A Sustainable Green Approach towards the Production of High Purity Alumina from Waste Aluminium Dross

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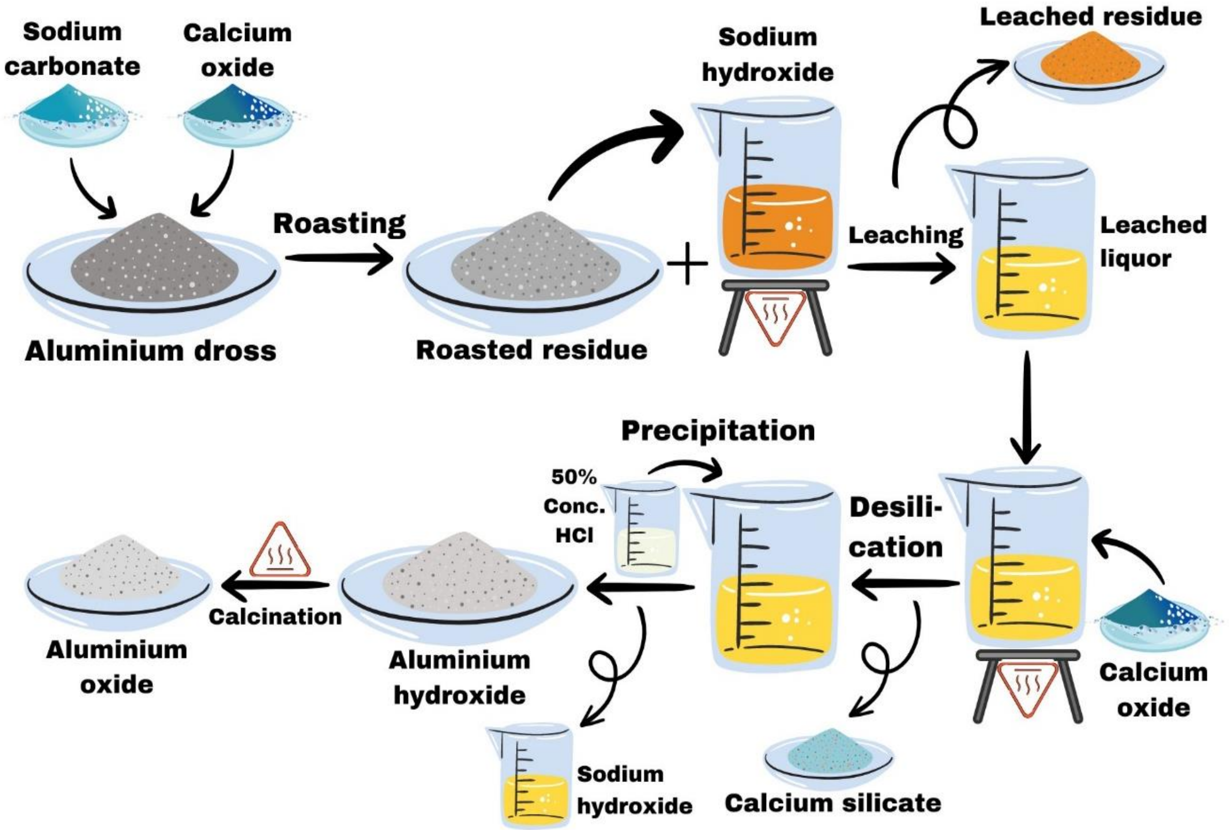
Abstract

Solid waste encompasses various materials discarded from homes, industries and construction including organic, paper, plastic, metal, glass and hazardous waste. Recycling is crucial for environmental preservation, involving the collection, processing and conversion of waste into reusable materials, mitigating greenhouse gas emissions and conserving landfill space. Bauxite, treated with alkaline leaching, yields aluminium and derivatives. Accumulation poses environmental risks, leaching harmful substances. Despite hazards, it is a valuable alumina source. Processing is challenging; remelting with salts recovers metal. A complex method involves soda-roasting, alkali leaching and purification, with around 90% alumina recovery. Chemical recycling is a key advantage.

Resulting aluminium hydroxide is primarily bayerite. The aluminium hydroxide was then put through calcinations, resulting in the formation of high purity alumina, aluminium chloride, aluminium sulphate water purifiers, steel making accelerators, building materials, refractory materials, composite materials etc.

The study involved examining the characterization of SAD and its refined products through a range of analytical techniques including thermodynamic analysis, X-ray diffraction analysis, X-ray fluorescence spectroscopy, Fourier transform infrared analysis and chemical analysis respectively. The results show that aluminium dross is not only be used only to efficiently extract high purity alumina compounds, but also to effectively achieve the green disposal of toxic components in SAD and to achieve its high-value utilization and thereby avoiding stockpiling.

Keywords: Aluminium dross, Sustainable green approach, Aluminium hydroxides and oxides.



Graphical Abstract

Introduction

Aluminium is a widely used industrial material. According to the World Primary Aluminium Production Report, the world primary aluminium production reached 67.243 million tons in 2021, of which China's primary aluminium production accounted for 57.79% of the total global primary aluminium production[1]. Using Bayer's procedure, an alkali leaching step, bauxite, an oxidic mineral, is primarily converted into aluminium and associated compounds[6]. Aluminium stands out as the most extensively employed non-ferrous metal, exhibiting remarkable performance and boasting a vast array of applications. A significant volume of industrial solid waste, known as aluminium dross, is generated from diverse processes involving primary aluminium smelting, casting, aluminium alloy production and the recycling of scrap aluminium[7].

