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SYNTHESIS AND CHARACTERIZATION OF PURE SILICA POWDER FROM A K-FELDSPAR SILICATE ORE FOR INDUSTRIAL VALUE ADDITION

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Abstract. Nigeria is one of the African countries endowed with abundant varieties of solid mineral resources. Despite huge mineral abundance in all States of the Federation, these resources to boost the country's economy, aid industrialization and revenue generation has been far less utilized. Thus, the utilization of a Nigerian k-feldspar ore containing an admixture of orthoclase (KAlSiO8 : 96-740-9031), albite (AlSiO8 : 96-940-0741) and quartz (SiO2 : 96-900-9667) impurities to produce pure silica powder (PSP) was investigated using roast-leaching and co-precipitation method. During leaching, parameters such as leachant concentration, effect of particle size and reaction temperature on the synthesis yield were accordingly examined. At optimal leaching conditions (2.0 mol/L NaOH, 90°C), 87% of the initial 10g/L ore reacted within 120 minutes. The un-reacted product (~13.0%) analyzed by XRD was found to contain siliceous impurities which could serve as a valuable by-product for some defined industries. The leachate at optimal leaching was accordingly co-precipitated using concentrated H2SO4 to obtain pure silica powder (PSP) of industrial value.

1. Introduction

Silica is a mineral that consists of silicon and oxygen, two of the most common elements on the planet. It comes in several forms, although by far the most common is crystalline silica [1]. The crystalline form of silica is so abundant that it makes up over 12% of the earth's crust, making it the second-most common mineral on the planet [2]. Silica occurs in nature as seven distinct polymorphs: quartz, cristobalite, tridymite, coesite, stishovite, lechatelerite (silica glass), and opal. Disordered cristobalite commonly occurs in soils; tridymite is rare in soils and usually associated with siliceous volcanic rocks; and coesite, stishovite, and lechatelerite are rare polymorphic forms. Opal is hydrated, amorphous silica and is not uncommon, but, of these minerals, quartz is the most abundant in soil environments [3,4].

Silica (SiO2) is so common in such a way that, it is known as a building block of modern life. Products containing silica are used in a vast array of industries, which has made it a key component in the manufacture of millions of products. It is an

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irreplaceable ingredient in lots of high technology applications such as: precision casting, fibre-optic cables, and the raw materials for computer chips. The presence of silica in our computers and phones cannot be over emphasize because it is a key to the infrastructure of the internet, renewable energy and telecommunications [5]. It is a key part of how we get around our cars and buses, roads and railways. Our homes are even made from it, rocks, glass and ceramics, and many of the things we use every day contain or rely on one form of silica or more [6].

To meet the increasing demand for pure silica powder, it is important to unravel variations of silica production precursor and cheaper production technologies. In the recent time two, main techniques are used for the manufacturing of PSP, this include: Pyrometallurgical method and hydrometallurgical or wet methods [7]. The pyrometallurgical method involved in large scale precipitation of PSP from sodium silicate which is continually produced by high temperature fusion of sodium carbonate and quartz sand while the hydrometallurgical method utilized different lixiviants such as acid/base and their salts to extract silica in the form either silica acid or alkali metal silicate solutions, which are later neutralized to give PSP. This method is of high advantages because it does not require elevated temperature reaction and thus require low energy input than the pyrometallurgical route [8].

Based on the aforementioned merit of hydrometallurgy over pyrometallurgy, various efforts have been exerted to find alternative raw materials for the production of PSP by this method. Many materials such as: rice husk, rice straw, bagasse ash, naturally occurring silicate rocks and photonic industrial waste have been employed [9-13]. However, the choices of these raw materials have been governed by various factors to achieve good commercially produced PSP. These factors include abundance, availability and relative concentration of impurity of the material [8]. Despite the abundance being recorded of rice husk, rice straw and bagasse, their use as a source of material for PSP production require high energy consumption and additional purification steps for the removal of impurities. Secondly, the use of photonic waste cannot meet the high global demand for PSP.

A good number of reports have been reported on the application of the hydrometallurgical process for silica recovery from various types of silicate rock using acid or alkaline solution through the optimization of reaction conditions such as: system temperature, concentration of acid or base solution and material particle sizes [7,14]. However, it has been reported by Chen and Brantley [14] reported that the dissolution of olivine in HCl depend on the pH and reaction temperature. In another report, sulphuric acid was adopted as a lixiviant to investigate the effect of olivine particle size on the porosity and surface area of nano-silica [15]. Their research gave silica with very small particle size ranging from 8-25 µm. But no attention was given over other kinetic parameters that affect the dissolution rate of olivine and the resulting texture properties of the produced silica powder. Lazaro et al., also achieve 95% purity of silica powder using sulphuric acid as lixiviant. They concluded that the olivine dissolution process in dilute acid could be a convenient alternative to the traditional pyrometallurgy method used for bulky production of nano-silica due to its fewer energy consumptions. Also, no attention was given to deduce the rate-determining step in the acid dissolution of olivine. Despite the fact that mineral acids as proven to be a good lixiviant they exhibit a greater tendency to dissolve impurities like iron from silicate minerals along with the compound of interest, leading to contamination of the product. This has made the use of an alkaline extracting agent is advantageous to compare to that of acid.

