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Production Of Liquid Alum Coagulant From Local Saudi Clays

A. AL-ZAHRANI & M. H. ABDEL-MAJID

Chemical & Materials Engineering Dept., King Abdul-Aziz University P.O. Box 80204, Jeddah 21589

ABSTRACT. This study was carried out to investigate the use of local clays extremely abundant in the Kingdom for production of liquid alum. Local kaolinitic clay containing 29.4% Al_2O_3 was ground, activated by calcination and treated with sulfuric acid solution to extract alumina. In the activation step, the effects of grain size of clay, temperature and period of calcination on the extraction of alumina and iron oxide are investigated. Leaching experiments were conducted using the stoichiometric requirement of 40 wt. % H_2SO_4 for one hour under boiling conditions. Clay samples of particle size ranging from 8 to 200 mesh standard Tyler screens, calcined at different temperatures ranging from 450 to 1000 ^oC and for different periods ranging from 15 to 180 minutes were used. The results revealed that 90.9% of Al_2O_3 can be extracted when using –65 mesh clay, calcined at 700 ^oC for one hour. Experiments on the effect of acid concentration on alumina extraction from clay under the above conditions indicate that 40 wt.% acid is the recommended as it gives high extraction of alumina and at the same time results in free flowing properties of the reaction mixture. Leaching experiments were performed to investigate the effects of acid to clay ratio (50 to 120%) and leaching period (0.25 to 3 hrs) on alumina extraction. The results indicate that 88.6% of Al_2O_3 could be extracted when using 88.1% of stoichiometric requirement of 40 wt.% acid for two hours leaching period under boiling conditions.

1. Introduction

Liquid Alum is a solution of aluminum sulfate having the general chemical formula $Al_2(SO_4)_3.xH_2O$. For many years, 17% alum in solid form was easy to package and economical to ship over long distances. However, liquid alum has become more popular now because of its lower cost in handling and storage.

Several processes have been suggested for producing aluminum sulfate from clay. Most of the reported processes include calcination of clay before extracting alumina by treatment with sulfuric acid. In most reported literature, the effect of clay grinding before calcination was not paid enough attention. Some of the reported literature recommended calcination temperature of 790°C [1] or 770- 820°C [2]. Others [3] reported a recommended temperature of 550°C and mentioned that if kaolin is not heated, the solubility vanishes. It was reported [4] that kaolin is not markedly affected by previous ignition of the sample, but ignition of the mineral pyrophyelite (Al_2O_3. 4 SiO_2.H_2O) causes a marked increase in alumina extraction. It was also reported that heating at 700 – 800°C for one hour could be practiced if alumina is extracted from highly aluminous clays [5]. When sulfuric acid was added to kaolin prior to heating, the calcination temperature was about 850°C [6]. Pressure was applied after heating kaolin to 600°C to extract aluminum oxide [7].

Different concentrations of sulfuric acid were reported for extraction of alumina from calcined clays. It was reported that, in general, the extraction of alumina from calcined clays increases with increasing duration of extraction at a fixed temperature and also increases by increasing temperature at a fixed duration of extraction. Concentration of about 70 wt.% H_2SO_4 has been patented to produce aluminum sulfate [8]. When kaolin was heated to 1100 – 1200°C for one hour, then boiling calcined kaolin with sulfuric acid of concentration 5 – 7 wt.% H_2SO_4 for one hour, alumina extraction was about 80% [9]. Leaching with sulfuric acid was practiced at 90°C and 10 – 20 % acid concentration [10]. Another patent reported 30 wt. % acid concentration as the recommended concentration [11]. A temperature of 105°C has been reported [8] as a recommended extraction temperature. Another patent recommended a temperature of 70 – 90°C for 10 – 12 hours to extract 70 – 80 % of alumina [12].

Other articles were reported for the production of aluminum sulfate for different types of clays [13-20], from aluminum industry wastes [21-24] or from different industrial wastes [25-29]. The use of amines for extracting iron from aluminum sulfate leach liquor was also reported [30-31].]

In this paper, the possibility of manufacturing liquid alum from Saudi clays to satisfy local demands of coagulant aluminum sulfate which are extremely abundant in the Kingdom will be investigated. A bench scale study was carried out to investigate the activation of Saudi clay by calcination method and the leaching of alumina from the calcined clay by treatment with sulfuric acid. The effect of different variables on both activation and leaching steps is investigated to recommend the most effective conditions for both processes.