Generally, dross is produced naturally when molten aluminium oxidises. When the metal encounters air, aluminium oxide is created on the melt's outer surface. Aluminium dross is commonly categorized into two groups: primary aluminium dross (PAD) and secondary aluminium dross (SAD). PAD is primarily derived from primary smelters and contains a significant amount of metallic aluminium[11]. On the other hand, SAD is acquired by removing metallic aluminium from PAD through specialized processes[5]. SAD exhibits characteristics such as reduced density, finely divided particle size and a significant specific surface area[2]. These distinct categories play a crucial role in recycling and waste management within the aluminium industry. Understanding their composition and characteristics is essential for developing efficient recycling techniques and promoting sustainability.

According to an acceptable estimate, India produces 75,000 tonnes of dross annually, the majority of which is either stored since there are no effective ways to deal with it or used to make crackers, impure chemicals and substandard refractory bricks[9]. This concerning trend not only poses significant environmental challenges but also signifies a missed opportunity to explore sustainable and innovative ways to manage this waste product. Addressing this issue could pave the way for developing eco-friendly solutions, promoting cleaner production processes and reducing the overall environmental impact.

The following primary reaction is involved in dross formation:

$$4Al + 3O_2 \rightarrow 2Al_2O_3 \quad (1)$$

During the formation of SAD, a considerable quantity of aluminium nitride (AlN) is generated as molten aluminium combining with nitrogen of the atmosphere[4]. This AlN compound exhibits reactivity with moist air, even when exposed to room temperature. AlN hydrolysis is a chemical process that yields colourless and highly toxic NH_3, emitting a pungent odour.

$$2Al + N_2 \rightarrow 2AlN \quad (2)$$

$$2AlN + 3H_2O \rightarrow Al_2O_3 + 2NH_3 \quad (3)$$

Detecting NH_3 at low concentrations poses significant challenges, yet prolonged inhalation of this noxious gas poses severe risks to public health. Moreover, NH_3 has the propensity to react with acidic gases in the atmosphere, resulting in the creation of ammonium aerosols, which exacerbate atmospheric pollution. The combination of these factors underscores the importance of understanding and mitigating the impacts of AlN hydrolysis, as it has implications not only for immediate health concerns but also for broader environmental considerations related to air quality and pollution control[3]. Additionally, allowing unregulated storage of SAD leads to the release of Cl and soluble F ions into both the soil and subsurface water. Coupled with its fine particle size, this practice gives rise to airborne dust pollution that can be inhaled. The repercussions are dire, posing significant threats to both public health and the environment.

As a result, the majority of Nations Currently categorise SAD as a hazardous solid waste. As people's consciousness of environmental protection has grown, there has been a lot of focus on the safe handling and efficient use of SAD[8].

The recycling and disposal of dross resulting from the melting of aluminium are pressing global concerns. The majority of this dross is currently being discarded in landfills, posing a serious risk of toxic metal ions leaching into groundwater, thereby exacerbating environmental problems. Furthermore, when aluminium dross comes into contact with water, it releases harmful gases like NH_3, CH_4, PH_3, H_2, H_2S and more, compounding the potential adverse effects[10-12]. Addressing this issue demands urgent attention and innovative solutions that prioritize eco-friendly recycling methods and sustainable management practices to safeguard our environment and future generations from the detrimental consequences of improper dross handling[21]. Remelting the dross using salt flux assists in minimising oxidation and removes the remaining metal component[28].

The oxide in the dross takes the shape of a long, continuous net that traps aluminium. This structure is also broken by the molten flux, which also helps the aluminium drops coalescence as they sink to the aluminium bath. Most of the time, a salt bath is used for maximising aluminium recovery[13]. It is crucial to create efficient recycling infrastructure and programmes as well as to increase awareness of the advantages of recycling among people, organisations and Governments in order to promote the recycling of solid waste[18]. Here, we are going to discuss about the solid aluminium dross and the method to produce high purity alumina from that waste dross.

Nevertheless, this study focuses on examining the exact dissolution characteristics of waste dross in base NaOH to identify optimal conditions for maximizing alumina recovery. The research aims to establish the waste dross as a valuable aluminium rich resource, thereby mitigating the need for stockpiling. The development of adequate treatment facilities for industrial wastes has been driven by the rising demand for valuable resources and the enforcement of environmental standards. Additionally, it has become necessary for sustainable development to replace main resources with secondary ones[14].

Material and Methods

Chemicals and reagents: Secondary aluminium dross (SAD), sodium carbonate (Na_2CO_3), calcium oxide (CaO), sodium hydroxide (NaOH), hydrochloric acid (HCl) are utilized in the present work. All reagents and chemicals, sourced from Merck, Germany, were of analytical reagent grade and were obtained from a local supplier.

Characterization of the materials: The synthesized materials underwent analysis to ascertain both their elemental composition and shape. To investigate the surface functional groups of SAD, leached residue, aluminium hydroxide and aluminium oxide, FTIR spectra were acquired using a Nicolet 6700 FTIR spectrometer from Thermo Fischer Scientific, USA. Additionally, the crystalline properties of the SAD, aluminium hydroxide and aluminium oxide were determined through powder XRD analysis using a D8 Advance Eco Make: Bruker, Germany.

Composition of Dross: Secondary aluminium dross is a byproduct that emerges during the process of melting aluminium. Its composition exhibits variability owing to the diverse alloying materials utilized during the re-melting of primary dross[17]. For this particular study, the researchers collected dross from a local producer, finding that it is primarily composed of alumina, with SiO_2 and also contains oxides of Mg, Ca and Fe. Characterization studies of aluminium dross were conducted utilizing various analytical techniques including chemical analysis, X-ray fluorescence spectroscopy, X-ray diffraction and Fourier transform infrared (FTIR).

