak	White
Crystal System	Monoclinic: 2/m: several polytypes 2MI. IM, 31; 2M12 common with space group C2/c
Crystal Habits	Distinct crystals rare; tabular with prominent faces (001); can have diamond shaped prism faces up to 60 degrees (110) mimicking orthorhombic symmetry; can also have hexagonal appearance (010); can have penetration twins with (310) at twin axis.
Gem Use	None
Field Indicators	Perfect cleavage, color and associations, elasticity, crystal habit

Associated Minerals	Beryl, tourmaline, quartz, feldspars
Associated Rock Types	Igneous, metamorphic, detrital sedimentary

1.1.2 Chemical properties of mica

Chemically, they contain complex silicate of aluminium and alkalis with hydroxyl. They crystallize in monoclinic system. Some varieties may contain iron, magnesium, lithium and rarely fluorine, barium, manganese and vanadium. Natural muscovite was expanded by thermal treatment and ion exchange [4] and dissolution of muscovite at acidic pH.[5]

1.2 Reactions of mica

The formation of muscovite requires high temperature of about 400 °C. At high temperature, muscovite will form from the hydroxides in acid solution as shown by the following equation [9]

$$3(Al_2O_3H_2O) + 6(SiO_2, 1/2H_2O) + 2KCl \rightarrow K_2O.\ 3Al_2O_3.6SiO_2.2H_2O + 2HCl + 9H_2O \qquad (1)$$

Conversion of Muscovite (a form of mica) to Kaolinite at 300 °C

$$2[K_2(OH)_4Al(Si_6Al_2)O_2] + 4HCl + 6H_2O \rightarrow 3(OH)_8Al(Si_4O_{10}) + 4KCl \qquad (2)$$

1.3 Industrial uses of mica

Mica is so unique with a lot of application either solely or in combinations with other minerals. Table 2 shows some of the most important uses of mica. Also, the degree of property improvement resulting from the addition of mica to a polymer is influenced by a number of factors which include its degree of crystallinity, particle morphology, method of grinding, polymer polarity.

Table 2: Typical Uses of Mica

Grade	Sieve Size (mesh)	Typical Uses
Coarse flakes	6	Oil well drilling. Artificial snow
Med-coarse flakes	10	Christmas ornaments, display material
Fine-coarse flakes	16	Concrete block fillers, refractory bricks, gypsum boards, asphalt roofing felts, shingles
Coarse-fine powder	30	Metal annealing, absorbent in explosives. disinfectants, automotive components
Med-fine powder	60	Welding electrodes, cables & wires, foundry works, pipeline enamels, mastics, lubricants, adhesives
Fine powder	100	Texture paints, acoustical plasters, ceiling tiles
Superfine powder	325	Paints, plastics, rubber products, paper

Though mica has numerous uses, the basic industry of application includes the electrical industry in which Nigeria has to brought up to speed. Therefore, the aim of the study is to quantitatively leach Awo mica using acetic acid and the objectives will therefore be focused on:

- Determination of elemental composition of the ore using XRF technique.
- The determination of mineralogical purity of the ore using XRD, SEM and EDS techniques.
- The determination of the dissolution rate using acetic acid so as to predict the optimum conditions for the beneficiation of the ore for industrial applications.
- To investigate the potential for developing flake muscovite deposits in Southwestern Nigeria.

2. LITERATURE REVIEW

Browning [12] Conducted laboratory and continuous process development work on weathered mica pegmatites and micaeous schist ores to determine the feasibility of recovering commercial grade mica concentrates by floatation. The research has resulted in the floatation of two processes for floatation of mica: an acid cationic method for recovery of coarse mica, and an alkaline anionic-cationic method for recovery of fine mica.

Kurganskaya et al., [13] worked on Mica dissolution mechanism, it was concluded that the layered structure radically changes the dissolution mechanism using data from VSI & AFM experiments involving muscovite to evaluate crystallographic controls on mica dissolution. This approach provides a basic mechanistic link between the dissolution kinetics and suggests a resolution to the general problem of mica reactivity.

Osman [16] used high cation-exchange capacity (CEC) muscovite mica with a homoionic surface which was prepared by replacing the Li surface ions of partially delaminated Li-mica with K. The exchange reaction with Cu(trien)2+, whose concentration can be photometrically measured, is a fast and convenient one-step method to determine the CEC of mica.

3. MATERIALS AND METHODS.

3.1 Sample location and collection

The mica ore used for this project was mined from Awo town, the Headquarters of Egbedore local government of the State of Osun in the south western part of Nigeria. Thus, mica ore sample used for this study was collected in Awo town in the state of Osun.

3.2 Apparatus

The following apparatus and instruments were used and were made available by the Department of Chemistry, Faculty of Physical Sciences, University of Ilorin, Nigeria. They include:

- Beaker
- Funnel
- Filter paper
- 100 ml measuring cylinder
- Stirring rod
- Conical flask
- 100 ml standard flask
- 120 ml plastic sample container
- 60 ml plastic sample container
- stop watch
- Muffle furnace
- Oven (Gallenkamp model DV-160 Oven BS)
- Desiccator (laced with silica gel)
- Hot plate equipped with magnetic stirrer
- Weighing balance (S. Mettler)
- Hanna pHEp pocket sized pH meter.

3.3 Reagents

The following reagents were used for the synthesis of Hydroxyapatite. Some were bought and the some were prepared in the laboratory:

- Distilled water
- Acetic acid
- Acetone

3.4 Sample preparation

The physical properties of the said sample were tested to ensure it correlates with what was needed. This also includes sorting out of the sample. The method used here is a dry separation process for mica developed by the U.S. Bureau of Mines. It involves crushing, grinding and screening the mica to close size fractions. Its advantages are that no liquids are used throughout the process and it is not capital intensive as well as the process is eco-friendly.

3.4.1 Crushing

The crushing process was embarked upon to make grinding easy which turns the lamination into thin films.

3.1.2 Grinding

The mica sample was broken into smaller pieces using hand and was crushed and grounded into particles using mortar and pestle. The sample was pulverized using a pulverizer from geology department to obtain fine form of mica. Scrap mica was ground by three processes, namely dry grinding, wet grinding and micronizing. Dry mica powder is prepared by grinding in high-speed hammer mills. Wet-ground mica is produced by grinding in water involving preferential delamination of flakes. Churn mills, with large wheels or rollers rotating on horizontal shafts are used for this purpose. The crushed ore is then pulverized to reduce the thin films into fine particles. Muscovite mica sheets with a thickness of 25 lm were irradiated by various kinds of swift heavy ions. Muscovite mica was also delaminated by heating with concentrated lithium nitrate solution according to [19]

3.1.3 Sieving/pulverization

The sample was sieved in the department of Geology, University of Ilorin into three particle size fractions (-90+75µm, -112+90µm, -250+112µm) which was done according to standard procedures.

