

Valorization of aluminium dross for the development of al-rich product

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Received: January 31st, 2024; Accepted: May 21st, 2024; Published: July 23rd, 2024

Abstract. In this study, aluminium dross undergoes a hydrometallurgical recycling process. Various chemical reagents are employed to treat the aluminium dross, facilitating the maximum extraction of aluminium content from the dross into leach liquor. The hydrometallurgical route ensures efficient aluminium leaching, forming a solution rich in Al, used as a precursor/raw material for valuable material generation.

α -alumina is obtained by leaching dross with alkalis at 323 K for 3 h and precipitating with acids followed by a calcination process at 1,473 K for 3 h. The parameters are optimized and multiple trials are done to ensure the reproducibility of the results. Morphological and topographical studies of the synthesized products are done by XRD and SEM-EDS characterizations. TGA analysis of the samples were also conducted. This work enables to recycling of aluminium dross and converts it into a valuable product which has high demand in refractory and high-temperature applications. By converting aluminium dross into a resource with intrinsic value, this process significantly mitigates environmental impact, aligning seamlessly with the principles of a sustainable and circular economy. In doing so, it exemplifies a proactive approach towards resource conservation, waste reduction, and the responsible utilization of materials within industrial processes.

Key words: aluminium dross, hydrometallurgy, recycling, recovery, valuable product.

INTRODUCTION

Each year Aluminium dross is generated in enormous quantities therefore disposing of it is a demanding endeavour to keep the environment clean. Aluminium dross is considered a waste which is generated when the molten bath comes into contact with surrounding atmospheric oxygen and gets oxidized. Approximately 8 wt.% per ton of aluminium production contributes to the generation of dross (Srivastava & Meshram, 2023b). Extrapolating from this estimate, it can be inferred that in 2023, primary smelters produced nearly 5.5 million tons of dross, considering that global aluminium production reached approximately 69 million tons (Primary Aluminium Production, International Aluminum Institute, 2023). China alone, in 2021, produced more than 3 million tons of dross (Wang et al., 2023). However, the dross generation is expected to increase 3% to 4% per annum in the coming years (Al Circle, 2023).

Aluminium dross is categorized into three types based on the aluminium content it contains. White dross, originating from primary smelters, has the highest aluminium content, ranging from 15–80 wt.%, along with 20–85 wt.% aluminium oxide and less than 5 wt.% salt fluxes. Black dross, also known as secondary dross, contains 7–50 wt.% aluminium, 30–50 wt.% aluminium oxide, and 30–50 wt.% salt fluxes. The third type, salt cake, is produced when white dross is processed in furnaces, and it has the lowest metallic aluminium content (3–10 wt.%) and the highest salt flux content (20–80 wt.%) (Manfredi et al., 1997; Mankhand, 2012).

The predominant method of disposing of the majority of dross involves depositing it in landfill sites, posing a risk of leaching toxic metal ions into groundwater and causing significant pollution issues (Das et al., 2007). Furthermore, when aluminium dross comes into contact with water, it releases harmful gases, including NH_3 , CH_4 , PH_3 , H_2 , H_2S , etc (Mahinroosta & Allahverdi, 2018; Meshram & Singh, 2018). This interaction underscores the potential environmental hazards associated with the disposal and contact of aluminium dross, emphasizing the need for more environmentally friendly disposal practices. The rising demand for valuable materials and the stringent enforcement of environmental standards have prompted the establishment of suitable treatment facilities for industrial wastes. Furthermore, in the pursuit of sustainable development, the substitution of primary resources with secondary resources has become increasingly essential.

To address this challenge, the consideration of recycling on a larger scale becomes essential. Among the various recycling methods, pyrometallurgy is a well-established industrial-scale approach for dross recycling. There are two routes in pyrometallurgy which are carried out at large scale. A rotary salt furnace (RSF) is a conventional method which uses various salt fluxes like a mixture of NaCl and KCl , MgF_2 , and CaF_2 (Alberto et al., 2002). These fluxes are esteemed for their ability to enhance aluminium recovery. However, a significant drawback of this method lies in the generation of salt slag or salt cake as a by-product due to flux usage. Therefore, a separate recycling process becomes necessary to manage these salt cakes.

The second route of pyrometallurgy is termed advanced techniques or salt-free techniques (SFT) which do not use salt fluxes and result in higher extraction yield therefore eliminating the generation of salt cakes. Although SFT have major advantages over RSF like high metal yield, and no use of salt fluxes yet these technologies are energy-intensive and require different types of fuels which increases the cost of the process (Ünlü & Drouet, 2002).

Apart from pyrometallurgy, hydrometallurgy and hydrothermal are two other routes for recycling leverage dross to yield valuable aluminium-rich products. The advantage of hydrometallurgy over pyrometallurgy is its applicability to low-grade ores (Fleming, 1992). Moreover, hydrometallurgical recycling allows direct utilization of dross in alkalis or acids to generate numerous value-added products (Srivastava & Meshram, 2023a).