2. Materials And Methods

2.1 *Materials*

The clay samples used in this study is native kaolinitic clay obtained from Az-Zabirah area. Chemical analysis of the ground clay sample was carried out according to the standard methods [32,33]. The analysis is given in Table 1. The sulfuric acid used was BDH general purpose reagent.

Table 1. Chemical Composition of Local Kaolinitic Clay

Compound	***Natural local clay (wt.%)***
SiO_2	47.25
Al_2O_3	29.4
Fe_2O_3	2.87
TiO_2	1.17
MgO	0.35
CaO	0.59
K_2O	0.17
Na_2O	2.11
MnO	< 0.05
SO_3	< 0.05
P_2O_5	< 0.05
L.O.I.	16.02

2.2 *Experimental*

The equivalent diameter of the raw local clay sample was about 2 to 3 inches. The clay sample was ground using a ball mill to pass a sieve of a specified mesh number ranging from 8 to 200 mesh (standard Tyler screens). For each standard sieve, the ground clay sample was placed on the sieve, and then mechanically shaked for 5 minutes. The oversize was further ground followed by sieving on the same sieve. The procedures were repeated till the entire clay sample passed through the sieve.

Twenty five grams of the ground clay sample were subjected to calcination. A muffle furnace with a maximum temperature of $1200^{o}C$ was used. The heating covered a range of temperature between 450°C to 1000°C with a heating interval of 50°C. The duration of calcination was varied from 15 minutes to 180 minutes.

The calcined kaolin samples were extracted by sulfuric acid of certain concentration. A range of acid concentration from 20 to 40% by weight was studied. This range was chosen to fulfill a relatively high acid concentration and at the same time to keep the fluidity of the resulting mixture. Leaching tests were then followed using 40% weight sulfuric acid leachant to investigate the effect of the following variables on the extraction step:

i- Reaction time: the effect of reaction time was studied over a range from 15 minutes to 3 hours at boiling conditions.

ii- Acid to clay ratio: the effect of acid to clay ratio was studied over a range from 50% to 120%. The acid to clay ratio equals the quantity of acid used divided by the stoichiometric quantity of acid required.

By the end of extraction, the resulting slurry was filtered then washed. The filtrate and washings are completed to a constant volume then diluted and analyzed for aluminum and iron by inductively coupled plasma – optical emission spectrometer [34].

3. Results and Discussion

3.1 *Effect of grain size*

Figure 1 shows the effect of grain size of clay sample on the percentage aluminum and iron oxides extraction. All samples were calcined at fixed temperature of 700 °C for a fixed period of one hour. Then the calcined samples were leached by sulfuric acid of 40 wt. % concentration using the stoichiometric acid requirement for one hour at boiling conditions under total reflux. The mesh number of the clay sample was varied over a wide range between –8 and –200 standard Tyler screen mesh. Figure 1 indicates the following points:

i- A relatively large increase in alumina extraction from 76.5 to 89.9% is obtained when the grain size is varied from –8 to 35 mesh.

ii- A comparatively slight increase in the % extraction of alumina is observed between the successive meshes –35 and –48 mesh.

iii- Nearly no significant change in the extraction of alumina is observed between –48 and –200 mesh.

iv- The percent extraction of iron oxide between –8 and –35 mesh lies between 40.8 and 47.8%. After –35 mesh, the effect of reducing the size of the clay sample on percent extraction is negligible.

