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Extraction of Aluminum and Iron from Boiler Slag by Sulfuric Acid

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Abstract: This paper presents a new method of recycling aluminum and iron in boiler slag derived from plants that use coal as fuel. The new method integrates efficient extraction and reuse of the leached pellets together. An elemental analysis of aqueous solutions leached by sulfuric acid was determined by EDTA- $\text{Na}_2\text{-ZnCl}_2$ titration method. The components and microstructures of the samples were examined by means of XRF, XRD and SEM. An aluminum extraction efficiency of 86.50% was achieved when the sintered pellets were leached using $4 \text{ mol} \cdot \text{L}^{-1} \text{ H}_2\text{SO}_4$ at solid/liquid [m(g)/V(mL)] ratio of 1 : 5 at 80°C for 24 h. An iron extraction efficiency of 94.60% was achieved in the same conditions for the maximum extraction efficiency of Al. The extraction efficiencies of Al and Fe increased with an increase in temperature, leaching time and acidity. The concentration of alumina and iron hydroxide in the final product was determined to be 99.12% and 92.20% respectively. This product of alumina would be used directly for the production of metallic aluminum.

Key words: boiler slag; Al extraction efficiency; Fe extraction efficiency; leaching; alumina

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0 Introduction

With the utilization of boiling furnace in plants using coal as fuel, boiler slag increases rapidly. The quantity of boiler slag produced in China is about 0.12-0.16 billion ton each year. Vast quantities of boiler slag have accumulated over the recent years. So much boiler slag causes many serious environmental problems^[1]. On the other hand, there is a growing concern for an imminent shortage of aluminum from domestic sources in China. Although leaching processes for the recovery of aluminum from coal fly ash(CFA) or clay have been developed a long time ago^[2], there have been few studies of the extraction aluminum from boiler slag. This produces the concept of recovering aluminum and some other metals from boiler slag.

Sulfuric acid is often used as a leachant for recovery of metals from ores, particularly using the direct acid leach(DAL) process. The DAL process is a most powerful method to reduce the metal content of whole ores, since low pH destabilizes many inorganic components. Nitric acid and hydrochloric acid leaching processes for the recovery of aluminum and other metals have been developed a long time ago. However, these processes found to be of little practical application due to the highly corrosive nature of concentrated chloride or nitrate solutions. In addition, hydrochloric acid and nitrate acid are expensive lixivants in terms of acid cost and large evaporative losses make these processes highly uneconomical. In contrast, sulfuric acid is cheaper than nitric acid or hydrochloric acid and can be obtained easily. There is sufficient industrial wastewater that contains sulfuric acid from chemical plants and can be recycled in

China^[3]. So, it is viable, from an economical point of view, to use sulfuric acid as a lixiviant for the leaching processes, especially for industrial scale processes.

Recovery of aluminum from boiler slag, CFA or residue, is based on the application of hydrometallurgical processes such as acid or base leaching, precipitation, solvent extraction, crystallization and re-crystallization^[4,5]. Leaching processes for recovery of aluminum and other metals from CFA can be described in the framework of heterogeneous non-catalytic reactions in conjunction with the shrinking core model. The shrinking core model assumes that the reaction products, and/or inert matter that remain in the solid phase, from a layer of ash that encapsulates the unreacted core. The process of aluminum leaching from CFA by sulfuric acid is considered elsewhere A. Seidel^[6]. The degree of aluminum recovery was found to decrease with an increase in the content of CFA in the leaching medium. It was found that the leaching process involved in a self-inhibition mechanism owing to precipitation of calcium sulfate on the surface of the CFA particles.

Boiler slag from plants using coal as fuel in China typically contains 40%-60% SiO₂, 18%-38% Al₂O₃, 2%-7% Fe₂O₃, 1%-4% CaO, 0.5%-3.0% MgO and 0.5%-2.0% TiO₂ (*m/m*, dried solid) according to literatures reported^[7,8]. The content of alumina is rich, so extraction of aluminum from this boiler slag is a valuable work.