On the other hand, hydrothermal technology represents an advanced approach which is used to synthesize valuable Al-rich products like zeolites (Bortolini et al., 2020; Hiraki et al., 2009), currently at the laboratory scale. Apart from the generation of valuable products, Tsakiridis et al. (2013) recovered aluminium using the hydrothermal method. Black dross was used to recover aluminium by reacting it with NaOH (260 g L^{-1})

at 513 K under pressure-leaching conditions. The leaching efficiency was achieved at around 58% (Tsakiridis et al., 2013).

Das et al. (2007) explored the hydrometallurgical route to produce η -alumina by using H_2SO_4 leaching of washed and unwashed dross samples at 363 K. Unwashed samples demonstrated a maximum recovery of approximately 88% using 30 mL of 30% concentration acid. In contrast, washed samples exhibited an efficiency of around 84% with a combination of 15% (v/v) acid and 10% dross (w/v). The leach liquor was neutralized with 10% aqueous ammonia solution to produce $\text{Al}(\text{OH})_3$ which was subsequently subjected to heating at 1,173 K, leading to the formation of η -alumina (Das et al., 2007). This study highlights the efficacy of washing aluminium dross samples, reducing the required acid concentration for leaching compared to unwashed samples. The simple washing step proves effective in achieving similar efficiency while using lower acid concentrations, emphasizing the economic and environmental benefits of this approach.

Similarly, employing black dross with 4 M H_2SO_4 and subsequent precipitation with propanol yielded α -alumina with a purity of 96.5%. The calcination temperature was 1,573 K. The reported yield for this process was 100%, and the synthesized α -alumina particles produced through this method were observed to be smaller than 100 nm (Meor Yusoff et al., 2011).

Table 1. Synthesis of different phases of alumina from aluminium dross using different recycling routes

Raw Material	Recycling route	Operating parameters	Synthesized products	References
Al dross	Pyrometallurgy	Radio frequency plasma synthesis system	Fine Al_2O_3	(Yang et al., 2014)
Al dross	Pyrometallurgy	Plasma arc melting	γ - Al_2O_3	(Saravanakumar et al., 2018)
Al dross	Pyrometallurgy	Sintering at 1,803 K with 10% dilute phenolic resin	High-alumina refractory	(Li et al., 2014)
Black dross	Hydrometallurgy	NaOH leaching for 1 h at 358 K, precipitation with 0.1 M HCl, calcination at 1,473 K	α - Al_2O_3	(Türk et al., 2020)
Al dross	Hydrometallurgy	NaOH leaching at 333 K for 40 min followed by desilicization, carbonization and calcination at 1,273 K	γ - Al_2O_3	(He et al., 2021)
Black dross	Hydrometallurgy	5 M HCl leaching at 358 K for 2 h, NaOH precipitation followed by re-precipitation with HCl and calcined at 873 K	Mesoporous alumina	(Mahinroosta et al., 2019)
Washed Al dross	Hydrometallurgy	H_2SO_4 leaching with a ratio of 1:4 at 333 K for 12 h followed by calcination at 873 K for 2 h	γ - Al_2O_3	(Roslan et al., 2019)

Different methods for synthesizing various alumina phases have been explored by several researchers, as detailed in Table 1. While some employed pyrometallurgy in alumina production, others chose hydrometallurgical processes. The advantage of

hydrometallurgy is that it enables various operational parameters like temperature, concentration, solid-to-liquid ratio etc. which results in different forms of products. Also, hydrometallurgy processes ensure a significant reduction in energy consumption, compared to pyrometallurgical processes.

Yang et al. (2014) employed an alternative methodology, utilizing radio frequency plasma as a high-temperature source with a system capacity of 1.2 kg h^{-1} . Vacuum conditions were sustained using argon, and a high current passed through six induction coils, generating an electromagnetic field. This process resulted in the creation of a thermal plasma flame as a high-temperature source. At such elevated temperatures, the dross underwent complete vaporization, and the formation of alumina occurred during the cooling phase through a nucleation process (Yang et al., 2014).

Similarly, the generation of $\gamma\text{-Al}_2\text{O}_3$ was achieved through plasma arc melting. The reaction chamber underwent purging with argon through a central opening in the graphite cathode. The dross was positioned within a crucible (anode), and as the cathode descended into the crucible, it contacted the anode, initiating an arc. This arc, propelled by extremely high temperatures (ranging from 6,000 to 14,000 K), vaporized the entire sample, leading to the formation of alumina during cooling (Saravanakumar et al., 2018).

However, an innovative hydrometallurgical method was employed to produce mesoporous alumina from black dross through leaching with 5 M HCl. The leaching process occurred for 2 hours at a temperature of 358 K. Subsequently, the extraction procedure was carried out with a solid-liquid ratio maintained at $1:20 \text{ g mL}^{-1}$. The addition of NH_3 facilitated achieving pH levels between 5.5 and 6.0, resulting in a milky white hydroxide. Introducing 3 N NaOH to the white hydroxide precipitate yielded an alkaline sodium aluminate solution, adjusting the pH to 12–13. Re-precipitation with HCl facilitated the formation of a filtrate solution, which was then calcined at 873 K for 2 hours to produce mesoporous alumina (Mahinroosta et al., 2019).