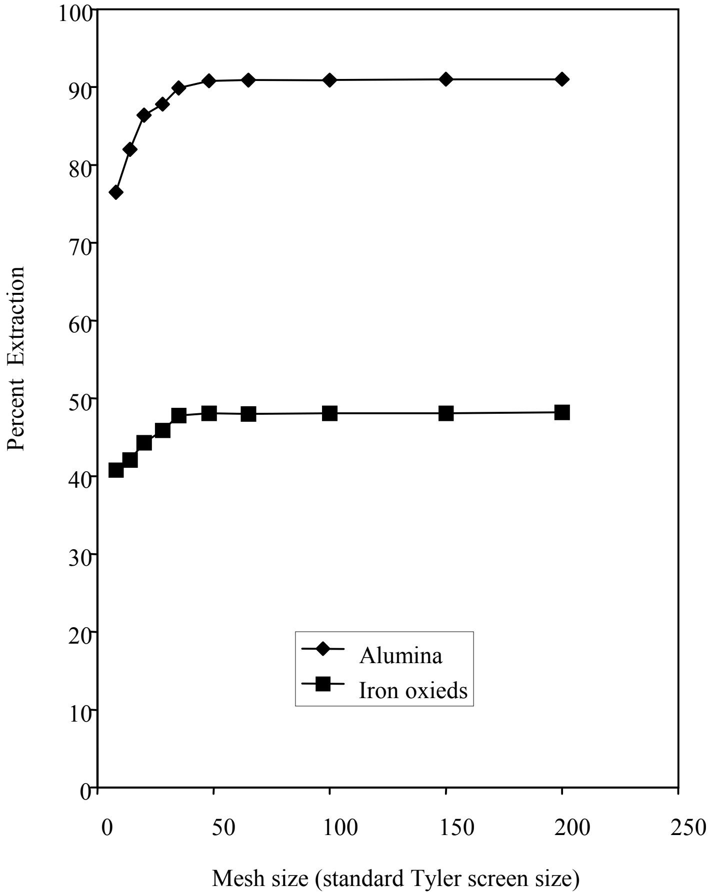

Figure1. Effect of grain size on percent extraction. Calcination temp. 700 ^{o}C for 1 hr. leaching with 40% by wt. H_2SO_4 at boiling conditions and a grain size of mesh no. –65 (standard Tyler screen mesh).

These results indicate that grinding the clay samples beyond –48 mesh has no significant effect on the extraction of alumina from local clay sample. This indicates that the grain size of -48 to-52 mesh is quite suitable for grain size to be used. This will also benefit in reducing excessive grinding cost. A grain size of –65 mesh was selected for further work.

3.2 *Effect of Temperature and Time of Calcination*

The effect of varying duration and temperature of calcination on the percent extraction of alumina and iron oxide is indicated in Tables 2 and 3 and Fig. 2. The data indicate the following points:

Table 2. Effect of Temperature and Time of Calcination on Al_2O_3 Extraction. Mesh no.: -65, Standard Tyler Screen; Concentration: 40 wt.% Sulfuric Acid; Extraction: at Boiling Temperature and Time of Extraction: 1hr.

Calcination temperature (°C)	450	500	550	600	650	700	750	800	850	900	950	1000
Calcination Time (hr)	% Extraction of Al_2O_3											
¼	17.5	70.9	78.8	82.2	84	84.5	84.5	84.4	82.6	25.1	10.9	5.6
½	22.7	75.2	83	87.8	88.1	88.2	88.1	88.1	84.8	25.3	11	5.6
1	26.8	78.2	84.9	88.3	90.3	90.9	91.1	89.7	85.1	25.1	11	5.5
2	27	78.1	84.9	88.2	90.4	90.9	91.1	89.7	85	25.2	10.9	5.5
3	26.9	78.2	84.8	88.3	90.4	91	91	89.8	84.8	25.2	11	5.5

Table 3. Effect of Temperature and Time of Calcination on Fe_2O_3 Extraction. Mesh no.: -65, Standard Tyler Screen; Concentration: 40% wt. Sulfuric Acid; Extraction: at Boiling temperature and Time of Extraction: 1hr.

Calcnation Temperature (°C)	450	500	550	600	650	700	750	800	850	900	950	1000
Calcination Time (hr)	% Extraction of F_2O_3											
¼	7.5	26.2	30.1	31.4	31.5	31.4	31.6	31.6	24.4	4.8	1.1	1
½	9.1	34.1	36.8	37.9	38	37.9	37.9	35	30.1	4.7	1.1	1.1
1	10.2	40.8	46.5	47.9	48.1	48	47.2	44.3	35.6	4.7	1.1	1
2	10.3	40.8	46.6	47.9	48	48.1	47.9	45.1	35.8	4.8	1.1	1
3	10.3	40.7	46.6	48	48	47.9	48.1	45	35.6	4.6	1.1	1.1