1 Experimental

1.1 Materials

The boiler slag used in this test came from Baotou Steel Plant, Inner Mongolia, China. The microstructure of the boiler slag was shown in Fig.1. A 50 kg sample of the boiler slag was collected for the test. The particle size distribution (PSD) and components of the sample were measured. The PSD was derived by physically screening the samples using standard screens. Sulfuric acid was purchased from market, which was produced in Tianjin. The washed coal came from a power plant near Wuhan city, Hubei province, China. The lime was purchased from market of Wuhan city, Hubei province, China.

1.2 Methods

In the proposed process, the boiler slag was pelletised together with washed coal and lime prior to sintering in order to decompose the mullite phase in the boiler slag. The boiler slag was pelletised with washed coal and lime at a ratio of 50 : 40 : 10 (*m/m/m* in all tests) and

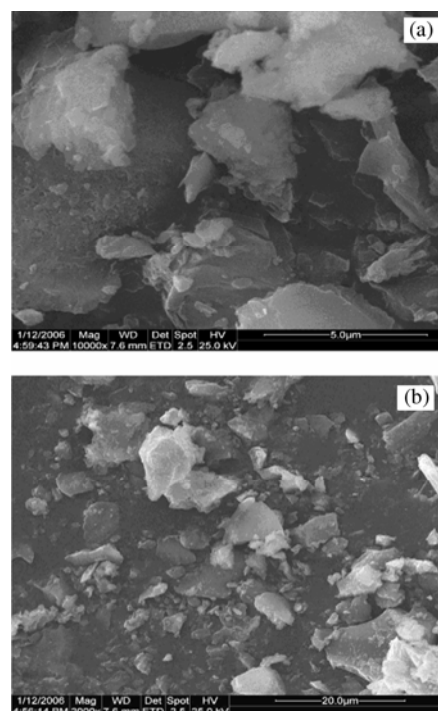


Fig. 1 SEM of boiler slag before leaching

(a) $\times 10\,000$

(b) $\times 2\,000$

some other different ratios, 40 : 40 : 20, 60 : 30 : 10, 70 : 20 : 10, to produce pellets ($D \approx 8$ mm) with a strong mechanical strength. The pellet size of washed coal is less than 1 mm. The washed coal and water mixed with the ratio of 4 : 1 (*m/m*) and the mixture was pelletised to form 5-10 mm diameter spherical pellets. The pellets of boiler slag mixtures were placed into a silicon crucible when the pellets were dried, which were then put into a muffle furnace (The type of the muffle furnace is XL-3-10, which was made in Wuhan, China), and then was sintered at 800-1 200 °C for different periods (in the range of 30-180 min) to produce sintered pellets for the leaching test. In the leaching process, sintered pellets were added into a 1 000 mL Pyrex beaker. H₂SO₄ solution was carefully transferred to the Pyrex beaker. The concentrations of sulfuric acid were 2, 4 and 6 mol · L⁻¹ respectively. The temperature was 20-95 °C and the leaching time was 2-24 h. Solubilization was conducted into by a 1 : 5 solid/liquid ratio (*m/V* in all tests) in all tests. The leached residues were filtrated from the leach liquor. A water-scrubbing must be done to remove the residual leach liquor absorbed by the pellets. The resulted solution after scrubbing was subsequently combined with a leach liquor to produce the final leach liquor. A solution of sodium hydroxide was added to the leach liquor in order to make precipitation of Fe(III) ions. A brown metal hydroxide precipitate was readily formed at

a pH of 3.0. The solution was then filtered to remove all of the insoluble metal hydroxide precipitate. The liquor from above filtration was prepared for precipitation of aluminum.

In order to remove some other metals, such as Ti, Mn, Mg, sufficient sodium hydroxide solution ($10 \text{ mol} \cdot \text{L}^{-1}$ NaOH) was added to the leach liquor in order to reach a pH value of 13.0. Following the addition of sodium hydroxide to the leach liquor, red metal hydroxide precipitate was readily formed. The precipitate formed was removed from the solution by filtration. The precipitate was then washed with distilled water to remove the residual filtrate containing sodium hydroxide and sodium aluminate solution that was absorbed by the metal precipitate during precipitation.

