STRAIGHT SYNTHESIS OF α AND γ ALUMINA FROM KAOLIN BY HCl ACID LEACHING

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Abstract

In this work the preparation of alumina from kaolinite by leaching with hydrochloric acid is studied. Crude kaolin was calcined at 850°C and then leached with 6 M hydrochloric acid at 90°C. Aluminum hydroxide was precipitated by adding 6 M HCl solution to the leaching water. Finally, the precipitated aluminum hydroxide was heated at 1100°C and converted to aluminum oxide. The materials were identified by XRF, XRD and SEM-EDX techniques. The percentage of alumina recovery was determined at different kaolin particle sizes (–200 to –400 standard Tyler sieve), different times (1, 2, 3 and 4 h), different leaching temperatures (80, 90, 100 and 104°C) and different concentrations of hydrochloric acid (20, 25, 30 and 37%). The phase changes of alumina were also investigated at two calcination temperatures (1100 and 1200°C). The purity of aluminum oxide obtained by this method is about 97% and the percentage of recovery is about 98%. It was observed that the kaolin granulation with dimensions smaller than 75 µm had no significant effect on the percentage of alumina recovery. It was also found that the percentage of alumina recovery increased with increasing leaching temperature.

Keywords: Alumina; Kaolin; Acid leaching; Hydrometallurgy; Hydrochloric acid

1. Introduction

Aluminum oxide (Al_2O_3) has a wide variety of technological and industrial applications. Depending on the synthesis route, the primary source of production and calcination temperature, it has different intermediate phases. Some of these phases are gamma (γ), delta (δ), kappa (κ), and theta (θ) alumina. All these intermediate phases have special industrial applications [1]. Thermodynamically, the most stable and abundant form of aluminum oxide is alpha alumina (α-Al_2O_3), which is formed at about 1050 to 1200°C [2]. Alpha alumina is used in industry as a catalyst substrate in automotive, oil, aerospace structural composites, thermal abrasion resistant coatings, catalyst supports, high-strength materials such as cutting tools, and in the manufacture of electronic devices [3, 4].

The γ-alumina (γ-Al_2O_3) phase is formed in the temperature range of 450 to 800°C and is used as a catalyst, catalyst substrate in automotive and petroleum industries, structural composites for spacecraft, and abrasive and thermal wear resistant coatings [2, 5]. Other states such as (χ), (η), (κ), (δ), and (θ) exist only transiently and are not used as a stable phase [2].

Bayer process is currently the most common method of alumina production in the world. This process is based on the leaching of crushed bauxite in alkaline solutions. After leaching, the aluminum hydroxide is separated from the aluminate solution and then calcined to obtain alumina. Only high quality bauxites with a mass ratio of Al_2O_3 to SiO_2 of more than 6 can be used as raw material for the production of alumina by the Bayer process [6, 7]. Therefore, the sources of high quality bauxite in the world are decreasing continuously [8]. On the other hand, the increasing global demand for aluminum has led to an increase in interest in the development of alternative methods for the production of alumina from low-grade minerals. In this regard, extensive research and engineering efforts have been made to extract alumina from various non-bauxite mineral resources, which are abundant in around the world [9-11].

Some examples of non-bauxite raw minerals are alunite [12], sillimanite [13], kaolinite [14-19], fly ash [20, 21], coal gangue [22], red-mud [23, 24], oil shale [25], anorthosite [26], andalusite, mica, kyanite, and local clays [27, 28].

Alumina synthesis has been reported from various chemical routes, some of which include hydrolysis [29], sol–gel [30], hydrothermal process [31], leaching process [32, 33], thermal treatment [34], and microwave roasting [35].

Among all non-bauxite raw materials, clay minerals which are mainly composed of aluminosilicate minerals with traces of iron oxide and alkali metal oxides are more abundant and inexpensive [36]. Due to the high content of aluminum in its structure (26-20%), kaolinite is an attractive candidate for alumina production and an alternative source of bauxite. Kaolinite with the chemical composition of $Al_2Si_2O_5(OH)_4$ is a layered silicate clay mineral [37, 38], and mainly contains quartz, mica (10-45%), and iron oxide (1-10%) as contaminants [11].