3.1.4 Preparation of standard solution

Several methods exist in the preparation of the solution which includes;

- Solution preparation using synthetic salts.
- Solution preparation using gravimetric factors where concentration of different molarities can be prepared through Gravimetric factor or Serial dilution approach.
- Preparation from liquid solutions in which different concentration from liquid chromogenic reagent can be prepared from the reagent assay with the following properties - percentage purity, specific gravity and molecular mass. It should be noted these properties vary depending on the nature of reagent as well as the manufacturer.

The last method was basically used in this project work for the preparation of the solutions used.

3.5 Physiochemical study

For this project work, the following studies were carried out:

- pH determination: The pH meter was calibrated with a buffer solution of pH 7.0. A 5.0 g of the sample was weighed and was dissolved in 50 mL of distilled water, the mixture was vigorously stirred for about 5 minutes and then allowed to equilibrate. The pH of the supernatant solution was then measured using Hanna pH meter pocket sized. The pH measurement was then monitored over a period of one week according to [3].
- Moisture content determination: 1.0 g of mica sample was weighed into a pre-weighed crucible and placed inside the Gallenkamp, model DV-160 oven BS for about 3 hours. The weight of the crucible and sample was then taken; the sample was placed in the oven again for an additional 1 hour to confirm the weight of the crucible and sample by checking for consistency in their weight. The difference in mass which was the mass of the moisture content was then determined. The moisture content determination of the mineral enabled us to find out the state of dryness or otherwise of the mineral and enable us to make appropriate corrections in the analytical results.
- Loss of mass on ignition (LOMOI): The weighed crucible and oven-dried calcite ore sample was put inside the muffle furnace and roasted at high temperature of about 500 °C – 600 °C for about 2 hours. After roasting, the sample was withdrawn from the furnace and allowed to cool for about an hour. Later, the cooled product was then weighed.

3.6 Processing

This can be done in many ways, for instance, the electrometallurgy and pyrometallurgy process. However, hydrometallurgy process was selected for this project work. Hydrometallurgical processes involve the removal of metals from different types of ores, concentrates and waste products by aqueous solutions containing different chemical reagents [8]. The process generally includes the following unit operations: (i) Roasting (much less frequent) (ii)Leaching by acids, bases or water, (iii)Removal of impurities, (iv)separation and recovery process and (v)refining of recovered metals [1].

3.7 Leaching procedure

The following study was conducted on mica sample to verify the effect of concentration with time at a constant temperature of 55 °C. The sets of concentration range used were; 0.1 M, 0.25 M, 0.5 M, 0.75 M, 1.0 M acetic acid solutions at a time interval of 10, 30, 60,120 mins.

- The solution was prepared using the above data calculations, an approximate 2.9 ml of acetic acid measured to prepare 0.1 M acetic acid solution.
- 100 ml was measured out into a reaction vessel, then 1.0 g was poured into the reaction vessel and immediately heated to about 55 °C for 10

mins. The same process is repeated for 30, 60, 120 mins.

- Filtration: After the time has elapsed, the solution was filtered out.

Similar pathway was continued to get other data for the effect of concentration, effect of temperature and the effect of particle size.

3.8 Instrumentation

The Mica ore was characterized by each of the following: Elemental analysis using XRF (fluorescence), Structural purity using XRD (X-ray diffraction) and Structural analysis through SEM (scanning electron microscope).

3.9 Leaching studies

Leaching is the process of extracting a soluble metallic compound from an ore by selectively dissolving it in a suitable solvent e.g., water etc. The leaching experiment was performed in a 250 ml glass reactor equipped with a mechanical stirrer. The study was carried out at different organic acid concentrations and at different temperatures and time of contact. The residue after leaching was filtered, washed, dried and weighed to determine the amount of the ore dissolved at various leaching times. Some selected residue after appropriate treatments was fully characterized by SEM, and XRD.

4. RESULTS AND DISCUSSION

4.1 Ore Characterization

4.1.1 Elemental composition by XRF

The elemental composition of the raw mica ore was determined using a MINI PAL 4 EDXRF spectrometer at geological survey unit, Kaduna, Kaduna State, Nigeria. The chemical composition of the Mica ore under study is shown in Table 3

Table 3: Elemental Analysis of Awo Mica Ore by EDXRF

ELEMENT	SiO_2	Al_2O_3	K_2O	Fe_2O_3	Na_2O	MnO
% COMPOSITION	58.3	18.70	8.25	7.51	1.74	0.496

It is evident from Table 1 that the calcite ore is comprised majorly of SiO_2 (58.30%). Other compounds detected from low to trace levels are Al_2O_3 (18.70%), K_2O (8.25%), Fe_2O_3 (7.51%) etc.

4.1.2 Morphological purity by XRD

The EMPYREAN X-Ray diffractometer at geological survey unit, Kaduna. Kaduna State, Nigeria was used in identifying the mineralogical purity and mineral phase of the ore. The XRD result of the Mica ore sample is shown in Fig. 3.

The Joint Committee on Powder Diffraction Standard file numbers (JCPDS) are put in curl brackets: (1) $Al_2H_2KO_{12}Si_4$ {58-2034} - Muscovite (2) SiO_2 {75-8320}- Quartz (3) MgO {71-6452}- Magnesium oxide. The result of the morphological purity by X-ray Diffraction (XRD) shows that the raw mica ore consists predominantly of Muscovite ($Al_2H_2KO_{12}Si_4$) and Silica (SiO_2) otherwise referred to as quartz.

