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Production of Alpha-Alumina from Black Aluminum Dross Using NaOH Leaching Followed by Calcination

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The aim of this work is to produce α-alumina particles from aluminum (Al) dross via leaching–precipitation–calcination processes. In the leaching test, the effects of parameters such as the temperature, NaOH concentration, and time were investigated based on the Taguchi approach. In addition, the effect of the particle size on the Al extraction rate was determined. The Al extraction rate ranged from 78.64% to 93.11% and from 78.35% to 91.99% for ground dross and as-received dross, respectively. In the precipitation test, the pH of the leachate solution was decreased using HCl to remove Al ions as Al hydroxide precipitate. The precipitate was calcined at 1000°C to 1200°C for 270 min. The morphological and polymorphic properties of the products were characterized by scanning electron microscopy, atomic force microscopy, and x-ray diffraction analysis. The experimental findings indicated that the calcination temperature and stabilization time were critical for producing α-alumina particles rather than other polymorphs.

INTRODUCTION

Aluminum (Al) dross is a by-product that is formed owing to the reaction between the surface of the molten metal and the furnace atmosphere during the Al melting process. The quantity of this by-product is high, and its disposal is an important problem because of its toxic nature that may create environmental pollution in the atmosphere and groundwater. Landfilling this type of waste is not a feasible option as Al demand from industry is expected to increase, resulting in the formation of considerably large quantities of such waste.[1]

The conversion of dross into valuable products could be an alternative to landfilling, and this choice would create new economic opportunities. Recently, the use of this waste as a raw material has been investigated to prepare ceramic materials,[2,3] ceramic membranes,[4] refractory materials,[5,6] and pure alumina particles with different polymorphs.[1,7–11]

Alkali or acid leaching can be performed to leach Al from the dross because of its amphoteric nature.[12] Various chemicals (HCl,[13] H_2SO_4,[14] NaOH,[15] NH_4OH,[11] and KOH[16]) have been used as leaching or precipitation agents depending on the leaching conditions. Notably, hydrogen gas released from the dross can be captured in a gas tank that contains water.[17,18] Prior to leaching, ball-milling treatment has been suggested to prepare mechanically activated Al dross that can be leached in short time with high recovery.[19] The use of ultrasound waves in leaching tests increased the Al extraction rate up to 60%.[20] The polymorph properties of the alumina particles were considerably influenced by the calcination conditions, i.e., temperature and time. The dross was converted to γ-alumina at 600°C to 800°C,[21–23] η-alumina at 900°C,[24] θ-alumina at 1000°C,[25] and α-alumina at 1000°C to 1400°C.[25]

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There are many alumina polymorphs (β, γ, δ, and κ) found in metastable phases. However, these polymorphs cannot be used in high-temperature applications (i.e., as ceramics or refractory materials) because of their transformation to α-alumina, which is the only thermodynamically stable phase.[26] In the work presented herein, production of α-alumina particles from Al dross was investigated using hydrometallurgical and pyrometallurgical processes divided into the following stages: NaOH leaching, precipitation, and calcination. The Taguchi approach was used to determine the effect of the experimental parameters on the leaching of Al from the dross. As-received and ground samples were used in the leaching test to evaluate the effect of the particle size distribution on the Al extraction rate. To determine the effect of the calcination conditions on the properties of the product, x-ray diffraction (XRD) analysis, scanning electron microscopy (SEM), and atomic force microscopy (AFM) were conducted. Furthermore, the influence of the stabilization time in the precipitation stage on the properties of the final products was determined.

EXPERIMENTAL PROCEDURES

Materials

Al dross waste was collected from an Al production factory in Adana, Turkey. XRD (Bruker D8 Discover) analysis (Fig. S1) revealed the following materials in the dross: aluminum nitride (AlN, 24.6%), metallic aluminum (Al, 22.3%), halite (NaCl, 18.1%), spinel ($MgAl_2O_4$/$MgO{\cdot}Al_2O_3$, 12.3%), aluminum oxide (Al_2O_3, 7%), calcite ($CaCO_3$, 6.8%), tricalcium aluminate ($Ca_3Al_2O_6$, 5%), and periclase (MgO, 3.9%). In addition, the chemical composition of the waste was as follows: 47.9% Al, 4.5% Mg, 7.1% Na, 8.4% N, 5% Ca, 0.8% C, 11.0% Cl, and 15.4% O. All chemicals used in this study were of analytical grade and used without purification.