In general, the leaching procedure is commonly used in mineral extraction due to its low cost, low energy consumption, and the ability to leach low-grade ores. Three types of leaching procedures have been considered for the recovery of alumina from mineral clay. The sulfurization method, which is sintering clay with ammonium sulfate and recovering alumina by leaching in hot water [39]. The alkali roasting method which is sintering clay with lime or soda ash and recovering alumina and silica through leaching in hot water [40]. In the acidic method, a variety of acids such as sulfuric, hydrochloric or nitric acids are used for the selective dissolution of alumina [41-44].

The expansion of a procedure for the acidic extraction of alumina from mineral clay was first offered by Hoffman *et al* [41]. The method involves several steps including the calcination of mineral clay, leaching the calcined product, filtering and separating the insoluble dealuminated material, making sedimentation the aluminum hydrochlorides, and finally calcining the hydrated chlorides to gain alumina [41].

From an industrial standpoint, one of the most promising processes for the extraction of alumina from mineral ores with high silica content is the leaching process by hydrochloric acid. [41].

Extraction of alumina from kaolin was performed by Hulbert and Huff [45] through leaching method using nitric, sulfuric, and hydrochloric acids. They found that the leaching rate of alumina decreased from hydrochloric to sulfuric and to nitric acid, respectively [45]. In general, the rate of alumina extraction from calcined kaolin under acidic leaching methods with hydrochloric acid depends on a range of variables including kaolin particle size, temperature and duration of kaolin calcination, acid concentration, and solid/liquid ratio as well as leaching temperature [46].

The aim of this work is to evaluate the feasibility of recovering high purity alumina by dissolution calcined kaolin in hydrochloric acid. The effect of kaolin granulation, leaching temperature and duration, acid concentration, and alumina calcination temperature on the recovery percentage and purity of alumina were also investigated.

2. Experimental
2.1. Materials

A sample of kaolin clay from the northeastern deposits of Iran was used as raw material. The chemical composition of kaolin is given in Table 1. Concentrated hydrochloric acid (37 wt%, Merck) and sodium hydroxide pellets (Merck) were used with analytical grade and without further purification.

Kaolin stone was ground by a jaw crusher to reduce the particles size to 1-2 in. The crushed samples were then milled using a planetary ball mill for 1 h to get dimensions smaller than 75 μm. Four samples of kaolin with dimensions of (53 to 74 μm), (44 to 53 μm), (37 to 44 μm), and (smaller than 37 μm) were selected for next steps.

2.2. Characterization

Phase analysis and chemical characterization of the samples were carried out by X-ray diffraction (XRD) and X-ray Fluorescence (XRF) techniques. Shimadzu X-ray diffractometer (MAXima XRD-7000) and Shimadzu X-ray Fluorescence (Lab Center XRF-1800) instruments were used for these purposes. The morphology of the samples was observed by Scanning Electron Microscope (Leo VP 1450 (Germany)).

2.3. Leaching and synthesis procedure

Kaolin was calcined in a muffle furnace at 850°C for 3 h and subsequently cooled in the furnace to room temperature resulting in the transformation of kaolin into metakaolin. Hydrochloric acid (37 wt%) was used as a leaching agent. 10 g metakaolin was added to 40 ml of the 6 M hydrochloric acid and leached for 3 h at 90°C under stirring at 500 rpm (solid/liquid ratio = 1:4 g/mL). The chemical equation is as follow:

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O + 6HCl \rightarrow 2AlCl_3 + 2SiO_2 \downarrow + 5H_2O \quad (1)$$
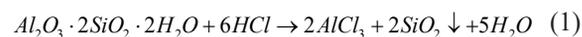
The slurry was filtrated and the residue was rinsed with hot distilled water (90°C). The leachate was heated to near boiling point (90°C) and then 7.5M NaOH solution was added to convert alumina-based compound to $NaAlO_2$ according to Equation 2.

**Table 1.** *The chemical composition of kaolin clay*

Oxide	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	Na_2O	MgO	K_2O	MnO	CaO	SO_3	LOI
Wt/%	47.14	33.76	2.27	1.38	0.15	0.15	0.15	0.01	0.22	0.03	14.65

$$AlCl_3 + NaOH + H_2O \rightarrow NaAlO_2 + 3HCl \qquad (2)$$

In the next step, 6 M HCl was added to the $NaAlO_2$ filtrate with stirring until the pH was adjusted to 7. The precipitated aluminum hydroxide (Equation 3) was filtered and then washed with distilled water and dried in an oven at 200°C for 2 h.

$$NaAlO_2 + HCl + H_2O \rightarrow Al(OH)_3 \downarrow + NaCl \qquad (3)$$
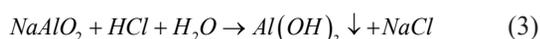
The resulted aluminum hydroxide was calcined for 2 h in a muffle furnace at 1100°C and converted to alumina (Al_2O_3). The synthesis procedure is summarized in Figure 1.