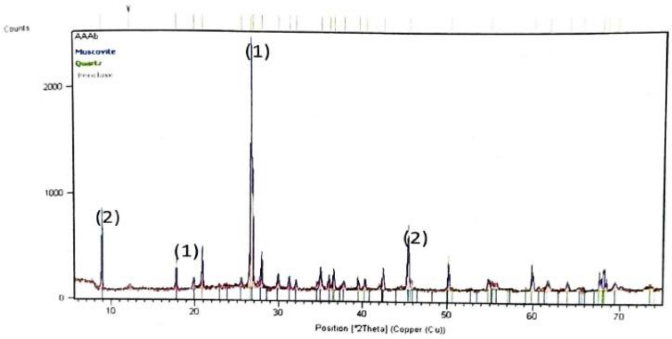


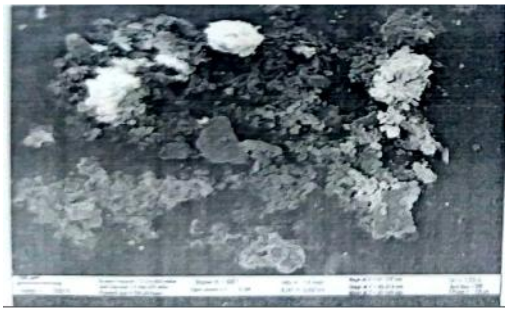
Fig. 3: XRD pattern of Awo (Nigeria) mica ore with important compounds detected.

4.1.3 Structural analysis by SEM

The mica ore and leached product morphology was determined using he FEI NOVA NanoSEM 230 with a field emission. The EDS detector is an Oxford x-max and uses INCA software to analyze the spectra. This is available at the electron microscope unit at the University of Cape Town, Rondebosch, South Africa. The SEM micrographs of raw mica ore is shown in Fig. 4.

The analysis of the SEM micrograph of the raw mica ore in Fig. 4 shows cluster-like structure before forming a molten magma type.

(a)



(b)

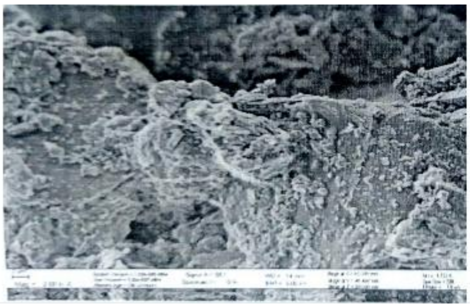


Fig 4 (a and b) SEM micrographs of raw mica ore

4.2 Leaching studies

4.2.1 The effect of acetic acid concentration

The effect of acetic acid concentration [0.1 - 1.0 mol/L] on the mica ore in solution at 55 °C; at different leaching time [10-120 mins] with moderate stirring. As concentration of acetic acid increases with time, the rate of dissolution also increases. Under optimum condition (55 °C, 1.0 M) with moderate stirring, the extraction rate was found to be 65%.

4.2.2 Effect of leachant concentration

Values obtained when 30 samples of 1 g of the Awo mica ore each was leached using 100 ml acetic acid at concentrations ranging from 0.1 mol/L - 1.25 mol/L at different Ieaching time. The effect of [CH_3COOH] on the dissolution of mica was studied using a moderate stirring speed and <90µm particle size at a temperature of 55 °C. Result showed that the percentage of ore reacted ranges from 10% to 65% as leaching experiment progresses from 0.1 mol/L to 1.5 mol/L. A plot of fraction of ore dissolved versus leaching time at different concentrations was obtained as shown in Fig. 5. From the graph, it is evident that an increase in acetic acid concentration causes a corresponding increase in the faction of ore dissolved with leaching time. Hence, the rate of ore dissolution is controlled by the hydrogen ion concentration.

4.2.3 Effect of temperature

The effect of temperature on the rate of mica dissolution was studied over the temperature range of 25 ^{o}C – 75 ^{o}C, using moderate stirring speed, liquid solid ratio 10 g/L and a particle size of 90 µm. The experimental result obtained showed that the fraction of ore dissolved increases from 0.32 g to 0.84 g at 120 mins which indicates that the temperature had a slight effect on the fraction dissolved. Therefore, a moderate temperature of 55 ^{o}C can be used for further experiments. A plot of Fraction of ore dissolved versus leaching time at different temperatures was obtained as shown in Fig. 6. The plot shows that increasing temperature of the system accelerates the reaction rate and this leads to the increase in the number of ore dissolved.

4.2.4. Effect of particle size

Three different particle sizes were examined using l g of mica ore, 1 mol/L acetic acid, temperature of 75 °C and leaching time of 5, 10, 30 and 120 mins respectively. From fig. 7, it is evident from the result that the rate of mica dissolution decreases with increase in particle size. This may be due to the increase in the active surface area. Therefore, 90 µm particle size is the preferred particle size for further experimental analysis.

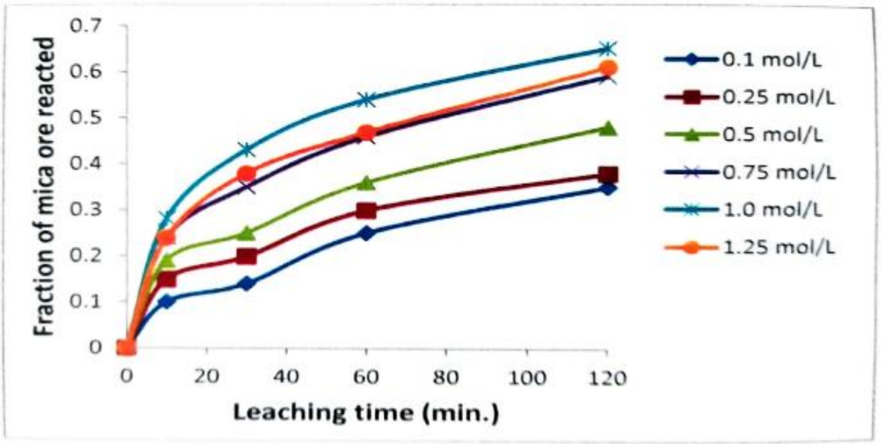


Fig. 5: Plot of fraction dissolved against leaching time at different concentrations

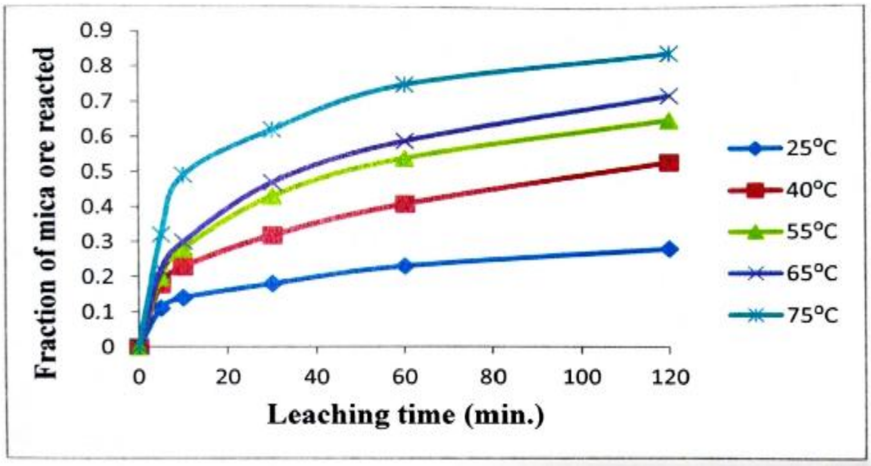


Fig. 6: Fraction of mica dissolved versus leaching time at different temperatures.