Experimental Methods

Leaching Test

The dross was prepared with size of less than 100 μm using a laboratory-scale jaw crusher followed by a laboratory-type ball mill. The dry milling conditions were as follows: 1000 g dross, 15 kg stainless ball mill, and 67 rpm circulation speed. Prior to performing the NaOH leaching test, the ground dross ($d_{100} <$ 100 μm) was leached using hot water at 70°C for 3 h to remove water-soluble elements, i.e., Na, K, and Cl, based on previous studies wherein the Al-extraction rate increased by 5% to 15% after a hot washing procedure.[14,24]

The solid-to-liquid ratio and stirring speed were 1:7 and 250 rpm, respectively. The slurry was filtered through filter paper (40 cm × 40 cm) and washed using hot water. The residues obtained after the water leaching were used as the starting material for the leaching test, which was performed in a 250-mL double-walled glass reactor equipped with a water circulator and condenser. The effects of the leaching temperature, reaction time, and NaOH concentration on the dissolution of Al from the dross were investigated based on the Taguchi approach (L9, 3^3). The dissolution of Al from the dross in the presence of NaOH obeys the following chemical reaction:

$$Al_2O_3 + 2NaOH \rightarrow 2NaAlO_2 + H_2O \qquad (1)$$

The experimental parameters and conditions are presented in Table I. The solid-to-liquid ratio and stirring speed were kept constant at 1:15 (i.e., 10 g dross and 150 mL solution) and 250 rpm, respectively. When the predetermined time had elapsed, the reaction had completed and the slurry was filtered using Whatman 1 filter paper. The amount of Al dissolved in the leachate solution was determined by atomic absorption spectrometry (AAS, 900 H; PerkinElmer).

Furthermore, the Al content in the residue was determined by AAS after digestion in a microwave digester (CEM, Mars 6), in which the residue was dissolved using HCl + HF + HNO_3 followed by boric acid neutralization. The Al extraction rate from the dross was calculated as

$$\mathrm{ER}\ (\%\ ) = \frac{A}{B} \times 100 \qquad (2)$$

where ER denotes the Al extraction rate from the dross (%), A is the Al concentration in the leachate solution (mg/L), and B is the initial Al concentration in the dross (mg/L).

For comparison, as-received dross waste (d_{100} 500 μm) was also leached using the aforementioned conditions to evaluate the effect of the particle size distribution on the Al dissolution rate. All the abovementioned procedures were performed separately for the leaching of the following two different sized samples: the as-received dross, denoted as sample B, and the ground dross (d_{100} 100 μm), denoted as sample A.

Precipitation of Al in Form of Hydroxide and Its Calcination Procedure

Stock solution was prepared considering the best leaching conditions prior to the precipitation test. In each precipitation test, 100 mL stock solution was used. The Al ions in the leachate solution were precipitated as hydroxide by adding the required amounts of HCl acid (0.1 M), thereby decreasing the solution pH from 13.5 to approximately 7. The solution was further stirred at 250 rpm for different times (10 min, 1 h, and 18 h). When the reaction time was over, the precipitate was recovered via solid/liquid separation. The following chemical

Table I. Experimental parameters with their levels and conditions (L9, 3^3)

Parameter	Symbol	Level 1	Level 2	Level 3
Leaching temperature (°C)	A	20	65	85
Reaction time (h)	B	1	3	5
NaOH concentration (M)	C	2	3.5	5
Experimental conditions	Experiment no.	A	B	C
	1	20	1	2
	2	20	3	3.5
	3	20	5	5
	4	65	1	3.5
	5	65	3	5
	6	65	5	2
	7	85	1	5
	8	85	3	2
	9	85	5	3.5

reaction represents the precipitation of Al ions in the form of Al hydroxide from the leachate solution:

$$NaAlO_2 + HCl + H_2O \rightarrow Al(OH)_3\downarrow + NaCl \qquad (3)$$

The obtained precipitate was calcined at 1000°C and 1200°C for 270 min to obtain a product as follows:

$$2Al(OH)_3 \rightarrow Al_2O_3 + 3H_2O \qquad (4)$$

The process parameters used in this study are denoted as S*Xy*-C*Z*, where S indicates stabilization, *X* is the stabilization time conducted after decreasing the pH to the desired level, *y* indicates minutes or hours, C indicates calcination, and *Z* is the calcination temperature; For example, S10m-C1000 means that the precipitate was stirred for 10 min after decreasing the solution pH to 7 and the calcination test was performed at 1000°C for 270 min.