## 3. Results and discussion
3.1. Calcination of metakaolin

The chemical composition of kaolin was evaluated by XRF spectroscopy. As shown in Table 1, kaolin is mostly composed of silica (SiO_2) and alumina (Al_2O_3), while other oxides such as Fe_2O_3 and TiO_2 are found to some extent, and the ignition loss is about 14%. The loss on ignition could be related to the removal of organic matter or some non-metals such as sulfur, which could exist as SO_2 [47, 48]. The XRD analysis was performed on kaolin and metakaolin samples and it was found that kaolin sample showed all the characteristic peaks of kaolinite (Figure 2). During kaolin calcination process, these peaks disappear and turn into irregular band of an amorphous metakaolin. Kaolin clays undergo a series of phase changes when heated in the air [49]. In general, clay matters have three kinds of water

molecules in their structure. The physisorbed and interlayer water that is loosely bound and can be removed under heating at temperature below 200°C [11]. The water molecules in the first coordination of the interlayer ions have strong bonds and need a higher temperature. Endothermic dehydroxylation/dehydration which removes these water molecules and produces irregular metakaolin ($Al_2Si_2O_7$) starts at 550-600°C, but gradual oxolation of metakaolin occurs at 800 to 900°C and is associated with persistent loss of hydroxyl (-OH) groups [50].

In this work, the kaolin samples were calcined for 3 h at 850°C to ensure the complete transformation to active metakaolin. This was confirmed by the XRD analysis of the calcined sample which showed no peaks of kaolinite. According to equation 4, during the calcination process, the kaolin structure is reduced and two water molecules are released:

$$Al_2Si_2O_5(OH)_4(Kaolin) \rightarrow$$
$$Al_2Si_2O_7(Metakaolin) + 2H_2O \qquad (4)$$
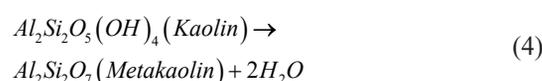
3.2. Effect of particle size of calcined kaolin on alumina extraction

Four kaolin samples in the particle size range of (53 to 74 μm), (44 to 53 μm), (37 to 44 μm), and (smaller than 37 μm) were used to measure the effect of granulation on the percentage of aluminum oxide extraction. After 3 h of calcination at 850°C, the samples were leached by concentrated hydrochloric acid (12 M) for 3 h at 90°C. Figure 3 shows the effect of particle size of kaolin on the percentage of
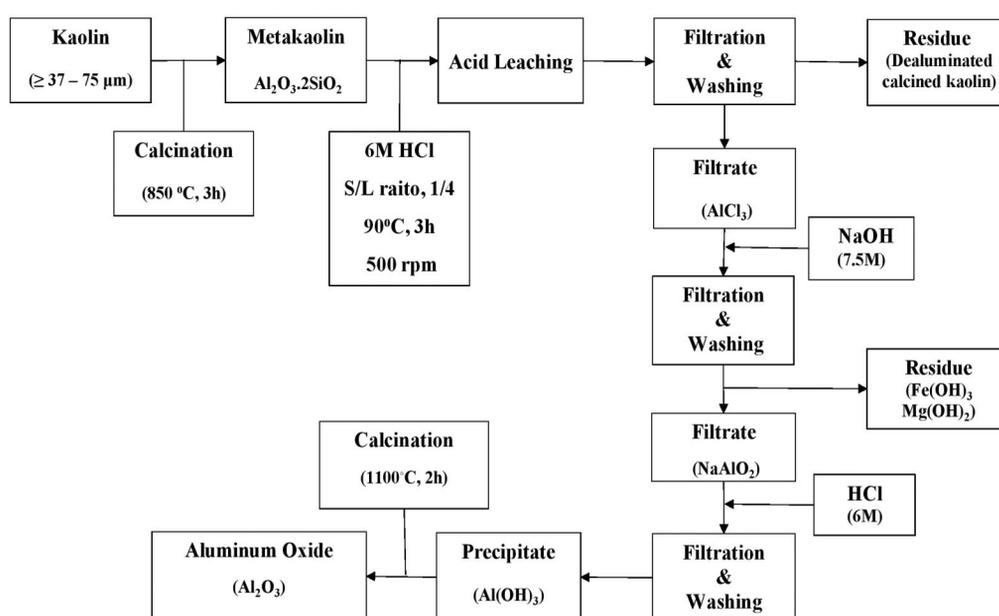