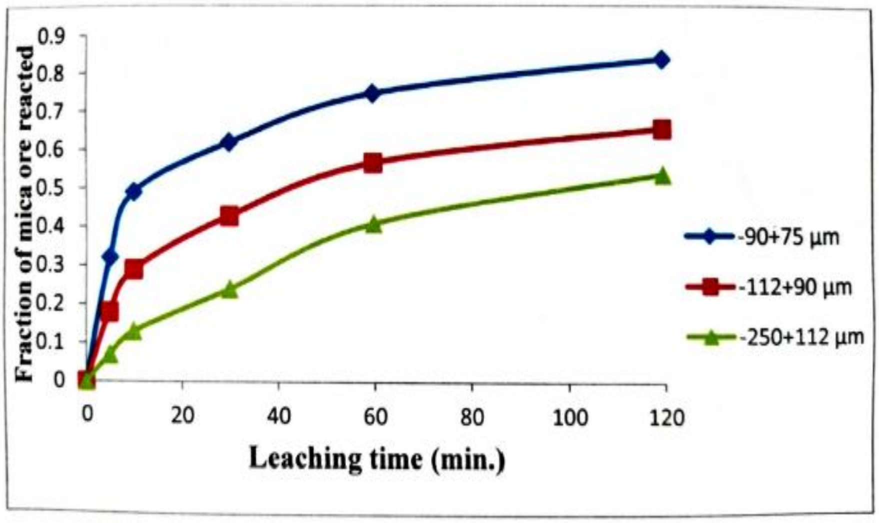


Fig. 7: Fraction of mica dissolved versus leaching time at different particle size.

4..3 Dissolution Kinetics Studies

4.3.1 Shrinking Core Model

The shrinking core model checks whether the leaching process is either controlled by the diffusion of the reactants through the solution boundary layer or through a solid product layer or by rate of the surface chemical reaction. The simplified equations of the shrinking core model when either diffusion or the surface chemical reactions are the slowest step i.e., rate determining step are expressed below as equations 1 and 2 respectively [15].

For the diffusion-controlled processed; $1-2/3\alpha(1 - \alpha)2/3 = k_d t$ (1)

For the Surface chemical reaction; $1-(1-)1/3 = k_r t$ (2)

Where;

α = fraction of mica ore dissolved,

t = reaction time,

k_d = rate constant for the diffusion-controlled process,

k_r = rate constant for the surface chemical reaction process.

