

PRODUCTION OF SODIUM SILICATE AND SODIUM ALUMINATE FROM ALUM PRODUCTION WASTE

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ABSTRACT

Industrial solid waste accumulation poses a great hazard for the environment. The aim of this work was the utilization of alum waste produced from the production of Alum { $\text{Al}_2(\text{SO}_4)_3$ } using kaolin to produce sodium aluminate and sodium silicate. This was carried out by the leaching of calcined Alum waste using several acids (H_2SO_4 , HCl , HNO_3) with different concentrations (2, 4, 6, 8 and 10 M) at 90 °C for 2 hrs to achieve maximum Al_2O_3 recovery followed by filtration process. The filtrate (concentrated Al_2O_3) was treated with NaOH to produce sodium aluminate while the precipitate (concentrated SiO_2) was treated with NaOH to produce Sodium Silicate via two different processes. The first process was by fusing the precipitate with NaOH with ratio 1:1 to 1:5 (Solid: NaOH) at 60 °C followed by dissolving in water at 60 °C. The second process was by treating the precipitate with several NaOH concentrations (1, 2, 3, 4, 5 M) at 90 °C for varying times (1, 2, 3 hrs). Results showed that maximum Al_2O_3 recovery was achieved with 10M H_2SO_4 leaching at 90 °C for 3hrs (89.28 %) which is used to produce Sodium Aluminate. Sodium Aluminate solution was dried at 130-150 °C and analyzed using X-Ray diffraction (XRD). Sodium Silicate was produced with $\text{SiO}_2/\text{Na}_2\text{O}$ weight ratio (WR) from 2.15 to 3.44 in the first process while was nearly 2 in the second process.

Key words: Kaolin; Sodium Silicate; Sodium Aluminate; Acid leaching; Alkaline dissolution

INTRODUCTION

Alum occurred naturally in the earth crust as a double salt with formula, $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{-}27\text{H}_2\text{O}$ (Kuranga *et al.*, 2018). It is used as a coagulant in water treatment plants for supply of safe domestic and industrial water. It is traditionally extracted from bauxite by the Bayer process or from other aluminosilicate minerals as kaolin (Chigondo *et al.*, 2015).

Kaolin is a clay mineral containing silica (SiO_2) and alumina (Al_2O_3) in a high percentage which is an unreactive mineral in its natural form (Bendary *et al.*, 2017; Rahayu *et al.*, 2018). The extraction of alumina from kaolin takes place by the activation of kaolin through the calcination process (de-hydroxylation), followed by leaching with mineral acids (Yahaya *et al.*, 2017; Ibrahim *et al.*, 2018; Rahayu *et al.*, 2018).

The production of alum using kaolin produces large quantities of partially dealuminated kaolin (Alum waste) which is mainly composed of silica and some un-leached elements. The main problems accounts to the amount of waste generated and air pollution which may cause direct environmental threats because of its acidic nature besides the fineness of the particles (Bendary *et al.*, 2017).

Sodium aluminate is an important industrial inorganic chemical. It is used in water treatment and acts as a coagulant to remove suspended solids and some metals (Cr, Ba, Cu) as well as for removing dissolved silica (Contreras *et al.*, 2006). It is considered a convenient source of aluminum in synthetic applications as in the production of zeolite and other catalytic materials (Kaduk and Pei, 1995; Caicedo-Realpe and Pérez-Ramírez, 2010). Also, it is used to accelerate the solidification of concrete, in paper industries, production of refractory brick and alumina production. It is mainly formed from Bayer process (Giels *et al.*, 2022; Blanks, 2000). Sodium aluminate can be prepared by the solid-state reaction of sodium hydroxide and subdivided aluminum hydrate at a temperature above the melting point of the caustic soda but below 600°C (Contreras *et al.*, 2006). It can be recovered from sodium dawsonite found in association with oil shales (Dyni, 1972), or it can be prepared from basic aluminum sulfate (Contreras *et al.*, 2006). In this context, aluminum hydroxide reacts with a concentrated NaOH solution to produce sodium aluminate.

Sodium silicate is the popular name for a compound with the formula $\text{Na}_{2x}\text{SiO}_{2+x}$, such as sodium metasilicate Na_2SiO_3 , sodium orthosilicate Na_4SiO_4 , and sodium pyro-silicate $\text{Na}_6\text{Si}_2\text{O}_7$ (Liu & Ott, 2020).

Over 100 years, sodium silicate have been produced by fusion of sand with sodium carbonate. This process is costly due to the consumption of more energy to reach high temperature for decarbonation of sodium carbonate beside the production of air pollutants such as dust particles, SO_x , NO_x , CO, and CO_2 (Cha and Park, 2001; Torres-Carrasco *et al.*, 2014). The hydrothermal process (Andreola *et al.*, 2020) is another process with the least energy requirement, no issue with dust emission (Owoeye and Isinkaye, 2017) and no emission of CO_2 (Rungronmitchai *et al.*, 2009). This process involves the reaction of silica (SiO_2) with aqueous NaOH in an autoclave under temperature and pressure or in an open system under atmospheric pressure (Ajayi and Owoeye, 2015; Owoeye & Isinkaaye, 2017; Owoeye *et al.*, 2021). The weight ratio of $\text{SiO}_2:\text{Na}_2\text{O}$ characterizes the grades of sodium silicate.

Every year, around 3–4 million metric tons of soluble silicate—including potassium silicate—are produced globally, estimated in 1998 (Liu and Ott, 2020). Sodium silicate is used in many applications such as detergents, paper, water treatment, construction materials, binders, adhesives, zeolites, catalyst bases, coagulant aids, corrosion inhibitors, coatings, deflocculants, cleaning chemical components and in oil, gas, and geothermal well operations. Also, it is considered as exogenous silicates which can help in increasing plant resistance to diseases such as powdery mildew and Fusarium wilt in cucumber, sheath blight and blast in rice and powdery mildew in wheat (Zhou *et al.*, 2018). *In vitro* tests also demonstrated that sodium silicate inhibited the mycelial growth of some fungi, such as *Fusarium semitectum*, *Alternaria alternata*, and *Trichothecium roseum* (Bi *et al.*, 2006).