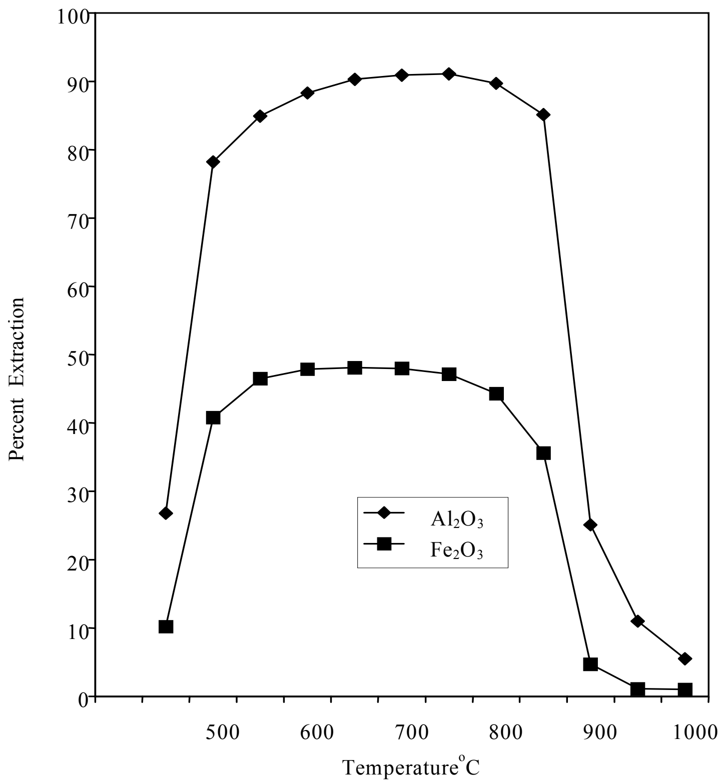

Figure 2. Effect of calcination temperature on percent extraction Calcination time of hr, leaching with 40% by wt. H2SO4 at boiling conditions and a grain size of mesh no. –65 (standard Tyler screen mesh).

i. An inert zone below 450 °C, during which the percent extraction of alumina is small.

ii. A promising zone from 500 °C to 750 °C during which percent extraction of alumina increases. The percent extraction decreases sharply when the temperature exceeds 800 °C.

iii. The highest alumina extraction (91.1%) is reached at a calcination temperature of 750 °C and for a duration time of one hour as can be seen from Table (1). However the increase in percentage of alumina extraction when temperature increases from 700 to 750 °C is only 0.2 %. This slight increase does not justify the corresponding increase in energy requirement. Therefore, a temperature of 700 °C is recommended for further experimental work.

3.3 *Effect of Acid Concentration*

The effect of acid concentration on the extraction of alumina is shown in Fig. 3, for –65 mesh clay calcined at 700 °C for one hour and extracted with the stoichiometric amount of sulfuric acid under boiling conditions. The alumina extraction increases moderately as the acid concentration increases. An acid concentration of 20% by weight corresponds to 88.6 % extraction while 40% concentration results in 90.9 % extraction and 60% concentration corresponds to 92.9 % extraction. The corresponding values for iron oxide extraction are 50.3, 48.0 and 55.7 %, respectively.

The difference in % alumina extraction between 40% and 60% acid concentration is not large. However at 60% acid concentration, the reaction mixture became pasty. During the course of the study of the time of the reaction, it was noted that when using 40% acid concentration and an acid to clay ratios below 100%, the reaction mixture was just free flowing. This makes it undesirable to increase the acid concentration beyond the 40% concentration.

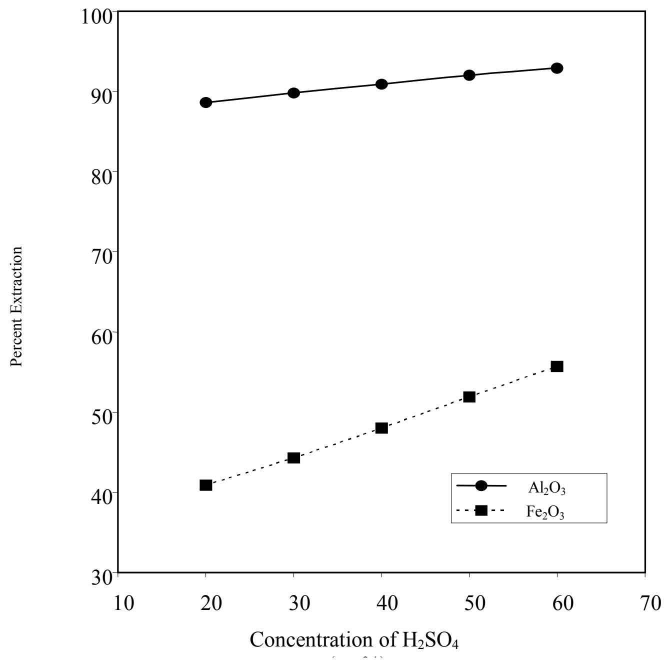

Figure 3. Effect of acid concentration on extraction of Al_2O_3 & Fe_2O_3. Grain size: -65 mesh standard Tyler screen and calcination temperature at 700 °C for 1 hr.