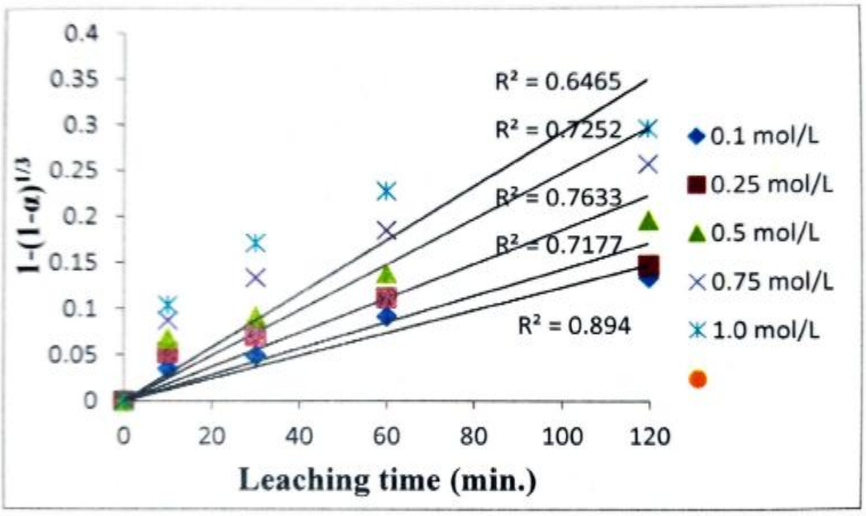


Fig. 8: Plot of Chemical Controlled reaction for EOC

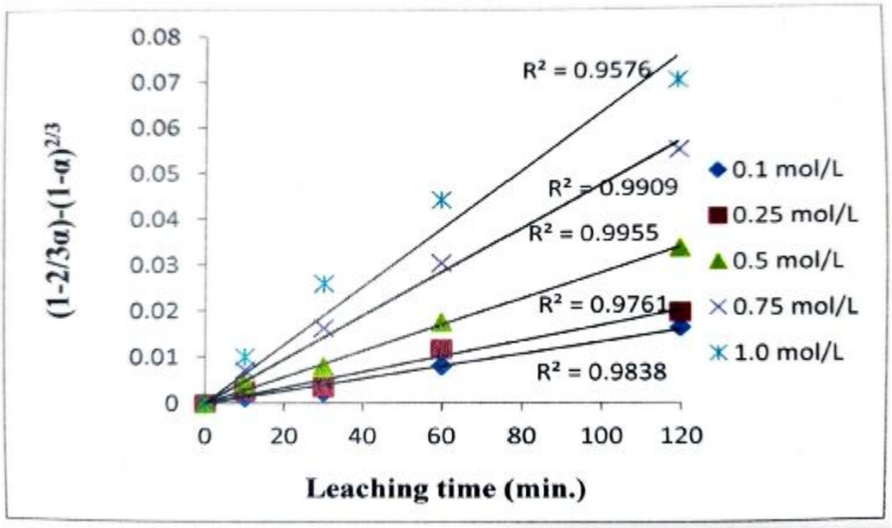


Fig. 9: Plot of Diffusion Controlled reaction for EOC

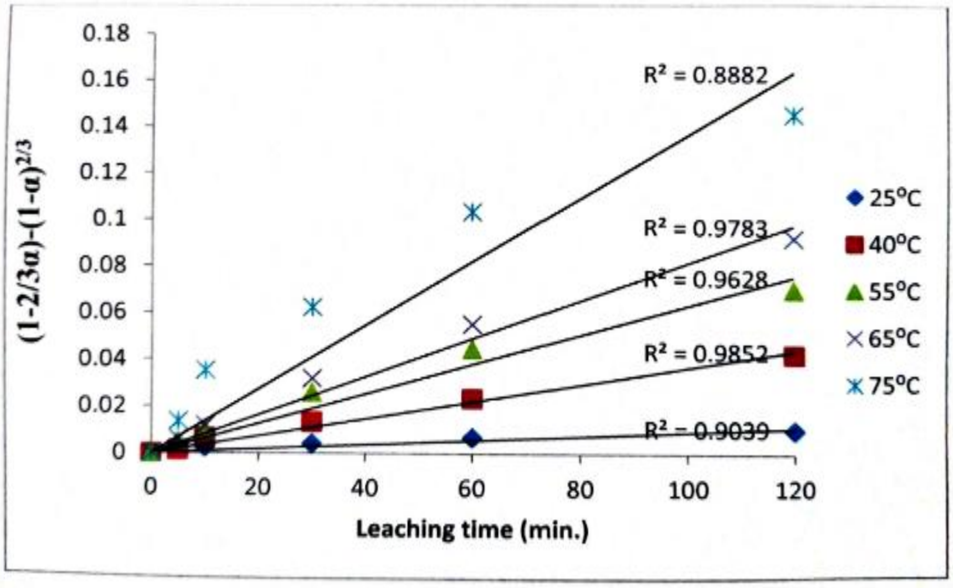


Fig. 10: Plot of Diffusion method for EOT

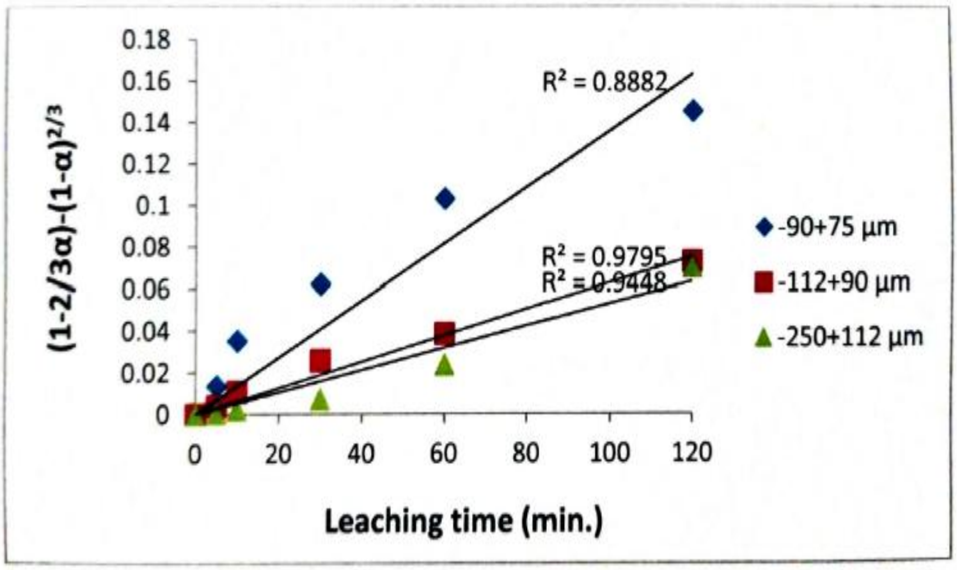


Fig. 11: Plot of Diffusion Control for EOPS

4.3.2 Determination of the reaction order

To determine the reaction order with respect to [H+] ion concentration, the shrinking core model 1-2/3α(1- α)2/3 versus leaching time at different concentrations was used in the linearization. The slope of each of the lines was calculated and recorded as apparent rate constants k_d from which a plot of InK versus In[CH_3COOH] was determined for the evaluation of the order of reaction.

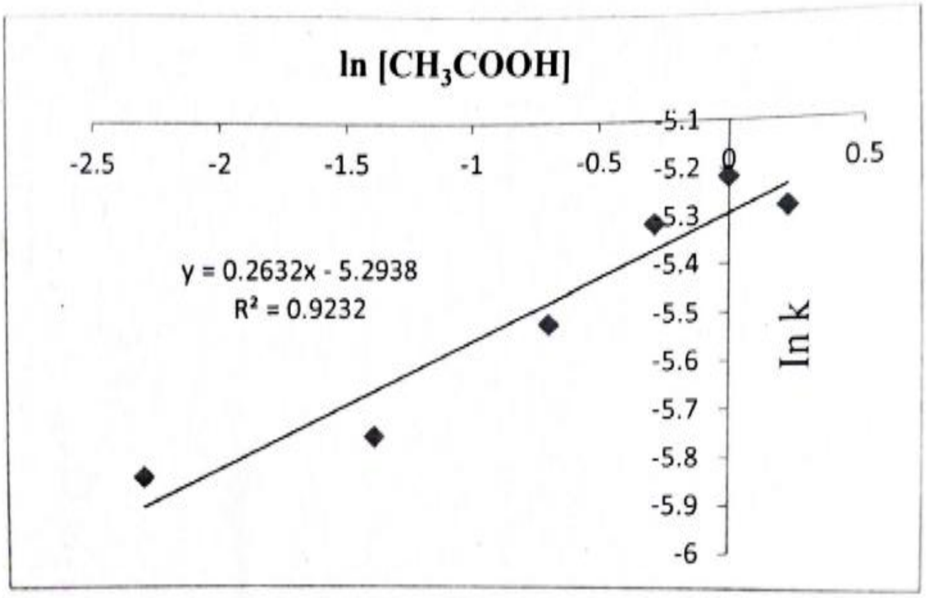


Fig. 12: Plot of Ink vs In[CH,COOH]

K which corresponds to the slope of each line in fig. 6 was obtained as well as InK and In[CH_3COOH].