The aim of the present work was to decrease the waste disposed to the environment by the utilization of waste produced from the production of aluminum sulfate (Alum) in the production of sodium silicate and sodium aluminate.

MATERIALS AND METHODS

Materials:

Partially dealuminated kaolin (PDK) (alum waste) sample was obtained from the aluminum sulfate Co. (ASCE) in Egypt. sulfuric acid (HKIN & WILLIAMS, 98%), hydrochloric acid (Merck, 37%), nitric Acid (Panreac Quimica, 69%) and sodium hydroxide pellets (POLSKIE ODCZYNNIKI CHEMICZNE S.A., >99%) were purchased. All the used chemicals were analytical grade.

Methods:

The alum waste (PDK) was sieved through 20 μ m mesh using wet sieving method to remove very fine particles, then dried at 105 °C for 5hrs. The sieved sample (SS) was grinded using disk mill to size between 74 – 20 μ m then calcined at 750 °C for 2hrs in electric furnace.

Second dealumination process

The calcined sieved sample (CSS) was treated with a series of different concentrations (2, 4, 6, 8 and 10 M) of mineral acids (H₂SO₄, HCl and HNO₃ acids) at 90 °C for 2hrs. 5 g of sample was weighed in beaker 250 mL, and 50 mL of acid was added (liquor ratio 1:10). The solutions were filtrated through Buchner Filtering Funnel with Medium Frit (G4) to produce silica-rich solid and filtrate. The solid was washed with boiling distilled water to be free from acid. The filtrate solutions were analyzed by AAS to determine the percentage of recovered aluminum.

The leaching process was carried out using 10M H₂SO₄ at 90°C for different time intervals (1, 2, 3, 4, and 5 hrs.), then filtrated. The solid was washed with boiling distilled water to be free from acid, then dried for 5 hrs. at 105 °C in a drying oven. The silica was determined by the gravimetric method, and the filtrate solution is analyzed by AAS to determine the recovery percentage of aluminum and the time that verified the maximum recovery percentage of aluminum.

Preparation of sodium Aluminate:

The leachate solution produced from the dealumination process (10 M H₂SO₄, 3hrs. at 90 °C) was neutralized using 10 M NaOH to pH 7; which caused the precipitation of Al, Fe, and Ti. The solution was filtrated through Whatman filter paper No. 40 and the precipitate was washed using hot distilled water until free from alkali. The precipitate was transferred to a glass beaker 250 mL containing 50 mL of distilled water. The beaker content was heated to about 50 °C while stirring and treated with 10M NaOH solution to pH > 12, which dissolve Al as Na₂Al₂O₄ (not Fe or Ti). This was followed by filtration through filter paper Whatman No. 1. This solution was dried in a drying oven at 130 – 150 °C, and the obtained solid was characterized by X-Ray Diffraction (XRD).

Preparation of sodium silicate:

The preparation process of sodium silicate was carried on silica obtained after acid leaching of (CSS) by two different methods: fusion and the alkaline dissolution methods.

1. Fusion method: a weight of silica was fused with different ratio of sodium hydroxide (Lázaro *et al.*, 2020).

In this method, 3g of silica was weighed in several polytetrafluoroethylene vessels (PTFE) and different ratios of NaOH to each vessel (w/w) 1:1, 1:2, 1:3, 1:4 and 1:5 (solid: NaOH) were added. The vessels were put on a hot plate for 15 min to fuse their content at about 60 °C. 50 mL distilled water was added to the vessels and stirred for 15 min. The solutions were filtrated through filter paper Whatman No. 50 in a volumetric flask 100 mL to filter out the unreacted silica from the solutions, washed with boiling distilled water and up to the mark. Then the concentrations of Na and Si were determined using AAS.

2. Alkaline dissolution method

In this method, 3 g of silica was weighed in several PTFE vessels and 30 mL of NaOH of different concentrations (1, 2, 3, 4, 5 M) was added to each vessel, heated with stirring at 90 °C for different time intervals (1hr, 2hr, 3hr). The filtration was carried through filter paper Whatman No. 50 in volumetric flask 100 mL and the residue was washed with boiling

distilled water up to the mark. Then the concentrations of Na and Si were determined using AAS (Ayegba *et al.*, 2015).

Figure 1 represents the whole process of producing sodium silicate and sodium aluminate.