The filtrate recovered from the precipitation at pH 13.0. This filtrate was combined with scrubbing solution. The solution obtained in this way was then bubbled with carbon dioxide gas until a white gelatinous precipitate was formed. In order to remove all of the insoluble precipitates from the solution, it was filtered off. The precipitate was thoroughly washed with distilled water in order to remove residual sodium carbonate solution that had been absorbed during the precipitation of metal hydroxides from the sodium aluminate solution. The washed metal precipitate was dried in an oven at $105 \text{ }^\circ\text{C}$ for 2 h to remove the absorbed free water. The white precipitate obtained after drying was calcined at $1200 \text{ }^\circ\text{C}$ for 5 h. Finally, the products of alumina and iron hydroxide were determined by XRF (X-ray fluorescence) fusion analysis.

The boiler slag was determined by XRF and XRD (X-ray diffraction) analysis and the sintered pellets and leached residue were determined by XRF analysis. In this way chemical components of the boiler slag and the types of elements present in the products could be

determined. Elemental analysis of aqueous solution was performed by EDTA- $\text{Na}_2\text{-ZnCl}_2$ titration method^[9]. The leached pellets were considered as a co-product and would be used as a solidified material in masonry concrete applications and highway construction^[10,11].

2 Results and Discussion

2.1 Characterization of Boiler Slag

XRF analysis shows that a representative sample of boiler slag used in this test consists of 42.60% SiO_2 , 32.60% Al_2O_3 , 2.43% Fe_2O_3 , 2.16% CaO and trace amounts of 0.74% K_2O , 0.44% MgO, 0.16% Na_2O , 0.88% TiO_2 , 0.66% S as SO_3 and loss of ignition (LOI) of 17.31% (m/m, dried solid). The PSD of this boiler slag is shown in Fig.2.

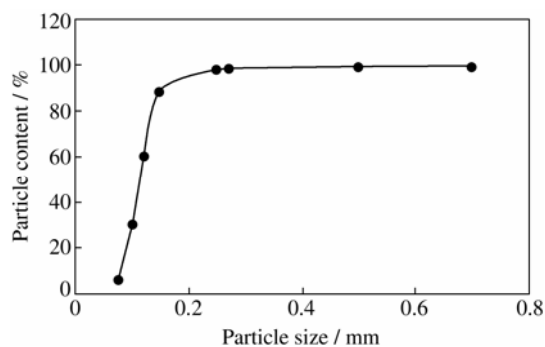


Fig. 2 The PSD curve of the boiler slag

From the curve presented in Fig. 2 it can be seen that about 85% of the total number of particles had a size of less than 0.15 mm, most of them falling in the range of 0.10-0.20 mm. The mean size of the particles was 0.18 mm.

XRD analysis of boiler slag was shown in Fig.3. The results indicate that predominate minerals such as mullite, quartz, calcium silicate and quicklime exist in this sample.

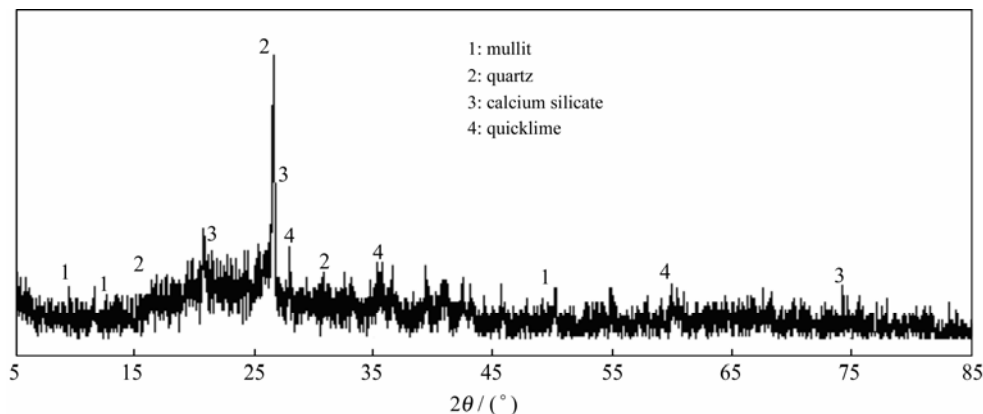
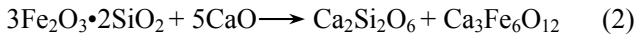
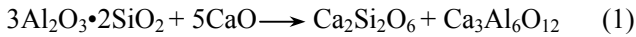