Ramaswamy et al. (2019b) employed aluminium dross to produce high-temperature resistant materials by agitating the dross in an aqueous medium at 373 K with a stirring speed of 1,200 rpm. The resulting slurry underwent calcination at 1,423 K, followed by compaction and sintering at 1,773 K for 6 hours. Additionally, they conducted thermal shock tests on their samples, observing either 100 cycles or thermal failure, whichever occurred first (Ramaswamy et al., 2019b).

Researchers have explored alternative applications for aluminium dross beyond alumina synthesis. One such process involved utilizing aluminium dross as a source material for producing aluminium and magnesium-based coating powders. Initially, the dross sample was ground to a particle size of less than $600 \mu\text{m}$ and subjected to leaching with water to extract aluminium nitride in the form of ammonia. Following the removal of aluminium nitride, the resulting slurry underwent calcination at 1,423 K for 2–6 hours. Subsequently, a ball milling process was employed on the calcined powder, which was then mixed with polyvinyl alcohol (PVA) acting as a binder. This amalgamation transformed the calcined powder into agglomerate powder suitable for plasma spraying. The resultant powder from aluminium dross leaching exhibited potential for application in coating the bond coating of NiCrAlY steel substrates (Gomes & Ramaswamy, 2022).

El-Amir et al. (2021) combined aluminium dross with soda-lime glass waste to create high-quality glass foam bricks suitable for thermal insulation purposes. They

prepared a mixture of dross powder and soda-lime glass, then dry-moulded the blend. The moulded samples were subsequently sintered in a muffle furnace for 30 minutes at temperatures ranging from 1,173 K to 1,273 K, with a heating rate of 5 °C min⁻¹. Following the sintering process, demolding, cutting, and finishing procedures were carried out to produce the glass foam bricks (El-Amir et al., 2021).

Hence, these studies emphasize the significance of the hydrometallurgical method, illustrating that valuable products can be efficiently synthesized with great effort. These products hold substantial value within the industrial market, making recycling a viable method for both disposing of dross and generating significant revenue.

Therefore, in the present study, a hydrometallurgical approach is chosen for dross valorization. The study employs a systematic approach to produce α -alumina, a material with diverse applications in refractory and ceramic industries. This involves the utilization of different alkalis in the synthesis process. This approach provides a comprehensive understanding of the effectiveness of different chemical reagents on aluminium dross. The ability to generate pure alumina with minimal effort enhances the applicability of this method for producing alumina on a larger scale to meet industrial demands.

MATERIALS AND METHODS

Aluminium dross sample, sourced from a casting unit at Indian Institute of Technology Kanpur, U.P., India, was acquired for the study. Initially collected sample was a solid, compact mass having white lusture. However, in the experiment solid sample was cut into smaller pieces, resulting in smaller chips. The studied dross sample was determined to be white dross after being characterized and considering its direct origin from the primary smelting unit. All the chemicals used in the study were of analytical grade (Rankem, Qualigens). NaOH and KOH solutions were prepared using deionized water.

A 100 mL solution of 1 M NaOH and KOH was prepared in conical flasks. The solutions were heated to a target temperature of 323 K using a Hotplate with a magnetic stirrer (Borosil HLS200). Once the desired temperature was reached, 2 g of pre-weighed dross chips were added to each solution. The system was left undisturbed with continuous stirring at 400 rpm for 3 hours. Throughout this period, the solution temperature was monitored using a thermometer. After completing the leaching process, the solution was filtered and the obtained mother liquor underwent further processing.

20 mL of mother liquor obtained post NaOH and KOH leaching were subjected to H₂SO₄ and HCl addition, dropwise, resulting in the formation of a thick white precipitate. The white precipitate underwent several rinses (minimum 5–6 times) with deionized water to thoroughly eliminate any residual salts from the precipitate. The precipitates were then oven-dried (Skylab with exhaust fan) at 323 K. Following this, the dried precipitates were subjected to calcination in a tubular furnace (Lenton Thermal Designs) at 1,473 K for a duration of 3 hours, utilizing a maximum operation voltage of 230 V and a power input of 5,500 watts. The furnace's heating rate was at 8–10 °C min⁻¹.

The calcined powders were characterized through X-ray diffraction (XRD) analysis and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS). XRD patterns were obtained using Rigaku MiniFlex 600 with a Cu target

(Cu K α = 1.54 Å), operating at tube voltage and tube current of 30 kV and 10 mA, respectively. SEM was employed to study the morphology of the product, while EDS revealed the elemental composition (Carl Zeiss EVO 50). XRD and SEM-EDS analyses were also conducted on the solid residue to gain a more comprehensive understanding of the remaining components after the leaching process. Fig. 1 shows a process flow chart which explains the synthesis route of α -alumina.