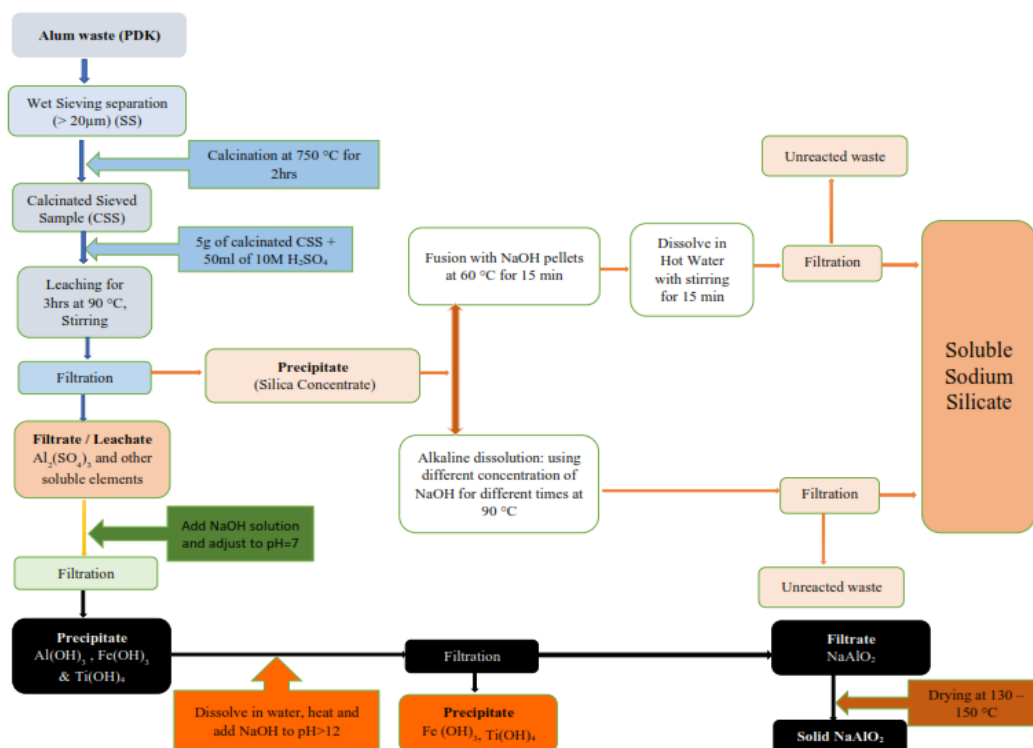


Figure 1. Flow diagram for the production of sodium silicate and sodium aluminate

RESULTS AND DISCUSSION

The alum waste (PDK) and sieved samples were characterized using chemical analysis and XRD. The results of chemical analysis (Table 1) showed that the samples contained mainly SiO_2 and Al_2O_3 and the sieving process didn't affect the main components of the main sample. XRD patterns of PDK and SS (Figs 2 & 3) emphasized that the samples consisted mainly of quartz and contain small amounts of anatase and aluminum silicate.

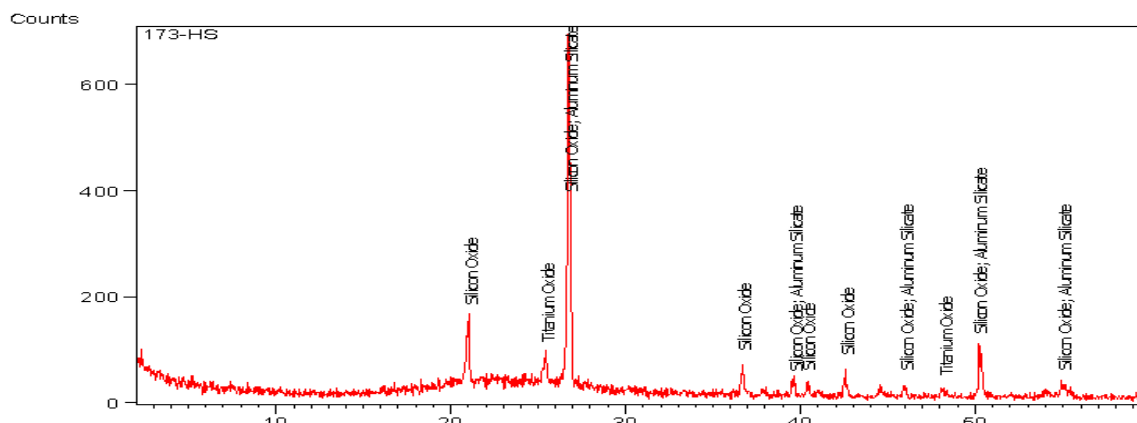


Figure 2. X-Ray Diffraction analysis of partially dealuminated kaolin, PDK sample

Table 1. Chemical analysis of partially dealuminated kaolin and sieved samples

COMPOSITION		PDK SAMPLE	SIEVED SAMPLE (SS)
SiO ₂	%	75.55	78.37
Al ₂ O ₃	%	5.30	4.59
Fe ₂ O ₃	%	0.28	0.25
TiO ₂	%	1.92	1.83
CaO	%	0.046	0.057
MgO	%	0.006	< 0.001
Na ₂ O	%	0.07	0.029
K ₂ O	%	0.046	0.050
L.O.I at 750 °	%	11.60	9.15
L.O.I. at 1000 °C	%	13.60	10.95

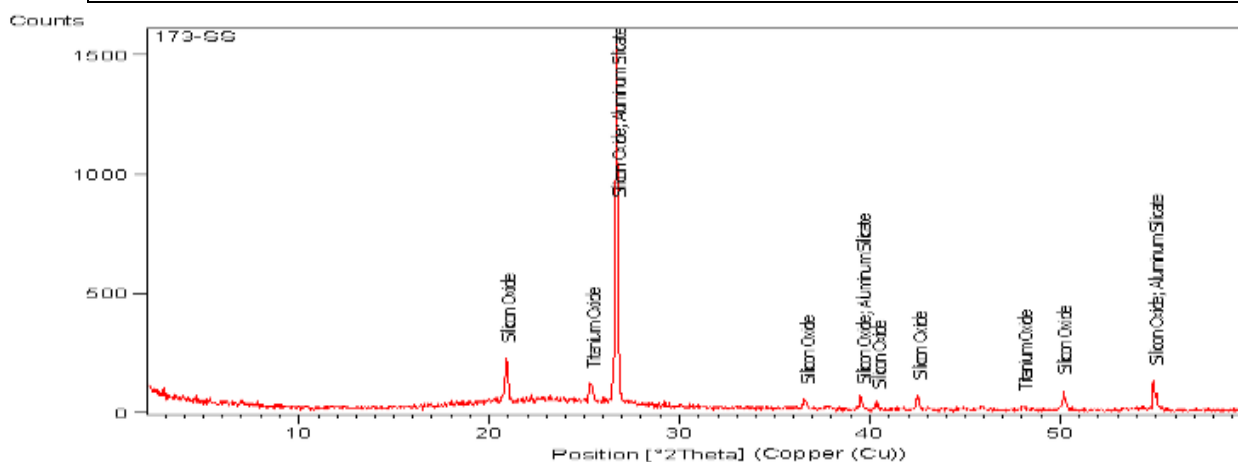


Figure 3. X-Ray Diffraction analysis for sieved sample

The sieved sample (SS) was calcined at 750 °C for 2hrs to obtain de-hydroxylation sample; which provides active Si-O and Al-O bonds (Rahayu *et al.*, 2018). The calcination process resulted in increase of SiO₂, Al₂O₃, Fe₂O₃ and TiO₂ contents (Table 2). XRD patterns (Fig. 4) showed that the calcination process didn't affect the main components of sieved samples but resulted in the losing of structural water to obtain a reactive phase.

