Study of alumina prepared from Iraqi kaolin

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Abstract

The current research was conducted to report the synthesis of alumina powder from Iraqi kaolin. The kaolin was transformed to metakaolin by calcinations at temperature 800 °C for three hours. Then the calcined kaolin was treated with (1.5 M) from H_2SO_4 for 6 hours to form $Al_2(SO_4)_3.12H_2O$ solution. The precipitate was dried at 80^oC for 10 hours and calcinations at different temperatures for two hours. The samples which result was characterized by X–Ray diffraction (XRD) and X–Ray fluorescence (XRF). The results indicate to the crystalline hydrate aluminum sulfate for the sample that be as – synthesis and when calcinations at 600 ^oC transformed into aluminum sulfate phase. The phases of alumina which we obtain consisted of a gamma alumina phase which getting at calcinations 1000 ^oC and an alpha alumina phase at calcinations 1300 ^oC.

The optimum conditions for preparation alumina from Iraqi kaolin is at reaction time 6 hours, particle size 75μm and concentration acid is (1.5 M) where was the highest percentage of extraction alumina is 98.8%. The effect of calcinations temperature on the rate of extracted alumina was studying in this research and found that the alumina ratio was extracted increasing with increase the calcinations temperature.

Key words

Alumina, Iraqi kaolin, sulfuric acid, X – Ray diffraction.

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دراسة الالومينا المحضرة من الكاؤلين العراقي

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الخلاصة

تهدف الدراسة الحالية إلى عملية تحضير مسحوق الالومينا من اطيان الكاؤلين العراقي. تم تحويل الكاؤلين الى ميتا كاؤلين بواسطة التحميص عند 800 ^oC لمدة 3 ساعات. تم معالجة الكاؤلين المكلسن بحامض الكبريتيك في تركيز 1.5 M لمدة 6 ساعات للحصول على محلول كبريتات الالمنيوم المائية. تم تجفيف الراسب عند 80 ^oC لمدة 10 ساعات ومن ثم تحميصه عند درجات حرارة مختلفة لمدة ساعتين. تم تشخيص العينات الناتجة بواسطة انحراف الاشعة السينية و تقنية تألق الاشعة السنية، حيث تشير النتائج الى بلورة كبريتات الالمنيوم المائية للعينة المحضرة وعند التحميص الى 600 ^oC تتحول الى طور كبريتات الالمنيوم. ان مراحل الالومينا التي نحصل عليها تشمل طور كاما الومينا عند التحميص الى 1000 ^oC و طور الفا الومينا عند التحميص الى 1300 ^oC.

الظروف المثلى لتحضير الالومينا من الكاؤلين العراقي هي عند زمن تفاعل 6 ساعات وحجم حبيبي 75 μm وتركيز حامض 1.5 M حيث كانت اعلى نسبة استخلاص للالومينا هي 98.8 %. في هذا البحث تم دراسة تأثير درجة حرارة التحميص على نسبة استخلاص الالومينا وقد وجد ان نسبة الاستخلاص تزداد بزيادة درجة حرارة التحميص.

Introduction

Aluminum oxide (Al_2O_3), which is called alumina consider from one of the most important oxide materials in industry because of their excellent physical and chemical properties. Alumina is a crystalline powder, white color, low cost, density (3.9 g/cm^3). Its melting point of (2055 ± 6^{o}C), does not dissolve in water or acid and is used as itchy as it comes after the diamond in hardness. Furthermore, it is a good insulator of electricity. Its energy gap (Eg > 8eV) [1- 4]. The importance of Alumina is two–fold; it is used as a starting material for the smelting aluminum metal, and used as a raw material for abroad range of advanced ceramic products and as an active agent in chemical processing [5]. Alumina have a wide range of the transfers stages such as γ , δ, θ, η, κ, χ and α phases [6]. Alumina is subject to a different of transitions these transformations ends to form alpha alumina at high temperatures which is considered the most stable situation [7]. Industrially, about more than 45 million tons of Al_2O_3 are produced in the world, which are mainly manufactured by the bayer method using bauxite, and about 40 million tons are consumed for refining aluminum [8].