3.4 ***Effect of Time of Extraction***

The effect of extraction time on the yield of alumina extracted from –65 mesh clay calcined at 700 ^oC for one hour using 40 wt.% sulfuric acid under boiling conditions and different acid to clay ratios is shown in Figure (4). The figure shows that for an acid to clay ratio greater than 80%, the percent extraction of alumina from the clay is almost constant after two hours. Therefore, a reaction time of two hours is recommended. The corresponding results for iron oxide are shown in Figure (5)

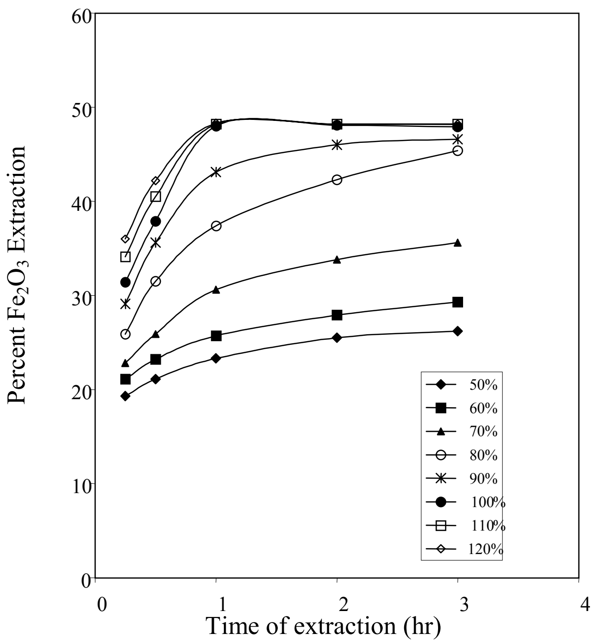

Figure 4. Effect of extraction time on % Fe_2O_3 extraction at different acid to clay ratios. Grain size: -65 mesh standard Tyler screens, calcination at 700 ^oC.

3.5 ***Effect of Acid to Clay Ratio***

The results of the effect of cid to clay ratio on –65 mesh clay calcined to 700 ^oC for one hour and extracted at the boiling conditions for one hour by using 40% concentrated acid are given in Figure (6). The data shows a large increase in the percentage extraction of aluminum oxide from 63.6% when half the stoichiometric quantity of the acid is used to 91.8% when 120% of the stoichiometric quantity is used. Corresponding values for extraction of iron oxide are 23.3% and 48.8%, respectively as shown in Figure (7).

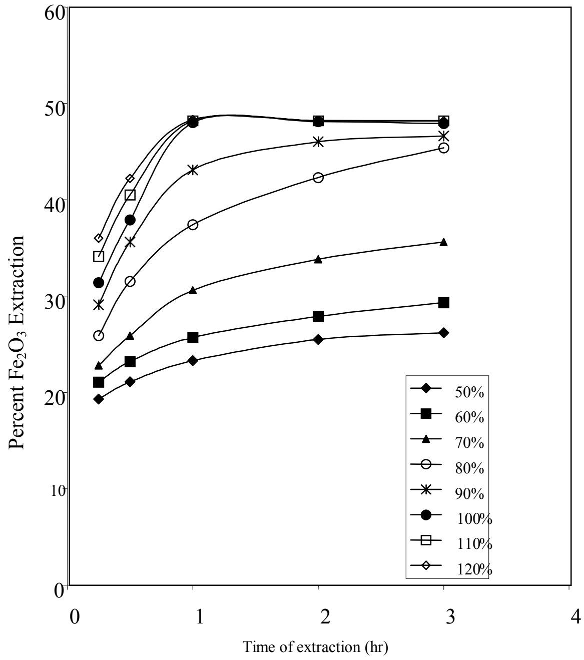

Figure 5. Effect of extraction time on % Fe_2O_3 extraction at different acid to clay ratios. Grain size: -65 mesh standard Tyler screens, calcination at 700 ^{o} C for 1 hr and leaching with 40% by wt. H_2SO_4 at boiling conditions.