Global market demand for silica powder has reported that the worldwide demand for precipitated silica is expected to expand at a 5.5% compound annual growth rate between 2015 and 2023. According to this report, the global precipitated silica market was US$2.22 billion in 2015 and will increase to US$3.49 billion by 2023 [16]. The prospect of this increase in demand is envisaged which is in collaboration with the present regime seven-point economic agenda to diversify Nigeria. Therefore, the study aimed to maximize the synthesis efficiency of PSP by focusing on the alkaline extraction efficiency through an evaluation of the effects of various experimental parameters, including the concentration of sodium hydroxide, liquid to solid ratio, temperature and particle sizes. The determination of optimal reaction conditions through reaction kinetics and rate-limiting steps is vital for the large scale commercial production of various chemicals. Therefore, the kinetics of K-feldspar dissolution in sodium hydroxide media are also analyzed to determine the rate-controlling step and to optimize the reaction conditions. Hence, this study is expected to contribute to economic diversification, as the country is working out some alternative proposal from the present crude oil exploration as a major means of foreign exchange earnings.

2. Materials and Methods

2.1. Materials

The K-Feldspar ore used for this study was sourced from Giri village, Municipal Local Government Area of Abuja (Federal Capital Territory) of Nigeria. The ore was crushed, grinded and pulverized into three different particle sizes: 75+63 µm, -63+75 µm and -75+90 µm fractions. All reagents used were of analytical grade (BDH grades) and deionize water was used to prepare all aqueous solution.

2.2. K- feldspar 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 aqueous sodium hydroxide solution of pre-determined molarity (0.5 – 2.5 M) and 2 g ore were mixed in the reactor vessel making 20 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. Sample was collected at an interval of 10, 15, 30, 60 and 120 min. after the desired reaction time, the leached slurry was immediately separated by filtration. The effect of concentration on the extent of K- feldspar dissolution was studied in the NaOH concentration range between 0.5 to 2.5 M at temperature 55°C, particle size -110+75 µm and 300 rpm stirring speed. Considering the temperature dependency, the effect of leaching temperature on phosphate leaching was studied in the temperature range 27 - 90 °C under standard condition of 2.0 M NaOH, -110 +75 µm particle size and atmosphere pressure. To ascertain the effect of particle size on the rate of K-feldspar dissolution, experiments were performed using three different size fractions in the range of -75+63 µm, -63+75 µm and -75+90 µm fractions. The chemical reaction that occurred can be written as follows:

$$KAlSiO_3(s) + 2NaOH(s) + H_2O(l) \rightarrow Al(OH)_3(s) + KOH(aq) + Na_2SiO_3(aq) \qquad (2.1)$$

2.3. Pure silica precipitation

The resulting slurry from optimal leaching process was then filtered through an ashless filter (Whatman No 41) and washed with the minimum necessary amount of boiled distilled water. Silica starts to precipitate when the pH of the mixture falls to less than 10 using 0.5 M Hydrochloric acid (HCl); thus, the acidic conditions were maintained until the complete precipitation of silica was achieved. An acidification and neutralization of the transparent filtrate solution was performed to form a silica gel; therefore, the solution was titrated with H2SO4 (1M) to pH 7 under vigorously stirring. The reaction that yields silicic acid can be written as follows:

$$Na_2SiO_3(aq) + H_2SO_4(aq) \rightarrow H_2SiO_3(s) + Na_2SO_4(aq) \quad (2.2)$$

The soft, white aqua gel that formed was allowed to stand at room temperature for 24 hours, before being filtered and washed with distilled water to remove the sulphate salt. The solid residue was dried at 80°C for 24 hours. The silica purification was carried out against impurity such as: Al, Ca, Fe and Mg, by refluxing the powder with a solution of 1M HCl at 110°C for three hours. The suspension was filtered and washed with copious amount of distilled water, and oven dried at 110°C for 2 hours, follow by Calcinations at 800 °C for two hours in a muffle furnace. The fine silica powder was obtained with an excellent mass yield of 94% [8]. The reaction which yields pure silica was given as:

$$H_2SO_4(s) \rightarrow SiO_2(s) + H_2O(g) \quad (2.3)$$

The percent recovery of PSP from K-feldspar was calculated using the following equation:

$$\%\ \text{recovery of SPS} = \frac{\propto silica\ obtained}{\propto silica\ calculated} \quad (2.4)$$

Where, α-silica obtained is the fraction of PSP obtained experimentally from the dissolution of K-feldspar, while α-silica calculated indicates the theoretical concentration of silica contained in given amount of K-feldspar based on the chemical analysis.

