Synthesis strategies of alumina from aluminum saline slags

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ABSTRACT

Aluminum saline slags is a waste of the metallurgical industry that presents serious environmental problems since it needs very extensive areas for its disposal, the toxicity it causes in the atmosphere and groundwater, in addition to high transportation costs. The valorization of this residue by the synthesis of alumina, a compound widely used in the chemical industry, generates a high impact and great interest. In this work, the strategies for synthesizing alumina from aluminum saline slags are reviewed in a context of growing demand for this metal and environmental crisis. The first sections present the aluminum production processes, both from natural bauxite (primary process) and from the recycling of materials with a high aluminum content (secondary process); paying attention to the waste generated and what environmental problems they produce. The main investigations that have allowed to address the recovery of the waste generated are described below, focusing on the processes of recovery/extraction of the aluminum present in its composition. The aluminum in these residues can be found as a metal or forming other compounds such as simple or mixed oxides. Chemical processes are the most relevant, especially those that deal with the acid and alkaline extraction of the metal. The most important section of the work reports on the methods of synthesis of Al_2O_3, highlighting the methods of precipitation, *sol-gel*, hydrothermal synthesis, and combustion, among others. The work ends with a summary and conclusions section.

1. Introduction

Aluminum, being the third most abundant element on Earth and the second most used metallic element after iron, cannot be found as a free element in nature (Abdulkadir et al., 2015). It is a non-ferrous metal, conductive, lightweight, malleable, water/smell-proof, easily alloyable and corrosion resistant with a strong affinity for oxygen (López-Delgado et al., 2009). It also has a low melting temperature ($\sim$ 660 ºC) and low density ($\sim$ 2.70 g/cm^3) (Gaustad et al., 2012). The need for new applications and improvements in mechanical properties has triggered a continuous development of new types of Al alloys with specific chemical compositions (Capuzzi and Timelli, 2018). Aluminum is generally produced from its oxide ores, primarily bauxite, refined by the Bayer leaching process, although there are other oxide feedstocks such as coal ash or clays that are currently being explored (Dash et al., 2008). The Hall-Héroult process was discovered simultaneously with the Bayer process and is based on obtaining aluminum from alumina by electrolysis (Nappi, 2013). Aluminum, unlike other metals, can be almost entirely recycled into melt products using tilting rotary furnaces and salts on an industrial scale (Tolaymat and Huang, 2016).

The demand for aluminum production is expected to increase in the next years. In 1990, 28 million tons of aluminum were produced and 8 of them were supplied from scrap recycling. In 2010, around 56 million tons of this metal were produced globally, recycling 18 million tons of scrap. In 2020 the annual world demand was expected to be 97 million tons and 31 million tons to be supplied from aluminum recycling (Mahinroosta and Allahverdi, 2018a; Meshram and Singh, 2018; Tsakiridis and Oustadakis, 2013).

Comparing with other materials, the aluminum industry is energy intensive with a notable difference between primary and secondary production (174–186 MJ/kg and 10–20 MJ/kg, respectively) (Abdulkadir et al., 2015). The reason by which secondary aluminum production consumes much less energy is because its raw material is aluminum scrap and primary aluminum metal (Tsakiridis and Oustadakis, 2013). That is, there are two current ways to manufacture aluminum. On the one hand, the primary production from alumina extracted directly from bauxite, and the secondary production from used aluminum. Recycling aluminum avoids 95% of emissions and requires only 5% of energy compared to producing it from bauxite (Hagelstein, 2009; Huang et al., 2014). The recovery of 1 kg of aluminum can save approximately four kg

of bauxite, 7.5 kWh of electrical energy and two kg of chemicals (AZO Materials, 2002; Barrera-Díaz et al., 2015; Hu and Bakker, 2015; Shinzato and Hypolito, 2005).

The global objectives, the government incentives and the ambitious energy objectives of the EU show the increase of a spirit towards the reduction of the consumption of natural resources and the reduction of pollution (Brough and Jouhara, 2020). It should be noted that recycled aluminum avoids the extraction of new raw materials and allows the manufacture of materials similar to those that would be produced from the original raw materials (López-Delgado et al., 2009). The raw materials used for the production of secondary aluminum are mainly made up of materials whose useful life has ended (López-Delgado et al., 2009). The accumulation of impurities is a major barrier in the material recycling industries. A growing number of studies and literature suggest that a number of unwanted elements from aluminum are highly problematic including but not limited to Si, Zn, Mg, Ni, Cu, Cr, Fe, V and Mn (Gaustad et al., 2012).

In a context of environmental crises and concepts of *Circular Economy*, the main objective of this study is to revise the newest and most innovative methods of recycling aluminum dross so that recycling this material is the only viable option for the scientific community.

2. Wastes from aluminum production

2.1. Aluminum slag

In general, the generation of waste and the depletion of natural resources are directly related to industrial production. These industrial wastes accumulate over time, causing very critical damage to the environment and public health (Xiao et al., 2005). Broadly speaking, waste materials can be classified into two broad categories: biodegradable waste and non-biodegradable waste (Mahinroosta and Allahverdi, 2018a). In addition, each type of waste has its physical and chemical properties and its value is determined by the amount of impurities and the cost of the recovery metal (Hwang et al., 2006).

Conventionally, there are two main categories of aluminum slag: (a) primary or white slag and (b) secondary or black slag (see Fig. 1). For 100 kg of molten aluminum, an average of about 15–25 kg of aluminum dross is generated (Mahinroosta and Allahverdi, 2018a). However, Al-dross is not completely a waste as it contains compounds of interest such as aluminum oxide (Al_2O_3) and magnesium aluminate ($MgAl_2O_4$) (Ramaswamy et al., 2019). About 1.5–2.5% and 8–15% of primary and secondary dross are produced annually per ton of molten metal, respectively. Therefore, a significant proportion of the aluminum slag is inappropriately landfilled (Meshram et al., 2020). In Europe, the

disposal of aluminum slag has been banned due to contamination of groundwater by soluble salts (Shinzato and Hypolito, 2005). The secondary aluminum industries in Europe and the United States have been forced to develop recycling technologies due to the increasing number of environmental laws (Mahinroosta and Allahverdi, 2018a; Tsakiridis and Oustadakis, 2013).

Recently, research has focused on the two forms of aluminum slag because their components are very similar to the constituents found at the very foundation of a refractory. White slag arises as a result of melting aluminum lumps for general use or recycling. On the other hand, black slag is the product of the fusion of aluminum (Ramaswamy et al., 2019). Secondary slag has a higher salt content and gas evolution than primary slag. The bulk density of the secondary slag is between 0.80 and 1.12 g/cm^3. Because metallic aluminum has a lower specific gravity than its oxide form, the greater the amount of metal in the slag, the lower the bulk density of the slag (Manfredi et al., 1997). Aluminum slag forms when molten aluminum comes into contact with air on the outer surface. Primary slag is typically recycled back to the smelter to recover residual aluminum metal by salt flux remelting to minimize oxidation (see Fig. 2). Sodium chloride and potassium chloride are salts used in rotary salt furnaces (Meshram et al., 2020). The extraction of the metallic aluminum and the separation of the non-metallic product is done thanks to the turning action of the rotary furnace (Drouet et al., 2000). The slag must be heated above the melting temperature of aluminum. Despite numerous studies on the effect of slag temperature, metal bath composition, salt addition or furnace rotation speed, it is still impossible to predict the recovery rate for a given slag. Duration of the rotating movement is important to achieve the transfer of energy from the refractory walls of the furnace to the load (Drouet et al., 2000). The
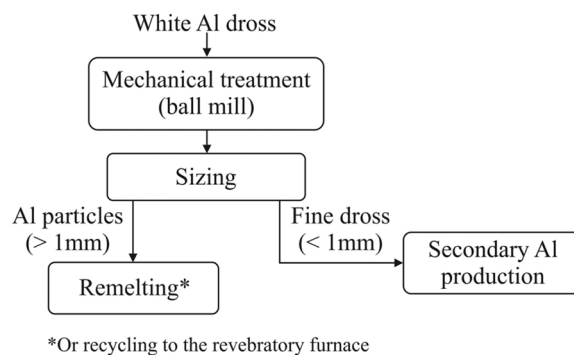
*Or recycling to the revebratory furnace

Fig. 2. Proposed treatment of white dross for Al recovery (Kudyba et al., 2021).
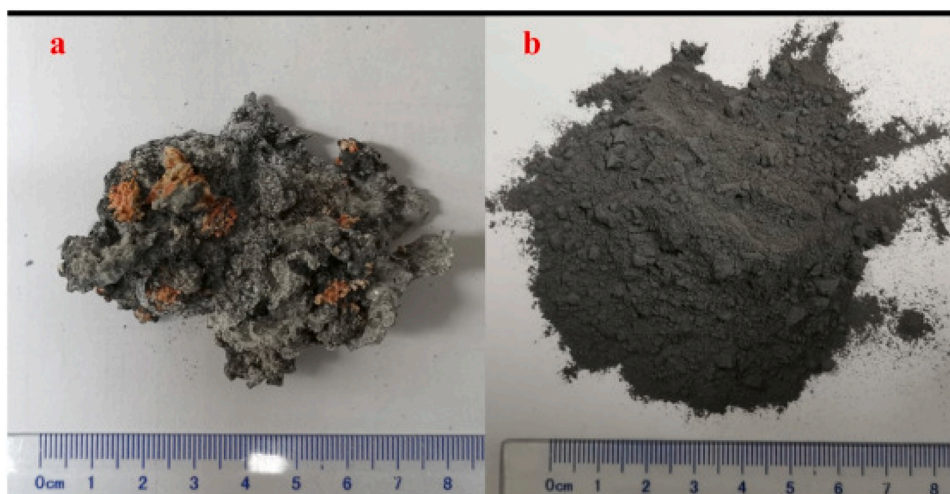
Fig. 1. (a) Primary aluminum dross and (b) secondary aluminum dross (Shen et al., 2021).

remaining residue, also called secondary slag or saline slag, is considered as hazardous waste and a rich source of aluminum (Soares and Romano, 2002; Mahinroosta and Allahverdi, 2018b; Matjie et al., 2005). During the secondary production of aluminum, a dust retained on the filters is generated. This type of waste is collected in filter sleeves mounted on the air extraction systems (Gil, 2005). The European Waste Catalogue (EWC) classifies this waste as hazardous (*Code 100321*) and its composition is similar to that of black slag (Galindo et al., 2015; Tsakiridis et al., 2016).