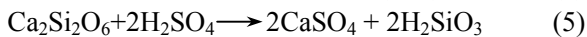
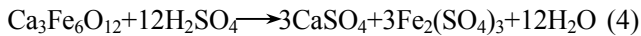
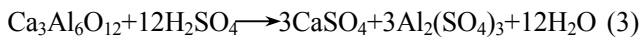
Fig.3 The XRD spectrum of the boiler slag

2.2 The Main Reactions in Leaching Process

The main purpose of sintering is to destroy the crystalline mullite phase ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) releasing free Al_2O_3 that is available to be leached^[12]. The aluminosilicate reacts with calcium oxide to form glassy phases such as calcium silicate ($\text{Ca}_2\text{Si}_2\text{O}_6$) and calcium aluminite ($\text{Ca}_3\text{Al}_6\text{O}_{12}$) during the calcination of the pellets prepared with lime, boiler slag and washed coal. The reactions occur during the sintering of pelletised boiler slag at elevated temperatures.



Thereafter the pelletised boiler slag was prepared for leaching with sulfuric acid solution. The H^+ ions from the acid react with solid aluminum and iron compounds that are progressively exposed on the surface and within pores of the pellets particles. The possible reactions that take place in the leaching of the pelletised boiler slag are as follows:



2.3 Flow Diagram

A flow diagram for the proposed process including leaching and separating described herein is shown in Fig.4. At the beginning of this work, sample was pelletised with washed coal, lime and water at different ratios to produce pellets. The pellets of mixtures then sintered in a muffle furnace for preparing the next step of leaching. Then the sintered pellets was leached with sulfuric acid at different temperature in order to dissolve Al, Fe and other species from them. The leached residual or

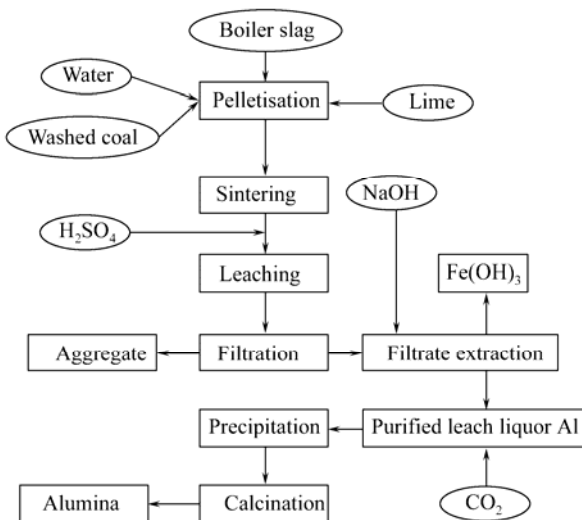


Fig. 4 A flow diagram of leaching the boiler slag

pellets were separated from the leach liquor by filtration and would be used as aggregate. Then the filtrate was prepared for purification including selective separation of aluminum ions, iron ions and some other species from the aluminum-containing aqueous solution. Finally, the purified solution containing aluminum ions was conducted by precipitation and then calcinations, from which the solid product of alumina was obtained.

2.4 Results of Leaching

2.4.1 Direct acid leaching

The direct acid leaching for this sample was performed with $4 \text{ mol} \cdot \text{L}^{-1} \text{H}_2\text{SO}_4$ at a solid/ liquid ratio of 1 : 4 yielded poor extraction efficiency of aluminum and iron even after 24 h of leaching. The results are presented in Table 1. These preliminary results are in agreement with those previously reported by A. Seidel^[13]. The author concluded that 90% of the aluminum originally exists in this boiler slag is constituted in the mullite phase with the balance located in the glassy phase. The aluminum that is in the mullite phase does not readily dissolve in mineralized acid such as sulfuric acid or hydrochloric acid. Therefore 10% extraction efficiency of aluminum by direct acid leaching is the achievable limit.