Table 2. Chemical analysis of calcined sieved sample (CSS)

COMPOSITION		CALCINED SIEVED SAMPLE (CSS)
SiO ₂	%	85.30
Al ₂ O ₃	%	5.13
Fe ₂ O ₃	%	0.31
TiO ₂	%	2.07
CaO	%	0.058
MgO	%	< 0.001
Na ₂ O	%	0.021
K ₂ O	%	0.050

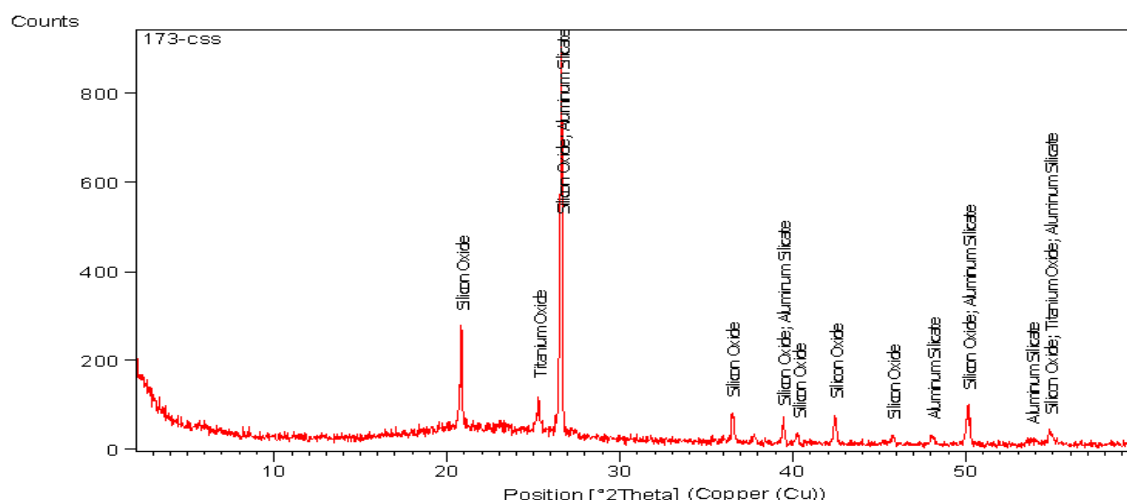


Figure 4. X-Ray Diffraction analysis for calcined sieved sample

Second dealumination process using different concentrations of different mineral acids at 90 °C for 2 hrs. was carried in order to determine the best acid and its concentration for maximum aluminum recovery condition. The AAS analysis of the leachate produced from the dealumination process using H₂SO₄, HCl and HNO₃ with different concentration (2, 4, 6, 8 and 10 M) at 90 °C for 2hrs, showed that the maximum recovery of Al₂O₃ achieved using 10M H₂SO₄ and it was 71.35 % as shown in Fig 5.

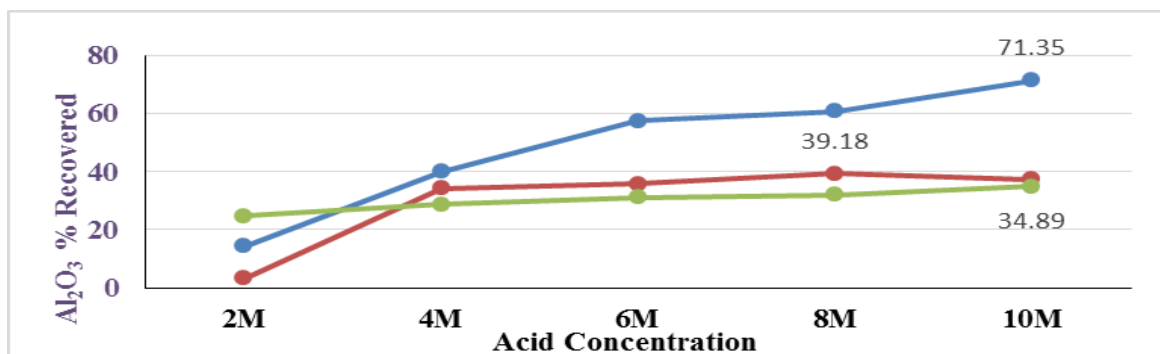


Figure 5. Effect of different acid concentration on recovery % of Al₂O₃ at 90 °C for 2hrs.

Effect of leaching time on yield of aluminum recovery:

Second dealumination process using 10M H₂SO₄ at 90 °C for different time intervals was carried to determine the time that verified the maximum recovery percentage of aluminum. The AAS analysis results of the produced leachate were presented in Fig 6. It showed that the maximum recovery of Al₂O₃ (89.28%) was verified by 10M H₂SO₄ acid for 3hrs. While maximum recovery of TiO₂ and Fe₂O₃ was 80.67% and 74.20% respectively. The decrease in Al concentration with time is due to the precipitation of Al as monobasic aluminum sulfite from the pregnant solution (Raddatz *et al.*, 1981). The achieved result is an effective result compared to the extraction of Al₂O₃ from kaolin using 40% wt H₂SO₄ at 140 °C for 3h 45 min where the maximum extraction was 79.28% (Ibrahim *et al.*, 2018).