Aluminum slag, as oxidized material, contains a complex mixture with a variable composition but mainly consisting of Al_2O_3, quartz (SiO_2), Na_2O, Fe_2O_3, metallic aluminum, spinel $(MgAl_2O_4)$, diaoyudaoite $(NaAl_{11}O_{17})$, hibonite $(CaAl_{12}O_{19})$, aluminum oxide nitride (Al_5O_6N), fluorite (CaF_2), calcite $(CaCO_3)$, among others. Other components including AlN, NaCl, KCl, Si, cryolite (Na_3AlF_6), aluminum carbide (Al_4C_3), aluminum sulfide, MgF_2, $NaAlCl_4$, $KAlCl_4$, periclase (MgO), $KMgF_3$, and K_2NaAlF_6 have also been reported. Salts derived from NaCl, KCl and CaF_2 are originated after their use as molten salt fluxes (Dash et al., 2008; Mahinroosta and Allahverdi, 2018a; Kudyba and Safarian, 2022; Jafari et al., 2014; Scharf and Ditze, 2015; Tsakiridis and Oustadakis, 2013). $Ca(OH)_2$ is formed as a result of the hydration of CaO (impurity in raw material), and its partial carbonation results in the appearance of calcite (Tsakiridis and Oustadakis, 2013). Na_3AlF_6 has been identified by XRD as a minor crystalline phase. Corundum $(α-Al_2O_3)$ results from the reaction of aluminum with atmospheric oxygen at high temperatures. Aluminum nitride oxide can be formed when molten aluminum reacts with aluminum nitride. Spinel is the consequence of the oxidation in the melting process of the magnesium existing as an alloying element in aluminum scrap. In fact, the black color of the slag is due to the formation of spinel (Ozerkan et al., 2014). When molten aluminum reacts with carbon particles from organic contamination of aluminum scrap with paints and plastic coatings, aluminum carbide is produced. The presence of aluminum carbide content can be quantitatively estimated by knowing the amount of methane released from the salt slag (Xiao et al., 2005). The slag that has a quantity of metallic aluminum greater than 53% tends to form granular slag particles, while the formation of oxide predominates when the metallic content is lower (Manfredi et al., 1997; Meshram and Singh, 2018).

The typical chemical and mineralogical composition of secondary aluminum slag is presented in Table 1. This composition varies depending on the applied production process and the raw materials (Das et al., 2007; Ewais et al., 2009; Ozerkan et al., 2014; Lucheva et al., 2003, 2005; Mahinroosta and Allahverdi, 2018a; Sultana et al., 2013). Similar results were reported by Huang et al. (2015).

Aluminum disposal and recycling slag produced during the melting of aluminum is a global concern (Reddy and Neeraja, 2018). Various pollution problems are associated with the slag because it is disposed of in landfills causing toxic metal ions to leach into groundwater (Pandidharadhya et al., 2018). After the disposal of aluminum solid waste in landfills, many operational problems have been reported such as baghouse dust, which is a solid residue generated by air pollution control systems (Huang et al., 2015). An increase in landfill temperature from 60 to 93 ºC over periods ranging from several months to years has also been recorded (Tolaymat and Huang, 2015). Safely disposing of aluminum slag is a costly challenge for producing industries (Adeosun et al., 2014; López-Delgado et al., 2009). Furthermore, slag can generate high safety risks due to its toxicity and high flammability in case of improper recycling and reutilization (Murayama et al., 2012). For example, the inhalation of slag particles dispersed in the air can cause diseases such as silicosis and bronchitis (Adeosun et al., 2014; Xiao et al., 2005). The environmental impact generated by aluminum slag is highly detrimental. In terms of chemical reactions, when aluminum slag comes into contact with water, water vapor or moisture, it releases dangerous and explosive gases such as gaseous ammonia (produced from the hydrolysis of nitrides present in the salt cake), phosphine, methane, hydrogen disulfide, according to the following reactions: (Abdulkadir

Table 1

Chemical and mineral phases of saline slags from Secondary Aluminum Production (SAP). Total AlN included AlN, and AlN content in their oxidation products ($Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$ and $Al_{2.78}O_{3.65}N_{0.35}$). Total Al_2O_3 included Al_2O_3, and Al_2O_3 content in $Al_{2.85}O_{3.45}N_{0.55}$, $Al_{2.81}O_{3.56}N_{0.44}$ and $Al_{2.78}O_{3.65}N_{0.35}$ and $Al_{2.667}O_4$) (Das et al., 2007; Ewais et al., 2009; Ozerkan et al., 2014; Huang et al., 2014; Lucheva et al., 2003, 2005; Mahinroosta and Allahverdi, 2018a; Sultana et al., 2013).

Compound/element	wt%	Mineral Phases (%)	Mean
Al_2O_3	42–88	Al	3.1
SiO_2	1.3–14.0	AlN	4.6
Fe_2O_3	0.2–2.5	$Al_{2.85}O_{3.45}N_{0.55}$	11.3
CaO	0.6–1.0	$Al_{2.81}O_{3.56}N_{0.44}$	7.3
MgO	1.8–4.2	$Al_{2.78}O_{3.65}N_{0.35}$	5.8
MnO	0.11	Total AlN	8.0
Na_2O	0.11–2.75	Al_2O_3	7.5
K_2O	0.26–0.51	$Al_{2.667}O_4$	9.9
TiO_2	0.13–0.31	Total Al_2O_3	41
P	0.01	K_2NaAlF_6	3.1
S	0.22–0.26	$MgAl_2O_4$	8.6
Cu	0.05–0.63	MgO	1.4
Zn	0.18	NaCl	18
Cl	3.90	KCl	8.8
C	1.25	CaF_2	5.0
		$CaSO_4$	4.7
		SiO_2	1.5

et al., 2015; Bruckard and Woodcock, 2009; Calder et al., 2010; Galindo et al., 2015; Gil, 2005; Laue et al., 1994; Li et al., 2012; Liu and Chou, 2013; López-Delgado et al., 2009; Padamata et al., 2021; Shinzato and Hypolito, 2005; Yoshimura et al., 2008; Xue et al., 2022; Yang et al., 2022).

$$AlN + 3H_2O \rightarrow NH_3\uparrow + Al(OH)_3 \tag{1}$$

$$2Al_5O_6N + 3H_2O \rightarrow 5Al_2O_3 + 2NH_3\uparrow \tag{2}$$

$$AlP + 3H_2O \rightarrow Al(OH)_3 + PH_3\uparrow \tag{3}$$

$$Al_2S_3 + 6H_2O \rightarrow 2Al(OH)_3 + 3H_2S\uparrow \tag{4}$$

$$NH_3 + H_2O \rightarrow NH_3(aq) \tag{5}$$

Ammonia increases the pH value to 9, causing metallic aluminum to react with water, forming hydrogen and Al_2O_3. On the other hand, hot hydrogen reacts with oxygen in the air and combusts (Padamata and Yasinskiy, 2021).

$$2Al + 3H_2O \rightarrow 3H_2 + Al_2O_3 \tag{6}$$

When the aluminum carbide in the salt cake reacts with water, methane and Al_2O_3 are formed:

$$Al_4C_3 + 6H_2O \rightarrow 3CH_4\uparrow + 2Al_2O_3 \tag{7}$$

As a possible solution, these gases can be burned and the generated heat can be recovered (Lucheva et al., 2005), ammonia can be absorbed by H_2SO_4 and phosphine can be catalytically oxidized to produce phosphate fertilizers (Mahinroosta and Allahverdi, 2018a). After this

processing, the air expelled into the atmosphere shows ammonia and phosphine values below 10 and 0.08 ppm, respectively (Xiao et al., 2005). Another possible solution for managing these emissions is the determination of the slag by pickling liquor (removal of oxides by immersing the methane in inorganic acids, simultaneously avoiding problems of harmful gases and the corrosivity of the liquors) (Mahinroosta and Allahverdi, 2018a).