Figure 1. Flowchart of the proposed alumina synthesis process

aluminum oxide extraction. The results showed that the particle size parameter with dimensions less than 75 μm had no significant effect on the extraction percentage of aluminum oxides. Al-Zahrani *et al.* [51] also reported that grinding kaolin samples beyond 65-mesh (smaller than 250 μm) has no effect on the extraction of alumina from local clay samples. Therefore, a combination of all 4 granulations was selected for the subsequent studies.

3.3. Effect of acid concentration on alumina extraction

The effect of different concentrations of

hydrochloric acid on the percentage of alumina extraction is shown in Figure 4.

It is observed that by increasing the concentration of acid and thus decreasing the pH of the reaction, the extraction rate of aluminum oxide increases. The maximum extraction efficiency of aluminum oxide was recorded at about 99%, which was obtained by the most concentrated hydrochloric acid (12 M). Similar results were reported by Al-Zahrani *et al.* [51] and Alaa *et al* [15]. Since the recovery percentage of alumina using 6 M hydrochloric acid was about 97%, from the economic point of view, 6 M hydrochloric acid was selected for the next studies.
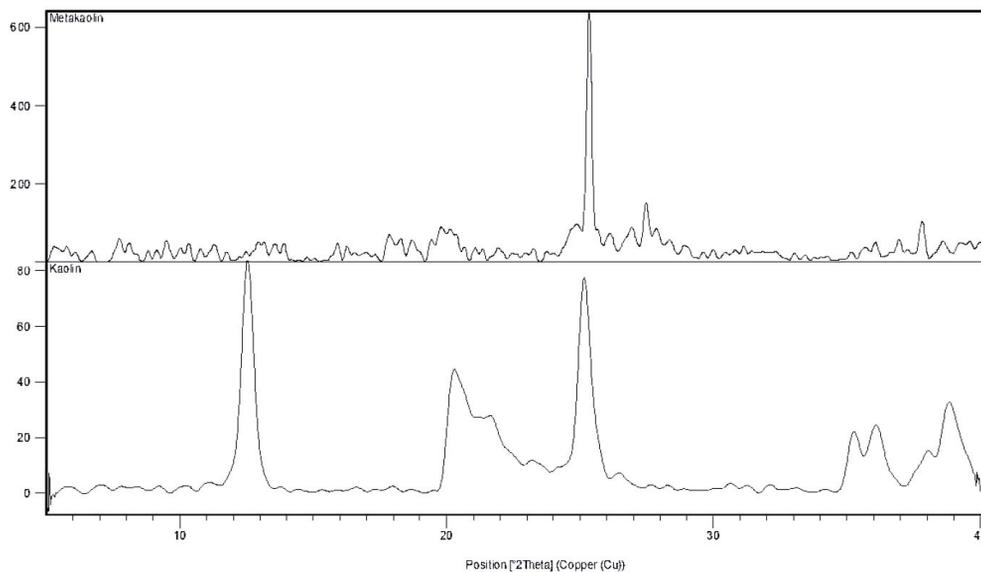

Figure 2. *XRD pattern of kaolin and metakaolin*
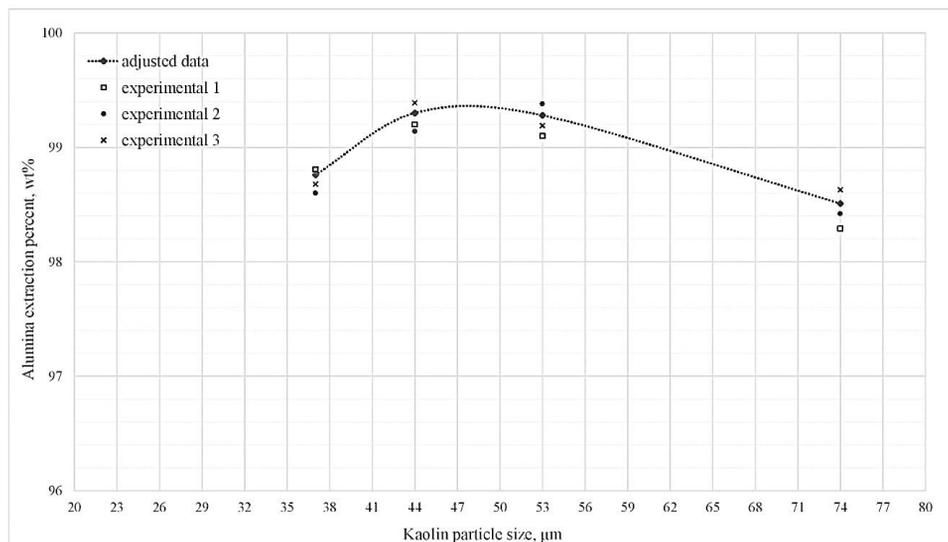