Understanding the presence of different forms of alumina and other metal oxides compounds is essential for optimizing production processes, as well as for ensuring the recovery, recycling, quality and purity of the final aluminium product as it poses a significant environmental challenge due to its composition and abundance. Effective management of this waste is crucial to minimize its environmental impact.

Experimental processes: The methods comprise of multiple stages, beginning with soda-roasting followed by diluted alkali leaching, precipitation, calcination and a series of purification steps.

(i) Roasting: The roasting process of dross with sodium carbonate and calcium oxide converts the aluminium containing waste material into sodium aluminate, calcium aluminate and calcium silicate. The mixtures were uniformly ground in an agate mortar and were placed in crucibles and then, subjected to a temperature of 1200^0C for a duration of 1 hour in a Muffle furnace and after that the resulting residue could be processed to obtain pure aluminium oxide, contributing to the recycling and recovery of valuable resources from aluminium production waste. Precise control over the proportion of these raw materials was crucial to ensure the desired compounds formation during the roasting process. Based on the Al and Fe composition of SAD, the mass ratio of Na_2CO_3 to SAD was determined to be 80%.

Additionally, considering the Si and soluble F composition of SAD, the mass ratio of CaO to SAD was chosen to be 2%. The important reactions involved in roasting process are as follows:

$$2Al_2O_3 + 3Na_2CO_3 + 3CaO \rightarrow 2NaAlO_2 + 3CaAl_2O_4 + 3CO_2 \quad (4)$$

$$SiO_2 + 2CaO \rightarrow Ca_2SiO_4 \quad (5)$$

The AlN present in SAD underwent a reaction with Na_2CO_3 during the roasting process, resulting in the formation of $NaAlO_2$ and non-harmful N_2. Meanwhile, the soluble NaF combined with CaO during roasting, leading to the creation of insoluble CaF_2 in the leaching residue. This approach not only accomplishes the eco-friendly disposal of hazardous substances in SAD but also optimally extracts valuable Al from SAD, enabling its high-value utilization.

(ii) Leaching: During the leaching process, the initial step involves immersing the waste aluminium dross in a leaching solution, typically composed of either acidic or alkaline properties. This solution has the ability to selectively dissolve the alumina present, while the impurities remain in the solid residue. As a solvent, the leaching solution effectively disintegrates the alumina into a soluble state, facilitating its separation from the remaining dross components. In this experiment, the roasted mass was subjected to leaching with 20% solid to liquid alkaline solution. The reactor was positioned on a magnetic surface and stirring was achieved using a magnetic stirrer. The leaching process took place at temperatures ranging from 80^0C to 90^0C with a duration of 1.5 hours. Thus, 1M NaOH solution is heated with continuously stirring so as to raise its temperature to 90^0C.

After achieving the required temperature, the roasted sample is mixed with the NaOH solution, then the initial and final pH are recorded. Following the completion of the reaction, the slurry mixture was subjected to filtration and the remaining solid was meticulously washed 3 to 4 times with hot water. The extracted solution obtained through leaching was subsequently examined for its aluminium and silica content and the percentage of alumina recovery was determined using established procedures.

Notably, the dominant element present in the leaching residue was active Ca_2SiO_4 which holds potential for utilization in cement production, thus offering an eco-friendly alternative to solid waste disposal[15,16,20,21].

(iii) Desilication: Desilication process using CaO, also known as quicklime or lime, in the production of pure alumina from waste aluminium dross is a chemical procedure aimed at removing excess silica (SiO_2) impurities from the liquor formed after leaching to obtain high purity alumina. The reaction between CaO and SiO_2 leads to the formation of calcium silicate ($CaSiO_3$), which is a slag-like material[19,20,22]. This calcium silicate slag can then be separated from the pure alumina through various separation methods such as settling or filtration. Laboratory experiments demonstrated that the most favourable conditions for this procedure involved employing 10 g/L of CaO at a temperature of 90^0C for a duration of 2 hours. Then, the slurry is filtered to separate out the calcium silicate residue and liquor is stored for further processes.

Under these conditions, approximately 92% of Si was successfully eliminated, with only a minimal co-precipitation of Al, amounting to 7%. In summary, the process of desilication plays a crucial role in eliminating silica impurities from waste aluminium dross. As a result, the extracted alumina achieves significantly higher purity levels, rendering it suitable for subsequent processing in the manufacturing of aluminium metal or for various high-end applications that demand pure alumina.

(iv) Precipitation: The utilization of 50% concentrated HCl in the precipitation of aluminium hydroxide plays a crucial role in transforming discarded aluminium dross into valuable alumina. The resulting alumina possesses wide-ranging industrial applications such as in the production of aluminium metal, ceramics and diverse materials[29]. The leached liquor obtained from the above leaching procedure is now being used to precipitate aluminium hydroxide $Al(OH)_3$ with the help of 50% conc. HCl at 90-100^0C with continuous stirring. The pH range of the precipitation of the aluminium hydroxide is in between 5.7 – 6.7. The main reactions involved in this process are as follows[23-26]:

$$NaAlO_2 + 4HCl \rightarrow NaCl + AlCl_3 + 2H_2O \quad (6)$$
$$AlCl_3 + 4H_2O \rightarrow Al(OH)_3 + HCl \quad (7)$$

After that, the white slurry solution was filtered and the residue was washed thoroughly for 3-4 times with hot water to remove excess of salts from the residue. The obtained liquor i.e. NaOH solution could be reused in the preceding leaching process.