2.4. Sample/product characterization

The initial raw k-feldspar ore and the PSP produced after precipitation was analyzed by X-ray fluorescent (XRF) with model No: X-MET800. X-ray powder diffraction (XRD) analysis of both k-feldspar and PSP was carried out using an APD 2000 diffractometer manufactured by Ital Structures (Riva Del Garda, Italy) and Scanning electron microscopy (SEM) techniques was used for microstructure analysis. pH values were measured with a pHM-26 instrument and a combined glass electrode with an operating range of up to pH ~ 14, both manufactured by Radiometer (Denmark). The Fourier transform IR spectrum was recorded using a Perkin-Elmer spectrometer (Model 2000). The specimen was pressed using a spectroscopically pure KBr matrix.

3. Results And Discussion

3.1. Ore characterization

The raw mineral used in this study as examined by XRF gave 18.61 % quartz, 1.32 % iron, 12.05 % aluminium, 12.15 % potasium, 0.69 % sulphur, 0.14 % calcium and 0.55 % calcium. The mineral phase of the ore detected by XRD supported the XRF result, comprised mainly of *quartz* ($Si_{3.00}$ $O_{6.00}$: 96-900-2700) albite ($AlSi_{3.00}O_{8.00}$: 96-900-9076), and orthoclase (KAlSi3.00O5.00 : 96-900-9567). The SEM analysis indicated that the raw ore consists of an-hedral to sub-hedral with two distinct scattered crystals of potassium and aluminium silicate (Fig. 1) varieties [18]

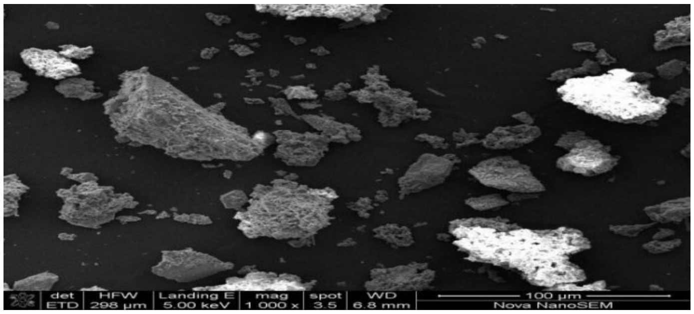

FIGURE 1. Micro structural image of raw K-feldspar.

The photomicrography of the thin section also indicated the main metallic minerals present in ore as aluminum oxide, potassium oxide, iron oxide (haematite) with associated gangues identified as quartz (Fig. 2).

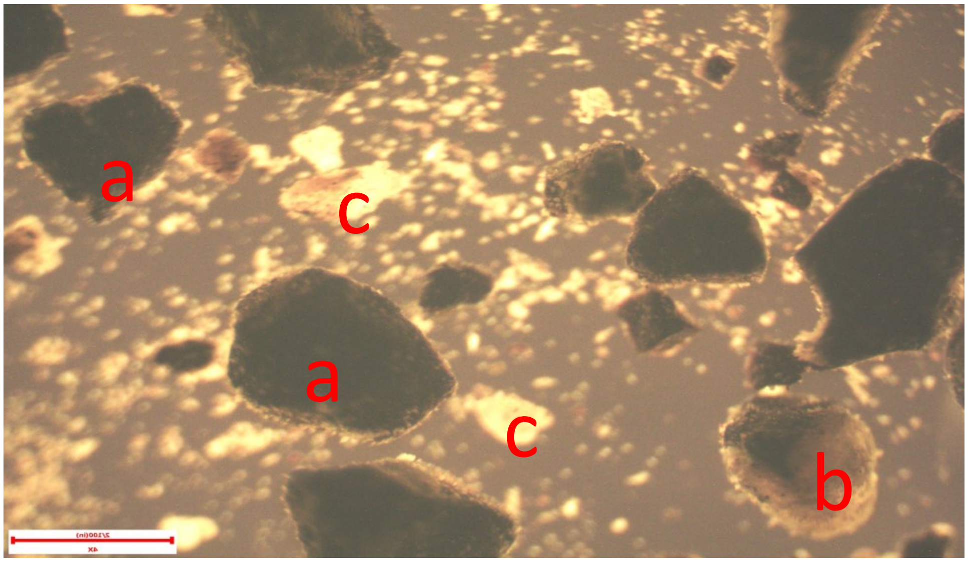

FIGURE 2. Photomicrography image of raw K-feldspar (a) quartz (b) albite (c) aluminium oxide.

3.2. Dissolution parameters optimization

3.2.1. Effects of sodium hydroxide concentration

A series of experiments was carried out to determine the influence of NaOH on the dissolution kinetics of K-feldspar. The concentration of NaOH varied from 0.5-2.5, as shown in Fig. 3. The highest SPS recovery was obtained from k-feldspar when 2.5M NaOH was used. This observation indicates that a highly concentrated alkaline solution is required to digest the K-feldspar and to extract silica.