The phase properties of the products were investigated by XRD analysis using Cu K_α radiation in the 2θ range from 5° to 80°. The obtained patterns were evaluated using Diffrac.Suite Eva software for mineral identification. In addition, SEM (Zeiss Gemini 300) was performed to evaluate the morphological properties of the selected product. The surface topology of the product ($2.5\ \mu m \times 2.5\ \mu m$) was determined by AFM (NanoSurf Flex Amf C3000) in static mode. Finally, the purity of the selected product was determined by AAS after digestion in the microwave digester.

RESULTS AND DISCUSSION

Leaching Experiments

Leaching experiments based on the Taguchi approach were conducted to determine the effect of the reaction temperature, time, and NaOH concentration on the dissolution of Al from the dross. In addition, the two samples identified as samples A and B in the leaching test were compared. During the leaching experiments, hydrogen gas was released from the dross and was captured in a laboratory-scale gas holder. However, no measurements were conducted to determine the amount of gas released, which will be investigated in future study. The use of NaOH as the leaching agent resulted in a purer leachate solution compared with acid leaching processes because impurities (Fe, Ti, Mg, and Ca) in the dross did not dissolve.[19] Therefore, no measurements of these elements in the leachate solution were carried out.

The experimental findings (Table II) show that the Al extraction rate from the dross varied between 78.64% and 93.11% for sample A and between 78.35% and 91.99% for sample B. These values are higher than those obtained in some previously conducted studies;[19,27] however, they are in good agreement with those of another previously conducted study.[9] The low Al extraction rate in the previously conducted study can be attributed to the high activation energy required to dissolve the AlN present in the dross. Comparison of the experimental results confirms that a decrease in the particle size increased the Al extraction rate from the dross. These findings are in line with those of previous studies[9,28–30] that explained in detail the effect of mechanical activation on the leaching of Al from different resources.

The aim of this work is to obtain a high Al extraction rate from the dross. Accordingly, the "larger is better" quality characteristic was selected to evaluate the effect of the leaching parameters on the Al extraction. By analyzing the S/N ratios, the most important factors for the Al extraction rate from sample A were determined to be, in descending order of significance, NaOH concentration, leaching temperature, and leaching time. Interestingly, the most important factors for sample B were, in descending order of significance, leaching temperature, NaOH concentration, and leaching time (Fig. 1).

Table II. Experimental results obtained in this study

Experiment no.	Temperature	Time	NaOH concentration	Al extraction rate for sample A (%)	Al extraction rate for sample B (%)
1	20	1	2	80.88	80.55
2	20	3	3.5	78.64	78.35
3	20	5	5	88.23	82.34
4	65	1	3.5	91.11	89.12
5	65	3	5	92.78	91.99
6	65	5	2	84.53	83.61
7	85	1	5	93.11	89.80
8	85	3	2	85.15	84.57
9	85	5	3.5	86.88	87.34

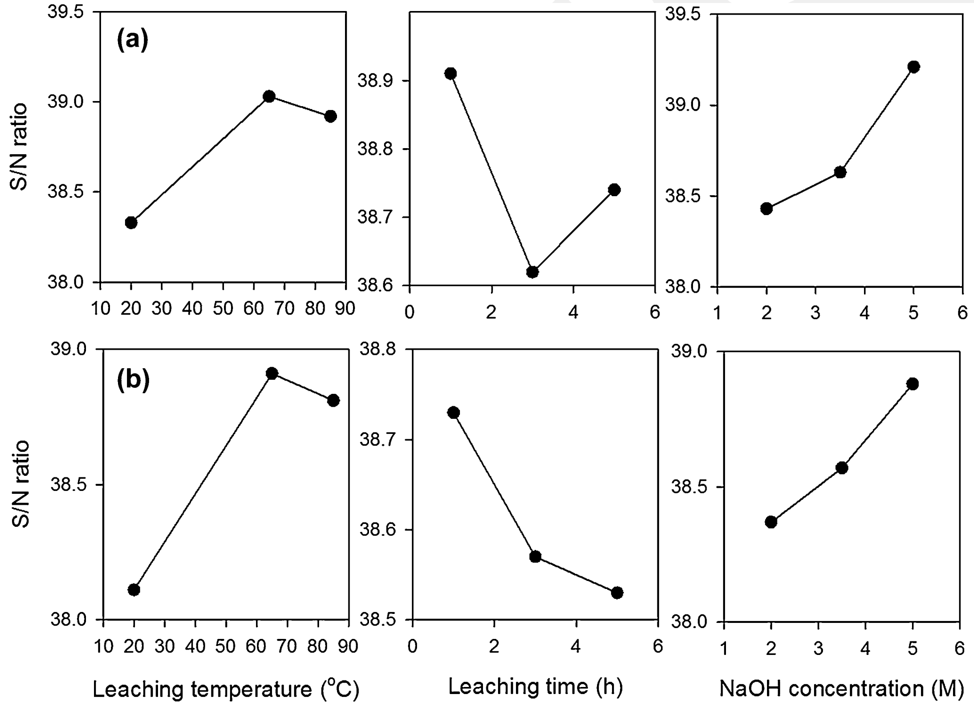

Fig. 1. Influence of leaching parameters on the extraction rate of Al from the dross: (a) sample A, (b) sample B.