(v) Calcination: Calcination refers to the method of subjecting a substance to elevated temperatures, with the purpose of eliminating impurities and moisture, ultimately leading to chemical or physical changes[29,30]. When it comes to aluminium hydroxide, calcination plays a crucial role in transforming it into alumina, which is the primary precursor for aluminium production. During the calcination of aluminium hydroxide, it is heated to temperatures around 1000-1200^0C, causing the release of water molecules and resulting in the formation of alumina.

The main reaction involves in this calcination process is given below:

$$2Al(OH)_3 \rightarrow Al_2O_3 + 3H_2O \quad (8)$$

Finally, the calcination stage during alumina production from waste aluminium dross plays a crucial role in converting aluminium hydroxide into its pure form, known as aluminium oxide or alumina[27]. This essential transformation turns the waste material into a valuable resource, fostering sustainable utilization of aluminium resources and minimizing environmental repercussions. All experiments were conducted at a laboratory scale. Each of the procedures, tests and analyses were meticulously performed within the controlled environment of a laboratory setting. This approach allowed for precise and detailed observations, ensuring that the results obtained were reliable and accurate.

Furthermore, this laboratory-based approach facilitated the exploration of different methodologies and the development of new techniques, laying a strong foundation for potential future research on a larger scale. In brief, a standard flowchart has been suggested to outline the entire aluminium dross processing procedure for alumina production. Figure 1 illustrates the sequence of operations required to treat aluminium dross.

Results and Discussion

Chemical analysis: Table 1 shows the chemical composition of the material consisting of alumina, silica and iron in the form of oxides obtained through chemical analysis. Furthermore, trace amounts of salts, like NaCl and KCl, were detected in the dross[30,32]. The particles comprising this dross vary in size, with larger ones ranging from 2 to 5 mm. These larger particles are mainly constituted with metallic aluminium or its alloys while the finer fractions consist predominantly of alumina (Al_2O_3)[2].

XRF analysis: Table 2 shows the elemental composition of SAD determined by using XRF. Apart from that, the nitrogen (N) content was assessed through chemical titration method[31]. Approximately 80% of SAD is comprised of aluminium (Al), oxygen (O) and nitrogen (N), with the remaining portion containing soluble fluoride, chloride and impurity elements[35].

XRD analysis: The mineralogical phases of the SAD were investigated by X-ray powder diffractometry (XRD). The XRD pattern of SAD (Figure 2a) suggests that the presence of the Al element can be observed in two main forms: metallic aluminium and a variety of compounds such as α-Al_2O_3, γ-Al_2O_3, β-Al_2O_3, $MgAl_2O_4$ and AlN.