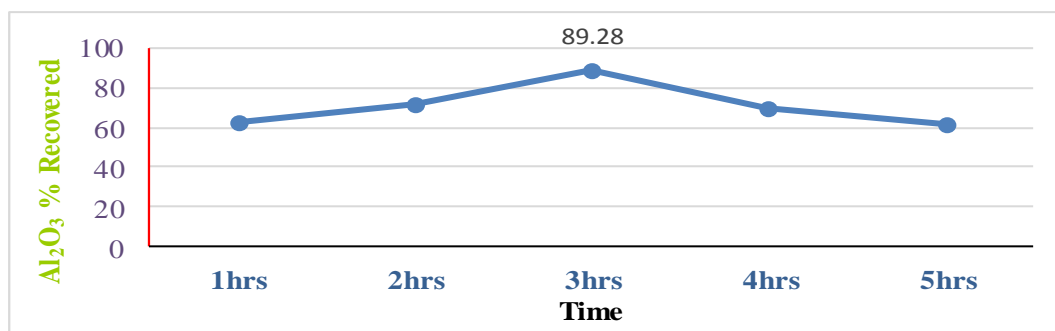


Figure 6. Effect of time on recovery % of Al₂O₃ using 10M H₂SO₄

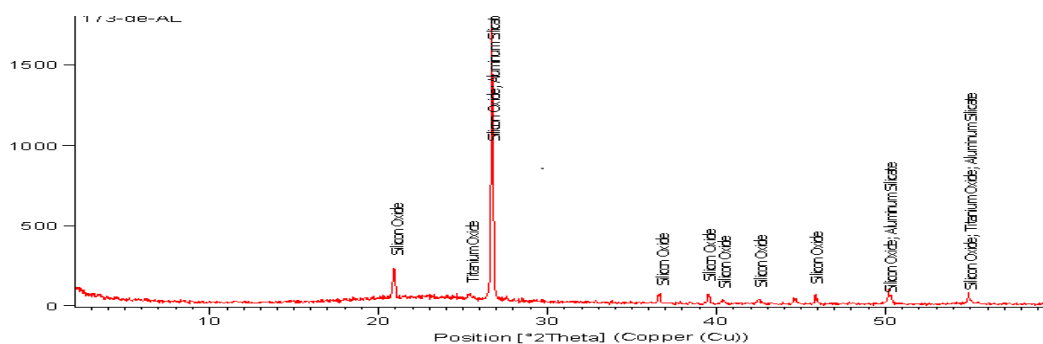


Figure 7. X-Ray Diffraction analysis for the dealuminated Sample using 10M H₂SO₄ for 3hrs

XRD pattern of the *dealuminated sample using 10M H₂SO₄ for 3 hrs* (Fig. 7) showed that the sample consists mainly of quartz and disappearance of some peaks of anatase and aluminum silicate compared to (Fig. 4) indicate decreasing in their percentage and emphasizes the effectiveness of the dealumination process.

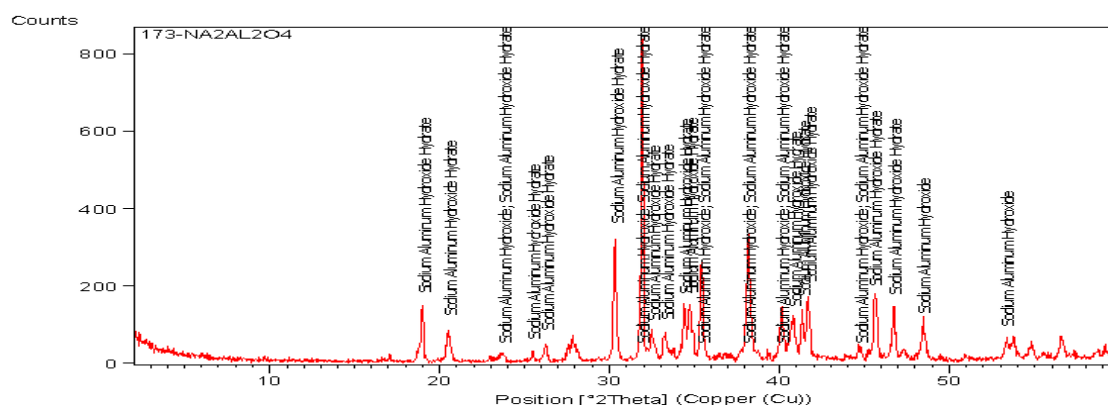


Figure 8. X-ray Diffraction analysis for Sodium Aluminate

The XRD pattern (Fig. 8) illustrates the formation of sodium aluminum hydrate, which was produced from the leachate solution treatment with 10M sodium hydroxide and dried at 130–150 °C.

Sodium silicate production:

The reaction between SiO_2 and NaOH require high temperature and pressure but dealuminated kaolin contained amorphous silica (Rahayu et al., 2018) which is reactive at low temperature with NaOH (Yadav and Azeem, 2023).

1. Fusion method

The AAS analysis results of sodium silicate produced by fused dealuminated sample with different ratios of NaOH showed that, the increasing in the dissolution of silica which ranged from 35.29 to 68.40% with increasing the ratio of NaOH (Fig. 9). The weight ratio (WR) of $\text{SiO}_2/\text{Na}_2\text{O}$ also increased with increasing NaOH ratio, which ranged from 2.15 to 3.44 (Fig. 10).