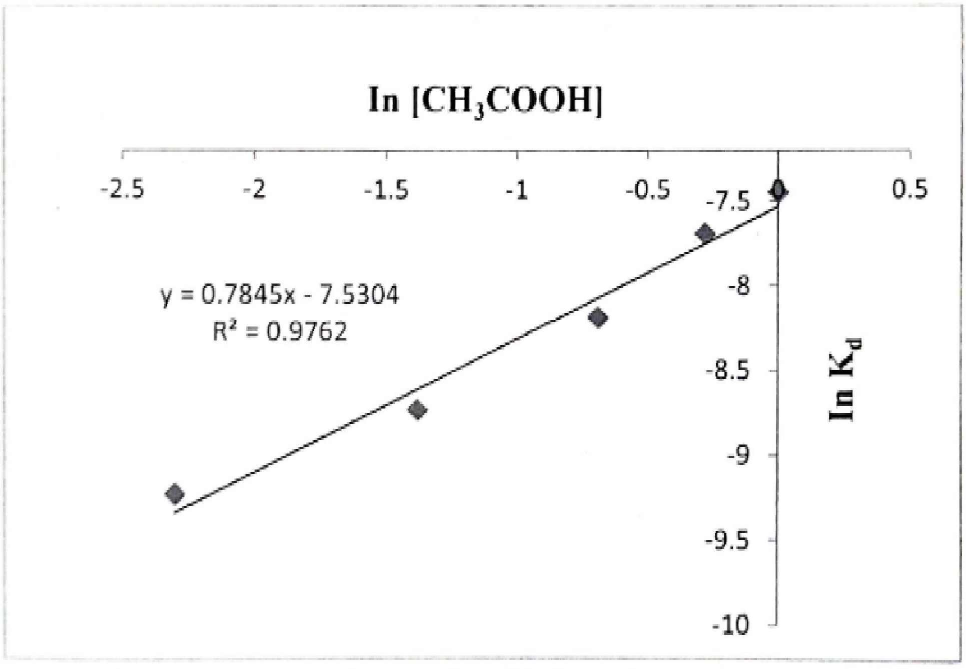


Fig. 13: The Arrhenius plot of InK_d versus In[CH_3COOH]

4.3.3 Determination Of Activation Energy

If a reaction is diffusion-controlled limited, the rate of the reactant is governed by the rate at which the reactant molecules diffuse through the solvent; thus, little activation energy will be required for this reaction. In the case of surface chemical-controlled reaction, substantial activation energy is required. Therefore, in order to determine the activation energy for the reaction, the Arrhenius equation was linearized as shown in appendices. A graph of 1-2/3 α- (1- α)2/3, which shows the best correlation versus leaching time (t) at different temperature (T) was used and the slope of each of the lines was obtained at apparent rate constants. The experimental rate constant, K was determined from the slope of chemical method and the plot of InK_1 versus In[CH_3COOH].

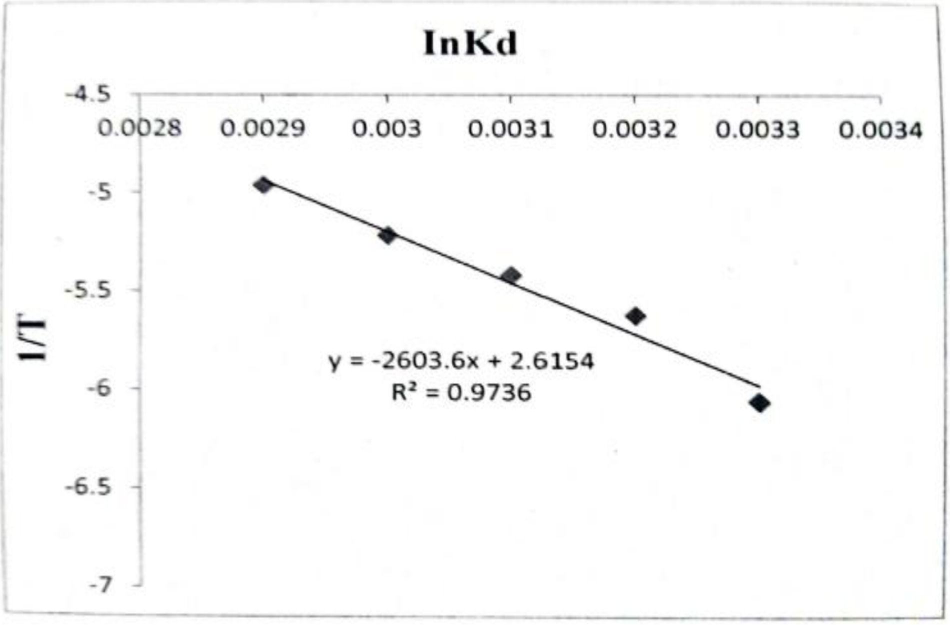


Fig. 14: Plot of Ink versus 1/T

From fig. 14, the activation energy of the reaction was calculated to be 21.64 kjmol^{-1}. The relative value of activation energy (21.40kjmol^{-1}) obtained from the kinetic model plot for the leaching of mica ore using acetic acid is most likely indicative of diffusion and not a chemically-controlled mechanism.

5. RESULT OF ANALYSIS

5.1. Analysis of residual product by XRD

The XRD spectra of the synthesized mica at 75 °C for 2 hours respectively was shown in Fig 15. It shows the XRD pattern of synthesized mica at the various reaction temperatures and time indicated above with other important compound detected. The Joint Committee on Powder Diffraction Standard file numbers (JCPDS) numbers are put in curl bracket: $Mg_3[Si_2\text{-}xO_5]$ (OH) {25-0645}, $KAlSi_3O_8$ {31-0966}, mixtures of impurities {$Mg_3Si_2O_5$ $(OH)_4$ $LiAlSi_4O_{10}$}.

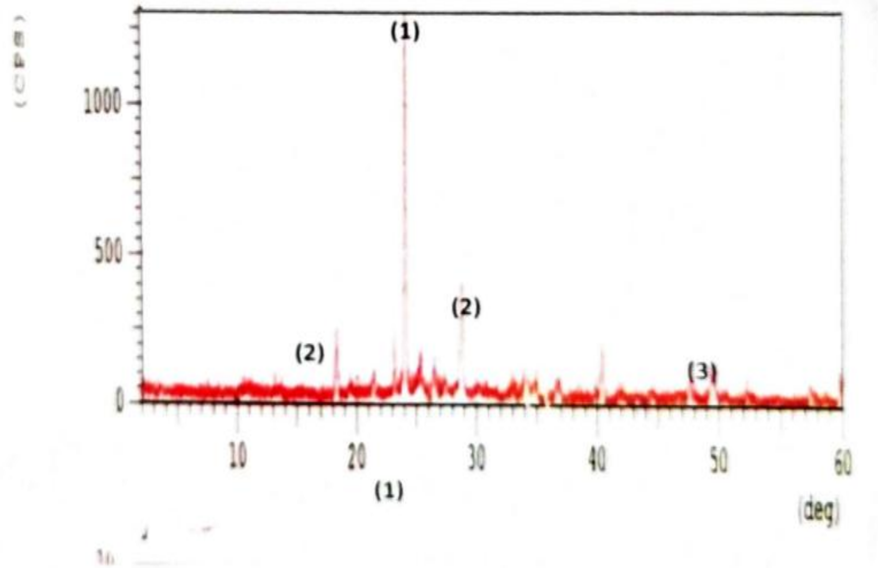


Fig. 15: XRD spectra of synthesized mica at 75 ^{o}C for 2 hours.