An increase in landfill temperature and pressure could cause aluminum carbide to react with nitrogen in the soil, generating aluminum cyanide. Aluminum cyanide is further hydrolyzed to produce HCN.

$$2Al_4C_3 + 6N_2 + 9C \rightarrow 4Al(CN)_3 \tag{8}$$

$$4Al(CN)_3 + 6H_2O \rightarrow 12HCN + 2Al_2O_3 \tag{9}$$
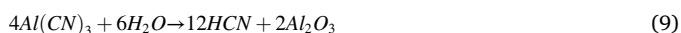
Tertiary aluminum treatment industries produce effluents that contain chemical species such as Na^+, Ca^{2+}, Mg^{2+}, K^+, NH_4^+ (Chieko and Raphael, 2016) and the problem lies when the effluents lead into water bodies. In the vicinity of aluminum treatment plants, the pH of groundwater drops to 4 due to ammonia decomposing bacteria increasing the percentage of aluminum in the form of $Al(OH)_3$ (Meshram and Singh, 2018). Therefore, the present laboratories have recently launched extensive research work on developing practical methods to recover catalytic-grade alumina (Al_2O_3) from aluminum dross tailings chemical waste (ADT).

Due to the increased use of designer products, the production of secondary aluminum has risen sharply (Meshram and Singh, 2018). New applications of aluminum slags have been used by investigations to synthesize other raw materials such as hydrogen gas (Nakajima et al., 2007), ceramic materials (Ewais and Besisa, 2018; Foo et al., 2019; Ibarra Castro et al., 2009; Verma et al., 2021), ceramic membranes (Aziz et al., 2019), alumina-pillared clays (Cardona et al., 2021), zeolites (Hiraki et al., 2009; Yoldi et al., 2020; Jiménez et al., 2021), composites, cement and concrete products, ion exchangers, generation of gases such as methane and ammonia (Meshram and Singh, 2018), adsorbents (Gil et al., 2018a), hydrotalcites (Gil et al., 2018b; Santamaría et al., 2022), synthesis of AlPO4-5 and CrAPO-5 (Kim et al., 2009), aluminum sulfate ($Al_2(SO_4)_3 \cdot 18\ H_2O$) (Sangita et al., 2018), catalysts (Benkhelif et al., 2022; El-Katatny et al., 2003), biomaterials (Sarker et al., 2015), La-hexaaluminates (Torrez-Herrera et al., 2021) and refractory materials (Adeosun et al., 2014; Lin et al., 2022; Maheswari et al., 2023). Shinzato and Hypolito (2005) recovered secondary aluminum slag leaching residues (non-metallic products-NMP and salts) for use in the manufacture of concrete blocks by adding two parts NMP to one-part cement and four parts sand. NMPs are composed of refractory and abrasive oxides such as α-Al_2O_3, $MgAl_2O_4$, SiO_2 and α-$Al(OH)_3$. In recent years, ceramic membranes have attracted a lot of attention as an alternative to polymeric membranes due to their robustness. Nowadays, ceramic materials such as alumina, zirconia and titania are very common materials in the manufacture of these new membranes. Tripathy et al. (2019) have proposed the use of aluminum residues to manufacture a new alumina-spinel composite hollow fiber membrane for use in pretreatment of produced water with oily saline solution. They used a water leaching of the powder from the alumina slag, using a 1:4 slag-water ratio for 8 days to remove the maximum of soluble impurities such as chloride salt, subsequently filter, dry the mixture at 100 °C overnight and calcined at 110 °C for 90 min to obtain Al_2O_3.

2.2. Salt flux and salt cake

The salt flux contributes in several aspects to the melting process such as promoting heat transfer to the metal, protecting the metal against the atmosphere, helping the distribution of oxides, metallic and non-metallic materials in the furnace, and some salt flux reacts with aluminum oxide and dissolve it in them (Gil, 2005; Xiao et al., 2005). In the primary slag smelting process, the amount of salt flux is equivalent to

the total content of oxides in the raw material and varies between 10% and 40%. In the scrap casting process, the amount of flux salt is equivalent to 30–50% of the weight of the recovered metal. In addition to recovering the metallic aluminum, it is also important to recover the salt flux (Mahinroosta and Allahverdi, 2018a). There are more than 100 varieties of fluxing salt compositions, although the most common has an equimolar NaCl-KCl base with 10% by weight of fluorinated additives such as CaF_2, NaF, Na_3AlF_6 and KF (Soares and Romano, 2002; Das et al., 2007; Dash et al., 2008; Scharf and Ditze, 2015; Xiao et al., 2005). The addition of these fluorides reduces the surface tension. The order of increasing efficiency of fluorides is as follows (Roy and Sahai, 1997; Van Linden and Stewart, 2013):

$Na_3AlF_6 < CaF_2 < NaF < KF$.
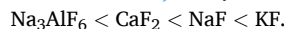
A large amount of salt is required in aluminum recovery processes from low-grade wastes, producing large amounts of salt cake or salt slag (Shinzato and Hypolito, 2005; Xiao et al., 2005). Typically, for every kilogram of low-grade waste, 1 kg of salt flux is needed in the rotary furnace (Nakajima et al., 2007; Shinzato and Hypolito, 2005).

3. Recovery of aluminum

Aluminum is an amphoteric metallic element capable of dissolving in both acid and alkaline solutions by formation of aluminum complexes. In general, there are two approaches to treating secondary slag: hydrometallurgical processes and pyrometallurgical processes (Mahinroosta and Allahverdi, 2018a; Srivastava and Meshram, 2023). Pyrometallurgical processes have a high energy consumption while hydrometallurgical processes are a promising way of recovering secondary slag and are generally carried out through alkaline or acid routes (Dash et al., 2008; Shi et al., 2023; Sultana et al., 2013; Tsakiridis and Oustadakis, 2013; Yoldi et al., 2019) (see Fig. 3). The recovery of aluminum in hydrometallurgical processes is complex because it is often recovered as aluminum hydroxide or alumina (Karuppuswamy et al., 2016). These methods have been developed as alternatives to the Bayer process in those countries with limited bauxite sources (Mahinroosta and Allahverdi, 2018a). The first method patented in the 1970s for treating salt cakes involved the use of paraffins. The primary slag was cooled with water or naturally, then ground and re-melted together with the addition of a salt flux (Papafingos and Richard, 1978). At this point, depending on the interest of the producing factory, other aluminum
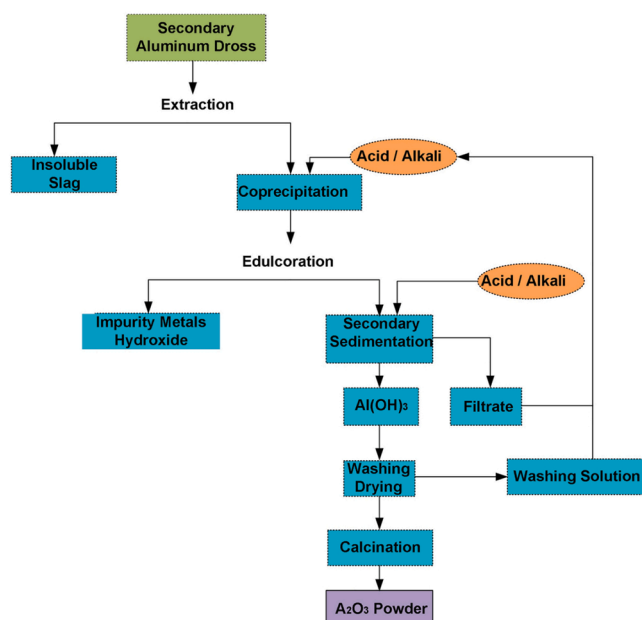

Fig. 3. Preparation of Al_2O_3 by hydrometallurgy (Huang and Yi, 2023).

alloy elements can be added. The solid residue known as black slag or salt cake is deposited in landfills (Lazzaro et al., 1994).

In any hydrometallurgical process, the elimination of soluble salts from the slag must be considered. During leaching, these salts go into solution and slow down the reaction rate. Significant changes have been reported in solutions in which salts have been washed out (Das et al., 2007; Dash et al., 2008; Teodorescu et al., 2014). Failure to wash the slag first results in contamination of the final aluminum hydroxide (Mahinroosta and Allahverdi, 2018a). In the event that the slag is washed at 80 °C for 1 h, 90% of the salt it contains will be eliminated. To achieve approximately 100% salt removal, a two-stage rinse at 80 °C is required (Das et al., 2007).

3.1. Alumina extraction

Alumina is one of the most widely used ceramic materials as catalyst, catalyst support and as a resistant coating (Farahmandjou et al., 2016). It is used industrially as a raw material in areas such as aerospace, automobile, construction, and semiconductor manufacturing (He et al., 2010; Liao et al., 2020; López-Alonso et al., 2019; Shanmugaselvam et al., 2020). Moreover, it is considered as one of the most important materials in the world for future development, for example in adsorbents or nanomaterials due to its specific surface area and high purity (Asencios and Sun-Kou, 2012; Davoodi et al., 2020; Galaburda et al., 2020; Zhao et al., 2020). The aluminum oxide exhibits the form of a long continuous network where the metal stays entrapped (Tripathy et al., 2019). Theoretically, Al_2O_3 contains approximately 53% aluminum, that is, the fractions with a lower content of aluminum are mainly composed of oxide. So, if the aluminum content is above 53%, metallic aluminum is present in the slag fraction (Manfredi et al., 1997).