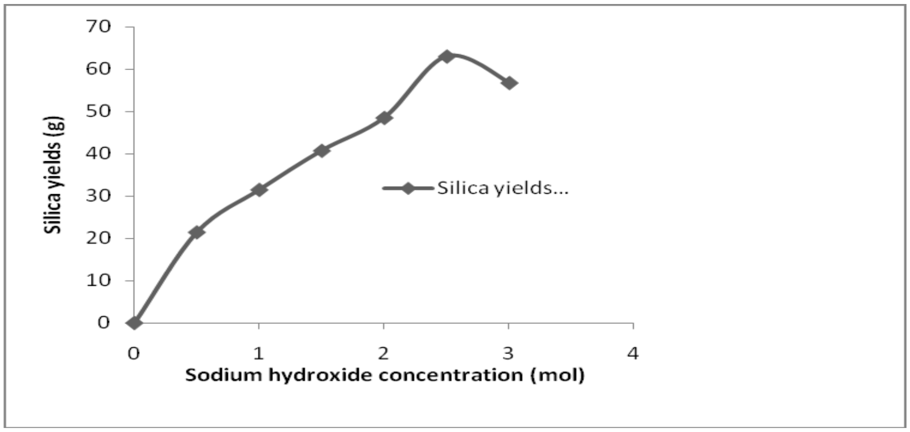

FIGURE 3. Profile of effect of concentration on silica extraction.

3.2.2. Effect of reaction temperature

The effect of leaching temperature (27–90°C) on the extent of k-feldspar ore dissolution was investigated. Other experimental conditions was kept constant at S/L of 20 g/L, 2.5 mol/L NaOH concentrations with moderate stirring (300 rpm) speed up to time interval of 120 minutes (Fig. 4).

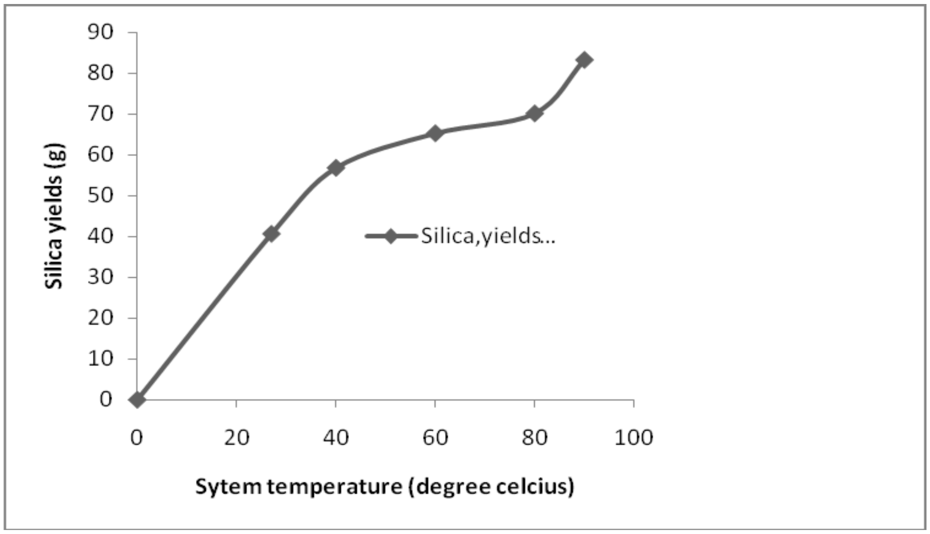

FIGURE 4. Profile of system temperature on silica extraction

From the result shown graphically in Fig. 4, it is evident that temperature plays an important role on the extent of K-feldspar ore dissolution. With increasing temperature, the dissolution increases. For example, at 90°C, about 83.3 % of the initial 20 g/L k-feldspar ore was reacted within 120 minutes of reaction time. This is in line with the result of other similar studies which affirmed that increasing temperature of a system during ore dissolution speed up the rate of a reaction process [8]. However in this study, temperature above 90°C was not considered.

3.2.3. Effect of particle size

Leaching experiment was carried out using three different particle size fractions of -75+90 µm, -90+112 µm, -112+145 µm. Other experimental conditions were kept constant at temperature 90 °C, S/L of 20 g/L, 2.5 mol/L NaOH with moderate stirring (400 rpm) speed up to time intervals of 120 minutes (Fig. 5). This result affirmed that as ore fraction decreases from -112+145 µm to 63+75 µm, dissolution efficiency increases moderately. This result support liberation properties by mechanical milling that improve the surface area for reaction enabling chances.