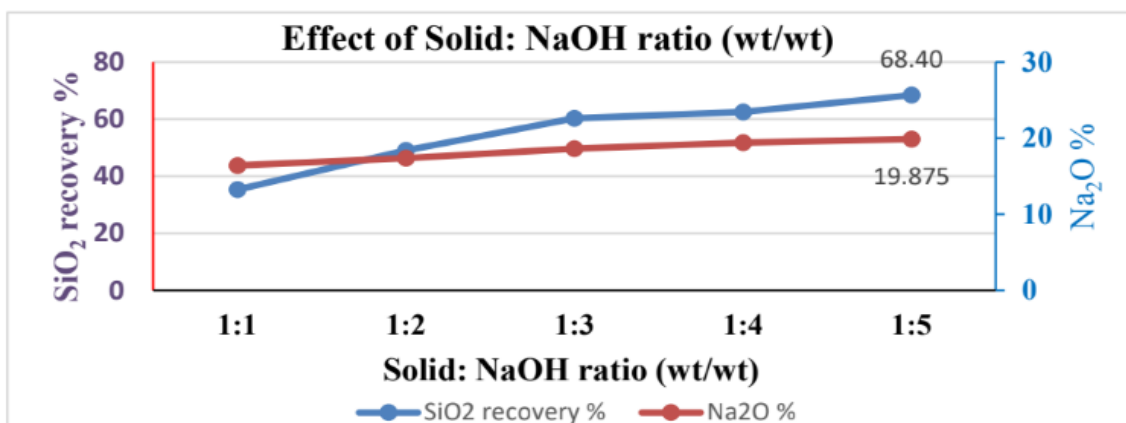


Figure 9. Effect of NaOH ratio on silica dissolution

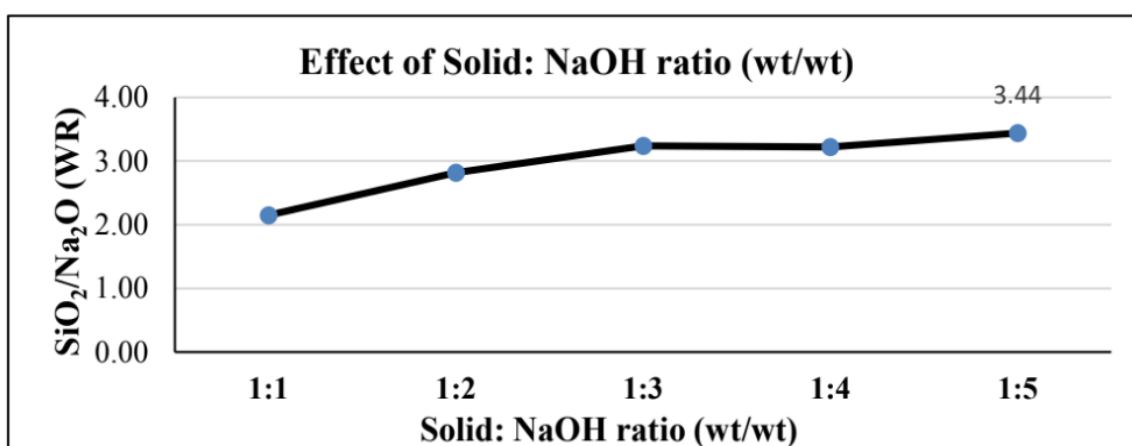


Figure 10. Effect of NaOH ratio on SiO₂/Na₂O weight ratio (WR)

2. Alkaline dissolution

The results of sodium silicate produced by treating dealuminated sample with different concentrations of NaOH at different time intervals represented in Figs 11, 12, 13, 14, 15 and 16.