Figure 3. *The effect of calcined kaolin particle size on alumina extraction by acid leaching process. Condition of experiment (solid/liquid ratio of 0.25 g/ml, leaching time: 3 h, leaching temperature: 90°c, Stirring rate = 500 rpm)*
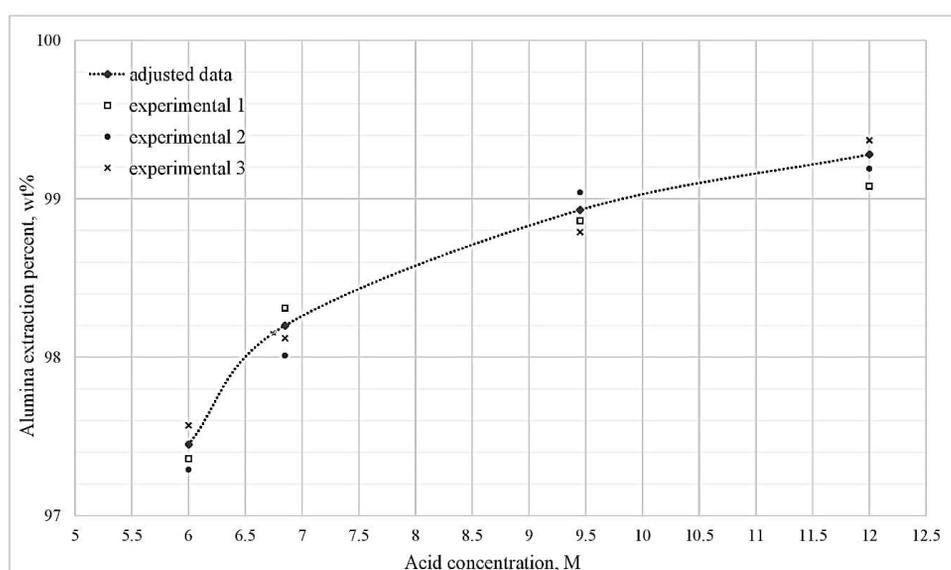
3.4. Effect of leaching time and temperature on alumina extraction

To investigate the effect of leaching time on the extraction of aluminum oxide, 10 g of calcined kaolin was leached at different leaching times of 60 to 240 min with 6 M hydrochloric acid at 90°C. During these experiments, the solid-to-acid ratio was kept constant (1:4). Figure 5 shows the effect of different leaching times (60-240 min) on the percentage of aluminum oxide extraction.

According to the results, with the increase of leaching time up to 120 min, the percentage of alumina extraction increases rapidly. The same observation was made by Wang et al. [18] and Aly et al. [6]. As the leaching time increases (up to 120 min), the number of acid molecules that react with the calcined kaolin particles increases, and then (from 120 to 240 min) the alumina extraction proceeds at a slower rate. This can be attributed to the slight evaporation of the acid, which is unavoidable as the leaching time increases from 3 h at high temperatures. Therefore, it is better for the leaching time not to be more than 3 h. The same observation was made by Tantawy et al. [39]. As reported by Al-Zahrani et al. [51] and Aly et al. [6], the extraction percentage of alumina decreases by reducing the acid-to-clay ratio.