These findings are in good agreement with the contribution ratio (CR) that indicates the ratio of the effect of each parameter on the leaching performance. The CR value was calculated based on analysis of variance (ANOVA) presented in Table - III. The CR for the Al extraction rate from sample A was 39.98% and 48.49% for leaching temperature and NaOH concentration, respectively. The CR for the Al extraction rate from sample B was 65.25% for leaching temperature and 24.06% for NaOH concentration. These results suggest that the most important parameter was the NaOH concentration for sample A and the leaching temperature for sample B. Because of the decrease in the particle size, the reaction could be realized at low temperature with high efficiency for sample A. Notably, the Al extraction rate for sample B was lower than that for sample A, due to its presence as an inclusion in the sample. The reaction rate changed with temperature, but not the barrier to the reaction happening, which corresponds to the activation energy. In addition, the ANOVA results showed that, in comparison with the other parameters, the reaction time did not substantially affect the Al extraction rate.

We observed satisfactory correlations between the experimental results and the values predicted using the regression models. The *R*-value for the model was 0.97 and 0.96 for samples A and B, respectively (Fig. 2a and b). The models could be fit with a satisfactory correlation. Figure 2c and d depict contour diagrams that show the effect of the reaction temperature and NaOH concentration on the Al extraction rate. Clearly, the performance of the

Table III. ANOVA results and model summaries for Al extraction rate from dross

Sample	Source	Degrees of freedom	Sum of squares (SS)	Mean square	*F*-value	*P* value	CR (%)[a]
Sample A	Temperature	2	82.26	41.131	7.30	0.120	39.98
	Time	2	12.44	6.220	1.10	0.475	6.04
	NaOH conc.	**2**	**99.76**	**49.880**	**8.85**	**0.101**	**48.49**
	Error	2	11.27	5.634			5.47
	Total	8	205.73				100.00
Sample B	**Temperature**	**2**	**108.723**	**54.362**	**9.89**	**0.092**	**65.25**
	Time	2	6.829	3.415	0.62	0.617	4.10
	NaOH conc.	2	40.092	20.046	3.65	0.215	24.06
	Error	2	10.990	5.495			6.59
	Total	8	166.635				100.00
Model summary (sample A)			*S*			R^2	
		2.37355				94.52%	
Regression equation Sample A (%) = 86.813 − 4.23 Temperature_20 + 2.66 Temperature_65 + 1.57 Temperature_85 + 1.55 Time_1 − 1.29 Time_3 − 0.26 Time_5 − 3.29 NaOH concentration_2.0 − 1.27 NaOH concentration_3.5 + 4.56 NaOH concentration_5.0							
Model summary (sample B)			*S*			R^2	
		2.344413				93.40%	
Regression equation Sample B (%) = 85.297 − 4.88 Temperature_20 + 2.94 Temperature_65 + 1.94 Temperature_85 + 1.19 Time_1 − 0.33 Time_3 − 0.86 Time_5 − 2.39 NaOH concentration_2.0 − 0.36 NaOH concentration_3.5 + 2.75 NaOH concentration_5.0							

Bold values show the parameters most strongly affecting the Al extraction rate.[a]Contribution ratio calculated as CR = (SS value of source/total SS value) × 100.

leaching experiments at ambient temperature (20°C) resulted in low extraction recovery of Al (< 80%). To obtain a higher extraction recovery, the leaching test should be conducted at higher temperatures; For example, the highest Al extraction rate for sample A could be obtained by applying the following conditions: temperature of 85°C, leaching time of 1 h, and NaOH concentration of 5 M. These findings are in good agreement with those of previous studies.[9,31] If the leaching temperature is decreased to 65°C, then to obtain a similar Al extraction rate, the leaching test should be conducted for 3 h.

Finally, we decided to prepare a stock solution using sample A, as the extraction recovery of Al was higher for sample A than for sample B. Therefore, 1000 g of sample A was ground to particle size below 100 μm. Thereafter, sample A was leached in the presence of NaOH under the following conditions: temperature of 85°C, leaching time of 1 h, and NaOH concentration of 5 M. The amount of Al dissolved in the leachate solution was 29.7 g/L.