Figure (8) shows a comparison between the theoretical and actual percent extractions for Al_2O_3 and Fe_2O_3. Acid to clay ratio above 120% does not appear to give any sign of increase in the yield of alumina. The point of intersection between the straight line, representing the theoretical conditions and the experimental curve, corresponds to 88.1% acid to clay ratio and 88.6% alumina extraction, which means complete reaction of the acid added with alumina in the clay. Acid to clay ratio above 88.1% results in the presence of free unreacted acid. Acid to clay ratio below 88.1% shows higher values than those expected theoretically which indicates that such solutions of extracted aluminum sulfate contain dissolved alumina. Analysis of the liquor obtained after filtration gives 17% Al_2O_3 content whereas analysis of combined filtrate and washing gives 8.1% Al_2O_3, which is within the commercial requirements for liquid alum to avoid crystallization of aluminum sulfate and therefore no concentration step needed.

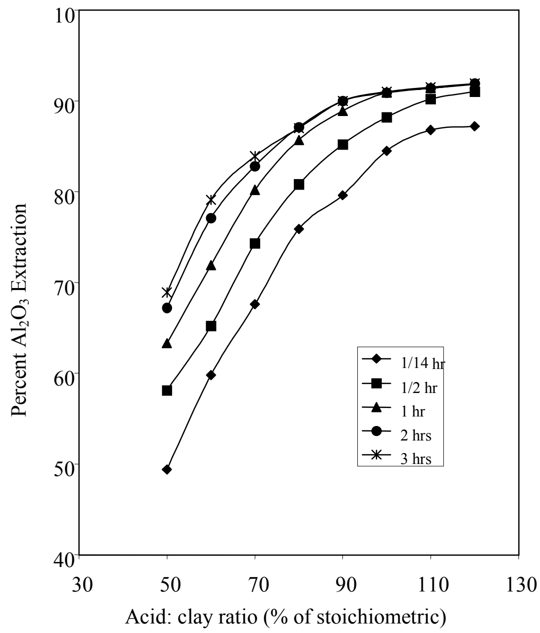

Figure 6. Effect of acid to clay ratio on extraction of Al_2O_3 at different leaching periods. Grain size: -65 mesh standard Tyler screens, calcination at 700 ^{o} C for 1 hr and leaching with 40% by wt. H_2SO_4 at boiling conditions.

3.6 *Effect of Temperature of Extraction*

The study of the optimum conditions of the temperature of extraction is limited by the need for conducting the extraction under boiling conditions to hydrolyze and therefore precipitates any dissolved titanium salts. Extraction of alumina from calcined clay at temperature below the boiling temperature results in rendering titanium impurities present in the clay soluble in solution. However, at the boiling conditions, soluble titanium sulfate is hydrolyzed to insoluble form and therefore prevent contamination with the product.

4. CONCLUSIONS

The optimum conditions for production of aluminum sulfate for Saudi clay are: grinding the clay to pass 65 mesh sieve (standard Tyler screens), calcination of the ground clay at 700 ^oC for one hour, extraction of alumina from calcined clay by leaching with 40% by weight sulfuric acid, using 88.1% of stoichiometric acid to clay ratio and extraction at boiling conditions for two hours

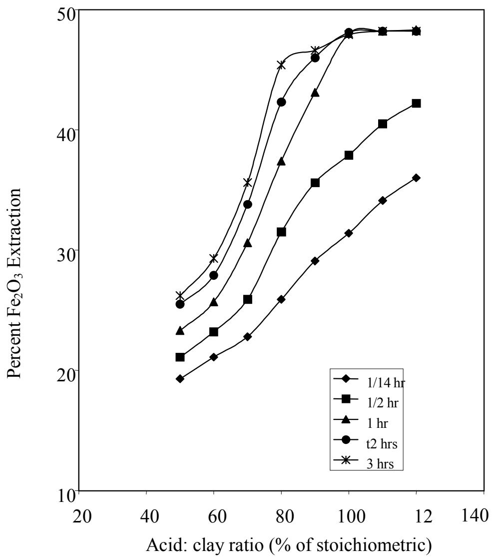

Figure 7. Effect of acid to clay ratio on extraction of Fe_2O_3 at different leaching periods. Grain size: -65 mesh standard Tyler screens, calcination at 700 ^{o} C for 1 hr and leaching with 40% by wt. H_2SO_4 at boiling conditions.