Table 1 Effect of leaching time

t/h	Al extraction efficiency/%	Fe extraction efficiency / %
2	4.70	5.20
4	8.50	10.10
8	10.10	13.50
12	10.20	13.50
24	10.20	16.70

The leaching of the unsintered pellets with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime using $4 \text{ mol} \cdot \text{L}^{-1} \text{H}_2\text{SO}_4$ at solid/liquid ratio of 1 : 4 for 24 h

2.4.2 Indirect acid leaching

In order to improve the extraction efficiency of aluminum, the pellets were placed into a silicon crucible and sintered at 1000°C for 2 h in all tests before the leaching processes. The leaching parameters such as the concentration of sulfuric acid, leaching time, different mixture of sintered pellets and solid/ liquid ratio are considered to find the optimal extraction efficiencies of aluminum and iron. The results are listed in Table 2-Table 6.

The results in Table 2 clearly show that the increase in temperature of $20\text{-}95^\circ\text{C}$ during the leaching of the sintered pellets with sulfuric acid resulted in a relatively significant improvement in the extraction efficiencies of Al and Fe. However, the extraction efficiency of metals remained less increase when the temperature exceeded 80°C .

Table 2 Effect of temperatures

Temperature / °C	Al extraction efficiency/%	Fe extraction efficiency / %
20	41.70	67.20
40	60.20	73.10
60	72.10	80.50
80	75.80	83.00
95	76.00	83.10

The leaching of the sintered pellets with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime using 4 mol · L⁻¹ H₂SO₄ at solid/ liquid ratio of 1 : 5 for 4 h

Table 3 Effect of acid strength

c/mol · L ⁻¹	Al extraction efficiency/%	Fe extraction efficiency / %
2	57.30	70.20
4	75.10	81.60
6	77.50	85.20

The leaching of the sintered pellets (with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime) using H₂SO₄ at solid/ liquid ratio of 1 : 5 and 80 °C for 4 h

Table 4 Effect of leaching time

t / h	Al extraction efficiency/%	Fe extraction efficiency / %
2	63.10	79.50
4	75.30	80.70
8	83.00	84.70
12	85.60	89.70
24	86.20	94.30

The leaching of the sintered pellets with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime using 4 mol · L⁻¹ H₂SO₄ at solid/ liquid ratio of 1 : 5 and 80 °C

Table 5 Effect of ratio of solid liquid

Solid(g)/ liquid(mL)	Al extraction efficiency/%	Fe extraction efficiency / %
1 : 6	83.90	85.00
1 : 5	83.10	84.90
1 : 4	76.00	71.20
1 : 3	53.10	55.60

The leaching of the sintered pellets with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime using 4 mol · L⁻¹ H₂SO₄ at different solid/ liquid ratios and 80 °C for 8 h

Table 6 Effect of ratio of different mixtures

Percentage of pellet composition	Al extraction efficiency/%	Fe extraction efficiency / %
70 : 20 : 10	72.60	61.10
60 : 30 : 10	71.50	57.90
50 : 40 : 10	83.70	84.00
40 : 40 : 20	84.00	90.20

The leaching of the sintered pellets using 4 mol · L⁻¹ H₂SO₄ at solid/ liquid ratio of 1 : 5 and 80 °C for 8 h

The results in Table 3 it show that the extraction efficiency of metal species increases with an increase of sulfuric acid concentration, particularly for extraction of Al. As the concentration of sulfuric acid increased from 2 mol · L⁻¹ H₂SO₄ to 4 mol · L⁻¹ H₂SO₄ the extraction efficiencies of Al increased significantly from 57.30% to 75.10% and Fe increased from 70.20% to 81.60% during the leaching of the sintered pellets with a solid/liquid

ratio of 1 : 5 at 80 °C for 4 h. However, the extraction efficiencies of Al and Fe remained limited increase during the leaching of the sintered pellets with 6 mol · L⁻¹ H₂SO₄.

From the results given in Table 4 it is clear that the extraction efficiencies of Al increased significantly from 63.10% to 86.20% and Fe increased from 79.50% to 94.30% as the leaching time increased from 2-24 h during the leaching of the sintered pellets using a solid/ liquid ratio of 1 : 5 at 80 °C. However, the extraction efficiency of Al remained limited as the leaching time increased from 12-24 h. But the extraction efficiency of Fe increased significantly as the leaching time increased from 12-24 h.