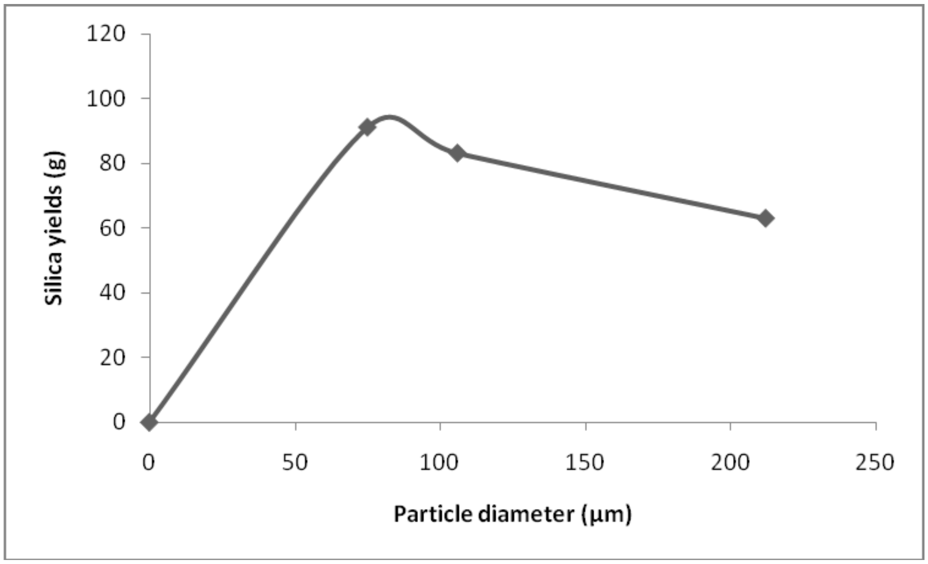

FIGURE 5. Effect of particle diameter on silica yield.

3.3. Kinetic analysis

In solid–fluid systems, the reaction rate is governed by one of the following kinetic mechanisms: diffusion through fluid film, diffusion through ash or product layer, or surface-controlled chemical reaction [16,17-18]. In a general form, the solid–fluid reaction can be represented as:

$$A_{(\mathrm{fluid})} + B_{(\mathrm{solid})} \rightarrow C_{(\mathrm{products})} \tag{3.1}$$

In acidic/basic leaching, the heterogeneous non-catalytic reaction for most mineral ore might be kinetically interpreted using the un-reacted Shrinking Core Model (SCM) [19,20]. The SCM is based on the description that solid particles are consumed by dissolution or reaction and as a result, the material being consumed is shrinking. Following the principle that dissolution process takes place from the outer surface of the ore particle, and as dissolution proceeds, the interface between the un-dissolved solid phase and solute free phase move towards the centre of the core, yielding the following three steps[21,22].

(i) diffusion of solute molecules from the solid-solid interface to the solid-liquid interface via the solute-free portion of the particle,

(ii) diffusion of solute molecules from the solid-liquid interface through the surface layer to the outer boundary of the surface layer; and

(iii) diffusion of solute molecules from the outer boundary of the surface layer to bulk liquid phase.

The rate equations for surface chemical reaction and diffusion through fluid film can be represented by eqn (3.2) and (3.3), respectively:

$$1 - 2/3\alpha - (1 - \alpha)^{2/3} = k_d t \tag{3.2}$$

$$1 - (1 - \alpha)^{1/3} = k_s t \tag{3.3}$$

Generally, it is assumed that α is the fraction of k-feldspar dissolved, and t is the reaction time (min), and k_S and k_d are the reaction rate constants for surface chemical reaction and diffusion through fluid film, respectively. In order to determine the rate limiting step for the leaching of silica from k-feldspar, the obtained experimental data were analysed using statistical and graphical approaches. From an analysis of correlation coefficients, it was determined that the leaching of k-feldspar in alkaline media follows a surface chemical reaction-controlled mechanism. Therefore, substituting the experimental data obtained from

Figures 3 and 4 into the kinetic equations (3.2–3.3). The calculated statistical average correlation of 0.65 and 0.94 respectively, showing that equation (3.3) gave better correlation. This indicates that the mechanism of k-feldspar leaching is closely follows the chemical controlled model and it was used in the estimation of some thermodynamics parameters such as reaction order and activation energy for the dissolution reaction as follows.

The treatment of dissolution data in Fig. 3 was done using (equation 3.3) to obtain the linear graph which later used to determine the order of reaction at defined concentrations of NaOH by plotting the value of $\ln k_s$ versus ln[NaOH] as depicted in Fig. 6.

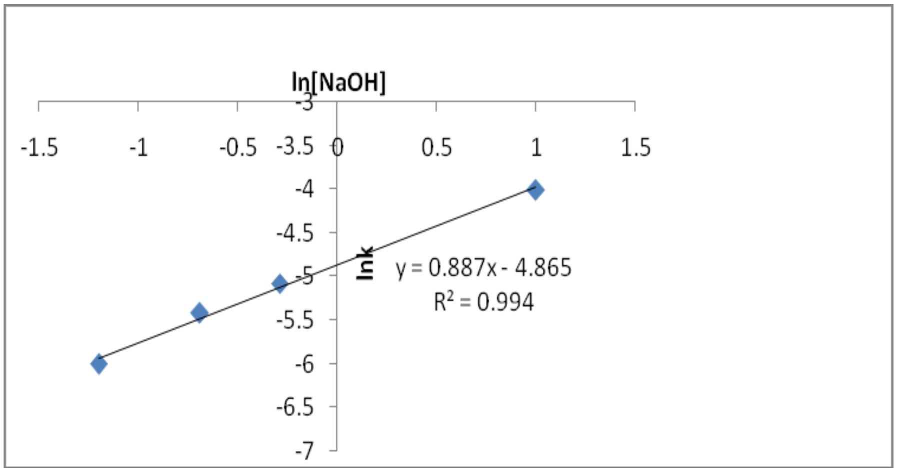

FIGURE 6. Relationship between ln*k* and ln*[NaOH]* for silica extraction.

