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ASSESSMENT OF A NIGERIAN TALC ORE FOR IMPROVED INDUSTRIAL APPLICATION BY SULPHURIC ACID LEACHING

ALAFARA A. BABA*[1], ABDULLAH S. IBRAHIM*[2],
RAFIU B. BALE[3], FOLAHAN A. ADEKOLA[1], AND ABDUL G. F. ALABI[4]
[1]Department of Industrial Chemistry, University of Ilorin, P.M.B. 1515, Ilorin-240003, Nigeria.
[2]Department of Chemistry, University of Ilorin, P.M.B. 1515, Ilorin-240003, Nigeria.
[3]Department of Geology and Mineral Sciences, University of Ilorin, P.M.B. 1515, Ilorin-240003, Nigeria.
[4]Department of Materials & Metallurgical Engineering, University of Ilorin, P. M. B. 1515, Ilorin-240003, Nigeria; now in Department of Material Science and Engineering, Kwara State University, P.M.B. 1530, Malete, Nigeria.
[alafara@unilorin.edu.ng +2348035010302]

ABSTRACT
Talc by its intrinsic properties is an important material with diverse industrial applications. In the present work, the evaluation of a Nigeria sourced talc ore for industrial utilization by sulphuric acid leaching was studied and accordingly characterized. During the experiments, it was found that the increase in acid concentration, reaction temperature and decreased particle size increase the extent of the ore dissolution. At optimal conditions (2.0 mol/L H_2SO_4, 75°C, -90+75μm), 71.2% of the ore reacted within 120 minutes with moderate stirring. The unleached product was analysed to contain silica. The estimated activation energy of 28.72 kJ/mol supported the proposed diffusion-controlled reaction kinetics. The leached product is hydrophilic having specific surface area increased from 0.15 m^2/g to 3.76 m^2/g with total cumulative pore volume of 0.010986 ml/g. Finally, a decrease in the ore's iron content from 3.75% to 0.16% yielded 95.73% product purity.

Keywords: *Talc ore; Nigeria; Leaching; Sulphuric acid; Dissolution kinetics; High surface area; Product purity.*

1. Introduction

Till date, the layer silicates minerals such as talc are of prime economic interest due to their extensive uses in industries (Nkoumbou et. al., 2008). Talc main industrial applications are in paper, paints, cosmetics, plastics, rubbers, refractory materials, pesticides, pharmaceuticals, agro-industries and ceramics depending on its physical properties, chemistry and mineralogy (Martin, et. al., 1999, Carretero, 2002, Yvon, et. al., 2002, Petit, et. al., 2004). Talc is widely used in industries due to its intrinsic properties such as chemical inertness, softness, high thermal stability, low electrical conductivity, high density, platy morphology, high crystallinity, wide range of particle size and high specific surface area (Nkoumbou et. al., 2008,Perez-Maqueda, et. al., 20005, Ersoy, et. al.,2013, Marzbani, et. al., 2013). However, talc surface area is composed of two types of surface area: the basal cleavage face and the edges. The face surface consist of a tetrahedral siloxane surface with –Si-O-Si links and has no charge. For that reason, the talc surface is believed to be non-polar and hydrophobic, while the edges are hydrophilic due to the presence of charged ions (Mg^{2+} and OH^-) (Ahmed, et. al., 2007). Thus, the surface of the talc layer often contains atoms of oxygen which causes the slow leaching behaviour (Zdralkova, et. al., 10].