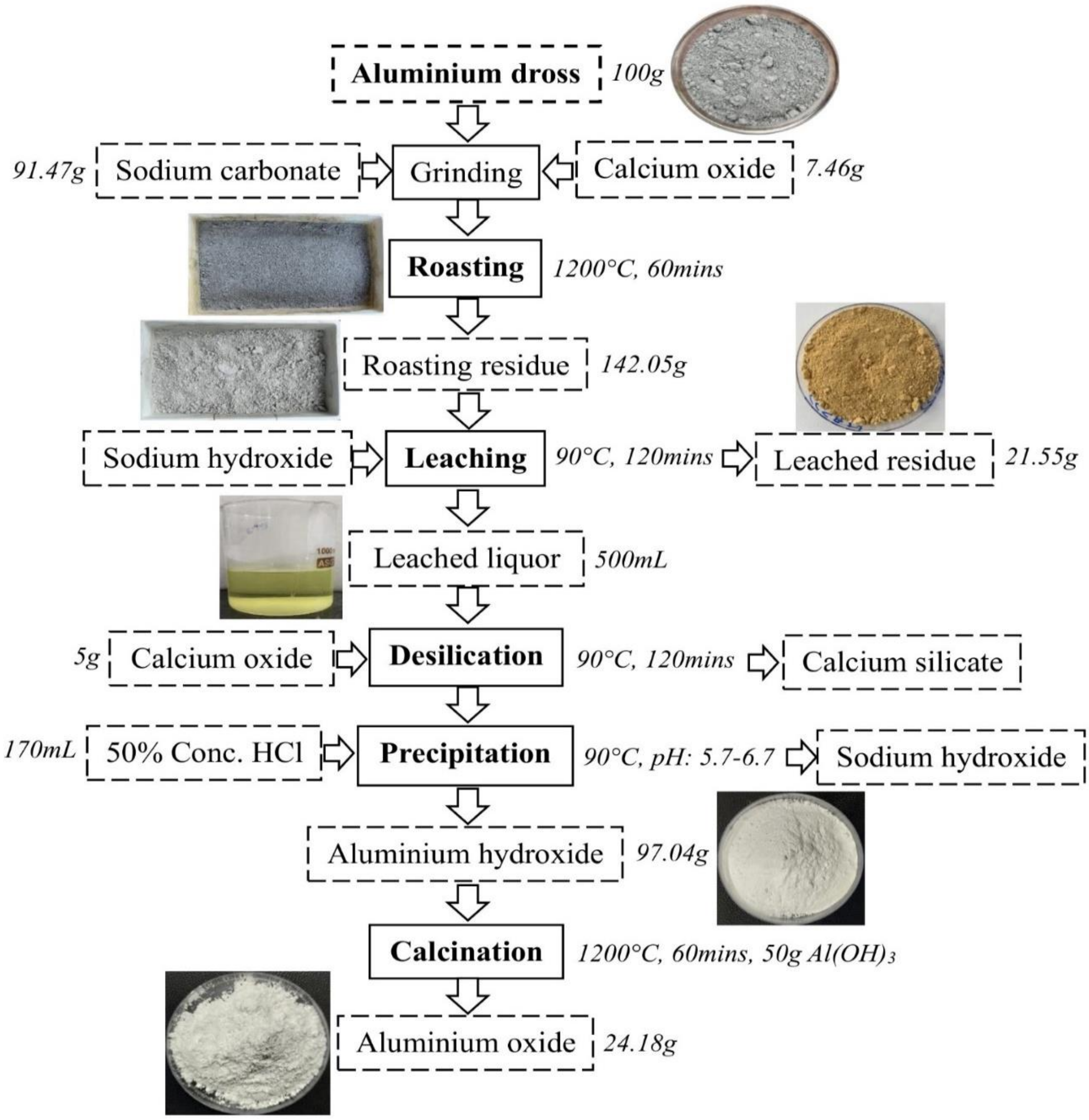


Figure 1: Flowchart for the preparation of alumina from SAD

Table 1
Composition of SAD by chemical analysis

Sample	Al_2O_3 %	SiO_2 %	Fe_2O_3 %
Aluminium Dross	80–90	4–6	1–2

Table 2
Composition of SAD by XRF

Element	Al	O	N	F	Na	Ca	Si	Mg	Fe	K	Cl
Content wt.%)	39.15	35.24	7.70	0.70	0.89	0.51	4.61	6.08	0.43	0.34	4.89

Table 3
Applications of the products formed

Products formed	Applications
Leached residue[26,27,34-36]	Cement Industry
Aluminium hydroxide[37-42]	Pharmaceuticals, Agate fillings, Paper Industry
Aluminium oxide[43-46]	Ceramic Industry
Sodium hydroxide[5]	Recyclable in leaching process

These compounds are the result of the reaction between molten aluminium and elements like oxygen, nitrogen and magnesium impurities. Additionally, there are traces of Fe and Si impurity oxides (Fe_2O_3 and SiO_2), as well as fluorides and chlorides (NaF, CaF_2 and NaCl). The generation of Fe_2O_3 and SiO_2 can be attributed to the oxidation of iron and silicon impurities during the production of primary aluminium.

The presence of chlorides can be attributed to the refining agent used during smelting, while the fluorides are likely derived from electrolyte inclusions[5]. Figure 2b and figure 2c are the XRD patterns of aluminium hydroxide and aluminium oxide respectively. It can be observed that the material exhibits a high crystallinity with characteristic peaks[33,34].

FTIR analysis: Secondary aluminum dross (SAD) typically comprises of aluminum oxides and other metal oxides, depending on the manufacturing process. The FTIR spectrum of SAD in figure 2d shows a notable peak at 569 cm^{-1} indicating the presence of various metal-oxygen bonds. This peak represents the primary functional group. Additionally, the broad band at 3390 cm^{-1} indicates the presence of hydroxyl groups, likely from absorbed water molecules or hydroxyl groups bonded to the metal centers.

In figure 2e, The FTIR spectrum of the leached residue reveals a peak at 600 cm^{-1} indicating the presence of various metal-oxygen bonds. This peak represents the most prominent functional group. Peaks observed between 1350-1600 cm^{-1}, while less distinct, may be linked to bending vibrations within the OH groups. Additionally, the broad band at 3410 cm^{-1} suggests the presence of hydroxyl groups, indicating O-H stretching likely from hydroxyl groups bonded to the metal centers.