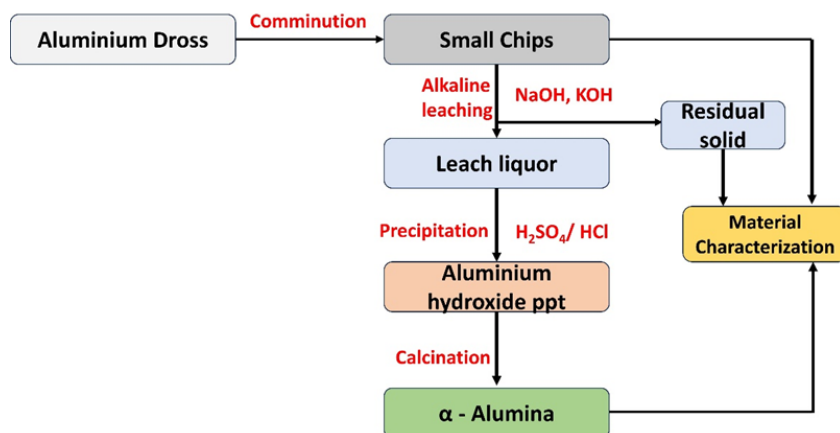


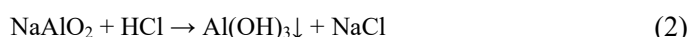
Figure 1. Flowchart illustrating synthesis of α -Al₂O₃ using alkaline leaching of dross.

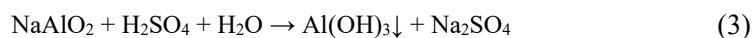
Calcined samples were also characterized by thermogravimetric analysis (TGA) (STA 500 Regulus; NETZSCH Instrument Inc., Germany). A platinum crucible with lid under an argon atmosphere (to maintain an inert atmosphere) with a heating rate of 25 °C min⁻¹ and a gas flow rate of 60 mL min⁻¹ was used. Platinum crucible with lid was used to minimize the volatile losses and a fresh baseline was established using another empty platinum crucible with lid. The TGA study was done from room temperature (298 K) to 1,673 K to measure the mass loss of the synthesized samples at different temperatures.

Recycling of aluminium dross enables us to produce various products like tamarugite (Meshram et al., 2019a; Meshram et al., 2019b), potash alum (Meshram et al., 2020), zeolites (López-Delgado et al., 2014) etc. All have their specific applications however alumina is a product which has vast applications like in refractory industries (Li et al., 2014; Ramaswamy et al., 2019a) and in various other ceramic applications (Abd Aziz et al., 2019). Therefore, the production of alumina through the recycling of aluminium dross is an attractive and viable solution on a large scale.

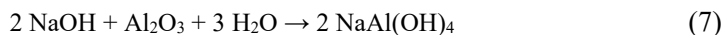
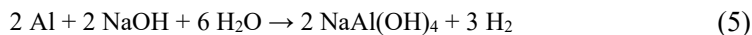
RESULTS AND DISCUSSION

The transformation of alumina at higher calcination temperatures can be elucidated by examining the following reactions (Breky et al., 2019):





However, some of the reactions can also be understood as follows (Birnin-Yauri & Musa, 2014; Singh et al., 2019):



Hence it is evident that when metallic aluminium reacts with NaOH, Al^{3+} ions are generated (Sipos, 2009) and hydrogen evolves as a by-product (Meshramet al., 2019b; Srivastava & Meshram, 2022). Similar reactions can be understood for KOH. Post leaching precipitated aluminium hydroxide powder shows amorphous behaviour as confirmed with XRD. However, this amorphous aluminium hydroxide goes under phase transformation at different calcination temperatures and forms different alumina phases. The particular type of phase transformation depends on the properties of the precursor material. Within the low-temperature range of 523–1,173 K, χ , η , γ and ρ alumina emerge, while at higher temperatures, α , δ , κ , and θ alumina become prevalent (Goodboy & Downing, 1990).

As received aluminium dross chips were analysed using XRD for phase determination. The XRD of dross (JCPDS 00-004-0787) indicates the presence of an aluminium phase. The dominance of the aluminium can also be confirmed with EDS. Peaks corresponding to aluminium were discerned at 38.7° , 44.9° , 65.3° , 78.4° , and 82.7° . XRD confirms that aluminium is the only detectable phase in the sample however some traces are evident of other elements found in EDS. This states that the sample was heterogenous in nature and XRD peaks of those elements were not identifiable. Fig. 2 illustrates the XRD pattern of the aluminium dross.

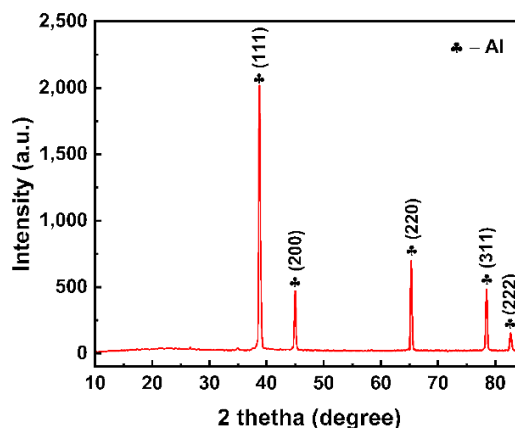


Figure 2. X-Ray Diffraction pattern of aluminium dross.