5.2 Analysis of residual product by SEM

The SEM micrographic of the synthesized mica powder after the heat treatment with acetic acid is presented in Fig. 16 (a) and (b). SEM micrograph of mica product obtained under hydrothermal conditions after reaction with 1 mol/L acetic acid solution for 2 hours at 75 ^{o}C.

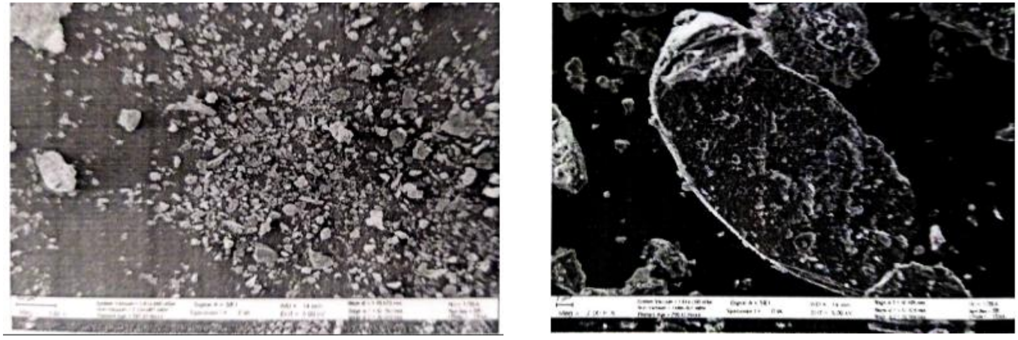

(a) (b)

Fig. 16: SEM micrograph of processed mica ore and acetic acid at 75 ^{o}C for 2 hours

Fig. 16: (a) Corresponds to SEM micrograph of processed mica ore and acetic acid at 75 ^{o}C for 2 hours. It shows a loosely packed macro particle of the ore flakes.

Fig. 16: (b) Corresponds to the SEM micrograph of processed mica ore and acetic acid at 75 ^{o}C for 2 hours. It shows a rocky-like structure in which we have big ones and smaller Ones.

5.3 Quantitative analysis by EDS

The EDS of raw Awo mica shown in Fig. 17 (a) indicates that the raw Awo mica contains compounds including aluminium (Al), Silicon (Si), Oxygen (O), Iron (Fe) and so on. They are better explained in table 4. Also, the EDS of processed Awo mica shown in Fig. 17 (b) indicates that processed Awo mica contains compounds including aluminium (Al), Silicon (Si), Oxygen (O), Iron (Fe) and so on. They are discussed better in table 5.

Overall, result obtained showed that there was improvement in the aluminium contents due to slight reduction in the silica content of the ore at optimal leaching.

Table 4: Quantitative Analysis of Raw Awo Mica by EDS

Element	Al	Si	O	Fe	K	Na
[Norm.wt%]	13.40	21.68	56.31	2.40	4.19	1.54

Table 5: Quantitative Analysis of Processed Awo Mica by EDS

Element	Al	Si	O	Fe	K	Na
[Norm.wt%]	18.55	19.15	51.31	1.63	6.91	2.46

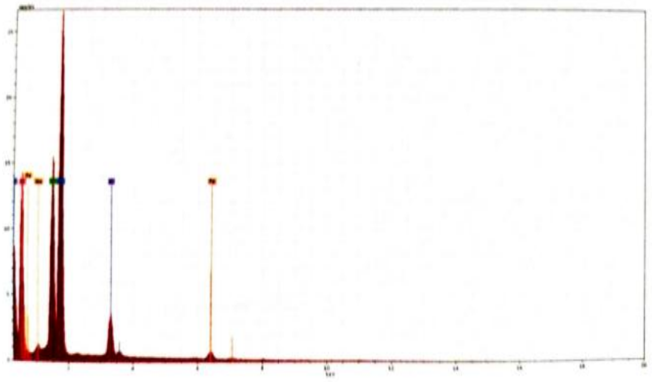


Fig. 17: (a) The EDS of raw Awo mica

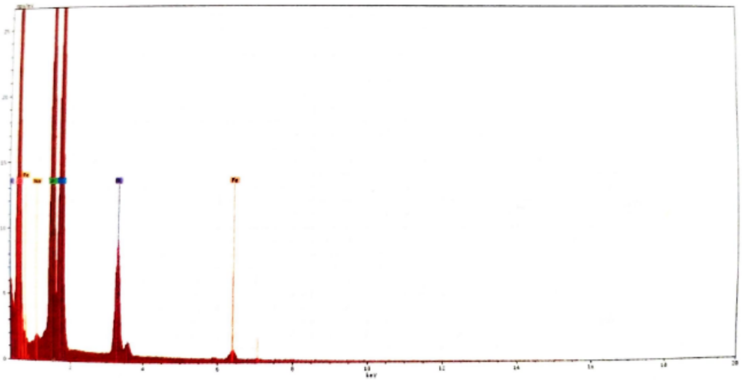


Fig 17: (b) The EDS of processed Awo mica

6. CONCLUSION

Mica ore has been leached successfully whose source is a Nigerian origin in acetic acid via leaching. The leaching plays a vital role in purification and separation of mica. The organic acid used has high selectivity, although their dissolving abilities are weak. The effect of temperature, solid-to-liquid, reaction time, stirring speed, particle size and acid concentration on the rate of dissolution of leaching was investigated. The concentration that gives maximum dissolution was used for the optimization of other leaching parameters including temperature and particle size. The activation energy and constant were evaluated from Arrhenius plots. The shrinking core method used was diffusion method which was found to be 0.9437 with the Ea been 21.64 kJ/mol. The EDS shows reduction of iron and give rise to increased Al content. The decrease in Fe and Si and increased Al content would probably make this processed mica be considered for use in some specific industrial applications.

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APPENDICES

Appendix 1: pH determination

The pH of the sample was 7.7. The value suggests that the ore is slightly basic and it is to be noted that the value increases slightly.

pH determination over a 3-day period.

Day	pH
1	7.7
2	7.8
3	7.9

The mean was calculated to be 7.7 as stated above.