To study the effect of leaching temperature on the percentage of aluminum oxide extraction, 10 g of calcined kaolin was leached with 40 ml of

Figure 4. The effect of acid concentration on alumina extraction. Condition of experiment (Particle diameter: (74–37 ≥ μm), solid/liquid ratio of 0.25 g/ml, leaching time: 3 h, leaching temperature: 90°c, Stirring rate = 500 rpm)
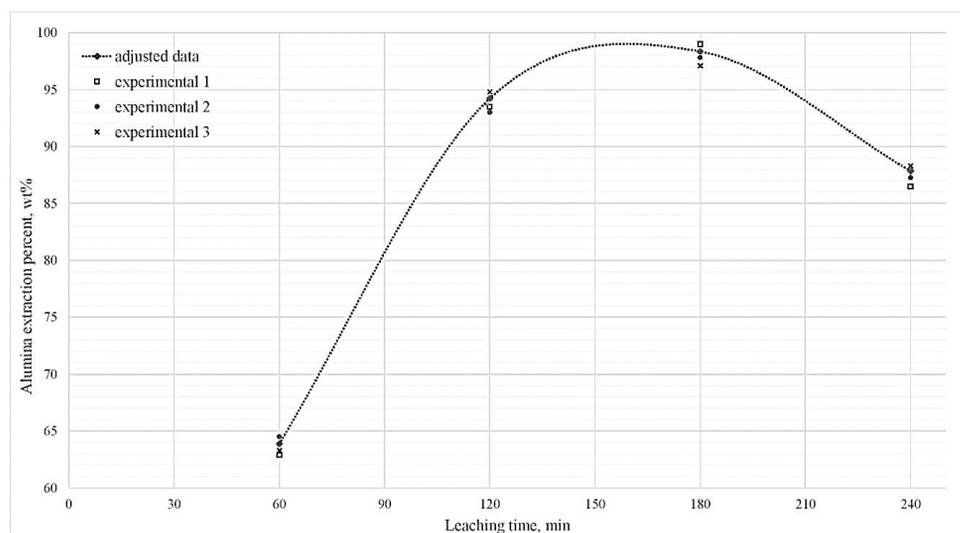
Figure 5. The effect of leaching time on alumina extraction. Condition of experiment (Particle diameter: (74–37 ≥ μm), acid concentration = 6 M, solid/liquid ratio of 0.25 g/ml, leaching temperature: 90°c, Stirring rate = 500 rpm)

hydrochloric acid (6 M) for three hours at four different temperatures (80, 90, 100, and 105°C).

The results show that the percentage of alumina extraction increases by rising (elevating) the leaching temperature, which presented a similar trend reported by Aly *et al.* [6]. The highest rate of alumina recovery is about 99% which is recorded at the highest leaching temperature (105°C). Figure 6 shows the percentage of aluminum oxide extracted from kaolin at different leaching temperatures. As shown in Figure 6, the percentage of alumina obtained at a temperature of 90°C is equal to (96%), which is higher than the extraction of alumina (75%) observed by Tantawy *et al* [39].

### 3.5. Effect of calcination temperature on alumina phase changes

Calcination of alumina particles was performed at two different temperatures of 1100°C and 1200°C for 2 h. Different calcination temperatures were selected to observe the phase transformations of the alumina. XRD analysis of the calcined sample at 1100°C confirmed the formation of stable γ-alumina (Figure 7). Close similarities were observed between the XRD patterns of kaolin-derived gamma alumina and commercial γ-alumina produced from bauxite. The standard characteristic peaks of γ-alumina ((311), (400), and (440)) in the XRD patterns of commercial γ-alumina and kaolin-derived alumina gamma are the same.