Kaolin, which is described as the earthy rock and characterized with white color and contains a large quantity of minerals, and this rock has a wide range of lithology also, these rocks are characterized as chemically inert and has a wide range of pH [9, 10]. The theory formula for kaolinite is $Si_2Al_2O_5(OH)_4$ and also there are other formulas are $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and $Al_2O_7Si_2 \cdot 2H_2O$, which has a molecular weight of 258.071 g/mol [11, 12]. Contains the kaolin on a high proportion of the aluminum oxide estimated about 39.8% therefore is considered suitable replacement for bauxite which is used to extraction alumina with purity high. Therefore a result of these high percentage for aluminum oxide in the kaolin, it is used to produce alumina [13, 14]. The installation of kaolinite is a 1:1 layer clay consists of a layer repeated from an alumina octahedral plate and a silica tetrahedron plate. Interlayer hydroxyl groups extend from the octahedral sheet into the interlayer zone which it is formed hydrogen links to basic of the oxygen for the opposing silica plate which is be tetrahedron [15]. The chemical analysis shows that different ions may be replaced in the installation, for instance Al^{+3} for Si^{+4} in the silica tetrahedral layer, and Mg^{+2} or Fe^{+2} for Al^{+3} in the alumina octahedral layer [16].

Material used

1. Iraqi kaolin extracted from Duwaikhla was used as a starting material.
2. Ethanol, used in process of precipitate.
3. Sulfuric acid was used to dissolution the kaolin to attain a solid\liquid, which have the density is 1.84 g\mol concentration assay is 95% and molecular weight is 98.08.
4. Distil water, was used to prepare (1.5M) from acid.

Experimental method

Iraqi kaolin extracted from Duwaikhla Mine was used as a starting material. Kaolin powder calcined even 800℃ for period three hours in an electrical furnace. After cooling in room temperature, the kaolin grinding by milling machine then sieving in sieve 75μm by sieve cheaker machine. Mix 8.3 g from kaolin powder and 276.6 ml from sulfuric acid with a concentration (1.5M) in a 1000 ml round phial and mixed by electric engine at the speed of 500 r.p.m for period 6 hours at 80^{o}C. Then leave the

mixture to cool down into chamber heat after that filtered to remove the residue whose mostly consist from silica, where obtain on leach liquor from aluminum sulfate. 152 ml from the filtered leach liquor added drop wise into 684 ml from ethanol at a rate 5ml\min where stirred with magnetic stirrer by magnetic bar then filtered and the precipitate separated was washed by using ethanol after that dries the precipitate separate at 80 ℃ for 10 hours. Calcinations the precipitate at various temperatures for period two hours in an electric furnace, finally we obtain alumina powder.

Results and discussions

Typically x-ray is an electromagnetic radiation having a wave length of 1 Å in between ultraviolet and gamma-rays. This non-destructive analytical technique is quite useful for studying chemical composition, crystal structures and their phases, size, symmetry of the unit cell, lattice constants of nanoparticles and physical properties of grown materials. It is important to point out that more than 90% solid materials are crystalline in nature [17]. Fig. 1 (a, b, c, d) showing x–Ray diffraction patterns results for the synthesized precipitate materials. The sharp peaks in the XRD patterns in the figure corresponding to the crystalline hydrate aluminum sulfate [$Al_2(SO_4)_3.12H_2O$]. When sample calcinations temperature at 600 ^oC, the powder of hydrate aluminum sulfate was dehydrated to aluminum sulfate $Al_2(SO_4)_3$. At high calcinations heat degrees, [$Al_2(SO_4)_3.12H_2O$] fully change into aluminum oxide as a result of molecules water removal, after that is removed of brimstone tri- oxide gas; these interactions are appeared during the equations below [18, 19].

$$Al_2(SO_4)_3.12H_2O\ (s) \longrightarrow Al_2(SO_4)_3\ (s) + 12H_2O\ (g)$$

$$Al_2(SO_4)_3\ (s) \longrightarrow Al_2O_3\ (s) + SO_3\ (g)$$

However, the XRD peaks for sample calcined at 1000 ^oC for period two hours are ascribed to the decomposition of aluminum sulfate into gamma alumina (γ–Al_2O_3), where believed that the structure of gamma alumina is spinel cubic deformation [20]. While the results appears the presence of alpha alumina (α–Al_2O_3) in the sample at calcinations temperature 1300 ^oC. In relation to the lattice parameters, it was observed that the gamma alumina (γ–Al_2O_3) affected by changes resulting from heat degree, while the alpha alumina (α–Al_2O_3) that do not show any effect as a result of thermal stability until at high heat degrees. The effect of calcinations temperature on the rate of extraction alumina was studying in this research by using technique of XRF and found that the alumina ratio was extracted increasing with increase in calcinations temperature where be high proportion extracted getting is 98.8 % as shown in Table 1.

Table 1: Calcinations temperature effect on the ratio of alumina extraction according to results X –Ray fluorescence (XRF).