The order of reaction derived from Fig. 6 gave 0.887 ~1.0 indicating a first order reaction at defined leachant concentrations. The temperature dependence of the reaction rate constant (dissolution activation energy) was calculated using the Arrhenius equation, the rate constant can be expressed as:

$$k_S = A_o e^{-Ea/RT} \tag{3.4}$$

Here, A_o is the Arrhenius constant, Ea the energy of activation, R the ideal gas constant, and T the reaction temperature. By comparing eqn (3.3) and (3.4), the integral rate expression can be written as

$$1 - (1 - \alpha)^{1/3} = A_o e^{-Ea/RT} t \tag{3.5}$$

In order to determine the values of the activation energy and Arrhenius constant, ln k_S and (1/T) values were plotted, as shown in Fig. 7.

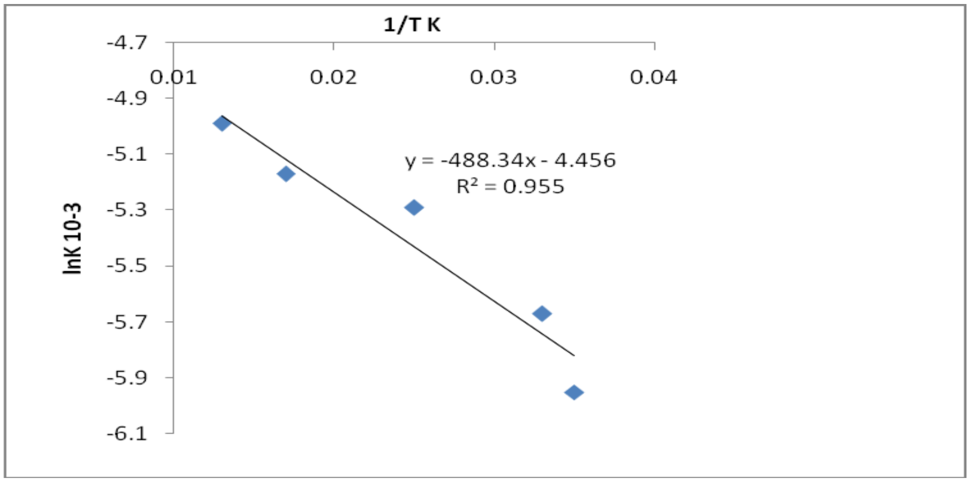

FIGURE 7. ln*k* versus 1/*T*.

Inserting the values for the activation energy and Arrhenius constant, eqn (8) can be written as

$$1 - (1 - \alpha)^{1/3} = 4.3357\ e^{-4063/RT}\ t \quad (3.6)$$

The value of the activation energy (40.63 $kJmol^{-1}$) in equation (3.6) for the alkaline leaching of k-feldspar is in agreement with values obtained in previous studies of fluid–solid reaction systems [8, 23-24].

3.4. Purity analysis of PSP

Pure silica precipitate obtained from alkaline dissolution of K-feldspar was characterized by Fourier Infrared (FT-IR), XRD, SEM and thermogravimetric analysis (TGA). Fig. 8 exhibits the FT-IR spectra of extracted silica obtained after calcinations at 800°C for two hours.

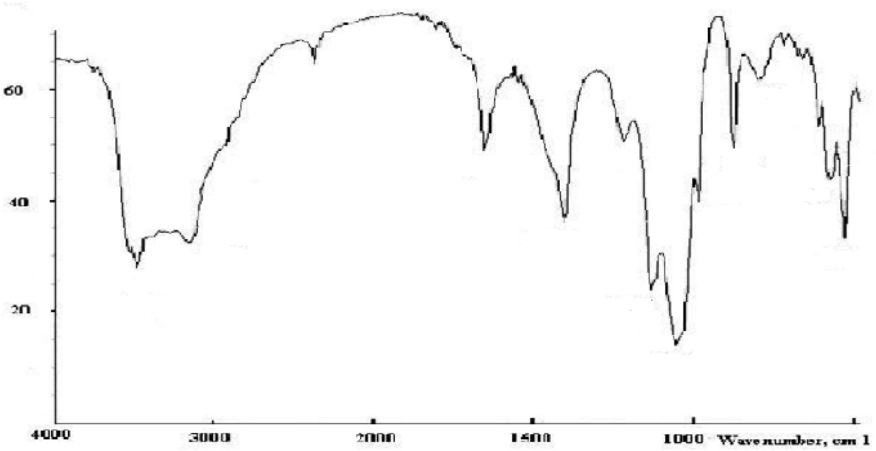

FIGURE 8. FT-IR spectral of precipitated silica.

The predominant band at 1100 cm^{-1} and the shoulder at 1189 cm^{-1} in Fig. 8 are associated with asymmetric stretching-vibrations of siloxane νs(Si-O-Si). The presence of bands at 474 cm^{-1} and 812 cm^{-1} was due to the presence of symmetric siloxane groups νs(Si-O-Si). The existence of a band at 950 cm^{-1} is associated with Si-OH groups; this band's existence is due to high concentrations of silanol groups with smaller particle sizes. The shoulder appeared at 3750 cm^{-1}, indicating the presence of hydrogen bonds that resulted from interaction between the silanol groups (Si-OH) located at the surface of the silica material. In the material, the band located at 1642 cm^{-1} is attributed to the O-H bending vibration of the adsorbed molecular water and its corresponding stretching vibration at 3452 cm^{-1}.