From the results presented in Table 5 it can be seen that at a solid/ liquid ratio of 1 : 5 and 1 : 6 there were almost same extraction efficiencies for both Al and Fe. From these results it is clear that solid/ liquid ratios below 1 : 4 yielded unacceptable levels of extraction efficiencies for both Al and Fe.

Table 6 shows that washed coal content has a marked effect on metal extraction efficiency in sulfuric acid leaching. Al and Fe extraction efficiencies of 84.00% and 90.20% were obtained respectively when leaching pellets, prepared with 40% boiler slag, 40% washed coal and 20% lime for 8 h at 80 °C with a H₂SO₄ concentration of 4 mol · L⁻¹. It can also be seen that the sintered pellets from the mixture of 40% boiler slag, 40% washed coal and 20% lime outperformed all of the sintered pellets from different mixtures in terms of the extraction efficiencies of both Al species and Fe species. Table 6 shows that acid leaching removed more metals from high CaO containing boiler slag than from low CaO containing boiler slag. The results show that high Al extraction efficiency was obtained for pellets (40% boiler slag, 40% washed coal, 20% lime, m/m/m) with high CaO content. An excess of CaO could reacted with mullite (3Al₂O₃·2SiO₂) sufficiently to form calcium aluminate (Ca₃Al₆O₁₂) that was soluble in acidic solution under the conditions used in this study. According to Gabler and Stoll^[14], the higher CaO content in the low rank bituminous coal fly ash was the major factor affecting the extraction efficiency of Al from coal fly ash. The results correspond to the conclusions of Gabler and Stoll but reversed to the conclusions of Matjie and Bunt^[15]. According to Matjie and Bunt, an excess of CaO could reacted with mullite(Al₆Si₂O₁₃) or free aluminum silicate to form anorthite(CaAl₂Si₂O₈) that is also insoluble in acidic solution.

The test results about the sintered temperature and time were listed in Table 7 and Table 8. Table 7 shows that the extraction efficiencies of Al and Fe increased with an increase in low sintering temperature within 800-1 000 °C, particularly for Al. The extraction efficiencies of Al and Fe decreased when the sintered pellets(sintered at 1 200 °C) were leached, particularly for Al. Sintering temperature has a marked effect on Al extraction efficiency in acid leaching. Table 8 shows clearly that the extraction efficiencies of Al and Fe increased with an increase in sintering time. However, the extraction efficiencies of Al and Fe decreased when the pellets were sintered for 180 min. It was shown that sintered pellets for the leaching step have an optimal condition, which depends on both the time and temperature for sintering.

Table 7 Effect of sintered temperature

Sintered temperature / °C	Al extraction efficiency/%	Fe extraction efficiency / %
800	53.20	80.60
1 000	86.50	94.60
1 200	60.50	92.10

The leaching of the sintered pellets with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime using 4 mol · L⁻¹ H₂SO₄ at solid/ liquid ratio of 1 : 5 and 80 °C for 2 h. The leaching time is 24 h

Table 8 Effect of sintered time

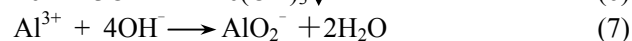
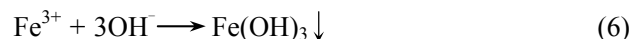
Sintered time/min	Al extraction efficiency/%	Fe extraction efficiency / %
30	43.80	84.90
60	62.00	87.20
90	71.30	90.40
120	86.50	94.20
180	76.20	92.70

The leaching of the sintered pellets with a weight ratio of 50% boiler slag, 40% washed coal and 10% lime using 4 mol · L⁻¹ H₂SO₄ at solid/ liquid ratio of 1 : 5 and 80 °C for 2 h. The pellets were sintered at 1 000 °C