Precipitation and Calcination Experiments

The pH of 100 mL leachate solution (containing 29.7 g/L Al) prepared at the optimal conditions was decreased to 7 by addition of HCl (0.1 M). After reaching the desired solution pH, the precipitate began to form in the solution. Three slurry samples were further stirred for 10 min, 1 h, and 18 h, respectively. The formed precipitate was recovered by solid-to-liquid separation. Subsequently, a calcination procedure was performed at 1000°C to 1200°C for 270 min to convert the Al hydroxide precipitate to Al_2O_3 product. The XRD patterns of the obtained products are shown in Fig. 3. Notably, Na-based minerals (such as sodium aluminum oxide and diaoyudaoite) were observed in some products due to the insufficient washing procedure that was conducted after the precipitation.

Alumina with different polymorphs (β, γ, δ, κ, and α) was produced according to the production conditions, which are critical in alumina production. From Fig. 3a, it is evident that the product that contained different polymorphs (β, γ, δ, and κ) was obtained at the S10m-C1000 condition. The amounts of AlO, β-Al_2O_3, γ-Al_2O_3, δ-Al_2O_3, and κ-Al_2O_3 were 26.9%, 19.2%, 16.4%, 12.9%, and 10.6%, respectively. The rest was identified as $NaAlO_2$ (13.9%). Upon increasing the calcination temperature from 1000°C to 1200°C (S10m-C1200), the obtained product mainly comprised β-Al_2O_3 (76.1%) and δ-Al_2O_3 (23.9%) (Fig. 3b). A mixture of AlO (47.9%), γ-Al_2O_3 (27.5%), and κ-Al_2O_3 (24.7%) polymorphs was observed in the product prepared at the S1h-C1000 condition (Fig. 3c). The stabilization time for the Al hydroxide precipitate was observed to be an effective parameter that affected the phase properties of the alumina products. An increase in the stabilization time resulted in the production of an alumina product with a single polymorph rather