Group No.	Sample No.	Calcinations temperature ^{o}C	Time of Reaction (hour)	SiO_2 %	Extracted Al_2O_3 %
S_1	E_o	As- synthesized	6	1.2	28.9
	E_1	600	6	0.9	40.3
	E_2	1000	6	-	92.6
	E_3	1300	6	-	98.8

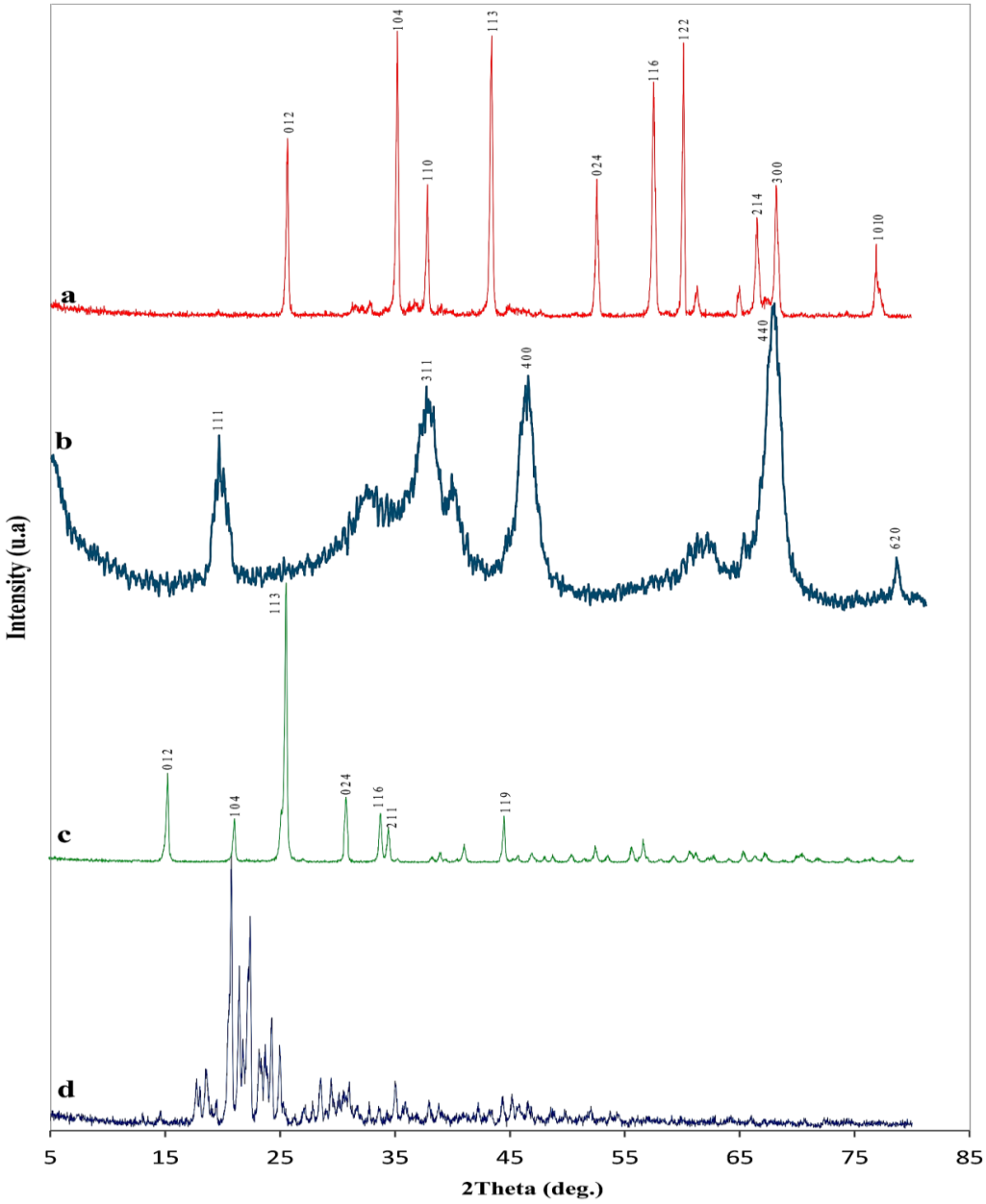

Fig. 1: XRD Pattern of group S_1, samples E_n. a. Alpha Alumina at 1300 ^{o}C. b. Gamma Alumina at 1000 ^{o}C. c. Aluminum Sulfate at 600 ^{o}C. d. Hydrate Aluminum Sulfate As-Synthesized.

Conclusions

The alumina powder (Al_2O_3) were successfully prepared by alumina extraction processes from Iraqi kaolin through mixed Iraqi kaolin which calcined at 800 ^oC for three hours with sulfuric acid (H_2SO_4). The highest proportion extraction of alumina is 98.8 where increasing the ratio of extraction with increase calcinations temperature.

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