The contained impurities in the γ-alumina are Na_2O (1.8 wt%), along with traces of SO_3 (0.15%), K_2O (0.1%), CaO (0.04%), Fe_2O_3 (0.03%), TiO_2 (0.02%), and MnO (0.01 %). γ-alumina, due to its high specific surface area and good thermal stability (up to 1030°C), is mostly used as a catalyst or catalyst support [2, 5]. According to Belver *et al.* [52], the thermal stability of γ-alumina can be improved by adding some materials including CaO, TiO_2 and SiO_2. Therefore, it can be concluded that the presence of such impurities in the synthesized γ-alumina will improve its thermal stability [14, 49]. But it should be noted that although Si is more effective than other elements in improving the thermal stability of γ-alumina, it significantly delays the conversion process of γ-alumina to α-alumina [49].
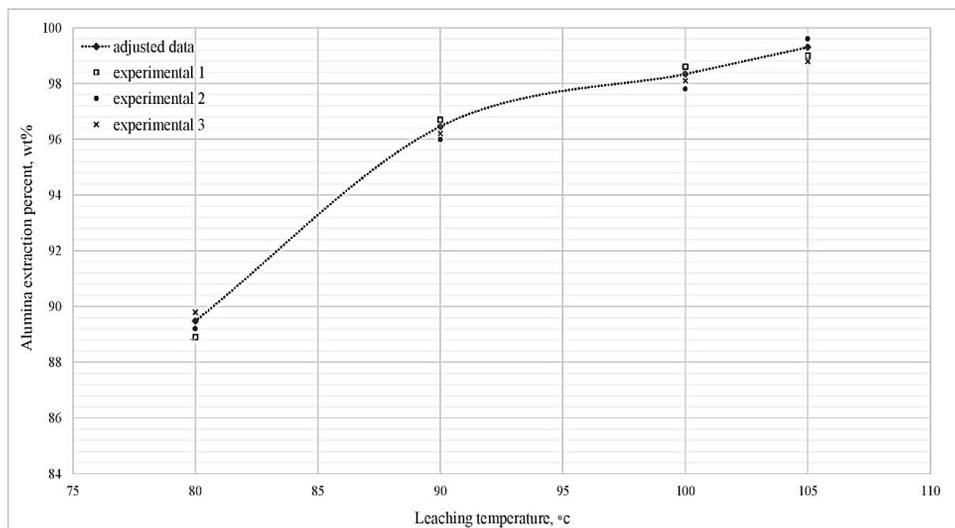
Figure 6. The effect of leaching temperature on alumina extraction. Condition of experiment (Particle diameter: (74–37 ≥ μm), acid concentration = 6 M, solid/liquid ratio of 0.25 g/ml, leaching time: 3 h, Stirring rate = 500 rpm)
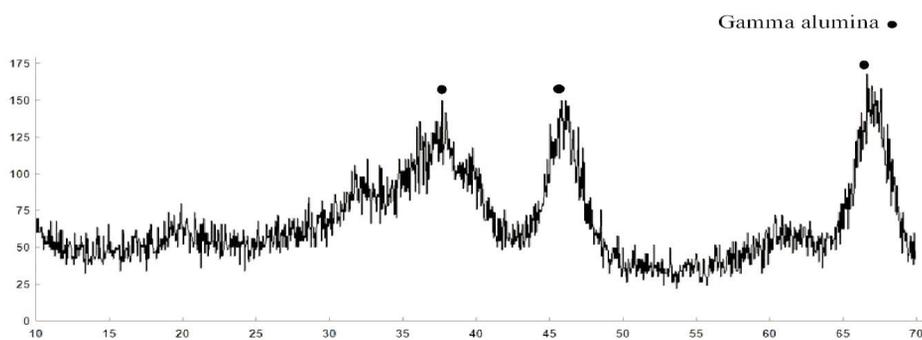
Figure 7. XRD pattern of extracted gamma-alumina

The morphology of the obtained γ-alumina was examined by SEM-EDX analysis (Figure 8). SEM micrographs show that the synthesized alumina particles have irregular and non-uniform shapes with sharp edges. Most particles are less than 10 μm in size. The grains size appears to be similar to those synthesized by Ibrahim *et al.* [14], but they are bigger than the grains synthesized by Hosseini *et al.* [49].

After 2 h of calcination at 1200°C, the alumina samples are completely converted to α-alumina. As shown in Figure 9, the XRD patterns confirm the formation of stable α-alumina phase with characteristic peaks of (012), (104), (113) and (116).

4. Conclusions

Alpha and gamma alumina particles were successfully prepared from kaolin deposits in Iran by calcination and leaching. The optimum extraction
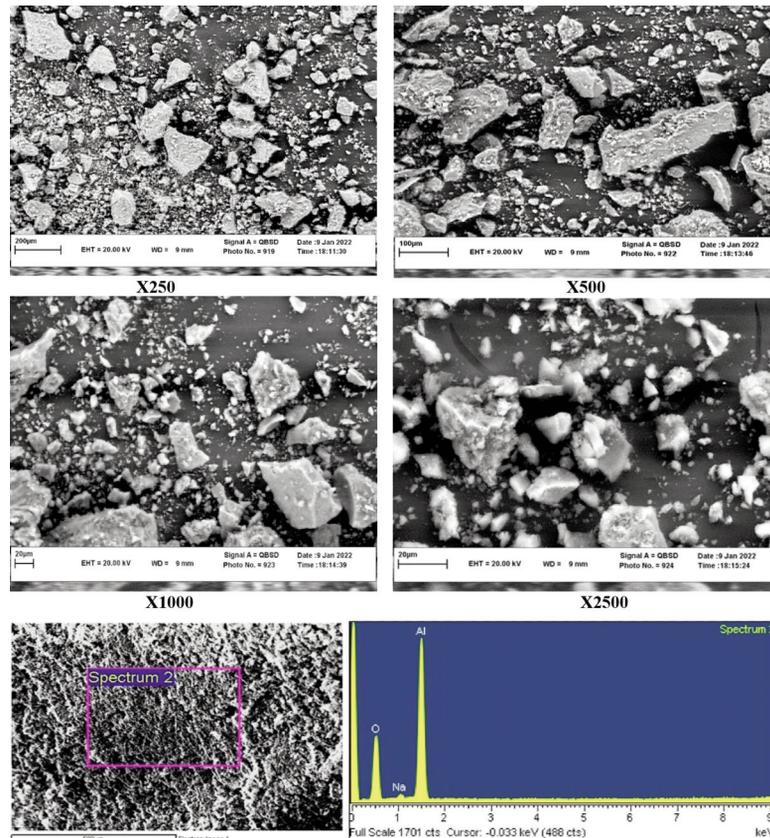