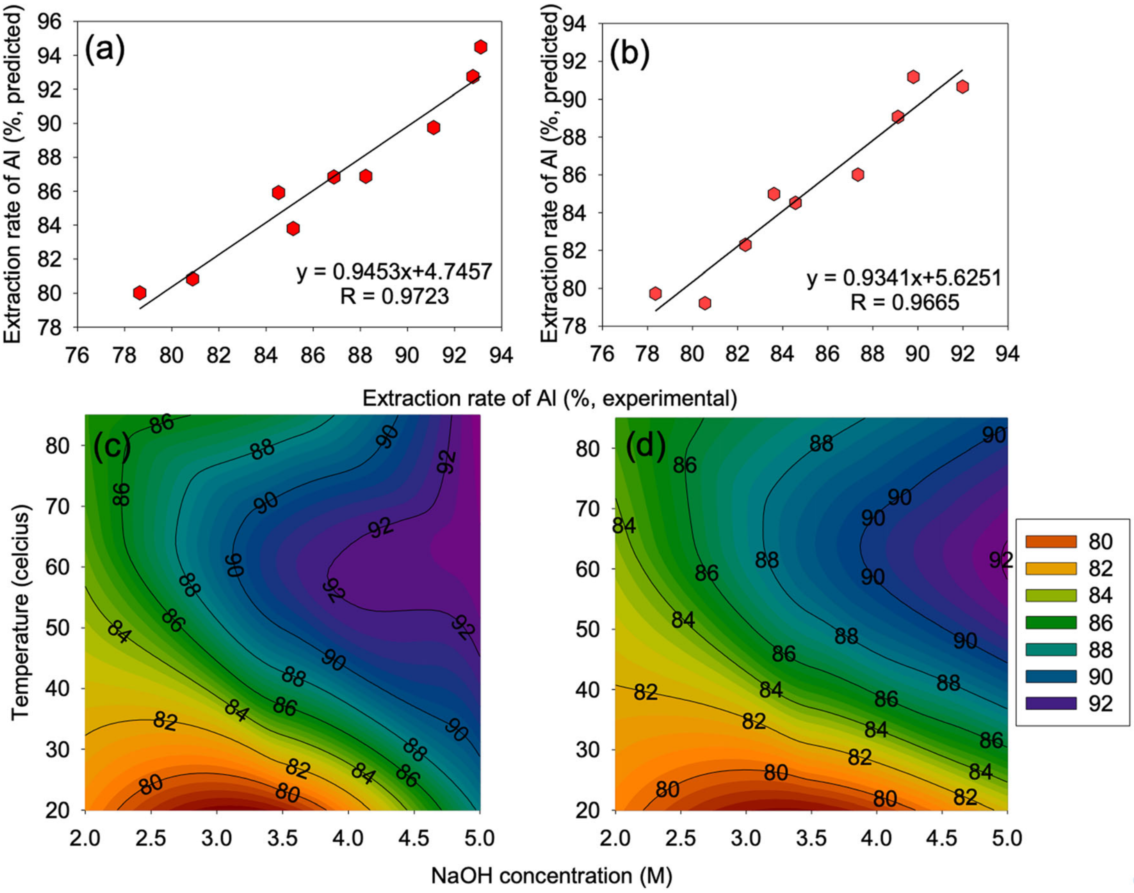

Fig. 2. Comparison of experimental and calculated results for Al extraction from (a) sample A and (b) sample B, and effects of NaOH concentration and temperature on Al extraction rate from (c) sample A and (d) sample B.

than a mixture of alumina polymorphs. The product with the α-Al_2O_3 polymorph was obtained at the S1h-C1200 and S18h-C1200 conditions.

The effect of the production conditions on the morphological properties of the product is depicted in Fig. 4. Polycrystalline crystals were observed when conducting calcination at 1000°C (Fig. 4a, c, and e). The product obtained at the S10m-C1000 condition had lumpy or balloon-type particles with rounded corners (Fig. 4a). These are similar to those obtained in previous studies.[32,33] The stabilization time in the precipitation stage might have affected the morphological properties of the products. An increase in the stabilization time resulted in the formation of agglomerated alumina particles at 1000°C (Fig. 4c–e). A mixture of tiny (nanosized) and large agglomerated crystals was observed in these products. The product S10m-C1200 (Fig. 4b) had layered plate structures, indicating the characteristic surface morphology of β-alumina.[34] This finding is in line with the XRD analysis (Fig. 3b).

The extended stabilization time in the precipitation stage might have triggered the formation of α-alumina that comprised sheet structures with sharper edges (Fig. 4d–f). These observed morphologies are in good agreement with those of a previously conducted study.[35] In another study, plate-like α-alumina crystals were prepared at 1100°C, but an increase in the temperature resulted in the disappearance of the sharp edges in these plate structures.[36] The EDX results (Supplementary Table S1) indicated that the purity of α-alumina was high (39.65% O, 52.29% Al, 7.61% Au, and 0.45% K). The presence of Au in the product was attributed to the coating of the product prior to the SEM examination. AFM images of the products are shown in Fig. 5, and the findings obtained from the