The major gangue minerals in talc ore include carbonates, magnesite, dolomite, serpentine, chlorite and calcite. These impurities apparently contribute to the production of undesirable characteristics, which are responsible for why talc ore do not meet some uses (Castro, 1996). For example, the presence of iron impurities both in the surface of talc, and in the structure of clinochlore and accessory minerals, reduces talc refractory properties and diminishes its whiteness which gives it an undesirable colour and thus limits its industrial applications Orosco, et. al., 2011). It is important to note that several methods including conventional flotation, magnetic separation, chlorination and leaching techniques have been proposed to remove gangue minerals from the talc ore surface. The acid leaching technique has been proposed as a method of removing gangues from the talc ore surface (Kho, and Sohn, 1989, Boghdady, et. al., 2005, Yehia, and AL-Wakeel, 2000, Piga, and Marruzzo, 1992, Hojamberdiev, et. al., 2010). However, the acid leaching technique has been reported to be effective due to its low cost, eco-friendly and relatively inert nature of talc to acids (Castillo, et. al., 2012a, Jamil, and Palaniandy, 2010, Helmy, et. al., 2005). Recently, we have reported the treatment of a Nigerian talc ore by HCl leaching (Baba, et. al., 2015a), but in order to produce a porous product with increased surface area, the use of sulphuric leachant was adopted (Okada, et. al., 2005a). Considering the recent focus of the Nigeria Government to grow and develop the nation's economy through the solid minerals sector reform, this study has been devoted to the purification of a Nigerian talc ore by sulphuric acid leaching for improved industrial applications.

2. Methodology

2.1 Materials

The talc ore for this investigation was obtained from Isanlu area of Kogi State, North Central Nigeria. This talc ore which have been previously studied (Baba, et. al., 2015a) was initially crushed and reduced to fine particles using an acetone-rinsed mortar and pestle. The pulverized ore was sieved into three different particle sizes: -90+75, -112+90, and -250+112 µm using American Society for Testing and Materials (ASTM) standard sieve. Sulphuric acid was used as the leachant in the present investigation. All experiments, unless otherwise stated were performed using the -90 +75 µm particle size fraction, due to its high surface area.

2.2 Leaching Experiments

Leaching experiments were performed in a 250 ml Pyrex glass reactor equipped with a mechanical stirrer. The required temperature of the reactor contents earlier used in our recent work using HCl leachant (Baba, et. al., 2015a), was within ±0.5 °C and was adjusted by a thermostatically controlled electric heating mantle. The reactor was filled with 100 ml leachant (sulphuric acid) with predetermined concentration ranges (0.01 - 2.0 mol/L). For all leaching experiments, 10 g/L of talc ore was treated with freshly prepared solution mixture heated from 25 °C to 75 °C between 5 to 120 minutes. Trial experiments were also performed in order to assess the optimal leaching conditions. The concentration that gives the maximum dissolution was used for the optimization of other leaching parameters including temperature, and particle size. The residue after leaching was washed with acidulated hot water and then with deionised water. The product was allowed to cool, filtered and dried to constant weight at 80°C overnight before being analysed. For each experimental run, the fraction of the talc ore dissolved was evaluated from the initial difference in weight of amount dissolved or undissolved at various leaching time in intervals up to 120 minutes. The product residue at optimal leaching was accordingly characterized for monitoring the extent of iron removal for product purity assessment (Castillo, et. al., 2012a, Baba, et. al., 2015a, Okada et. al., 2005a, Sanchez, et. al., 1996, Sarquis, and Gonzalez, 1998, Okada, et. al., 2003b).

2.3 Characterization

The ground raw talc and leached talc residues at optimal leaching were subjected to various characterization techniques. The mineralogical crystalline phases in the sample were studied by XRD (EMPYREAN) with monochromated CuKα radiation. The elemental and chemical compositions were measured by EDXRF (MINI PAL 4), the microstructure morphology of the raw and leached talc product were analysed using FEI Nova NanoSEM 230 with an Oxford X-max EDS detector using INCA software to analyse the spectra. Nitrogen gas adsorption-desorption isotherms were measured at -196°C using a micromeritics BET, Tristar II 3020. The measurements were performed after degassing overnight at 90 °C. The specific surface area (SSA) of the talc ore was calculated using the Brunauer-Emmet-Teller (BET) method (Brunauer, et. al., 1938) and the Barrett-Joyner-Halenda (BJH) method was used for determining the pore size distribution from the adsorption-desorption isotherms (Barrett, et. al., 1951). The total pore volume (V_p) of the raw and leached talc ore were obtained from the maximum amount of N_2 adsorption at a partial pressure (P/P_0) of 0.99. The raw and the leached product were further subjected to hydrophobicity/hydrophilicity tests using de-ionized water and n-hexane (Baba, et. al., 2015a, Castillo, et. al., 2011b).