Alumina (Al_2O_3) can exist in many crystalline phases although only two of them are commercially available. Among these polymorphs, α-Al_2O_3 (corundum form) is the only thermodynamically stable crystalline phase. It has excellent properties and a Mohs hardness of 9.0, which makes it a great candidate for electrical, structural and refractory applications (López-Delgado et al., 2009; Lumley, 2011; Tsakiridis and Oustadakis, 2013). The other commercial form is γ-alumina which is used primarily for catalyst and filtration applications. It is a metastable material that is easily transformed into several alumina phases by applying temperature (López-Delgado et al., 2009; Mahinroosta and Allahverdi, 2018a). The metastable Al_2O_3 structures can be divided into two broad categories: a face-centered cubic (fcc) or a hexagonal close-packed (hcp) arrangement of oxygen anions (Levin and Brandon, 1998). All these metastable phases irreversibly transform into alpha alumina (α-Al_2O_3) if heated above 1200 °C. Submicron and nano-sized α-alumina has a great demand in catalysis as a thermally stable alternative to transition alumina, which undergoes phase transformations at high temperatures (Benkhelif et al., 2022). α-alumina crystallizes in the form of a close-packed hexagonal structure of oxygen ions and exhibits high thermodynamic stability (Piconi et al., 2014). Gibbsite, $Al(OH)_3$, and boehmite, $AlO(OH)$, are the most widely used precursors for its preparation (Wefers and Misra, 1987).

The polymorph properties of the Al_2O_3 particles are significantly affected by the calcination conditions, i.e., time and temperature (Türk et al., 2020). During the heat treatment, the aluminum hydroxides are transformed into metastable forms of alumina (χ-, κ-, γ'-, γ-, δ-, θ-, η- and ρ-Al_2O_3) before obtaining the thermodynamically stable phase α-Al_2O_3 (Lamouri et al., 2016) (see Fig. 4).

Due to environmental regulation policies, the need for a world economy to recover aluminum and the demand for valuable materials, researchers are looking for several applications of alumina-rich materials (Mahinroosta and Allahverdi, 2018a) such as acid-base leaching processes (Li et al., 2014), lime sintering methods (Wahab et al., 2014) or sintering of soda and lime (Pedersen, 1927) for extracting alumina from solid wastes.

Currently most of the world's commercial alumina is produced by the Bayer process using bauxite as raw material (Manivasakan et al., 2009). Bayer process is the primary process by which alumina is extracted from bauxite ore and separated from red mud (Capuzzi and Timelli, 2018), which is a waste by-product from alumina extraction from bauxite with caustic soda in the Bayer process (Yang and Xiao, 2008). Bayer process is a fairly selective process for aluminum because iron is almost insoluble in alkaline solutions, but when treating aluminosilicates, silica can only be removed at the expense of extracted aluminum (Sarker et al., 2015). On the other hand, if aluminum is introduced into an aqueous solution as Al^{3+}, the silica remains substantially insoluble in the acid pathways but, however, it is not a selective leaching process for aluminum as iron, potassium, magnesium, sodium and calcium are generally co-dissolved (Hussain and Jamal, 2000; Mahi et al., 1985).

The leaching process from aluminum slag has been reported with both acid or alkaline routes due to its amphoteric nature (Feng at al, 2020; Kelmers et al., 1982; Sarker et al., 2015; Yang et al., 2019). Various chemicals (HCl, $NaOH$, H_2SO_4, NH_3, KOH) are used for leaching or as precipitating agents depending on the desired conditions (Türk et al., 2020; Dash et al., 2008; David and Kopac, 2012; Tsakiridis and Oustadakis, 2013). A disadvantage of digestion is that iron is also leached (unless there is little iron slag) and it is difficult to separate iron from aluminum hydroxide (Tripathy et al., 2019). Technically, an acidic process might be more attractive than an alkaline process due to the low iron content and high silica content of aluminum ash (Sibanda et al., 2016). In the case of alkaline leaching, the recovery of the metallic part of aluminum only takes place. Knowing that the amount of metal in the slag is low compared to its oxide, the alkaline processes are less efficient with respect to the complete utilization of the slag (Tripathy et al., 2019). In the leaching with sulfuric acid, aluminum sulphate is
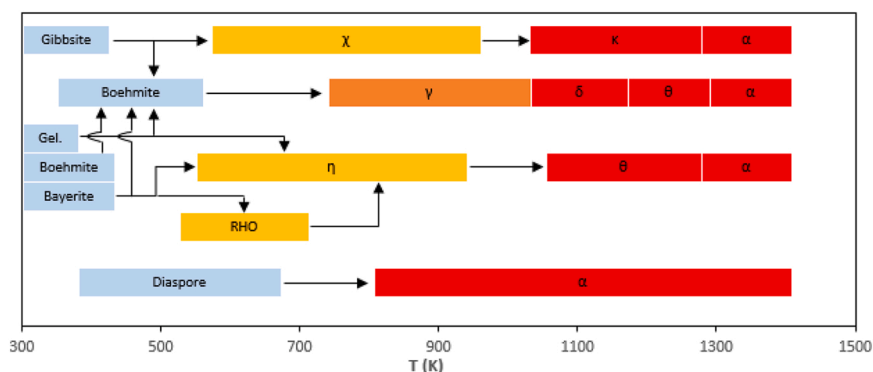

Fig. 4. Alumina phases formed, under thermal treatment, from the (oxi)hydroxides of this element (Lamouri et al., 2016; Wefers and Misra, 1987; Trejo-Trejo et al., 2021).
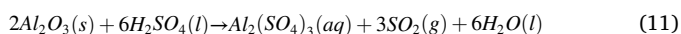
produced, which often contains impurities (Tripathy et al., 2019). With 15% sulfuric acid, 85% of the alumina present in the slag can be leached (Dash et al., 2008) and with alkaline leaching, high purity alumina extracted (Asencios and Sun-Kou, 2012). However, achieving a high alumina extraction rate and high purity at the same time is only achieved with a few methods because of the silica and salt contents of the slag (He et al., 2021). The investigations carried out on the leaching of aluminum from slag in an acid medium and in an alkaline medium are summarized in Table 2.
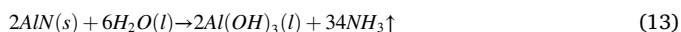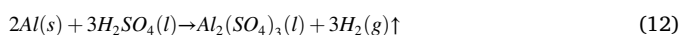
3.1.1. Acid route

Several acid routes have been reported for the recovery of alumina from aluminum slag. H_2SO_4, HCl, HNO_3 or H_2SO_3 have been used as agents in the acid route leaching process (Mahinroosta and Allahverdi, 2018a; Jiménez et al., 2022). The most versatile and most available acid is hydrochloric acid, since it is extracted as by-products in many industrial processes (Sarker et al., 2015). The study of the leaching kinetics of aluminum slag under acid extraction with HCl revealed that the removal of particles was controlled by diffusion in layers presenting an apparent activation energy of around 10.5 kJ/mol. The dissolution data presented a best fit with the following kinetic model (Mahinroosta and Allahverdi, 2018b):

$$[1 - (1 - \alpha)^{\frac{1}{3}}]^2 = 0.0449 \cdot e^{\frac{-10490}{RT} \cdot t} \tag{10}$$
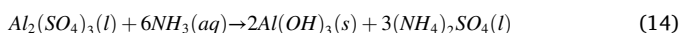
The following reactions take place during sulfuric acid leaching (How et al., 2017) (although in some cases Al^{3+} may be in solution, forming the soluble complexes stables at a given pH, as $[Al(H_2O)_6]^{3+}$ or $[Al(OH)_4]^-$, all the reactions are balanced in molecular form):

$$2Al_2O_3(s) + 6H_2SO_4(l) \rightarrow Al_2(SO_4)_3(aq) + 3SO_2(g) + 6H_2O(l) \tag{11}$$

During the reaction, flammable and toxic gases are released:

$$2Al(s) + 3H_2SO_4(l) \rightarrow Al_2(SO_4)_3(l) + 3H_2(g)\uparrow \tag{12}$$

$$2AlN(s) + 6H_2O(l) \rightarrow 2Al(OH)_3(l) + 34NH_3\uparrow \tag{13}$$

Ammonia solution and leached liquor react in the following way:

$$Al_2(SO_4)_3(l) + 6NH_3(aq) \rightarrow 2Al(OH)_3(s) + 3(NH_4)_2SO_4(l) \tag{14}$$