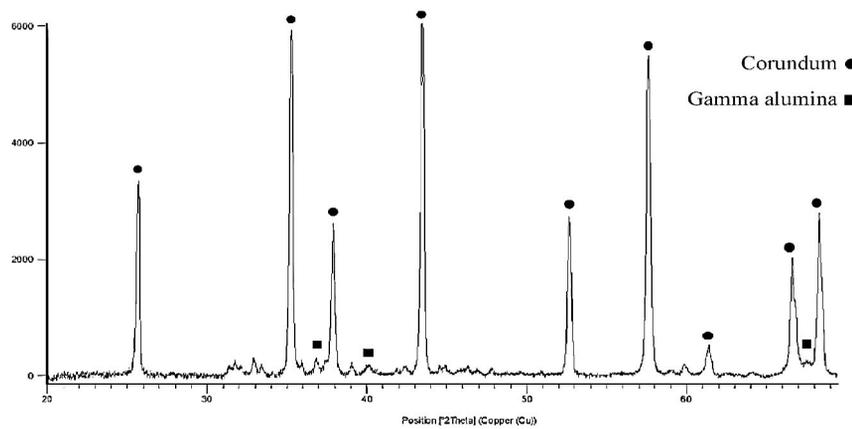*Figure 8.* SEM-EDX images of synthesized gamma alumina

Figure 9. XRD pattern of extracted alpha alumina

method involves grinding kaolin clay, then calcining for 3 h at 850°C and finally leaching with hydrochloric acid (6 M) with a solid to liquid ratio of 0.25 for 3 h at 90°C. The alumina produced by this method has a high purity of about 97%. The percentage recovery of alumina from calcined kaolin under the conditions studied was about 98%.

Author's contributions

Miss Bagherzadeh is MSc Student of Metallurgy, and Dr M.H. Golmakani is supervisor and Dr E.Z. Karimi is advisor in this investigation.

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IZRAVNA SINTEZA α I γ GLINICE LUŽENJEM HCL KISELINOM

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Apstrakt

U ovom radu proučava se priprema glinice iz kaolinita postupkom luženja hlorovodoničnom kiselinom. Sirovi kaolin je kalcinisan na 850°C, a zatim lužen 6 M hlorovodoničnom kiselinom na 90°C. Aluminijum hidroksid je taložen dodavanjem 6 M HCl rastvora vodi za luženje. Na kraju, precipitirani aluminijum hidroksid je zagrevan na 1100°C i pretvoren u aluminijum oksid. Materijali su identifikovani pomoću XRF, XRD i SEM-EDX tehnika. Procenat dobijanja glinice je određen pri različitim veličinama čestica kaolina (-200 do -400 standardne Tyler sito mreže), različitim vremenima (1, 2, 3 i 4 sata), različitim temperaturama luženja (80, 90, 100 i 104°C) i različitim koncentracijama hlorovodonične kiseline (20, 25, 30 i 37%). Takođe su proučene fazne promene aluminijum oksida pri dve temperature kalcinacije (1100 i 1200°C). Čistoća aluminijum oksida dobijenog ovom metodom iznosi oko 97%, a procenat dobijanja je oko 98%. Utvrđeno je da granulacija kaolina sa veličinom manjom od 75 μm nema značajan uticaj na procenat dobijanja glinice. Takođe je utvrđeno da procenat dobijanja glinice raste sa povećanjem temperature luženja.

Ključne reči: *Glinica; Kaolin; Kiselo luženje; Hidrometalurgija; Hlorovodonična kiselina*