3. Results and discussions

3.1 Geochemical and Mineralogical Compositional Characterization

The ground raw talc ore is composed of 52.9% SiO_2, 27.2% MgO, 6.32% CaO, 7.4% Fe_2O_3 and 2.17% Al_2O_3 along with minor to trace amounts of TiO_2 (0.23%), MnO (0.43%), V_2O_5 (0.05%), Cr_2O_3 (1.48%) and NiO (0.51%). The EDX analysis and EDXRF gave very comparable composition of the ore as C (16.38%), O (45.5%) Mg (8.38%), Al (2.17%), Si (17.71%), Ca (6.12%) and Fe (3.75%) . However, from our recent report (Baba, et. al., 2015a), the Powder X-ray diffraction of the raw talc shows the dominating talc constituents as having sharp diffraction peaks indicating well-crystallized phases. The associated clinochlore variety of chlorite and the actinolite variety of amphibole are magnesium-rich silicate minerals which would have formed along with talc in the retrograde metamorphism of ultramafic protoliths. Also, the scanning electron microscopic studies revealed the raw talc ore to be comprised by prismatic and fibrous actinolite, fine crystallite of chlorite and tabular, platy talc crystals.

3.2 Leaching Studies

3.2.1 Effect of acid concentration

The effect of H_2SO_4 concentration on the extent of talc ore dissolution has been examined at H_2SO_4 concentration range (0.01 – 2.0 mol/L). The results of talc ore reacted as a function of contact time is given in Figure 1.

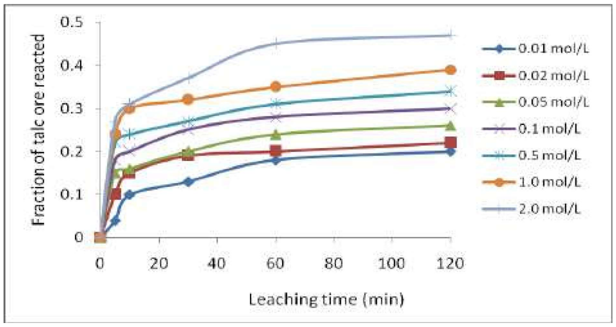


Figure 1: Effect of H_2SO_4 concentrations on talc dissolution at various leaching time (min).

It is evident that higher H_2SO_4 concentration leads to the increase of talc dissolution rate. The increase in the H_2SO_4 concentration makes more reactants to attack the talc ore, thus, increasing the leaching rate. The H_2SO_4 acid concentration of 2.0 mol/L was found to be optimum with 47% dissolution achieved at the end of 120 minutes of leaching, as compared to the result of 1.0 mol/L H_2SO_4 solution where the dissolution reached 37.1%. For subsequent investigations, the experiments were performed with 2.0 mol/L H_2SO_4 solution. This condition as previously suggested by some authors has ability of increasing the ore surface area as well as its silica content required for its unique application as fillers for paints, paper and rubbers (Temuujin, et. al., 2002).

3.2.2 Effect of reaction temperature

The effect of reaction temperature on the rate ore of talc ore dissolution was studied over the temperature ranges: 25-75°C as shown in Figure 2.

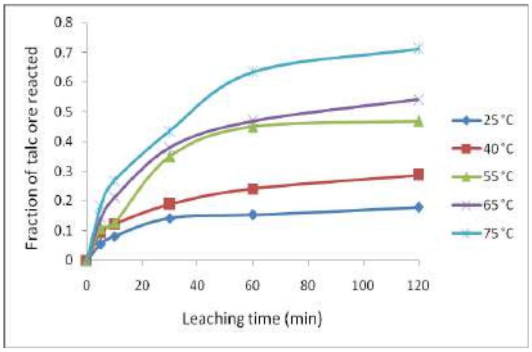


Figure 2: Effect of temperature on talc ore dissolution by 2.0 mol/L H_2SO_4 solution.