During calcination process:

$$2Al(OH)_3(s) \rightarrow Al_2O_3(s) + 3H_2O(l) \tag{15}$$

A possible acid route using hydrochloric acid could be the following (Adans et al., 2016; Kozakova et al., 2015; Li et al., 2013; Mahinroosta and Allahverdi, 2018c; Ohki et al., 2004; Raschman et al., 2010; Raschman and Fedoročková, 2008):

$$2Al(s) + 6HCl(aq) \rightarrow 2AlCl_3(aq) + 3H_2(g)\uparrow \tag{16}$$
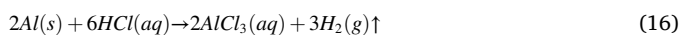
After the leaching process, all the possible reactions in the first stage are as follows:
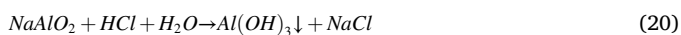
$$M_xO_y + 2_yHCl \rightarrow xMCl_{2y/x} + yH_2O \; (M : metal cation) \tag{17}$$
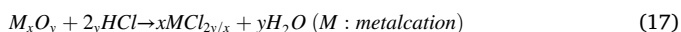
During the second stage, a hybrid hydroxide precipitate is formed from the metal chlorides according to the reaction:

$$MCl_y + yNH_3(aq) \rightarrow M(OH)_y\downarrow + yNH_4Cl \tag{18}$$

NaOH is added in order to separate $Al(OH)_3$ from the other metal hydroxides:

$$Al(OH)_3 + NaOH \rightarrow NaAlO_2 + 2H_2O \tag{19}$$

$Al(OH)_3$ is re-precipitated by adding HCl:

$$NaAlO_2 + HCl + H_2O \rightarrow Al(OH)_3\downarrow + NaCl \tag{20}$$

Dehydroxylation was performed through calcination:

$$2Al(OH)_3 \rightarrow Al_2O_3(aq) + 3H_2O \tag{21}$$

In the study carried out by Roslan et al. (2019), γ-Al_2O_3 was extracted from aluminum slag by acid leaching with 2 M sulfuric acid with a slag-acid ratio of 1:4 for 1 h, 70 °C and ignition speed of 200 rpm. Subsequently, a 10% ammonia solution was added dropwise to the solution to reach pH = 9. The precipitate was collected, washed, dried at 60 °C overnight and subsequently calcined at 600 °C for 2 h. With this, a γ-alumina with a specific surface area of 157 m^2/g was obtained, which was successfully used as a catalytic support for a 10% Ni catalyst in the dry reforming reaction of glycerol. Foo et al. (2019) verified that an adequate mixing ratio and acid leaching have positive effects on the mineralogy, macromorphology and crystallinity of the synthesized material. How et al. (2017) mixed the aluminum slag with distilled water in order to remove the chloride salt at 200 rpm and 30 min. The mixture was separated by vacuum filtration and the chloride content in the effluent was analyzed by Mohr's method. In order that the salt does not affect the acid solution, this process was repeated until the chloride salt was completely eliminated. For leaching, 50 g of washed slag were obtained with 200 mL of 2 M H_2SO_4 at 70 °C for 1 h with a centrifugation of 450 rpm. The precipitation of the final leachate was carried out with 10% ammonia solution at pH = 4.4 and the different solutions were evaluated at target pH (5, 6, 7, 8 and 9). The white precipitate was centrifuged and washed with deionized water to remove excess ammonia. It was dried for 18 h at 60 °C and, finally, the product was calcined for 2 h to obtain γ-Al_2O_3. A crystalline particle size of 5 nm and a specific surface area between 111 and 128 m^2/g with various pore sizes were obtained.

Dash et al. (2008) carried out the extraction of aluminum from the waste slag by means of acid dissolution. The slag used contained 65% alumina, 4% SiO_2 and the rest was a mixture of oxides of magnesium, calcium or iron together with some salts such as NaCl and KCl. The dissolution of 10 g of slag was carried out in H_2SO_4 (36 N) in a flat-bottomed glass reactor which was placed on a hot plate under vacuum for 3 h. The temperature during the leaching was kept at 90 ± 2 °C. The liquor was analyzed by the conventional EDTA-ZnSO$_4$ method. The liquor responded favorably to the acid solution and the results obtained showed that the aluminum slag could be used to obtain alumina, achieving 85% alumina under stoichiometric conditions. The results are shown in Fig. 5. It is important to note that for all these acid concentrations, with increasing acid volume, the efficiency initially decreased and then remained constant beyond a certain amount.

David and Kopac (2013) carried out the extraction of alumina at low temperature by leaching with H_2SO_4. In each experiment, 100 g of aluminum slag and a slag/acid ratio of 5:8 were used, with several diluted solutions of sulfuric acid at a temperature of 90 °C, achieving a purity greater than 99%. The liquor obtained in the leaching contained Al^{3+}, Na^+, Fe^{3+}, Ca^{2+}, Mg^{2+}, among other impurities. Since one of the main objectives of this study was to obtain high purity Al_2O_3, the rest of the ions must be eliminated. An important conclusion reached was that selective ion precipitation by adjusting the pH value was not successful in separating Al^{3+} and Fe^{3+}. Instead, to remove Fe^{3+} and other impurity ions, EDTA was used as a complexing agent. The success of this process was due to the fact that the complexation capacity of each metal in the EDTA leach liquor was different. NH_4HCO_3 was used as a precipitating agent and the precipitate was decanted, filtered and washed. The filtrate was concentrated by heating in order to obtain density values of 1.35 kg/dm^3. Finally, the product was dried at 120 °C for 30 min and calcined at temperatures between 850 and 1050 °C. On the other hand, Sulaiman et al. (2008) described a solvo-thermal process for the recycling of aluminum slag powder nano α-alumina. This process included washing with water to remove unwanted sodium and leaching with H_2SO_4. Later propanol was added for the precipitation (crystallization) of aluminum hydroxide. The precipitate was dried at 70 °C and subsequently calcined at 1300 °C for 3 h, where the final white powder was obtained. The XRD, SEM and EDX analyzes showed a single phase of α-alumina crystal with a spherical shape and an average particle size of 3.80 μm, which was decreased by wet grinding at high speed to 0.49 μm.

Table 2

Summary of the experimental studies carried out by leaching aluminum slags in acid and alkaline route.

Route	Alumina phase	Leaching agent	Leaching conditions	Alumina synthesis method	Synthesis method conditions	Results	Reference
Acid	γ-Al$_2$O$_3$	H$_2$SO$_4$	1 h, 70 °C, 200 rpm, slag-acid ratio 1:4	Precipitation	pH 9 (with 10% ammonia solution). Precipitate is washed, dried at 60 °C overnight and calcined at 600 °C 2 h.	Specific surface area 157 m^2/g	(Roslan et al., 2019)
	γ-Al$_2$O$_3$	H$_2$SO$_4$	50 g washed slag + 200 mL H$_2$SO$_4$ (2 M) at 70 °C, 1 h and 450 rpm	Precipitation	pH 4.4 (with 10% ammonia solution). Precipitate was centrifuged, washed, dried 18 h at 60 °C and calcined 2 h.	Specific surface area 111–128 m^2/g, crystalline particle size 5 nm.	(Yang et al., 2019)
	-	H$_2$SO$_4$	10 g slag + H$_2$SO$_4$ (20% solution) at 90 °C	Vacuum drying	Flat-bottomed glass reactor which was placed on a hot plate under vacuum for 3 h	With 20 mL acid, 75% of aluminum extracted. With 30 mL, 80% and with 40 mL 85% extracted.	(Dash et al., 2008)
	γ-Al$_2$O$_3$, α-Al$_2$O$_3$	H$_2$SO$_4$	100 g aluminum slag; slag-acid ratio 5:8; 90 °C, pH 3 with EDTA.	Precipitation	NH$_4$HCO$_3$ as precipitating agent. Precipitate was decanted filtered and washed. The filtrate was concentrated by heating to achieve final density 1.35–1.40 kg/dm^3. Dried at 120 °C 30 min and calcined at 850–1050ºC 4 h.	Purity of product was Al$_2$O$_3$ 99.28%, MgO 0.63% and 0.09% other impurities.	(David and Kopac, 2013)
	α-Al$_2$O$_3$	H$_2$SO$_4$	-	Precipitation + solvothermal	Propanol as precipitating agent. Precipitate was dried at 70 °C and calcined at 1300 °C for 3 h.	Particle size 3.80 μm, decreased by wet grinding at high speed to 0.49 μm.	(Sulaiman et al., 2008)
	γ-Al$_2$O$_3$	H$_2$SO$_4$	275 °C 8 h. Hydrochlorination (-10 ºC to 20 °C). The leachate was cooled at $-$ 10 °C and treated with gaseous HCl to caused aluminum chloride to precipitate.	Hydrochlori-nation + pyrolysis, pyrohydrolization	Leachate was filtered under vacuum. Aluminum chloride was heated at 500 °C 2 h in order to convert the aluminum chloride into aluminum hydroxide. The product was heated for 15 min at 105 °C.	Purity > 98%	(Beaulieu et al., 2011)
	α-Al$_2$O$_3$	H$_2$SO$_4$	10 g slag + 100 mL H$_2$SO$_4$ (several concentrations) 90 °C 3 h.	Precipitation	40 °C, 10% ammonia solution was added dropwise. pH> 10 (maximum precipitation rate). Precipitate washed, dried at 80 °C for 24 h and calcined 1200 °C for 2 h.	99.2% purity of the alumina with H$_2$SO$_4$ 15%.	(Benkhelif et al., 2022)
	α-Al$_2$O$_3$	H$_2$SO$_4$	170–230 °C (10–15 h)	Precipitation	Ammonia as precipitating agent (ratio 1:1). Precipitate was dried at 100–200 °C	Particle size 200–700 nm. > 90% yield of α-Al$_2$O$_3$. High crystalline single phases having purity of 99.5%.	(Datta et al., 2014)
	α-Al$_2$O$_3$	HCl	25 g aluminum slag + 500 mL 1–6 M HCl at various reaction times (15–240 min) and different leaching temperatures (20–100 °C)	Precipitation	25% NH$_3$ was added to the solution to precipitate Al (OH)$_3$ which was purified with 5% NaOH solution and reprecipitated with a 10% NH$_4$Cl solution. Calcined at 1000 °C (20 °C/min).	Rates of diffusion of Al^{3+} species were supposed to block hydronium ion diffusion at HCl concentration above 6 M.	(Sarker et al., 2015)
	γ-Al$_2$O$_3$	HCl	5 g slag + 100 mL HCl. 500 rpm at several temperatures, concentrations and reaction times.	Precipitation	Precipitate of all metal hydroxides at pH 3–5. Al (OH)$_3$ is diluted with 3 M NaOH and the rest of the hydroxides are eliminated. Al(OH)$_3$ is precipitated by dilute HCl, dried at 105 °C and calcined at 700 °C for 2 h.	Optimal conditions leaching: 85 °C, HCl 5 M and 2 h reaction.	(Mahinroosta and Allahverdi, 2018b)
	γ-Al$_2$O$_3$, α-Al$_2$O$_3$	H$_2$SO$_4$	-	Precipitation	Ethanol as precipitating agent. Calcined at 500–1300 °C	Crystal size for γ-Al$_2$O$_3$ was 6.5 nm and for α- Al$_2$O$_3$ was 67 nm.	(Muslim and Paulus, 2009)
	-	H$_2$SO$_4$	100 g dross. Dross/acid ratio 100/160. Aluminum was recovered in the form of a double sulphate [(NH$_4$)$_2$SO$_4$. Al$_2$(SO$_4$)$_3$·24 H$_2$O]	-	-	Optimum conditions leaching: 100 °C, H$_2$SO$_4$ 10%, 5 h reaction and solid/liquid ratio 0.111.	(Amer, 2002)
	γ-Al$_2$O$_3$	HCl	120 min, 85 °C, HCl 5 M and 500 rpm.	Co-precipitation	Hybrid hydroxide product precipitate was formed from	-	