On the other hand, scanning electron microscopy reveals a non-uniform structure of the dross chips. The morphology indicates that the chips were not entirely flat; rather, some areas exhibited undulations. The EDS analysis affirms the prevalence of aluminium in the dross, constituting 80 wt.%, although traces of other phases such as Si and Mg are also detected. The sample exhibits an oxygen content of approximately 17.7 wt.%. Hence, the presence of these additional phases indicates the heterogeneous nature of the sample. Fig. 3 shows the SEM-EDS of white aluminium dross chips. The observed chip was having multiple undulations on its surface hence the morphology was not uniform.

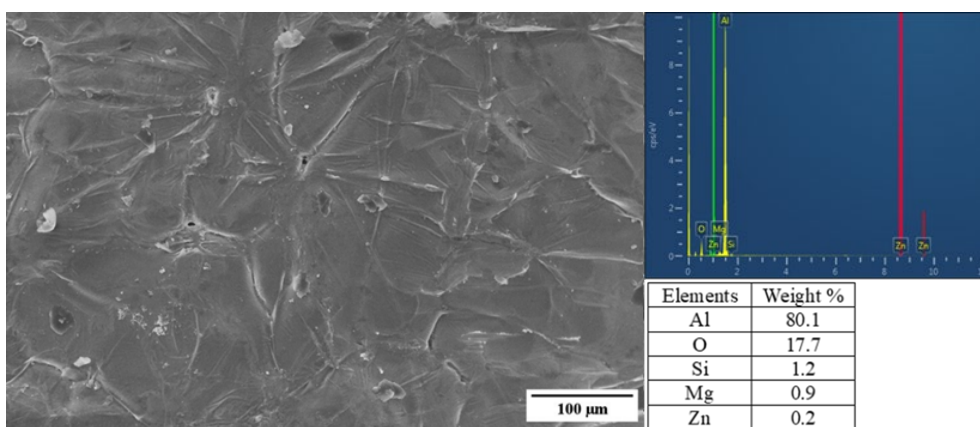


Figure 3. SEM-EDS of white aluminium dross chips.

In this current investigation, precipitated aluminium hydroxide powder underwent calcination at 1,473 K for a period of 3 hours. Previous studies have reported a wide range of calcination temperatures, varying from 473 to 1,473 K. According to literature, lower temperature calcination, up to 1,073 K, results in amorphous structures, while crystalline structures begin to emerge around 1,173 K (Bhattacharya et al., 2004). Hence, the choice of calcination temperature and duration was determined based on information gathered from previous studies (Das et al., 2007; Zhu et al., 2022). This procedure also signifies the valorization of aluminium dross, wherein valuable α -alumina is synthesized without the need for pretreatment operations of the dross. The approach chosen for this study is straightforward, and devoid of complicated steps, making the process easy and simple. Fig. 4 illustrates the XRD patterns of the synthesized alumina powders, subjected to leaching with NaOH and KOH, and precipitated with H_2SO_4 and HCl.

Observing the XRD patterns above (JCPDS 00-046-1212), it can be deduced that the resulting product aligns with the desired $\alpha\text{-Al}_2\text{O}_3$. Although the peaks exhibit a slight leftward shift (~ 0.0023 d-spacing) with standard data, they remain within the acceptable range. The characterization of the synthesized product shows no other phase formation apart from α -alumina. The SEM-EDS of the synthesized powders from NaOH and KOH leaching are shown in Fig. 5 and Fig. 6, respectively. Morphology of

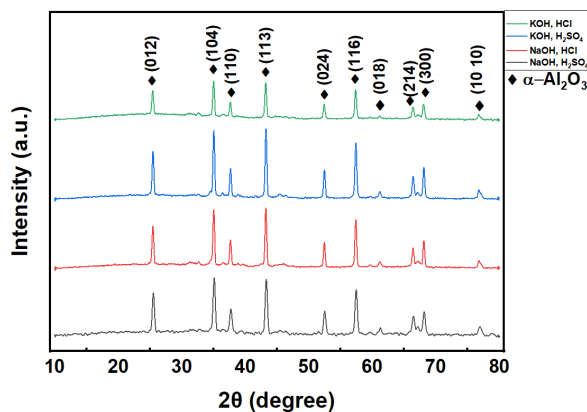


Figure 4. XRD patterns of $\alpha\text{-Al}_2\text{O}_3$ synthesized by reacting dross with NaOH, and KOH and precipitated with H_2SO_4 and HCl.

formed α -Al₂O₃ shows that there is no uniformity in the structure. The morphology of the synthesized powder resembles as dense agglomerates.