A. Dissolution of SiO₂ using different concentration of NaOH at 90 °C for 1hrs.

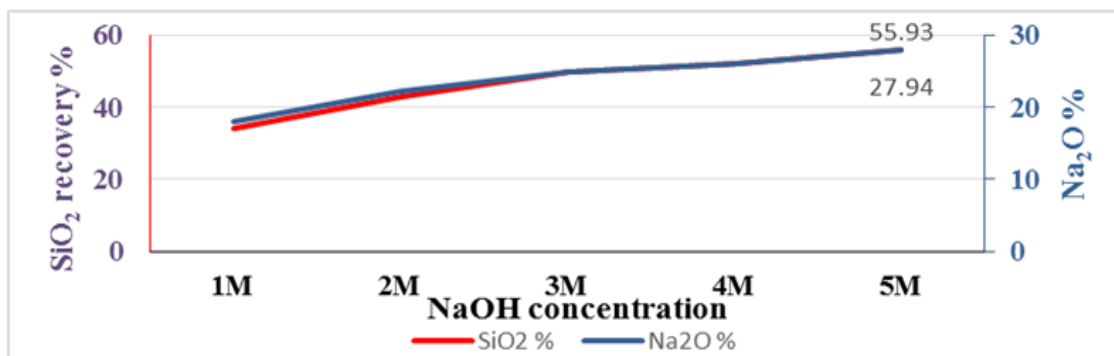


Figure 11. Effect of NaOH Concentration on SiO₂ dissolution at 90 °C for 1hrs

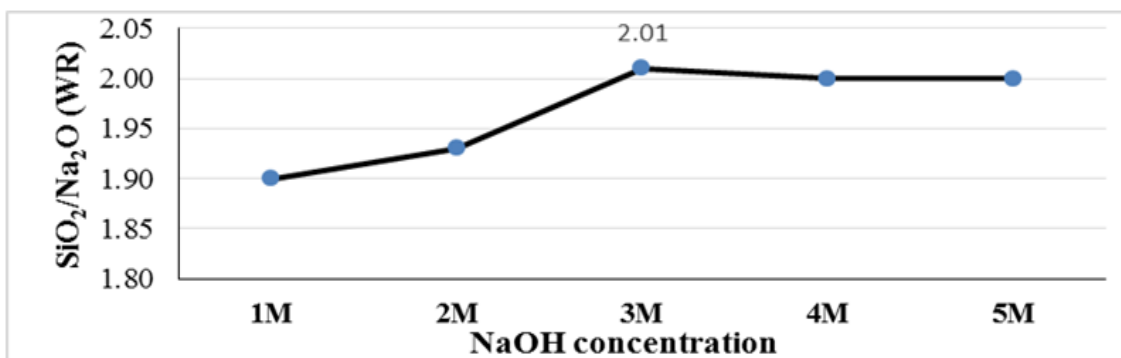


Figure 12. Effect of NaOH Concentration on SiO₂/Na₂O ratio at 90 °C for 1hrs

B. Dissolution of SiO₂ using different concentration of NaOH at 90 °C for 2hrs.

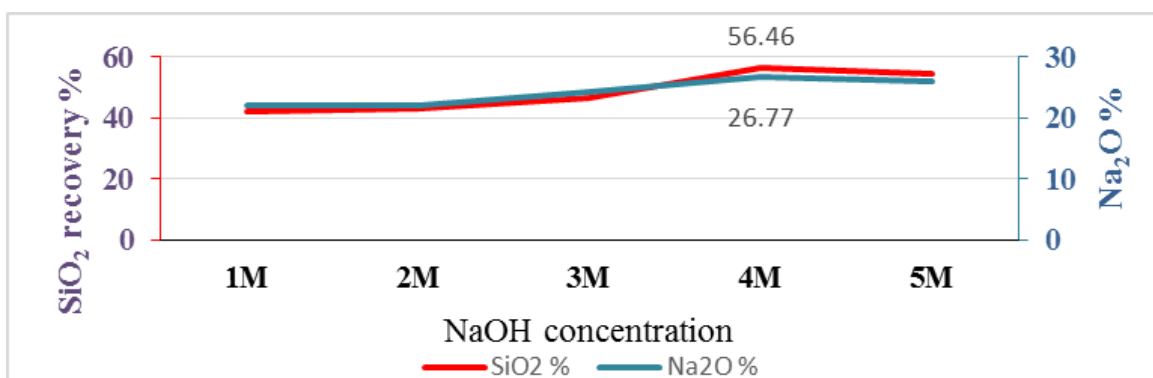


Figure 13. Effect of NaOH Concentration on SiO₂ dissolution at 90 °C for 2hrs

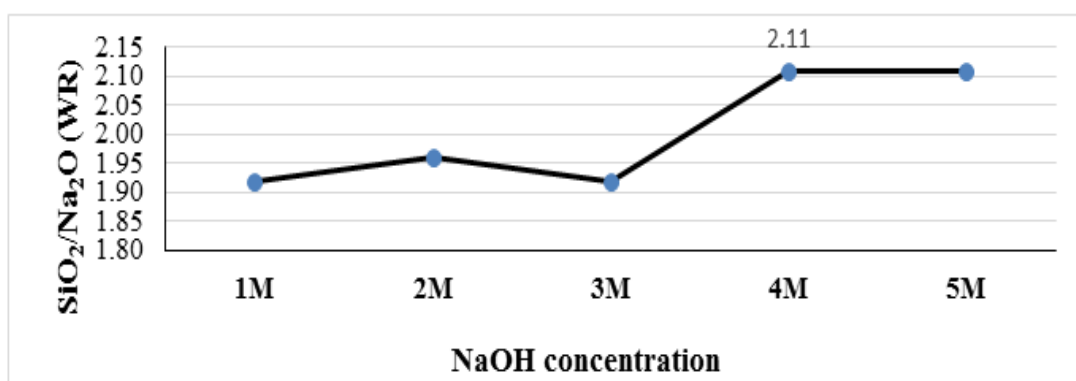


Figure 14. Effect of NaOH Concentration on SiO₂/Na₂O ratio at 90 °C for 2hrs

C. Dissolution of SiO₂ using different concentration of NaOH at 90 °C for 3hrs.

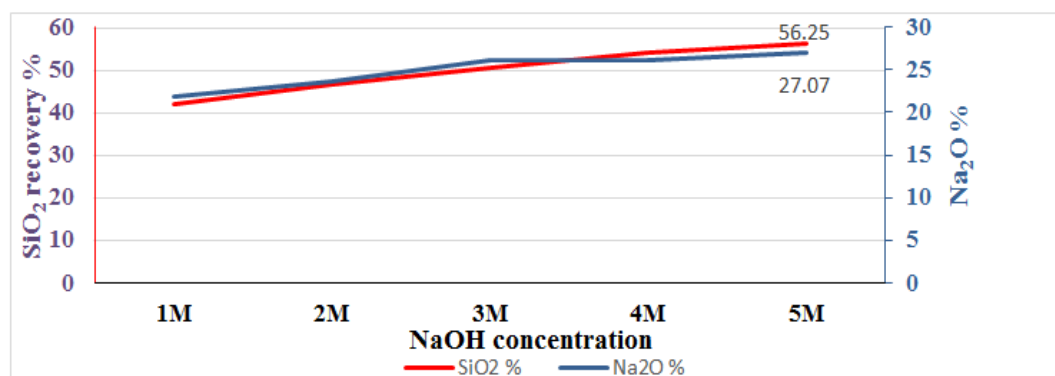


Figure 15. Effect of NaOH Concentration on SiO₂ dissolution at 90 °C for 3hrs

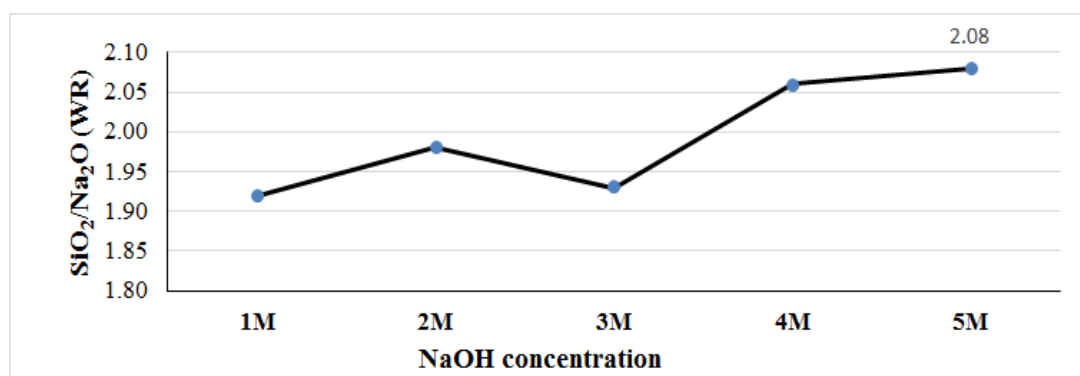


Figure 16. Effect of NaOH Concentration on SiO₂/Na₂O ratio at 90 °C for 3hrs

The results presented in Figs (11, 12, 13, 14, 15 & 16) showed that the extracted yield of SiO₂ increased with increasing concentration of NaOH from 34.24 to 55.93 at leaching for 1hrs, 42.20 to 56.46 at leaching for 2hrs and 41.85 to 56.25 at leaching for 3hrs, while the SiO₂/ Na₂O weight ratio shows a very small variation from 1.90 to 2.11. The highest ratio was achieved using 4M NaOH at leaching for 2hrs. The formation of sodium aluminum silicate is the key to decreasing the solubility of silica at 3M NaOH due to the presence of Al, causing a decrease in the SiO₂/Na₂O ratio, which increases with increasing the concentration of NaOH at 4M and 5 M (Zeng and Li, 2012).

The maximum dissolution achieved with 4M NaOH for 2hrs was effective compared to other studies such as the production of sodium silicate by dissolution of quartz sand in sodium hydroxide solutions which shows the highest extraction obtained (11%) with 7.5M NaOH at 90 °C for 3 hrs. (Febriana *et al.*, 2020), while the production of sodium silicate using the hydrothermal process in autoclave produced SiO₂/Na₂O with limited ratios of 2.5-2.7 or lower and the production of sodium silicate using fusion method of sand with Na₂CO₃ at temperature ranged from 1100 °C -1500 °C which produced solutions with ratios of SiO₂/Na₂O up to 4.0 (Weldes and Lange, 1969). Another result of sodium silicate production by dissolution of soda-lime glass and rice husk ash with sodium hydroxide produced the highest SiO₂/Na₂O ratio 2.27 and 2.45 using 4M NaOH at 200 °C (Owoeye *et al.*, 2021).