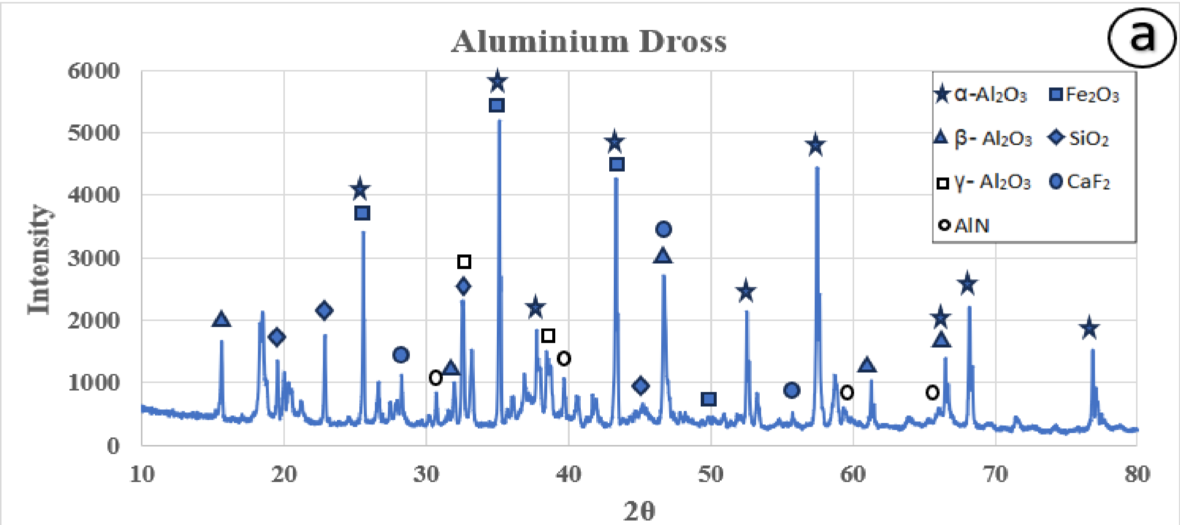


Figure 2a: XRD pattern of aluminium dross

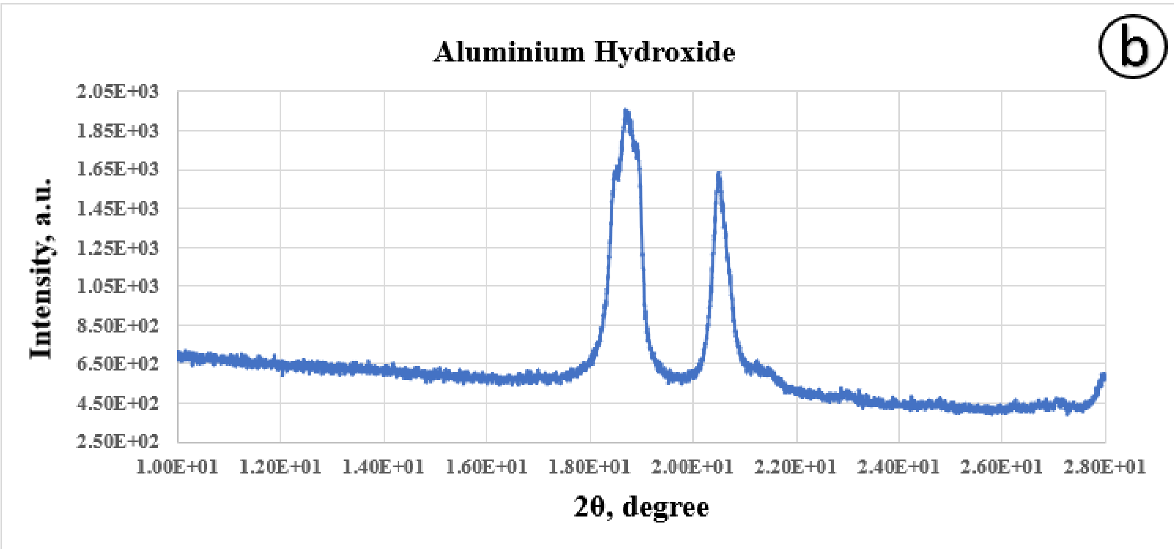


Figure 2b: XRD pattern of aluminium hydroxide

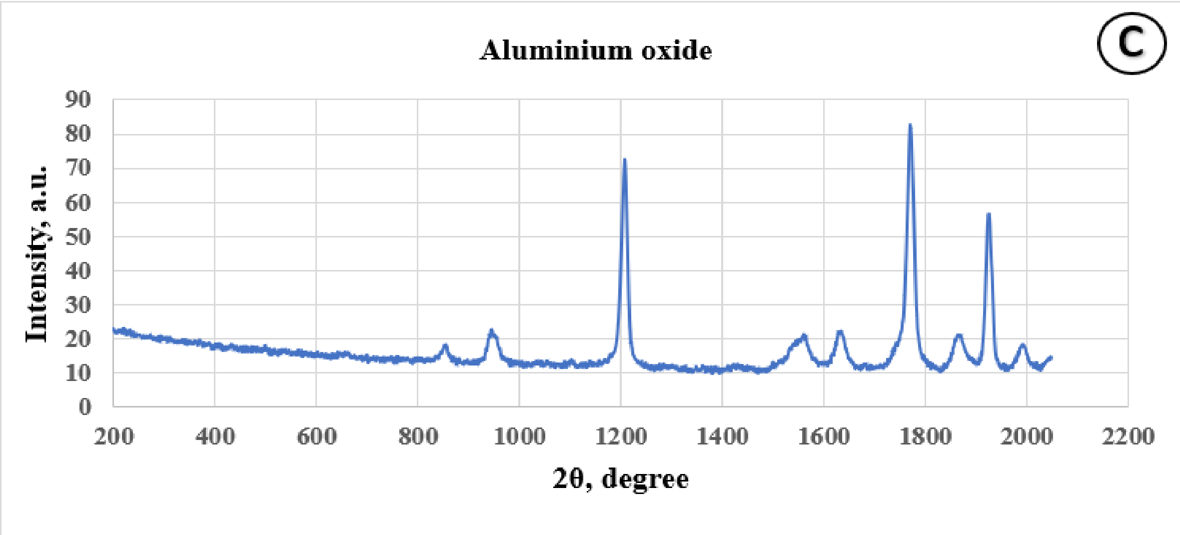


Figure 2c: XRD pattern of aluminium oxide

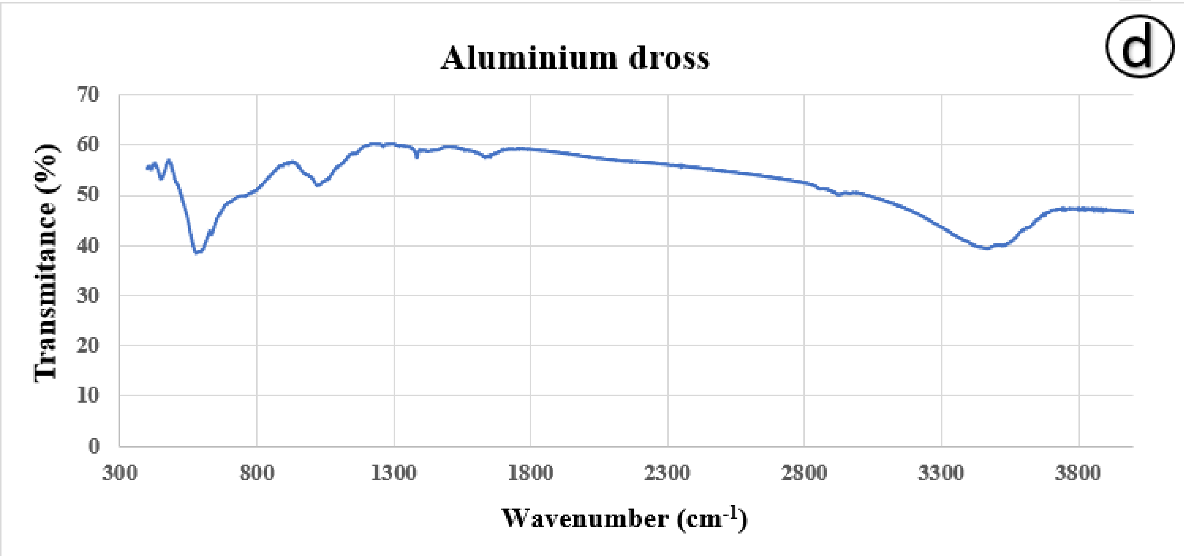


Figure 2d: FTIR spectra of aluminium dross

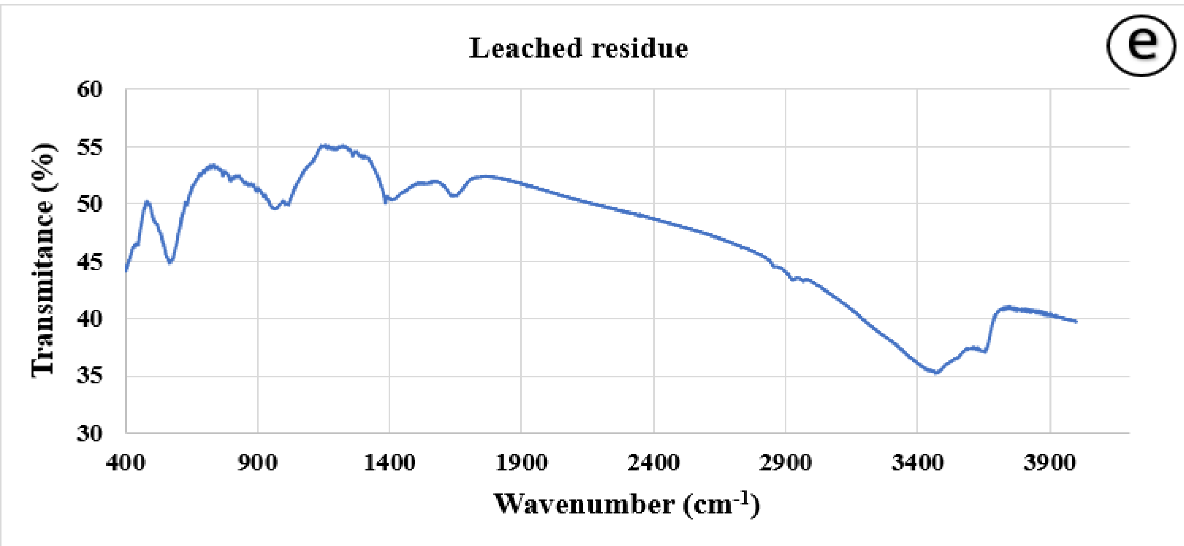


Figure 2e: FTIR spectra of leached residue

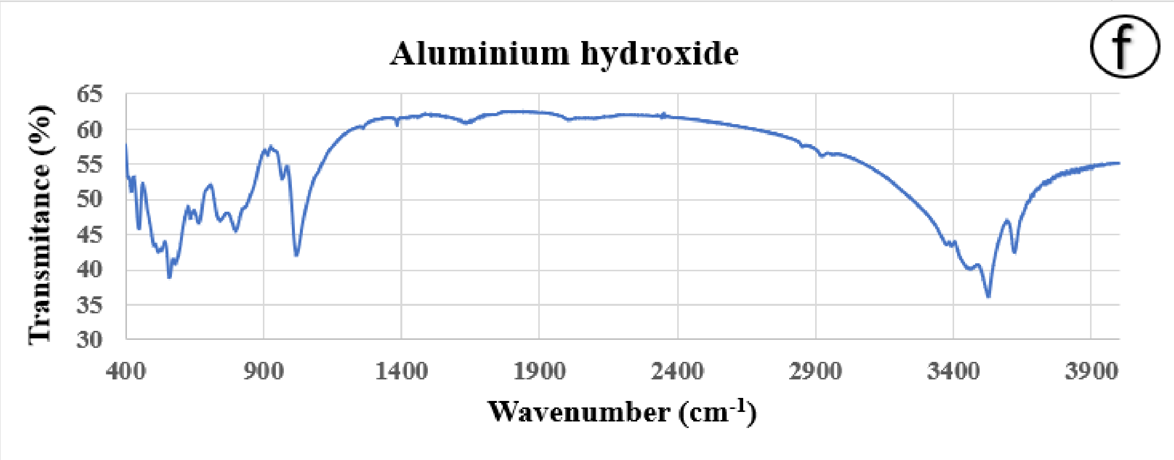


Figure 2f: FTIR spectra of aluminium hydroxide

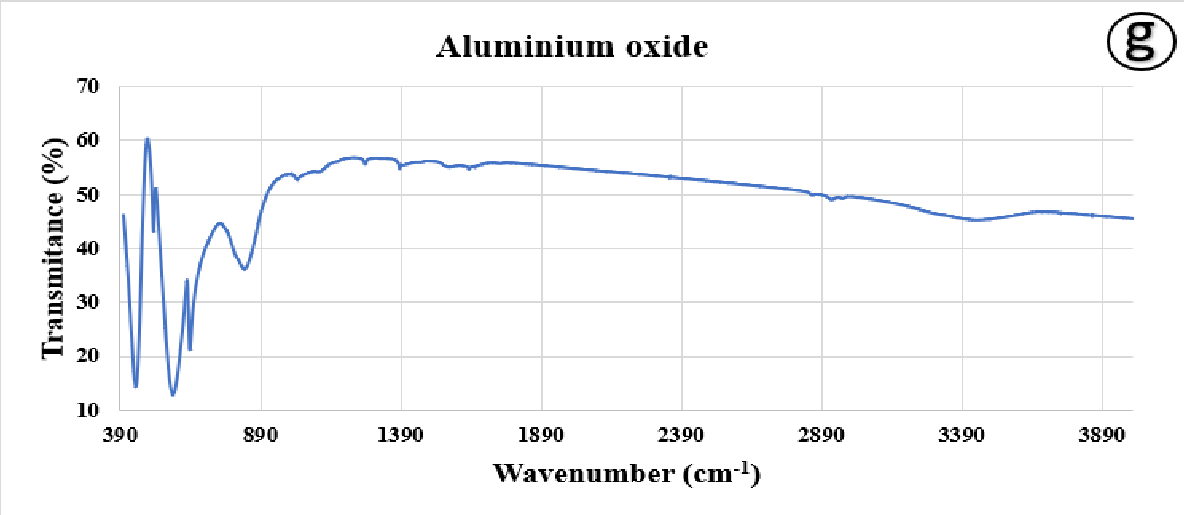


Figure 2g: FTIR spectra of aluminium oxide

The FT-IR spectra of $Al(OH)_3$ nanoparticles in figure 2f exhibit a peak at approximately 668 cm^{-1}, likely representing the Al-O stretching vibration, the primary functional group peak in aluminum hydroxide. In the Al-O-H bending region, bands were observed at 1080 cm^{-1}. Peaks at 1453 cm^{-1}, though less distinct, may denote bending vibrations within OH groups or skeletal vibrations involving Al-O bonds. The low-frequency band at 2530 cm-1 corresponds to OH stretching in water in the second coordination sphere of aluminum, while the 2965 cm^{-1} band relates to OH stretching in water coordinated to aluminum. The high-frequency band at 3580 cm^{-1} is attributed to OH stretching bound to two aluminum atoms.

FT-IR spectrum of synthesized aluminum oxide particles (Figure 2g) reveals distinct features. The peak at approximately 465 cm^{-1} signifies the asymmetric stretching of Al-O bonds while the peak at 590 cm^{-1} predominantly represents the symmetric stretching of Al-O bonds. Peaks within the range of 700-900 cm^{-1} are linked to the corner-sharing octahedral linkages of Al-O-Al bonds. Smaller frequency bands around 1100-1200 cm^{-1} are attributed to the asymmetric stretching of Al-O in hydroxyl groups. Additionally, the peak at 3480 cm^{-1} indicates the stretching vibration of water molecules present in the sample.