From Figure 2, it can be seen that increasing temperature improves the talc ore dissolution rate with time after 120 minutes of acid leaching. At 75 °C, the ore dissolution reached 71.2% within 120 minutes. Hence, high temperature favours the gangue mineral dissolution to a greater extent and this shows that the ore dissolution is a thermally activated phenomenon (Castillo, et. al., 2012a).

3.2.3 Effect of particle size

The effect of particle size on the extent of talc ore dissolution was studied using three particle sizes (-90 + 75 µm, -112 + 90 µm, -250 + 112 µm) by 2 mol/L H_2SO_4 solution. The results obtained are summarized in Figure 3.

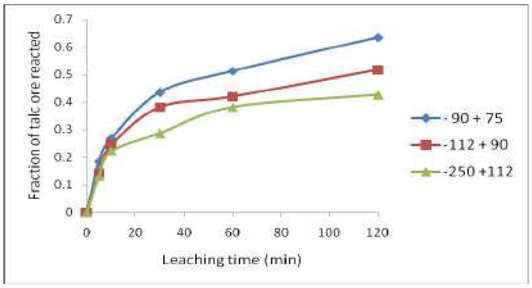


Figure 3: Effect of particle size on talc ore dissolution by 2 mol/L H_2SO_4 solution at 75°C.

From Figure 3, the rate of talc ore dissolution reached 42.6%, 51.7% and 71.2% for the -250 +112, -112 + 90 and -90 +75µm particle size respectively at a set of experimental conditions. The rate of dissolution increases with decrease in particle size of talc ore used, which can be attributed to increase of the contact surface with decrease of the particle size per unit weight of the solid. Thus, larger particle size fraction may need more time to complete the removal of gangue minerals contents and may not be thermodynamically favourable.

3.3 Discussion

3.3.1 Composition of leached product

The leached product formed after optimal leaching conditions (temperature = 75°C, acid concentration = 2 mol/L leaching time = 120 min.) was appropriately characterized. The X-ray diffraction analysis of the 2 mol/L H_2SO_4 leached product at 75°C and 120 minutes mainly showed the presence of degraded, broad peak of pure talc (**T**) and loss of the associated gangue mineral phases as actinolite $\{(Na_{0.11}K_{0.04})(Ca_{1.68}Na_{0.04}Fe_{0.28})(Mg_{3.65}Fe_{1.14}Al_{0.21})(Si_{7.38}Al_{0.62})O_{22})(OH)_2\}$: *(85-2157)* and clinochlore$\{(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8\}$: *(29-0701)* (Figure 4).

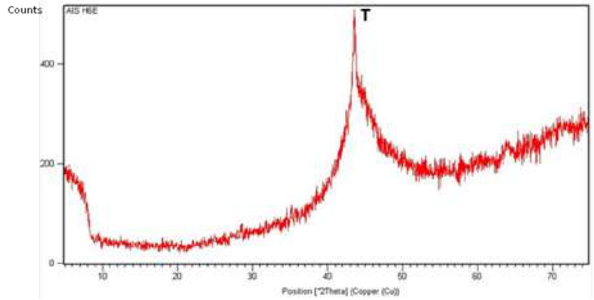
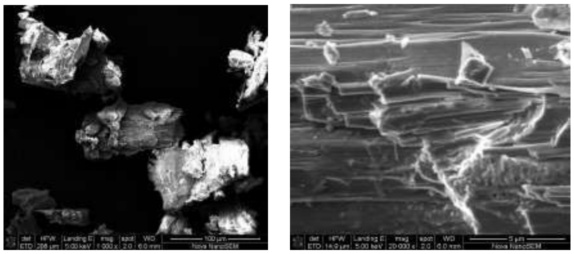


Figure 4: XRD pattern of leached talc product showing degradation of the talc crystallinity and destruction of actinolite and chlorite peaks: **T** = Talc $\{Mg_3Si_4O_{10}(OH)_2$: *19-0770*$\}$.