The α -Al₂O₃, synthesized using H₂SO₄ as the precipitating agent, exhibits certain areas with a dense air bubble-type structure, visible on the larger particles, as depicted in Fig. 5, a. Upon magnification to 2,000x, illustrated in Fig. 6, a, these bubbles appear as protrusions growing on the larger particles. Conversely, when precipitated with HCl, the structure appears in the form of flakes as shown in Fig. 5, c and 6, c. The particles exhibit irregular shapes, with some having sharp-edged corners while others have blunt corners. The size of each particle varies, but all exhibit tiny protrusions. Fig. 5, b and 5, d show the EDS map of α -alumina synthesized using NaOH leaching and precipitated using H₂SO₄ and HCl, respectively. Similarly, Fig. 6, b and 6, d show the EDS map of synthesized product from KOH leaching.

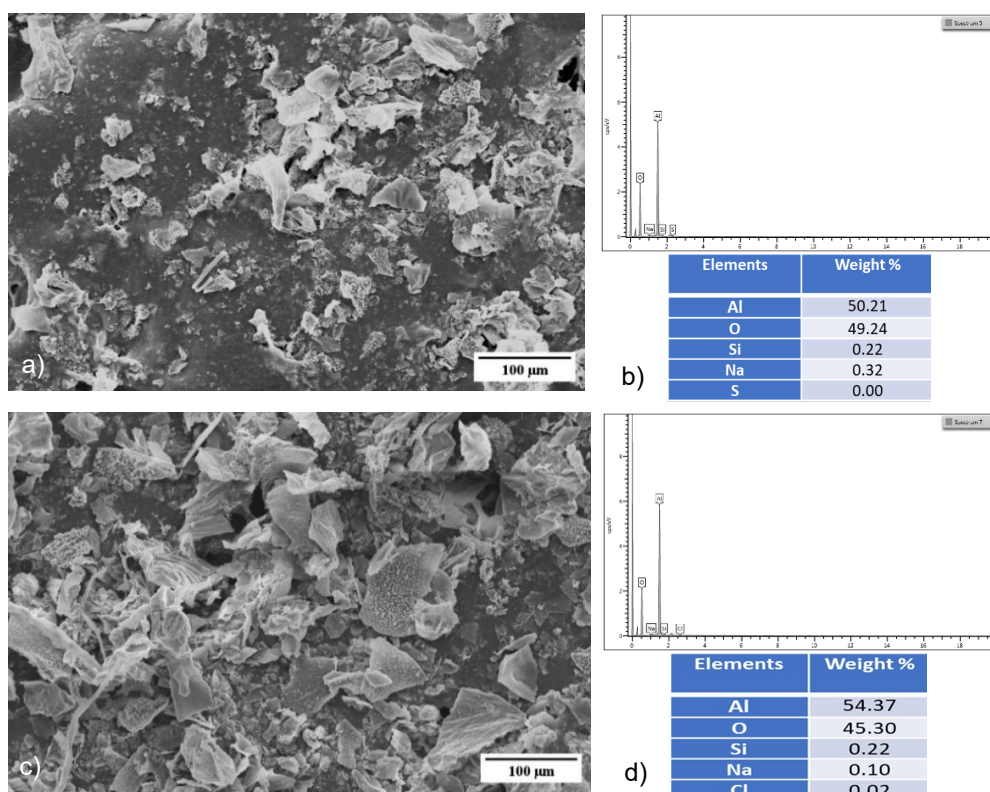


Figure 5. SEM micrographs and EDS reports of α -Al₂O₃ synthesized by NaOH leaching and precipitated with: H₂SO₄ (a, b) and HCl (c, d).

The solid residual following leaching was stored and subjected to characterization through XRD and SEM-EDS. For both NaOH and KOH residues, the predominant phases identified were sodium aluminium silicate and potassium aluminium silicate respectively, with an additional phase of silica i.e., SiO₂. Fig. 7, a and 7, b illustrate the XRD patterns of the distinct solid residues obtained from NaOH and KOH leaching,

respectively. The determined phase with a chemical formula of sodium aluminium silicate is $\text{Na}_w\text{Al}_x\text{Si}_y\text{O}_z$, where w falls within the range of 1–1.96, x ranges from 1–2, y varies between 0.35–6, and z varies from 2–8.