Applications: The active Ca_2SiO_4 compound discovered within the leaching residue exhibits highly favourable attributes that make it a compelling candidate for integration into the cement production process[36-40]. This breakthrough not only advances the field of construction materials but also aligns with global efforts to minimize the environmental impact of various industries. As we continue to explore innovative ways to utilize this resource, we pave the way for a more sustainable and responsible approach to cement production and waste management. The highly coveted high-grade white ultrafine aluminium hydroxide is renowned for its exceptional benefits arising from its expansive specific surface area and outstanding filling capabilities[23,33]. Its multifunctional characteristics render it indispensable across various industries with a significant role in pharmaceuticals where it enhances drug formulations and bioavailability[37,40,41].

Additionally, it finds widespread applications in the paper manufacturing sector, bestowing improved strength and texture upon paper products[43]. In high-temperature settings, its refractory properties make it a valuable component, ensuring durability and heat resistance[46]. Moreover, it adds both beauty and stability to decorative items during agate filling. Furthermore, when utilized as a toothpaste friction agent, it elevates dental care products, highlighting its pervasive impact on numerous applications. Ceramic stands out as an outstanding refractory material, displaying its exceptional capacity to endure elevated temperatures while maintaining its structural integrity[24]. This distinctive quality makes it a perfect candidate for utilizing α-Al_2O_3, a compound abundantly present in aluminium dross, to produce a diverse range of ceramic products[42,44]. These newly developed ceramics showcase remarkable durability and stability under extreme thermal conditions, surpassing conventional refractory bricks in both performance and cost-effectiveness[47].