The SEM images of leached residues with the diameter -90 + 75µm at 75⁰C by 2 mol/L sulphuric acid leaching are presented in Figures 5a, b.



(a) (b)

Figure 5: SEM images of leached talc ore by H_2SO_4 solution at optimal conditions at different magnifications showing crystal etchings and corrosion surface. The residual leached product has a granular morphology with broken flakes. They are rhomboic layer with fine powdered shreds. Few broken plates are also present. The EDX spectrum corroborated the presence of talc in the leached product with Si (25.69%), Mg (14.01%), O (52.54%), Al (1.22%), Ca (0.94%) C (5.23%) and Fe (0.16%).

3.3.2 Porous properties assessment

The -90+75µm sized fraction was used to determine the specific surface area properties for both the raw and leached talc ore products. The specific surface area by the BET method S_{BET} of raw talc ore apparently increased from 0.152 m^2/g to 3.760 m^2/g for the leached product. The surface area of the sulphuric-leached product gave higher surface area than the hydrochloric-leached product (Baba, et. al., 2015a). The value of the total pore volume obtained from the N_2 gas absorbed at a partial pressure of 0.99 for the leached product is 0.01 ml/g with a total cumulative pore volume of 0.010986 ml/g is shown in Figure 6.

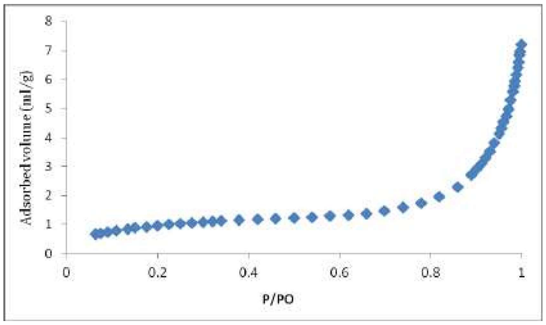


Figure 6: N_2 adsorption-desorption isotherm of leached talc ore at optimal conditions

The pore size distribution (PSD) determined from the adsorption isotherm by the BJH method is summarized in Figure 7.

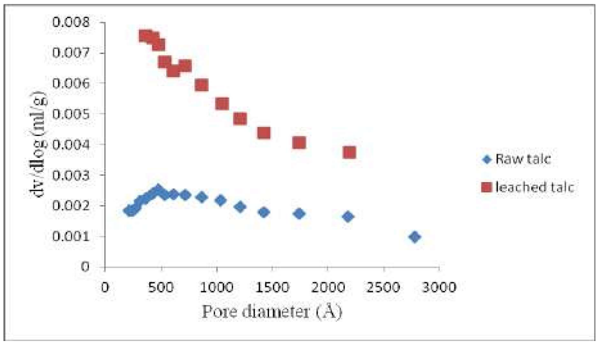


Figure 7: PSD curve of leached talc product by BJH method

From Figure 7, the pore size distribution of the leached product indicates a clear peak, and thus explains the porous nature of the talc product. The leached product shows a high baseline which apparently corresponds to the formation of stable micropores on the talc surface (Baba, et. al., 2015a, Okada, et. al., 2005a, Rouquérol, et. al., 1994).

3.3.3 Surface Modification Studies

Surface affinity of the treated talc was evaluated by a hydrophobicity/hydrophilicity test to characterize the surface modifications (Castillo, et. al., 2011b). The photographs of the leached talc sample inside beakers containing water and n-hexane are shown in Figure 8. Densities of talc, water, and n-hexane are 2.7, 1, and 0.675 g/cm^3, respectively. Considering these properties, it was observed that the acid treated talc sinks in n-hexane **(A)**, but due to the etched, more porous nature of the treated talc it floats on water **(B)**. Hence, the development of a hydrophobic property on the processed talc surface apparently supported the product quality, as the extent of iron removal during the process was 95.73%.