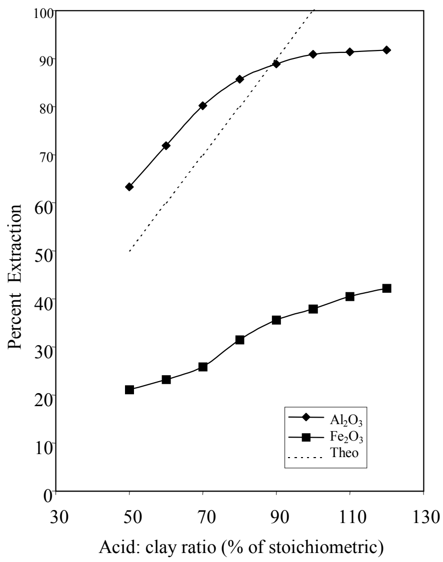

Figure 8. Effect of acid to clay ratio on extraction of Al_2O_3 & Fe_2O_3 .
Grain size: -65 mesh standard Tyler screens, calcination at 700 ^{o} C for 1 hr and leaching with 40% by wt. H_2SO_4 at boiling conditions.

Leaching the Saudi clay at the above conditions will result in 89.5 % extraction of the alumina content in the clay. The product liquid alum contains 8.1% Al_2O_3 which corresponds to about 23.4% $Al_2(SO_4)_3$.

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إنتاج مروب الشبه السائلة من الطفلة السعودية

عبد الرحيم بن أحمد الزهراني و محمد حسين عبد المجيد

قسم الهندسة الكيميائية وهندسة المواد - كلية الهندسة - جامعة الملك عبد العزيز

ص.ب. ٨٠٢٠٤ جدة ٢١٥٨٩

المستخلص: تم تنفيذ هذا البحث لدراسة استخدام الطفلة المحلية المنتشر تواجدها في المملكة العربية السعودية في إنتاج الشبه السائلة. وقد تم إجراء البحث باستخدام طفلة محلية تحتوى على ٢٩,٤% أكسيد ألومنيوم بعد طحنها وتنشيطها بالكلسنة ثم معالجة الطفلة المنشطة بحامض الكبريتيك لاستخلاص الألومنيا . وبالنسبة لخطوة التنشيط فقد تم دراسة تأثير كلا من حجم الحبيبات ودرجة حرارة وزمن الكلسنة على درجة استخلاص الألومنيا وأكسيد الحديد وأوضحت تجارب الاستخلاص والتي تمت باستخدام الكمية النظرية من حامض الكبريتيك تركيز ٤٠% عند درجة الغليان لمدة ساعة باستخدام طفلة مطحونة ما بين ٨ إلى ٢٠٠ مش نظام تايلر للمناخل، مع تغيير درجة حرارة الكلسنة من ٤٥٠ إلى ٩٠٠ درجة مئوية وتغيير زمن الكلسنة من ١٥ – ١٨٠ دقيقة انه يمكن استخلاص ٩٠,٩% من الألومنيا باستخدام طفلة لها حجم حبيبات – ٦٥ مش منشطة بالتسخين عند درجة ٧٠٠ مئوية لمدة ساعة ٠ وأوضحت التجارب التي أجريت حول تأثير تركيز حامض الكبريتيك على درجة استخلاص الألومنيا باستخدام الكمية النظرية من الحامض والاستخلاص عند درجة الغليان أن تركيز ٤٠% هو التركيز المفضل الذي يحقق نسبة عالية من استخلاص الألومنيا ويعطى خليط تفاعل ذو خواص سريان مناسبة ٠ وبالنسبة لخطوة الاستخلاص فقد أوضحت التجارب التي تم إجرائها لتعيين تأثير تغيير نسبة الحامض إلي الطفلة من ٥٠-١٢٠% من الكمية النظرية وتغيير زمن الاستخلاص من ٠,٢٥ إلى ٣ ساعات . إنه يمكن استخلاص ٨٨.%

من الألومنيا باستخدام ٨٨,١% فقط من الكمية النظرية من الحامض
تركيز ٤٠% وزمن استخلاص ساعتين .