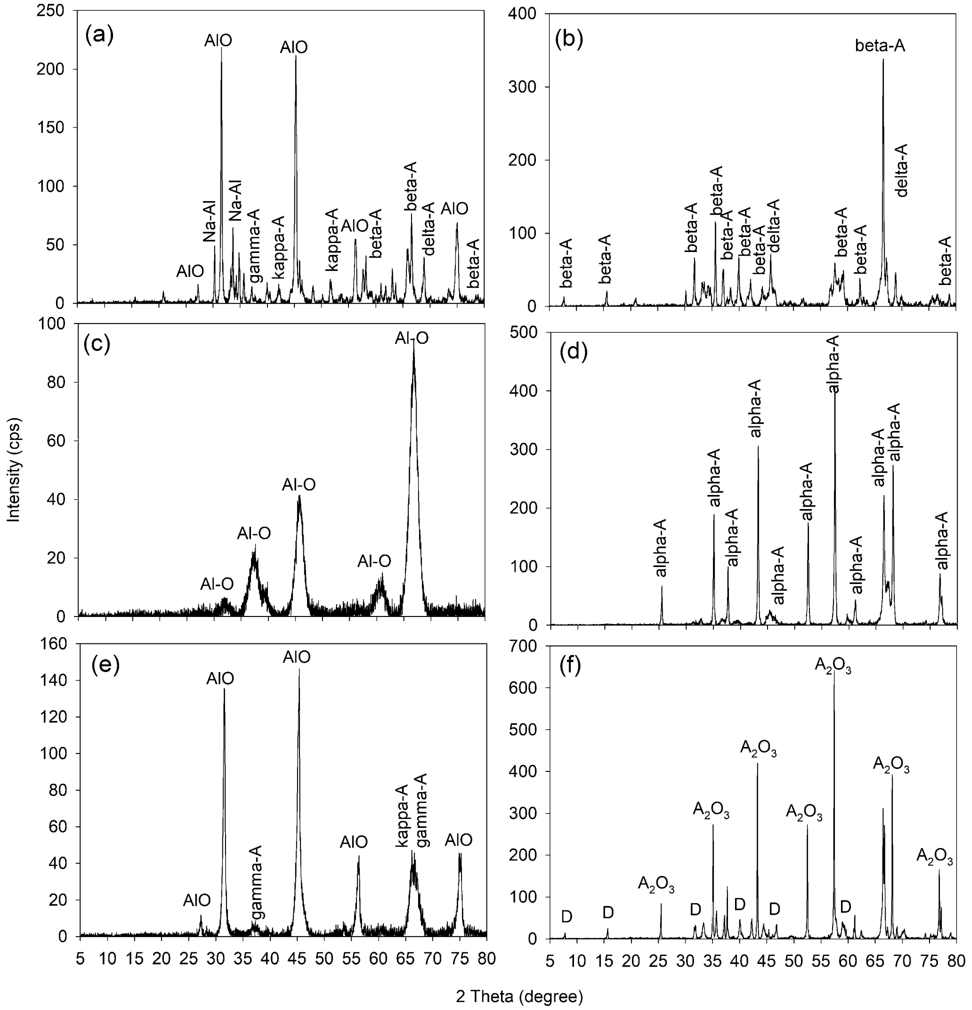

Fig. 3. XRD patterns of (a) S10m-C1000, (b) S10m-C1200, (c) S1h-C1000, (d) S1h-C1200, (e) S18h-C1000, and (f) S18h-C1200 products [AlO: aluminium oxide (card no. 75-0278), Na-Al: sodium aluminum oxide (card no. 33-1200), Al-O: $(Al_2O_3)_{5.3333}$ (card no. 74-2206), alpha-A: α-Al_2O_3 (card no. 82-1468), beta-A: β-Al_2O_3 (card no. 10-0414), kappa-Al: κ-Al_2O_3 (card no. 52-0803), gamma-A: γ-Al_2O_3 (card no. 46-1131), delta-A: δ-Al_2O_3 (card no. 46-1131), and D: diaoyudaoite "$NaAl_{11}O_{17}$" (card no. 21-1096)].

AFM observations are provided in Supplementary Table S2. The surface roughness of the product varied widely from 4.64 nm to 268.21 nm.

CONCLUSION

The aim of this study is to produce high-purity α-alumina from Al dross using leaching–precipitation–calcination processes. Prior to performing the NaOH leaching, the dross was washed using hot water. The effect of the particle size on the Al extraction was investigated using the following parameters: leaching temperature, leaching time, and NaOH concentration, based on the Taguchi approach. The experimental findings indicated that the extraction recovery increased as the particle size was decreased. The parameters in the leaching test were, in descending order of significance: for sample A (d_{100} 100 μm), NaOH concentration, leaching temperature, and leaching time; for sample B (d_{100} 500 μm), leaching temperature, NaOH concentration, and leaching time.

Prior to performing the calcination process, the stabilization time in the precipitation stage should be at least 1 h to obtain an alumina product with a single polymorph. Furthermore, the calcination