CONCLUSION

The fusion method of silica with sodium hydroxide showed an increasing in the dissolution of silica with an increasing in the weight ratio (WR) of SiO₂/Na₂O which ranged from 2.15 to 3.44. While the alkaline dissolution method shows a positive effect of sodium hydroxide concentration and time on the dissolution of silica; where the maximum dissolution of silica was achieved using 4M NaOH for 2hrs with a weight ratio (WR) of SiO₂/Na₂O nearly to 2.00. Due to the high alkalinity of the sodium silicate solutions produced in this study, they can be recommended for use in industrial applications such as soaps and detergent, xerogel, silica extraction and as a clay deflocculant. Finally, the

production of sodium silicate and sodium aluminate using alum waste (PDK) as a source of aluminum and silica aggregate value to this waste.

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إنتاج سيليكات الصوديوم وألومينات الصوديوم من مخلفات صناعة كبريتات الألومنيوم

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المستخلص

يشكل تراكم النفايات الصلبة الصناعية خطراً كبيراً على البيئة. وعليه فإن هذا البحث يدرس الاستفادة من المخلفات الناتجة من إنتاج كبريتات الألومنيوم (الشبة) باستخدام الكاولين لإنتاج ألومينات الصوديوم وسيليكات الصوديوم. حيث تم معالجة مخلفات الشبة المكسنة بالعديد من الأحماض المعدنية (حمض الكبريتيك، حمض الهيدروكلوريك، حمض النيتريك) باستخدام تركيزات (2، 4، 6، 8، و10مولر) عند درجة حرارة 90 مئوية لمدة ساعتين لتحقيق أقصى قدر من استخلاص أوكسيد الألومنيوم، ثم ترشيحه. تمت معالجة الرشيح بهيدروكسيد الصوديوم لإنتاج ألومينات الصوديوم، بينما تمت معالجة الراسب (السليكا) بهيدروكسيد الصوديوم لإنتاج سيليكات الصوديوم حيث تمت عملية الإنتاج بطريقتين مختلفتين. الطريقة الأولى تمثلت في صهر السليكا مع هيدروكسيد الصوديوم بنسبة 1:1 إلى 5:1 (سليكا: هيدروكسيد الصوديوم) عند درجة حرارة 60 مئوية يليه الذوبان في الماء عند درجة 60 مئوية ثم ترشيحه خلال ورق ترشيح 40. الطريقة الثانية تمثلت في إذابة السليكا في محاليل مختلفة التركيز من هيدروكسيد الصوديوم (1، 2، 3، 4، كمولر) عند درجة حرارة 90 مئوية بزمان متغير (1، 2، 3 ساعات). أظهرت النتائج أن الحد الأقصى لاستخلاص أوكسيد الألومنيوم تم تحقيقه باستخدام حمض الكبريتيك بتركيز 10مولر عند درجة حرارة 90 مئوية لمدة 3 ساعات والذي تم استخدامه في إنتاج ألومينات الصوديوم. كما تم إنتاج سيليكات الصوديوم بنسبة تتراوح من 2.15 إلى 3.44 لأوكسيد السليكا؛ أوكسيد الصوديوم؛ أما النسبة في الطريقة الثانية كانت تقريبا 2.

الكلمات المفتاحية: الكاولين؛ مخلفات الشبة؛ سيليكات الصوديوم؛ ألومينات الصوديوم، الترشيح الحمضي، الذوبان القلوي.