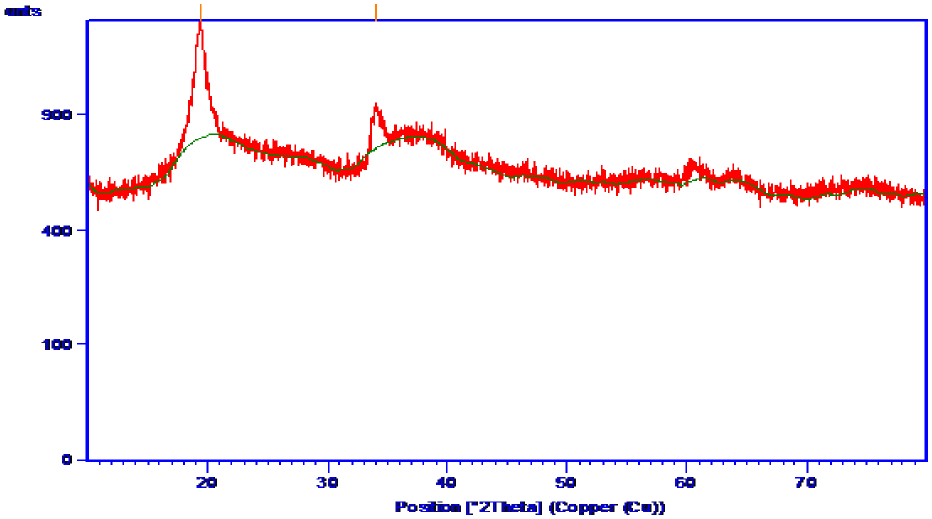

FIGURE 9. The XRD spectra of precipitated crystalline phase.

The XRD pattern of PSP shown in Fig. 9 shows that the PSP sample lacked any crystalline phase and exhibited an amorphous nature. The morphology of the PSP was studied by SEM (Jeol-5800LV). Fig. 10a shows the fluffy and porous nature of PSP, and that the most of the PSP particles were smaller than 1 mm. The EDX spectrum of APS (Fig. 10b) shows that the PSP sample was devoid of any inclusions or impurities. The thermal stability of PSP was determined by TGA using a heating rate of 10 °C min^{-1}. The TGA curve in Fig. 11 indicates two regions of weight loss, a lower temperature weight loss due to evaporation of weakly bonded (physisorbed) water molecules and a higher temperature loss during condensation of silanol groups.4 The overall weight loss of the APS sample was 11%; 8% weight loss occurred below 200 °C, while the remaining 3% took place between 200–500 °C.

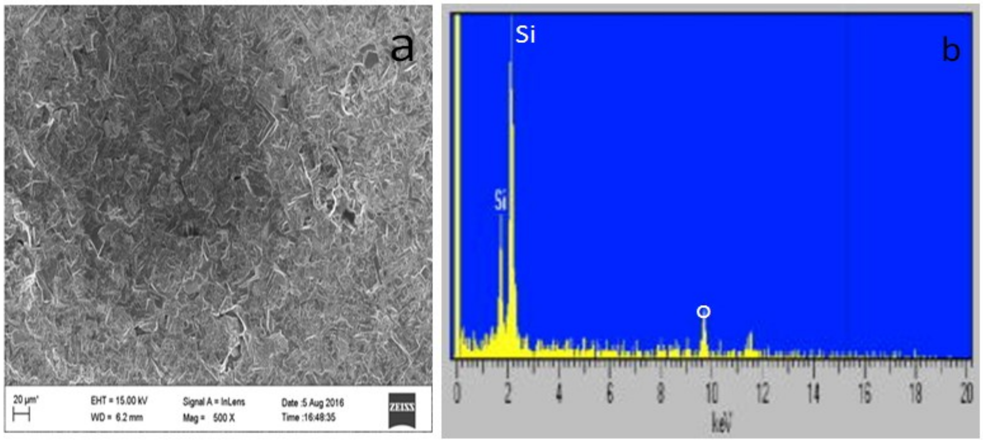

FIGURE 10. Microstructural Morphology of the PSP (a) SEM image (b) EDS of targeted point.