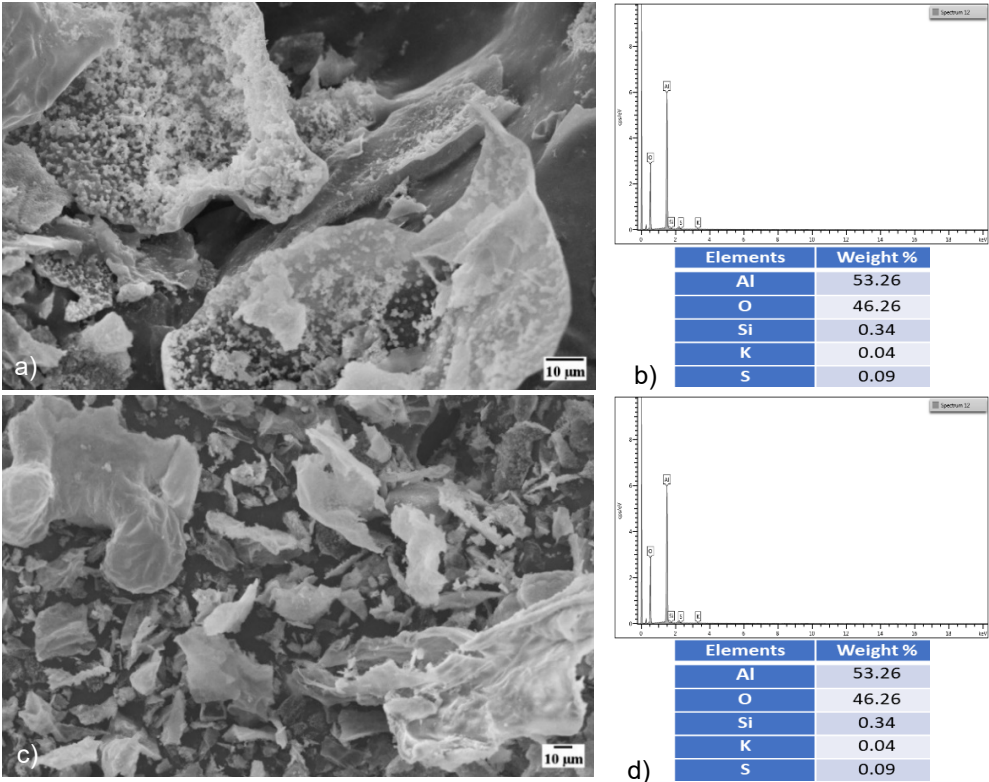


Figure 6. SEM micrographs and EDS reports of $\alpha\text{-Al}_2\text{O}_3$ synthesized by KOH leaching and precipitated with: H_2SO_4 (a, b) and HCl (c, d).

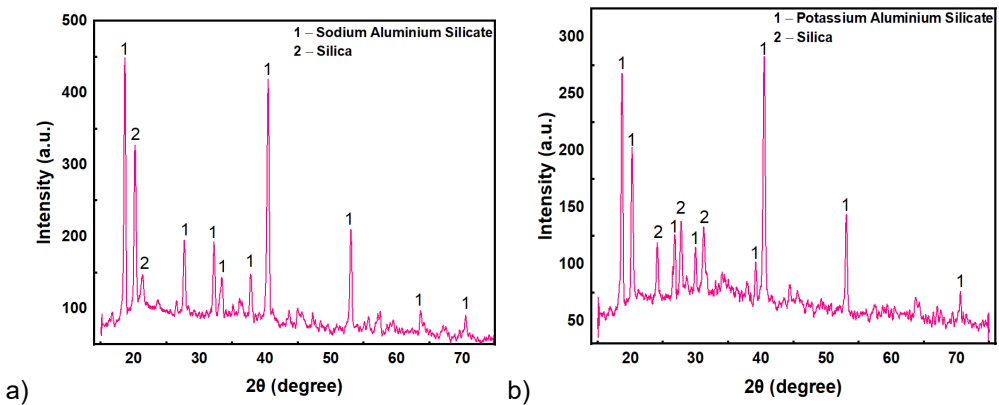


Figure 7. XRD patterns of solid residue post: a) NaOH leaching; b) KOH leaching.