Appendix 2: Moisture content determination

W_g = Weight of muscovite sample = 1g

Weight of crucible = 44.023g

W_i = Weight of crucible + sample = 45.025g

W_f = Weight of crucible + sample after heating = 43.993g

$$\% \, moisture\ content = \frac{W_i - W_f}{W_s} \times 100$$

$$\% \, moisture\ content = \frac{(45.025 - 44.993)g}{(1)g} \times 100 = 3.24\%$$

Appendix 3: Loss of mass on ignition (LOMOI)

W_g = Weight of muscovite sample = 1g

Weight of crucible = 23.951g

W_i = Weight of crucible + sample = 24.951g

W_f = Weight of crucible + sample after heating = 24.908g

$$\% \, L.O.I = W_i - W_f \times 100$$

$$\% \, L.O.I = (24.951 - 24.908) \times 100 = 4.3\%$$

Appendix 4: Sample preparation/preparation of standard solution

To prepare 0.1 M CH_3COOH in 500 ml distilled Water

$$Concentration = \frac{\%Purity \times Density}{Molar\ mass}$$

Where;

% Purity of acetic acid = 99.6%

Specific gravity = 1.049 g/L

Molar mass = 60.05 g/mol

$$Concentration = \frac{99.6\% \times 1.049\, g/L \times 1000}{60.05\, g/mol} = 17.40\frac{mol}{L} = 17.40M$$

Using $M_1V_1 = M_2V_2$

Where;

M_1 = Molarity of CH_3COOH = 17.40 M

V_1 = Volume of standard flask = 500 ml

M_2 = Molarity sought for = 0.1 M

V_2 = Volume of acetic acid required = ? ml

$$V_2 = \frac{0.1 \times 500}{17.40} = 2.87\ ml$$

Summary of the five concentrations used.

Solution Concentration (M)	Volume of acetic acid required to prepare solution (ml)	Volume of distilled water used (ml)	Total volume (ml)
0.10	2.87	497.13	500.00
0.25	7.18	492.82	500.00
0.50	14.37	485.63	500.00
0.75	21.43	478.45	500.00
1.00	28.74	471.26	500.00

Appendix 5: Effect of concentration with time.

Fraction of muscovite ore dissolved versus leaching time at different acetic acid concentrations.

Leaching time (Mins)	0.1 mol/L	0.25 mol/L	0.50 mol/L	0.75 mol/L	1.0 mol/L	1.25 mol/L
0	0	0	0	0	0	0
10	0.10	0.15	0.19	0.24	0.28	0.24
30	0.14	0.20	0.25	0.35	0.43	0.38
60	0.25	0.30	0.36	0.46	0.54	0.47
120	0.35	0.38	0.48	0.59	0.65	0.61

Appendix 6: Kinetic model.

1. **Chemical controlled reaction $[1-(1-\alpha)^{1/3}] = k_r t$ for EOC:**

Leaching time (Mins)	0.1 mol/L	0.25 mol/L	0.50 mol/L	0.75 mol/L	1.0 mol/L
0	0	0	0	0	0
10	0.0345	0.0527	0.0678	0.0874	0.1037
30	0.0490	0.0717	0.0914	0.1337	0.1708
60	0.0914	0.1120	0.1382	0.1856	0.2280
120	0.1337	0.1472	0.1958	0.2571	0.2952

2:Diffusion controlled reaction $[(1-2/3\ \alpha)-(1-\alpha)^{1/3}] = k_d t$ for EOC.

Leaching time (Mins)	0.1 mol/L	0.25 mol/L	0.50 mol/L	0.75 mol/L	1.0 mol/L
0	0	0	0	0	0
10	0.0012	0.0027	0.0044	0.0071	0.0100
30	0.0023	0.0035	0.0079	0.0162	0.0258
60	0.0079	0.0116	0.0173	0.0302	0.0441
120	0.0162	0.0195	0.0333	0.0547	0.0700

Appendix 7: Estimation of reaction order

1. Chemical method: Ink VS In[CH_3COOH]

In[CH_3COOH]	Ink$_1$
-2.30	-9.230
-1.38	-8.740
-0.69	-8.190
-0.28	-7.693
0	-7.447

In[CH_3COOH]	Inkd
-2.30	-9.230
-1.38	-8.740
-0.69	-8.190
-0.28	-7.693
0	-7.447

Appendix 8:

1. Effect of temperature with leaching time

Leaching time (mins)	25 ^{0}C	40 ^{0}C	55 ^{0}C	65 ^{0}C	75 ^{0}C
0	0	0	0	0	0
5	0.11	0.18	0.20	0.22	0.32
10	0.14	0.23	0.28	0.30	0.49
30	0.18	0.32	0.43	0.47	0.62
60	0.23	0.41	0.54	0.59	0.75
120	0.28	0.53	0.65	0.72	0.84

2. Diffusion controlled reaction $[(1-2/3\ \alpha)-(1-\ \alpha)_{2/3}] = k_dt$ for EOT

Leaching time (mins)	25 ^{0}C	40 ^{0}C	55 ^{0}C	65 ^{0}C	75 ^{0}C
0	0	0	0	0	0
5	0.0014	0.0014	0.0049	0.0060	0.0133
10	0.0023	0.0066	0.0100	0.0116	0.0350
30	0.0040	0.0133	0.0258	0.0317	0.0620
60	0.0066	0.0232	0.0441	0.0547	0.1031
120	0.0100	0.0421	0.0700	0.0920	0.1452

3. Activation Energy

Ink$_d$	1/T
0.0033	-6.0604
0.0032	-5.6223
0.0031	-5.4182
0.0030	-5.2159
0.0029	-4.9618

Appendix 10:

1. Effect of particle size with leaching time

Leaching time (mins)	90 ^{0}C	112 ^{0}C	250 ^{0}C
0	0	0	0
5	0.32	0.18	0.07
10	0.49	0.29	0.13
30	0.62	0.43	0.24
60	0.75	0.57	0.41
120	0.84	0.66	0.54

2. Diffusion controlled reaction $[(1-2/3\ \alpha)-(1-\alpha)^{2/3}] = k_dt$ for EOPS

Leaching time (mins)	90 ^{0}C	112 ^{0}C	250 ^{0}C
0	0	0	0
5	0.0133	0.0040	0.0005
10	0.0350	0.0108	0.0020
30	0.0620	0.0258	0.0072
60	0.1031	0.0384	0.0232
120	0.1452	0.0728	0.0700