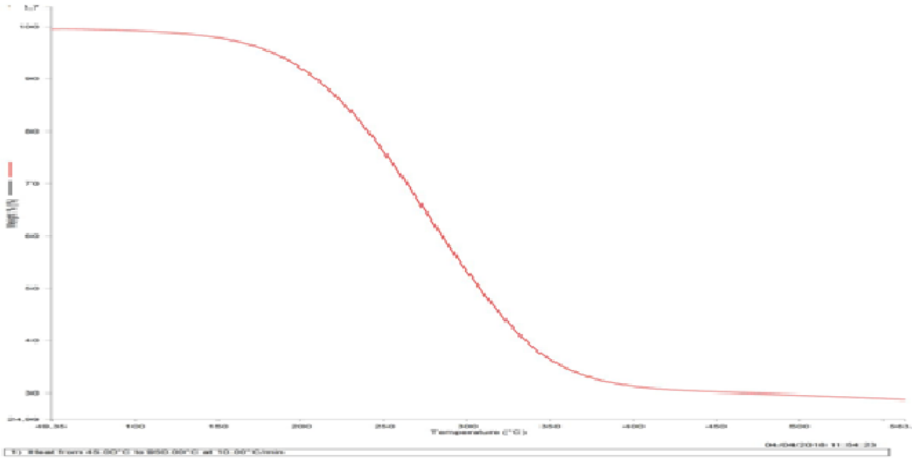

FIGURE 11. TGA spectra of precipitated silica heat from 45 °C – 950 °C at 10 °Cmin^{-1}.

3.5. Schematic diagram of pure silica precipitated process

A simple hydrometallurgical scheme was proposed for the extraction of silica from a Nigerian origin k- feldspar ore for industrial value addition (Fig. 12).

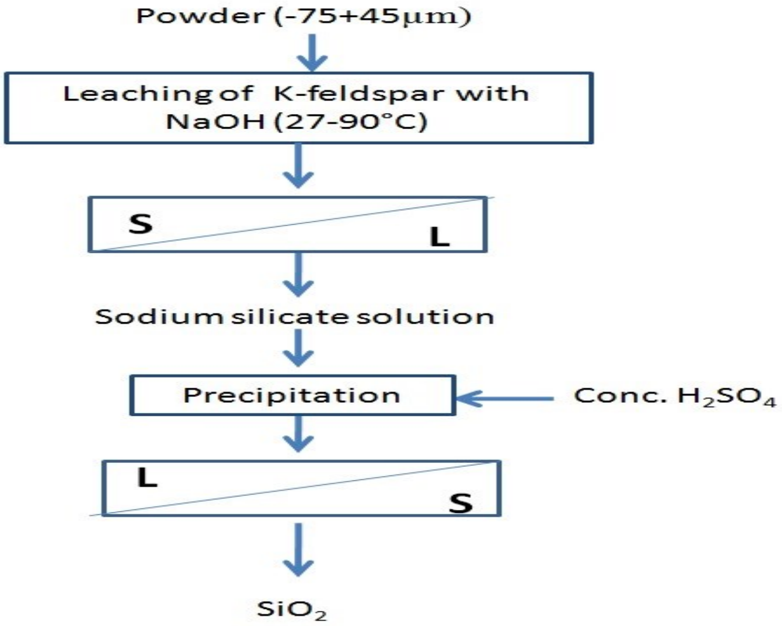

FIGURE 12. Schematic diagram of silica extraction from k-feldspar.

4. Conclusion

The process development for the extraction of pure silica from k-feldspar silicate mineral is unique in nature. Highly pure silica (SiO_2) powder was obtained through roast leaching and precipitation treatment of k- feldspar of Giri village, Nigeria, which contains an appreciable amount of silicate minerals. The conversion of k-feldspar powder into silica reached 83.3% as the process progresses. The leaching data obtained was analyzed by suitable Shrinking Core Model and the chemical controlled mechanism was found appropriate as the rate determine step for the dissolution process and the calculated activation energy (E_a) of 40.63 kJmol^{-1} supported the proposed model. The FTIR and XRD technique of the synthesized product confirmed production of pure silica at optimal conditions and expected to improve revenue generation to aid Nigeria economic diversification and sustainability.

ÖZET

Nijerya, çok çeşitli mineral kaynağı olan Afrika ülkelerinden birisidir. Ülkenin tüm bölgelerindeki büyük mineral bolluğuna rağmen, bu kaynaklar ülke ekonomisine katkı sağlamak, sanayileşmeye yardımcı olmak ve gelir yaratmak için daha az kullanılmıştır. Bu nedenle, *ortoklaz* ($KAlSiO_8$: 96-740-9031), *albit* ($AlSiO_8$: 96-940-0741) ve *kuvars* (SiO_2 : 96-900-9667) safsızlıklarının bir karışımını içeren bir Nijerya k-feldispatının saf silika tozu (PSP) elde etmek için kullanımı, kavurma liçi ve birlikte çöktürme yöntemi ile incelendi. Liç sırasında, sızıntı suyu konsantrasyonu, parçacık büyüklüğü ve tepkime sıcaklığı gibi parametreler incelendi. Optimum liç koşullarında (2.0 mol/L NaOH, 90 °C), başlangıçta 10g/L mineralin %87' si 120 dakika içerisinde tepkimeye girdi. XRD ile analiz edilen tepkimeye girmemiş ürünün (~% 13.0) bazı tanımlanmış endüstriler için değerli bir yan ürün olarak hizmet verebilen silisyumlu safsızlıklar içerdiği bulundu. Optimal liçteki sızıntı suyu, endüstriyel değerde saf silika tozu elde etmek üzere derişik H_2SO_4 kullanılarak çöktürüldü.

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