3.4 Dissolution kinetics analysis

The kinetics of dissolution of the talc ore was assessed on the basis of the shrinking core models (SCM). The SCM used in this study considers that the leaching process is controlled either by diffusion of reactants through the solution boundary layer or through a solid product layer (equation (1)), or by surface chemical reaction (equation (2)), and consisted with the following stoichiometry (Baba, et. al., 2015a, Dreisinger, and Abed, 2002, Habashi, 1979, Baba, et. al., 2009b, Sohn and Wadsworth, 1979):

$$1-\frac{2}{3}\alpha-(1-\alpha)^{2/3}=\frac{k_c M_B C_A t}{\rho_b a r_0}=k_d t \qquad \textbf{(1)}$$

$$1-(1-\alpha)^{1/3}=\frac{k_c M_B C_A t}{\rho_b a r_0}=k_r t \qquad \textbf{(2)}$$

where α is the fraction of talc ore reacted, k_c is the kinetic constant, M_B is the molecular weight of the solid, C_A is the concentration of the dissolved lixiviant, a is the stoichiometric coefficient of the reagent in the leaching reaction, r_0 is the initial radius of the solid particle, t is the reaction time, ρ_b is density of the solid and k_d and k_r are rate constants. Therefore, the dissolution data in this study (Figures 1 – 3) fitted perfectly to the shrinking core model expressed by equation (1) with average correlation of 0.989 as compared to model equation (2) giving correlation coefficient of 0.488

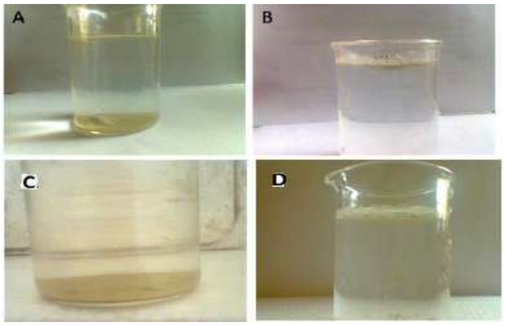


Figure 8: Hydrophobicity/hydrophilicity tests for raw and treated talcs [**A:** Raw talc in n-hexane **B:** Raw talc in water **C**: Sulphuric acid leached product in n-hexane; **D**: Sulphuric acid leached product in water]

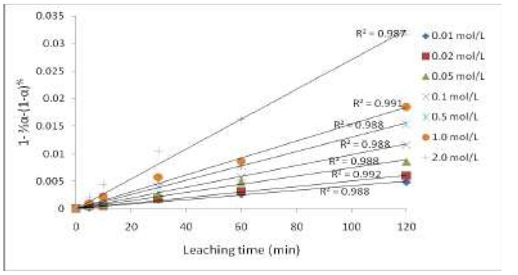


Figure 9: Plot of $1-\frac{2}{3}\alpha-(1-\alpha)^{2/3}$ versus leaching time at different H_2SO_4 concentration.

The experimental rate constants, k_d, were determined from the slope in Figure 9 and the plot of $\ln k_d$ versus $\ln[H_2SO_4]$ were made as shown in Figure 10.

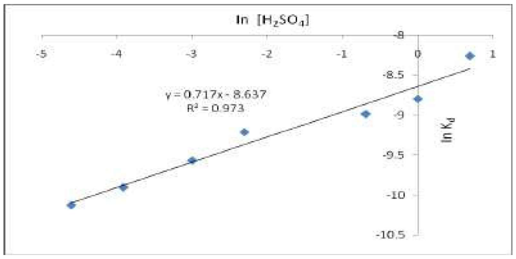


Figure 10: Plot of $\ln k_d$ versus $\ln[H_2SO_4]$.

As seen from Figure 10, the slope of the resulting plot indicates that the reaction order is 0.72 with respect to H^+ ion with correlation coefficient 0.973. For the reaction temperature analysis, the extracted data from Figure 2 was linearized using equation (1) to obtain the following results depicted in Figure 11.