Consequently, these advanced ceramics offer a compelling solution for various industries seeking reliable and efficient materials to withstand high-temperature environments[38,45]. Furthermore, it is worth noting that the resulting NaOH solution possesses inherent recyclability. This means that after its initial use in the leaching process, it can be effectively reintroduced into subsequent stages of the methodology. This recyclable attribute not only enhances the sustainability of the overall process but also contributes to cost-efficiency by reducing the need for fresh NaOH, making it an environmentally and economically sound choice for the extraction and separation processes[5]. The ability to reuse the NaOH solution underscores the importance of resource conservation and minimizes waste generation.

Conclusion

This study proposes an innovative approach to address issues related to solid and hazardous waste management by leveraging their unique properties and toxic characteristics. By extracting valuable elements from these wastes through eco-friendly techniques, it ensures safe handling and promotes responsible waste management. The method offers efficient management of various industrial wastes, reducing environmental hazards and fostering a circular economy by recycling and reusing valuable substances. A notable achievement is the successful extraction of high-purity alumina from waste aluminium dross, marking significant progress in resource recovery from industrial by-products. Implementing this reclaimed alumina in industries can decrease reliance on traditional raw materials, reducing environmental impact.

With the country facing challenges in managing escalating dross volumes, this study not only addresses environmental concerns but also offers a promising avenue for aluminium waste management. It presents a greener and economically viable solution, contributing to sustainable development and resource utilization. Further research and implementation of such innovative approaches are crucial for promoting environmental sustainability and mitigating the adverse effects of industrial waste.

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