(continued on next page)

Table 2 (*continued*)

Route	Alumina phase	Leaching agent	Leaching conditions	Alumina synthesis method	Synthesis method conditions	Results	Reference
					the metal chlorides. To separate Al(OH)$_3$ from the others, 2 M NaOH was added (pH 13.5). Al(OH)$_3$ re-precipitated by HCl (pH 9–10). Calcined 700 °C 2 h.		(Mahinroosta and Allahverdi, 2018c, 2017a)
Alkaline	γ-Al$_2$O$_3$	NaOH	Atmospheric and high-pressure leaching.	Precipitation	Six agents (H$_2$O$_2$, CO$_2$, ammonium carbonate, ammonium bicarbonate, ammonium aluminum sulphate and active seed). Precipitate was filtered, washed, dried at 120 °C for 12 h and calcined at 600 °C for 3 h.	Well-defined crystalline alumina with 99% purity. Surface area > 250 m^{2}g^{-1} and uniform crystallite sizes (60 Å). High pressure leaching achieved a higher recover value (73%).	(El-Katatny et al., 2000)
	-	NaOH	2 h, 240 °C, 700 rpm.	Hydrothermal	Leached residue dried at 110–260 °C.	All the metallic Al (10.25%) was recovered. Average composition of the produced sodium aluminate had Al^{3+} 28.64 g/L	(Tsakiridis and Oustadakis, 2013)
	Potash alum	KOH	2 g dross + 50 mL 3 M KOH, 45 °C, and 60 min	Precipitation	Leached was filtered. 3 M H$_2$SO$_4$ 70 mL was added to the solution filtrate slowly using a burette. The solution was boiled for 5, 8, 12 and 15 min. Solution was quickly transferred to an ice water bath to produce potash alum.	20 g of potash alum produced by using 2 g of white aluminum dross.	(Meshram et al., 2020)
	γ-Al$_2$O$_3$	NaOH	1 h at 500–750 °C. NaOH/dross ratio: 2–4.	Fusion method	Dross was mixed with a specified amount of NaOH. Mixture was put in a stainless-steel crucible. The fused sample was cooled to room temperature and dissolved in distilled water.	Optimum conditions leaching: NaOH/dross ratio 3.5. 750 °C and 60 min. Apparent activation energy 6.452 kJ/mol.	(Sultana et al., 2013)
	α-Al$_2$O$_3$	NaOH	150 g secondary aluminum slag + 1 L NaOH (10%) during 2 h.	Precipitation	Precipitation of Al(OH)$_3$ with CO$_2$ for 2 h at room temperature. Precipitate residue was washed, dried and calcined at 1000 °C 1 h.	Recovery rate 80%.	(Lucheva et al., 2005)
	γ-Al$_2$O$_3$, α-Al$_2$O$_3$	NaOH	100 g solid waste + NaOH (10%, 20%, 30% and 40%). Several solid/solution ratios (1:1; 1:2; 1:3 and 1:4).	Precipitation	Al(OH)$_3$ was precipitated from NaAlO$_2$ solution injecting CO$_2$ gas at pH 7. Precipitate was filtrated, washed, dried at 105 °C and calcined at 600 or 1000 °C for 5 h.	8% of pure alumina (97.8%) was recovered from the solid waste.	(Ghoniem et al., 2014)
	α-Al$_2$O$_3$	NaOH	112.5 g aluminum dross (<20 mesh) + NaOH (10%) during 2 h.	Precipitation	Precipitation with 1.7 g Al(OH)$_3$ seed	Precipitated solid was washed, dried at 110 °C 12 h and calcined at 1000 and 1350 °C.	(Park et al., 2001)
	α-Al$_2$O$_3$	NaOH	Prior to leaching: roasting at 800 °C was carried out in atmospheric conditions. Leaching: roasted mass was subjected to leaching with NaOH (2%) at 50–90 °C during 15 min–3 h.	Precipitation	Precipitation with CO$_2$ obtaining Al(OH)$_3$. Precipitate was washed, dried and calcined at 1200 °C during 2 h.	Recovery percentage was maximum at 70 °C. Beyond this temperature no significant enhancement in recovery was noticed.	(Tripathy et al., 2019)
	α-Al$_2$O$_3$	NaOH	NaOH 0.5 M (pH 13) 72 h	Precipitation	Addition of HCl up to pH = 7 in order to precipitate Al(OH)$_3$.	1.44 g of α-Al$_2$O$_3$	(Trejo-Trejo et al., 2021)
	-	NaOH	37.5 g saline slag + 750 mL 2 M NaOH during 2 h at 90 °C	-	-	2 M NaOH was 20% more efficient than 1 M NaOH.	(Yoldi et al., 2019)
	α-Al$_2$O$_3$	NaOH	Before leaching, the ground slag (d$_{10}$ < 100 μm) was leached with hot water at 70 °C for 3 h. 1000 g dross, 250 rpm. Solid/liquid ratio 1:7	Pyrometallurgical + precipitation	pH of the leached solution was decreased from 13.5 to 7 using HCl 0.1 M to remove Al ions as Al(OH)$_3$ precipitate. Precipitate was calcined at 1000–1200 °C for 270 min	Surface roughness of the product varied from 4.64 nm to 268.21 nm. The EDX results indicated that the purity of α-Al$_2$O$_3$ was high (39.65% O; 52.29% Al, 0.45% K)	(Türk et al., 2020)
	γ-Al$_2$O$_3$	NaOH	Na$_2$O/Al$_2$O$_3$ molar ratio 1:4 at pH 8–9. Temperature 60 °C for 40 min. Extracting rate 86.7%.	Precipitation	CaSO$_4$ was added to the leached to precipitate silicate. Al(OH)$_3$ was	Kinetic leaching test: dissolution of alumina and silica were controlled by a layer diffusion process with	(He et al., 2021)

Table 2 (*continued*)

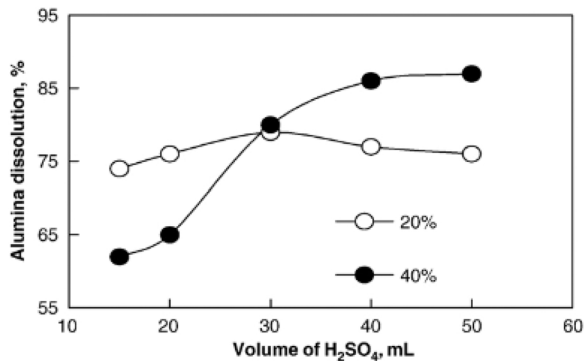| Route	Alumina phase	Leaching agent	Leaching conditions	Alumina synthesis method	Synthesis method conditions	Results	Reference
					precipitated and calcined at 900 °C for 2 h.	an apparent activation energy of 11.4010 kJ/mol. Purity> 98%.	

Fig. 5. Correlation between the volume of sulfuric acid and the extraction of alumina during acid extraction from a waste slag (Dash et al., 2008).

In the patent filed by Beaulieu et al. (2011), aluminum chloride was produced through the following steps: (1) leaching of aluminum slag residues with H_2SO_4, (2) hydrochlorination of the leachate to obtain aluminum chloride, (3) precipitation of aluminum chloride by HCl gas, and (4) pyrohydrolysis. The leaching must be carried out at a temperature of at least 70 °C. The hydrochlorination can be carried out at a temperature of about -10 - 20 ºC. The conversion of aluminum chloride to alumina was carried out by pyrolysis or pyrohydrolization. Alumina purities of at least 80% were obtained. On the other hand, it provided a process for the preparation of alumina comprising the optional treatment of slag residues to reduce the amount of aluminum nitride by converting it to aluminum hydroxide. First, it reduced the amount of aluminum nitride of the aluminum slag residue by 80–95% by converting aluminum nitride to aluminum hydroxide. For this, the slag residues were treated with water at a temperature of at least 80 °C. The recovered aluminum hydroxide solid was leached with H_2SO_4. The process carried out by Benkhelif et al. (2022) was based on the acid leaching of the slag followed by an alkaline precipitation (see Fig. 6). First, 10 g of slag were mixed with 100 mL of H_2SO_4 at several concentrations (3%, 5%, 10%, 15%, 20% and 25%) and the mixture was placed on a hot plate with stirring, maintaining a temperature of 90 °C for 3 h. Thus, the aluminum compounds (Al, $Al(OH)_3$ and Al_2O_3) present in the slag dissolved in the acid giving aluminum sulphates. The solution rich in aluminum sulfates was filtered to separate it from the solid residue. Subsequently, precipitation was performed at 40 °C by adding a

Fig. 6. Summary of the process carried out by Benkhelif et al. (2022) for the extraction of alumina from a waste slag.