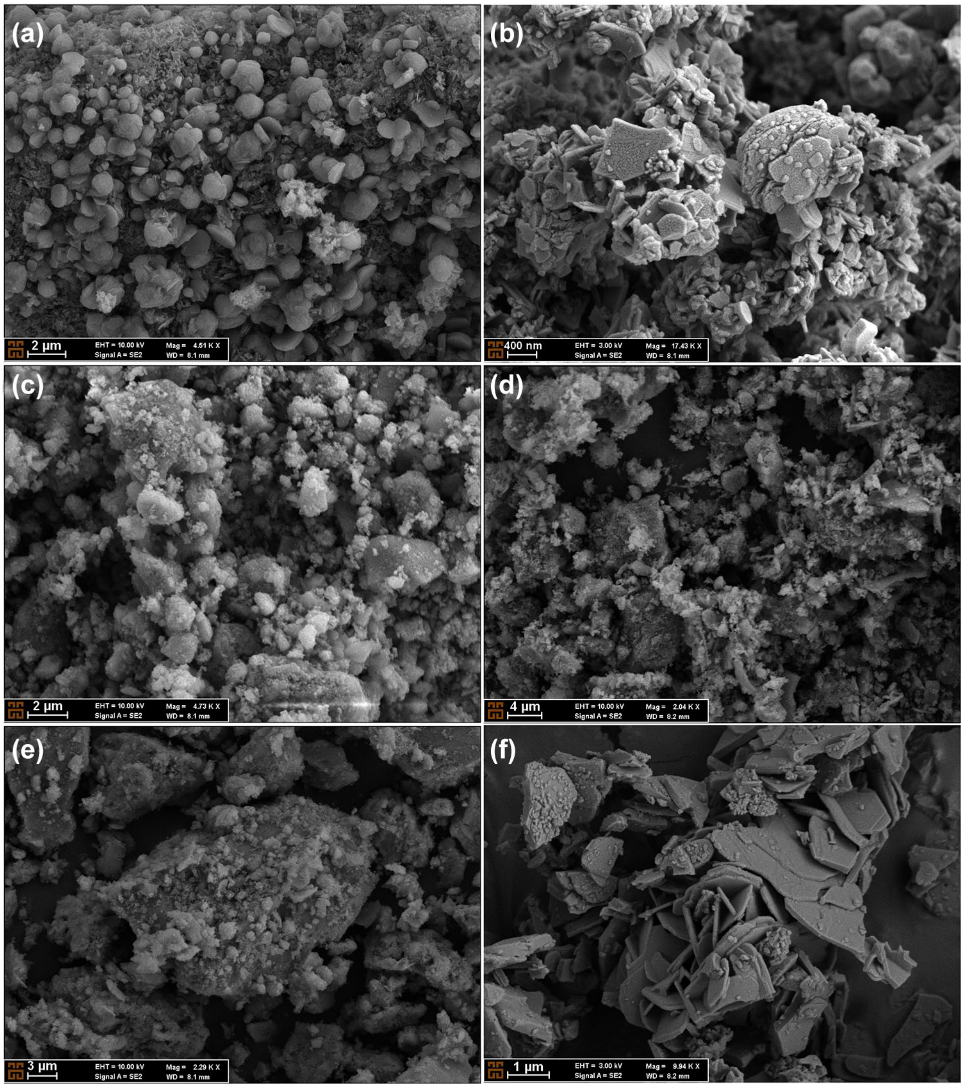

Fig. 4. SEM images of (a) S10m-C1000, (b) S10m-C1200, (c) S1h-C1000, (d) S1h-C1200, (e) S18h-C1000, and (f) S18h-C1200 products.

temperature significantly affected the morphological and crystal properties of the product. A mixture of alumina polymorphs (β, γ, δ, κ, and α) was obtained at 1000°C. To obtain α-alumina particles with high purity from the dross, the calcination should be performed at a temperature of 1200°C after stabilization of the Al hydroxide precipitate for 1 h or more in the present study. The obtained results suggest that Al dross can be used as a raw material to produce high-purity α-alumina particles, thereby solving the Al dross disposal problem.

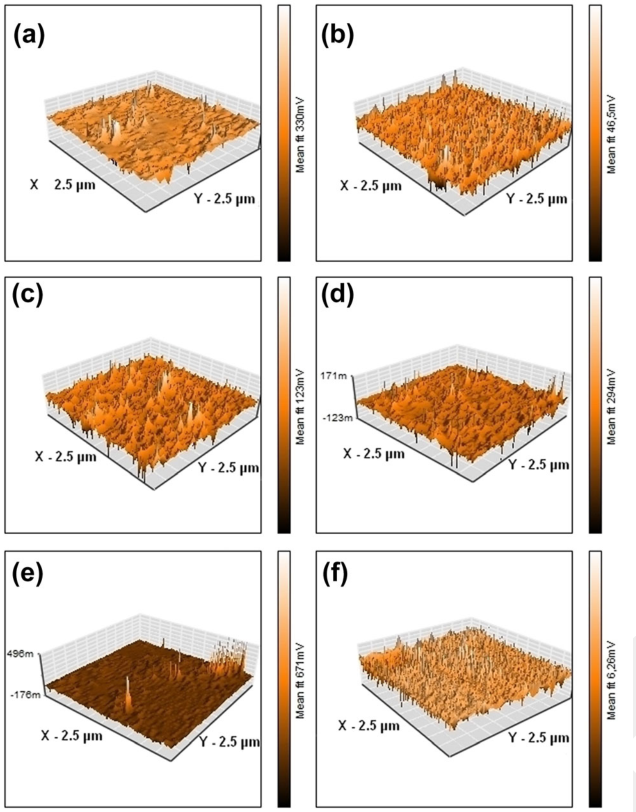

Fig. 5. Surface topography (2.5 μm × 2.5 μm) of products obtained under different conditions: (a) S10m-C1000, (b) S10m-C1200, (c) S1h-C1000, (d) S1h-C1200, (e) S18h-C1000, and (f) S18h-C1200.

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ELECTRONIC SUPPLEMENTARY MATERIAL

The online version of this article (https://doi.org/10.1007/s11837-020-04281-7) contains supplementary material, which is available to authorized users.

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