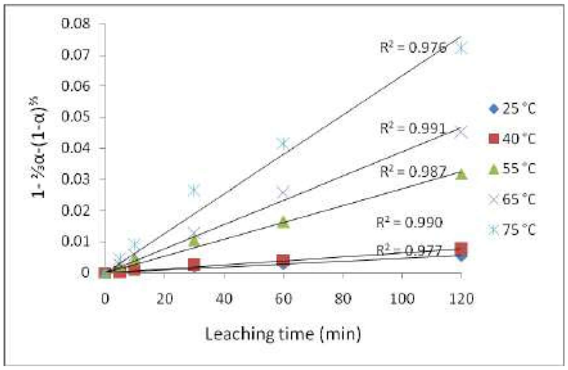


Figure 11: Plot of $1-\frac{2}{3}\alpha-(1-\alpha)^{2/3}$ versus leaching time at different temperatures.

The reaction temperature analysis can be used in the prediction of the apparent activation energy. It is widely accepted that systems with activation energy greater than 40 kJ/mol are controlled by a chemical reaction, while those with an activation energy less than 40 kJ/mol are controlled by a diffusion-controlled process whether in the product layer or a boundary fluid film (Levenspiel, 1992, Abdel-Aal, and Rashad, 2004). Thus, the apparent rate constants, K_d was calculated from the slope of straight lines in Figure 11. These values and their correlation rate coefficient were used to estimate the apparent activation energy of talc ore dissolution from Arrhenius relation:

$$k_d = A\exp^{\left(-Ea/_{RT}\right)} \qquad (3)$$

From equation (3), A is frequency factor, E_a is activation energy of the reaction. R is universal gas constant and T is absolute temperature [21].

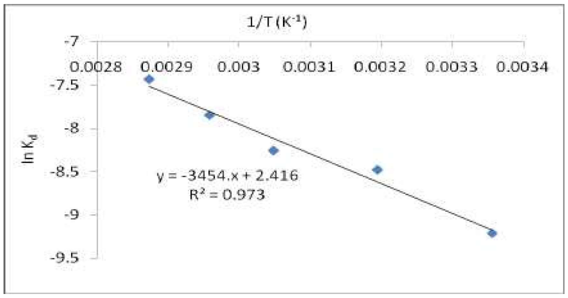


Figure 12: Plot of $\ln K_d$ versus 1/T

From the Arrhenius plot of Figure 12, the calculated activation energy for the process gave 28.72 $kJmol^{-1}$ and this clearly suggests diffusion control reaction for the dissolution process. This calculated activation energy is consistent with the values of activation energy previously reported for the diffusion-controlled reactions (Yang, et. al., 2006, Aydogan, et. al., 2005 Ucar, 2009).

4. Conclusion

In this study, sulphuric acid leaching was used in the treatment of a Nigeria sourced talc ore for improved industrial applications. The effects of leaching parameters such as acid concentration, reaction temperature and particle size were studied to assess the level of ore product purity. It was found that dissolution rate increased with sulphuric acid concentration, temperature and decreasing particle diameter. By fitting the shrinking core model to the kinetic data, it was found that the apparent activation energy for the dissolution process gave 28.72 kJ/mol and supports the proposed diffusion control reaction. Also, the extent of the ore dissolution reached 71.2% using 2 mol/L H_2SO_4 within 120 minutes at 75 °C. The analysis by BET showed that the maximum S_{BET} achieved for the purified leached talc is 3.760 m^2/g with a total cumulative pore volume of 0.010986 ml/g. The pore size distribution results indicate the presence of micropores on the leached talc surface and the surface modification tests indicates that the leached product has hydrophobilic property. The process recorded 95.73% iron removal efficiency as Energy dispersive spectra data showed a decrease in ore iron content from 3.75% to 0.16. Hence, the product obtained in this study could be found applicable in paints, rubber, paper, plastics and textiles industries.

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