10% NH_3 solution dropwise (Das et al., 2007) increasing the pH up to 10 which corresponds to the pH of maximum precipitation rate. The operation was carried out by controlling the particle size by measuring the zeta potential of the solutions. The obtained gel was washed with deionized water to remove impurities. Subsequently, it was dried at 80 °C for 24 h and calcined at 1200 °C for 2 h with a heating rate of 5 °C/min. Alumina with a purity of 99.2% can be reached with a concentration of H_2SO_4 in the leach of 15%.

In order to obtain α-alumina, Datta et al. (2014) reported a method in which the first step consisted of sieving the aluminum slag to achieve a medium particle size, then an acid leachate with H_2SO_4 that was treated with a solution of ammonia at acidic pH (4−6) to facilitate the precipitation of iron hydroxide. The iron-free liquid solution was treated with aqueous ammonia at alkaline pH (7.5–8.5) to facilitate precipitation of aluminum hydroxide. This precipitate was separated and calcined at 1100–1200 °C during 15 min - 1 h, obtaining α-Al_2O_3 with a highly crystalline single phase, 99.5% of purity and a particle size of 200–700 nm. In the study carried out by Sarker et al. (2015), HCl was used as a leaching agent, claiming its advantages such as its mild action, easy availability and by-product in numerous chemical industries. 25 g of ground aluminum slag were introduced into a flat-bottomed glass reactor placed on a stir plate and leached using 500 mL of 1 – 6 M HCl for several reaction times (15–240 min) and several leaching temperatures from 298 to 373 K. After separation of the solution from the solid residue, 25% NH_3 was added to the solution to precipitate $Al(OH)_3$. Subsequently, $Al(OH)_3$ was purified with a 5% NaOH solution and reprecipitating it with a 10% NH_4Cl solution. Finally, the solid was calcined at 1000 °C with a heating ramp of 20 °C/min. The results obtained determined that the initial extraction rate was higher and that it became almost constant after 1 h. Regarding the concentration of acid used, it was assumed that the rates of diffusion of Al^{3+} ions from the solid to the solution increased with increasing acid concentration. However, Al^{3+} species were also supposed to block hydronium ion diffusion at HCl concentration above 6 M, a phenomenon that has been reported by various researches (Arabia, 2009; Eisele et al., 1983). Mahinroosta and Allahverdi (2018b) presented a novel process based on HCl leaching for the recovery of γ-alumina nanoparticles as a high-value material from aluminum slags. In order to optimize the parameters, liquid/solid ratios were initially studied at various temperatures and reaction times, finding that the leaching was controlled by layered diffusion. First, the slag was dried at 105 °C in the oven in order to neutralize the effect of sample humidity on the dissolution efficiency calculations. Subsequently, 5 g of slag were taken in the reactor at atmospheric pressure and 100 mL of HCl were added under stirring at 500 rpm. Since aluminum hydroxide is an amphoteric material, it can be separated from the rest of the hydroxides by adding 3 M NaOH, thus remaining in the liquid phase. The other hydroxides that remained as precipitates were easily removed by filtration. Dilute hydrochloric acid was then added to precipitate aluminum hydroxide. Several temperatures, concentrations and reaction times were tested, finding the following optimal conditions: 85 °C, 5 M acid concentration and 2 h reaction time. The alumina obtained had a crystal size of 15.90 nm and was mainly composed of gamma phase, obtaining a product purity greater than 98%.

Muslim and Paulus (2009) worked out a process to produce two types of alumina powders, γ-alumina and α-alumina. The stages that followed in the process were: leaching, precipitation and calcination. Leaching was carried out using diluted sulfuric acid, precipitation with
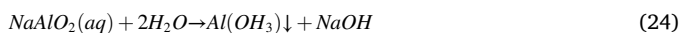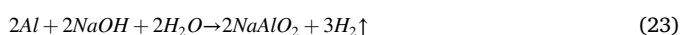
ethanol and finally calcination between 500 and 1300 °C depending on the alumina phase to be obtained. The size of the crystals obtained was 6.5 nm and 67 nm for gamma and alpha-alumina, respectively. Amer (2002) has carried out the leaching process in two stages in order to prepare a purified product. In the first stage, the impurities were removed by means of sulfuric acid at various temperatures and solid/-liquid proportions. With this, it was possible to dissolve the impurities of the starting material in the form of sulphates. In the second stage, the extraction of aluminum took place, in the form of aluminum sulphate, also studying the effect in leaching of time, acid concentration and temperature.

Mahinroosta and Allahverdi (2018c) (2017a) demonstrated the synthesis and characterization of activated alumina nanopowders slag that exhibited purity greater than 97 wt% from secondary aluminum. They studied several parameters that directly affected the efficiency of alumina extraction, including particle size, leaching time and temperature, and the acid concentration used. The optimal conditions studied were: particle size between 38 and 75 μm, leaching time of 120 min at 85 °C and 5 M acid concentration. In another work, the same research group synthesized and characterized nanocrystalline aluminum hydroxide gel (AHG) from secondary aluminum slag through a novel leaching process at atmospheric temperature and pressure. The arithmetic mean particle diameter (D_{50}) of aluminum hydroxide was 514 nm (Mahinroosta and Allahverdi, 2017b). The synthesis of alumina from aluminum slag has been scaled up to a pilot plant by Mahinroosta and Allahverdi (2021) based on previous studies carried out on a laboratory scale by the same authors (Mahinroosta and Allahverdi (2018c). The first step was the washing of the aluminum slag with water at 95 °C for 60 min and then leaching with 5 M HCl at 85 °C for 2 h. The chloride solution was clarified with a filter press. 25% ammonia solution was added to the obtained hydroxide to obtain a pH value of 9.5 ± 0.5. Subsequently, in order to purify the product, a 2 M NaOH solution was added to the precipitate, pH of 13.5 ± 0.5, achieving an effective separation of aluminum from the other metal hydroxides. The resulting sodium aluminate was separated from the formed sol and used as precursors to synthesize γ-Al_2O_3 by rapidly adding 3 M HCl to the sodium aluminate solution until a pH value of 8.0 ± 0.5 was achieved. Subsequently, it was washed with distilled water 3 times and the sol was dried in an oven for 220 °C for 5 h. Finally, it was dehydrated by calcining at 550 °C for 3 h to obtain γ-Al_2O_3 of high purity. The pilot plant was designed in order to obtain 5 kg/day of this alumina.

Paper alum or filter alum [$Al_2(SO_4)_3 \cdot 12 \ H_2O$], also known as pure aluminum sulphate, can be produced by acid leaching of primary (black) aluminum slag in hydrometallurgical leaching processes. Filter alum is widely used in water treatment processes, fireproof fabrics or cellulosic insulation (Amer, 2002; Mukhopadhyay et al., 2005; Huckabay and Skiathas, 1982).

3.1.2. Alkaline route

The alkaline leaching process is based on the dissolution of Al and Al_2O_3 in a strong sodium hydroxide solution under atmospheric or high pressure conditions. Aluminum can be recovered as aluminum hydroxide by crystallization or precipitation. Subsequently, an alumina with a high degree of purity can be produced by calcination at high temperatures (Das et al., 2007; Dash et al., 2008; Tsakiridis and Oustadakis, 2013). The chemical routes of the alkaline leaching process were the following (El-Katatny et al., 2003; Lucheva et al., 2005; Mahinroosta and Allahverdi, 2018a; Sultana et al., 2013):

$$Al_2O_3 + 2NaOH \rightarrow 2NaAlO_2 + H_2O \tag{22}$$

$$2Al + 2NaOH + 2H_2O \rightarrow 2NaAlO_2 + 3H_2\uparrow \tag{23}$$

$$NaAlO_2(aq) + 2H_2O \rightarrow Al(OH_3)\downarrow + NaOH \tag{24}$$

$$2Al(OH_3) \rightarrow Al_2O_3 + 3H_2O \tag{25}$$

In order to recover the content of aluminum in the form of alumina from aluminum slag, El-Katatny et al. (2000) proposed atmospheric and high-pressure leaching with NaOH adopting the Bayer process. The sodium aluminate solutions obtained were precipitated by six agents. Subsequently, the precipitates were filtered, washed, dried and calcined at 600 °C to produce alumina. The precipitating agents used were H_2O_2, CO_2, ammonium carbonate, ammonium bicarbonate, ammonium aluminum sulphate and active seed (freshly prepared aluminum hydroxide gel). The results showed well-defined crystalline gamma-alumina, with a purity of 99%, surface areas greater than 250 m^2/g and uniform crystallite sizes (60 Å). An important conclusion was that with the high-pressure leaching technique a higher recovery value (73%) was obtained compared to the atmospheric leaching technique (50%). The precipitation methods used affected, to a certain extent, the crystalline modification of the obtained precipitate. When using as precipitating agents H_2O_2, CO_2, ammonium carbonate and ammonium bicarbonate, crystalline boehmite, γ-AlO(OH), was obtained; while using ammonium and aluminum sulphate, gelatinous boehmite (pseudoboehmite) was obtained; and crystalline hard gibbsite (γ-Al(OH)$_3$) was the precipitation product when using active seed. El-Katatny et al. (2003) carried out the comparison of alumina products recovered from aluminum slag with alumina recovered by the same process as bauxite. After calcination of the precipitates, 95% γ-Al_2O_3 resulted. This is the most promising transition form of alumina with respect to surface area and catalytic activity (Levin and Brandon, 1998). The synthesis process consisted of the following steps: (i) leaching of aluminum in the form of soluble sodium aluminate adapting Bayer's method, (ii) precipitation, (iii) filtration, washing, drying (120 °C, 12 h) and calcination at 600 °C for 3 h. The three methods applied were: Method-I used H_2O_2 solution as precipitating agent; Method-II used ammonium aluminum sulphate; Method-III with active seeds of aluminum hydroxide. Reported results showed that the synthesized slag product was very similar to that obtained from bauxite. Specific surface areas obtained were highly dependent on the precipitating agent used, H_2O_2 leading to the highest surface area value.