2.5 Precipitation and Separation of Al(III) from Sodium Aluminate Solution

At pH value of 13.0, the Al³⁺ ions in the filtrate react with sodium hydroxide to form soluble sodium aluminate. White gelatinous metal hydroxide precipitates were formed after bubbling the filtrate, recovered from the precipitation of Fe³⁺ ions at pH 13.0, with CO₂. These white gelatinous precipitates indicated that Al³⁺ ions from the filtrate precipitated in the form of aluminum hydroxide. The separation efficiency of Al³⁺ ions from the filtrate was 87.20%. This process seems effective in separating Al³⁺ ions from this solution. However, the disadvantage of adjusting the pH of the leach liquor to a value of 13.0 is the consumption of large amounts of expensive sodium hydroxide for the precipitation of metal ion hydroxides. The sodium hydrogen carbonate

obtained from the precipitation of aluminum hydroxide can be converted to sodium hydroxide^[15]. This sodium hydroxide can be recycled to the sodium hydroxide leaching process to reduce the cost of the sodium hydroxide reagent. The chemical reactions described above are as follows:



2.6 Characterization of the Final Product of Alumina

The XRF fusion analysis of the precipitate formed after precipitation of Fe(III) indicates that the concentration of iron hydroxide in the precipitate was 92.20%. The XRF fusion analysis of the precipitate formed after bubbling the filtrate with carbon dioxide also indicates that high levels of the impurities such as iron oxide(0.12% Fe₂O₃), calcium oxide(0.40% CaO), silicon dioxide (0.20% SiO₂), sodium oxide(0.10% Na₂O), sulphur trioxide(1.70% SO₃) with LOI of 3.10% are still contained in the precipitates. The presence of these impurities in the alumina can also be attributed to the presence of hydroxide in the filtrate. The concentration of alumina in the final product after calcination was determined to be 99.12%. This product would be suitable for the production of aluminum metal(the concentration of alumina in the production of aluminum metal must be 99%^[16]).

2.7 Characterization of the Sintered Pellets and Leached Residue

The XRF fusion analysis of the sintered pellets indicates that the sintered pellets contained 30.16% SiO₂, 18.40% CaO, 26.70% Al₂O₃, 1.20% Na₂O, 2.10% Fe₂O₃, 3.81% S as SO₃, some trace elements such as 0.35% K₂O, 0.28% MgO, 0.38% TiO₂ and LOI 16.60%. The results indicate that the content of CaO and S as SO₃ was higher than that in the leached pellets. The XRF fusion analysis of the leached residue that was washing at ambient temperature also revealed that the leached residue consists of 45.20% SiO₂, 29.40% CaO, 3.90% Al₂O₃, 2.20% Na₂O, 0.20% Fe₂O₃, 0.82% S as SO₃, some trace elements such as 0.30% K₂O, 0.24% MgO, 0.41% TiO₂ and LOI 17.32%. It's clear that the sintered pellets contain high aluminum oxide in comparison with the leached residue. The LOI, which is now out of specification, can be used in structural concrete. However, a LOI of 25% is permissible when aggregates having SO₃ contents below 1% to be used in masonry concrete^[17]. So, the leached residue is considered as a co-product in this process having suit-

ability as a lightweight aggregate and can be used in Masonry concrete applications or cement production.

3 Conclusion

The objective of this study was to develop a new process for extracting aluminum from boiler slag. The high temperature calcination of the pellets was used to break the crystalline mullite phase rendering soluble alumina for leaching. The XRD analysis indicates that predominate minerals such as mullite, quartz, calcium silicate and quicklime exist in boiler slag. The leached residue is suitable for Masonry concrete application or cement production. The final product obtained after the calcination of pure aluminum hydroxide contains 99.12% of alumina and would be suitable for the commercial process.

Direct acid leaching yielded an Al extraction efficiency of only 10.2%. This is in agreement with previously published findings. The extraction efficiencies of Al and Fe increased with an increase in temperature, leaching time and acid concentration. The best conditions for leaching Al and Fe from boiler slag are as follows: ① Leaching temperature range of 60-80 °C; ② concentration of H₂SO₄ during the leaching step range within 4-6 mol • L⁻¹; ③ leaching time range from 4 to 8 h; solid/liquid ratio range from 1 : 5 to 1 : 6; ④ better pellet composition with a weight ratio of boiler slag, washed coal and lime was 40 : 40 : 20 or 50 : 40 : 10; ⑤ the maximum extraction efficiency of Al was 86.50% using 4 mol • L⁻¹ H₂SO₄ at solid/ liquid ratio of 1 : 5 at 80 °C for 24 h; The maximum extraction efficiency of Fe was 94.60% in the same conditions of that for the maximum extraction efficiency of Al.

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