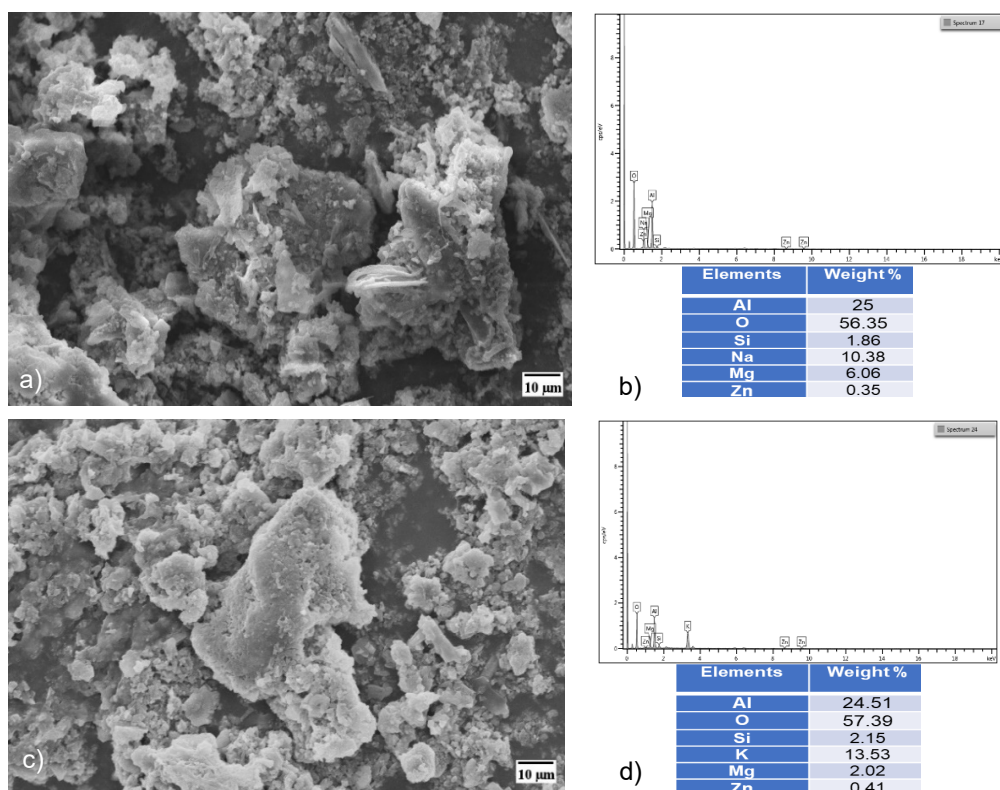


Figure 8. SEM-EDS of solid residues after: a,b. NaOH leaching and c,d. KOH leaching.

SEM-EDS of solid residues, as depicted in Fig. 8, indicates that the composition of the remaining aluminium in the residues post-NaOH and KOH leaching is nearly identical. Agglomerates of significant size emerge post-leaching and contain minute, finely distributed particles all over the surface. These conglomerates can be distinguished as either sodium aluminium silicate or potassium aluminium silicate, depending on the leaching agent employed.

Fig. 9 elucidates the TGA curve obtained for α -alumina samples which were synthesized using NaOH and KOH leaching. TGA for aluminium hydroxide was also done to get the total mass loss during the heating cycle. The total mass loss for $\text{Al}(\text{OH})_3$ was obtained around 27% while for alumina sample obtained

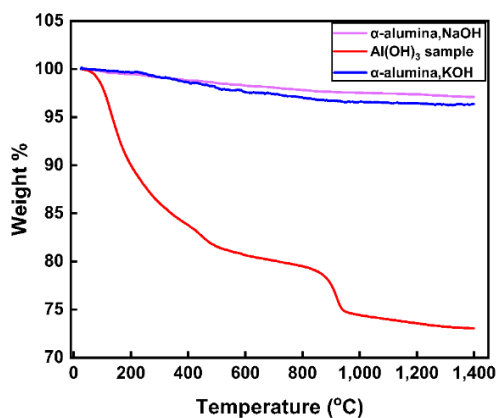


Figure 9. Thermogravimetric analysis (TGA) showing mass loss of α - Al_2O_3 samples synthesized from NaOH and KOH leaching and $\text{Al}(\text{OH})_3$ sample.

post NaOH leaching was around 3% and for KOH leaching was around 3.5%. Mass loss reported in literature (Sarker et al., 2015) falls in the range of 25–30% for $\text{Al}(\text{OH})_3$ which is seen in the result obtained in present study.

Upon analyzing the results presented above, it becomes evident that aluminium dross can be transformed into valuable products. The methodology employed in this study emphasizes that the generation of α -alumina from dross does not necessitate any pre-treatment. This not only simplifies the entire process but also highlights the efficacy and ease with which aluminium dross can be repurposed to yield materials of significant worth. The straightforward nature of the steps involved in this approach not only enhances the overall process but also signifies a practical and approachable method for harnessing the potential of aluminium waste in the production of valuable end products.

CONCLUSIONS

The recycling of aluminium dross emerges as a highly viable and advantageous solution. Given the substantial global volume of generated waste, recycling becomes imperative for its effective management. This study specifically focuses on synthesizing α -alumina directly from untreated aluminium dross, showcasing its potential minimal need for preliminary treatments.

The alkali treatment applied to the waste aluminium content within the dross efficiently produces valuable products. Leaching experiments using NaOH and KOH at 323 K for 3 hours, followed by precipitation with HCl and H_2SO_4 , and subsequent calcination at 1,473 K, resulting in the production of $\alpha\text{-Al}_2\text{O}_3$. Characterization analyses confirm the formation of α -alumina with high purity, free from any other phases.

The produced alumina can be used for making Al-rich products like refractory applications, zeolite synthesis and other value-added materials. The authors believe that there is a wide scope of research in the hydrometallurgical recycling route as there are multiple factors like temperature, solid-to-liquid ratio, agitation speed and calcination temperature which can be optimized to generate numerous products.

This research emphasizes the significance of aluminium dross as a substantial source of metallic aluminium, typically lost in disposal sites. The recycling of this waste not only yields valuable materials but also reintroduces the previously discarded metallic aluminium back into the production cycle. Hence, aluminium dross recycling not only lessens environmental impact but also contributes to the sustainable utilization of resources in a variety of industrial applications.

ACKNOWLEDGEMENTS. The authors revere the Head, Department of Materials Science and Engineering, IIT Kanpur for providing the necessary facilities to conduct the present research. The support from the institute in the form of an Initiation Grant (No. IITK/MET/2021324) is deeply thanked.

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