Tsakiridis and Oustadakis (2013) extracted 57% of aluminum from black dross using a sodium hydroxide solution for 2 h in a temperature range between 433 and 533 K. The process was based on four stages: (i) initial screening of the aluminum slag into particles smaller than 1 mm, (ii) crushing of particles to a size less than 100 μm, (iii) washing of the slag at 90 °C with distilled water, (iv) recovery of aluminum at high pressure and temperature (240 °C) using NaOH solution. Average composition of the produced sodium aluminate solution was Al^{3+} 28.64 g/L; Ca^{2+} 0.28 g/L; K^+ 0.044 g/L; Mg^{2+} 0.12 g/L; Si^{4+} 0.18 g/L and Fe^{3+} 0.007 g/L. Meshram et al. (2020) carried out the production of potash alum from white aluminum slag. The leaching was carried out in a basic medium using 3 M potassium hydroxide at 45 °C for 60 min. Precipitation was produced slowly adding 70 mL sulfuric acid 3 M using a burette, allowing the crystallization of tamarugite, NaAl(SO$_4$)$_2$·6 H$_2$O. Through continuous stirring and addition of acid, precipitation of aluminum hydroxide was achieved. Subsequently, the solution was boiled for several times (5, 8, 12 and 15 min) to eliminate excess acid, saturate the solution and calculate the total mass of potash produced. Sultana et al. (2013) molten an aluminum slag with 72% Al_2O_3 and 14% SiO_2 with NaOH for 1 h at a temperature range of 500–750 °C in a stainless-steel crucible for melting treatment. The fused mixture was subsequently cooled to room temperature and washed with distilled water. The optimal conditions were as follows: weight ratio of NaOH to aluminum slag = 3.5 and temperature 750 °C.

Lucheva et al. (2005) proposed a method similar to the Bayer process for the recovery of aluminum from slags. For leaching, 150 g of secondary aluminum slag were treated in 1 L of 10% NaOH (with a molar ratio of Na_2O:Al_2O_3 = 2) for 2 h, injecting carbon dioxide for the formation of aluminum hydroxide. Leached residue was washed with water, dried and calcined at 1000 °C for 1 h in order to produce α-Al_2O_3. The recovery rate was 80%. Along the same lines, Ghoniem et al. (2014)

focused their work to the production of alumina (Al_2O_3) from solid residues of the cake produced as a by-product in some small aluminum factories. Aluminum ions were separated from this cake as a solution of sodium aluminate ($NaAlO_2$) by leaching the aluminum cake with commercial caustic soda. Subsequently, the hydroxide was precipitated by $NaAlO_2$ solution by injecting CO_2 gas through the hot solution. Finally, the precipitate $Al(OH)_3$ was separated by filtration, washed with hot water, dried at 105 °C and calcined at 600 or 1000 °C to produce pure alumina in gamma or alpha forms, respectively. After leaching aluminum slag with NaOH solution, Park et al. (2001) filtered the hydrolyzed solution containing $NaAlO_2$. At this time a small amount of Al $(OH)_3$ seeds was added to promote $Al(OH)_3$ precipitation. The precipitated solid was washed and dried, while the liquid phase resulting from leaching with NaOH can be reused as raw material for ceramics. The dried $Al(OH)_3$ precipitate was calcined for being used as a high refractoriness alumina castable refractory. Tripathy et al. (2019) proposed an efficient recovery of alumina by roasting with soda in a leaching route with sodium carbonate and leaching with water. The sodium aluminate underwent a precipitation process with CO_2, obtaining the aluminum hydroxide phase and, after calcination, corundum (α-alumina), although various phases of metastable or activated alumina can be obtained depending on the needs of the user.

Trejo-Trejo et al. (2021) obtained α-Al_2O_3 from an aluminum slag (Fig. 7). The procedure was based on the leaching of the aluminum slag in 0.5 M NaOH basic medium at pH = 13 for 72 h, followed by the addition of HCl up to pH = 7 in order to obtain precipitated $Al(OH)_3$. The solid was recovered by vacuum filtration and analyzed by X-ray scattering (EDS), and the liquid was analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES). The solid was dried at 90 °C for 24 h and 1.93 g were obtained, which were subsequently calcined at 1200 °C, obtaining 1.44 g of α-alumina, which was analyzed by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

Another work carried out by Yoldi et al. (2019) presented an improvement in the extraction of aluminum from saline slags. The solution was treated under reflux with two NaOH concentrations, various extraction times and four extraction steps to obtain Al^{3+} solution (see Fig. 8). The reference conditions for this process came from the previous experience of the same research group (Gil et al., 2018a). 37.5 g of milled saline slag were treated with 750 mL of 2 M NaOH at reflux for 2 h at 90 °C. The results showed that the first extraction dissolved most of the aluminum and the extraction with 2 M NaOH was 20% more efficient than with 1 M NaOH. On the other hand, the highest values of aluminum were obtained at 1 h of reflux. In the work presented by Türk et al. (2020), the synthesis of α-Al_2O_3 particles from aluminum slag was carried out using pyrometallurgical and hydrometallurgical processes.

The first step of the process was the leaching with NaOH, precipitation and finally calcination at 1000–1200 °C. It was found that a smaller particle size increased the extraction rate of aluminum from the slag, which was confirmed by other researches (Alex et al., 2014; Erdemoğlu et al., 2018; Taşkin et al., 2009; Tripathy et al., 2019). Leaching tests were carried out on two samples. Test parameters on sample A were (in descending order of importance): NaOH concentration, leaching temperature, and time; while for sample B they were leaching temperature, NaOH concentration and time. The precipitation step must be stabilized for at least 1 h before calcination in order to obtain a single polymorph alumina product. The surface roughness of the product varied widely from 4.64 nm to 268.21 nm. EDX results indicated that the purity of α-Al_2O_3 was high (39.65% O; 52.29% Al; 7.61% Au (used in the preparation of the sample for SEM studies) and 0.45% K).

Recently, a NH_4HSO_4 roasting and water leaching process has been proposed to efficiently extract aluminum from secondary aluminum dross, SAD, (Shi and Li, 2022). During roasting, NH_4HSO_4 reacted with the aluminum and formed $NH_4Al(SO_4)_2$. The aluminum was recovered in the form of $Al(OH)_3$ after precipitation by adjusting the pH = 6.0 of the leaching liquid. Al_2O_3 of 98.8% of purity was obtained by roasting the $Al(OH)_3$ at 1200 °C (see Fig. 9).

Solid waste and flue gas discharge issues are generally not addressed and most researchers only focus on the alumina extraction rate. He et al. (2021) explained a novel extraction process of high purity alumina (Al_2O_3) from aluminum slag with a high extraction rate without producing exhaust or residual gases. The leaching process used NaOH whose mass depended on the Na_2O/Al_2O_3 molar ratio at pH = 8–9. The optimal parameters for this stage were a molar ratio of 1.4 Na_2O/Al_2O_3 and a temperature of 60 °C for 40 min, achieving an extraction rate of 86.7%. The results of the kinetic leaching test showed that the dissolution of alumina and silica were controlled by a layer diffusion process with apparent activation energies of 11.40 kJ/mol and 2.06 kJ/mol, respectively. To purify the alumina, $CaSO_4$ was added to the leachate to precipitate the silicate, achieving alumina with purity greater than 98%. Subsequently, the $Al(OH)_3$ precipitated and was calcined at 900 °C for 2 h to obtain γ-Al_2O_3.

3.1.3. Alternative extraction routes

Other novel research works have proposed alternatives to the acid and alkaline extraction routes. Yoo et al. (2011) dissolved aluminum slag in sec-butyl alcohol with a molar ratio of 3 mol SBA/mol Al, with HgI_2 as catalyst. They obtained aluminum sec-butoxide, a high-cost Al alkoxide used as a precursor of high purity activated alumina. López et al. (1994) described a preliminary study for the production of a mixture of alumina and spinel by sintering residues from the hydrolysis of remelted aluminum slag in rotary furnaces. The proposed route
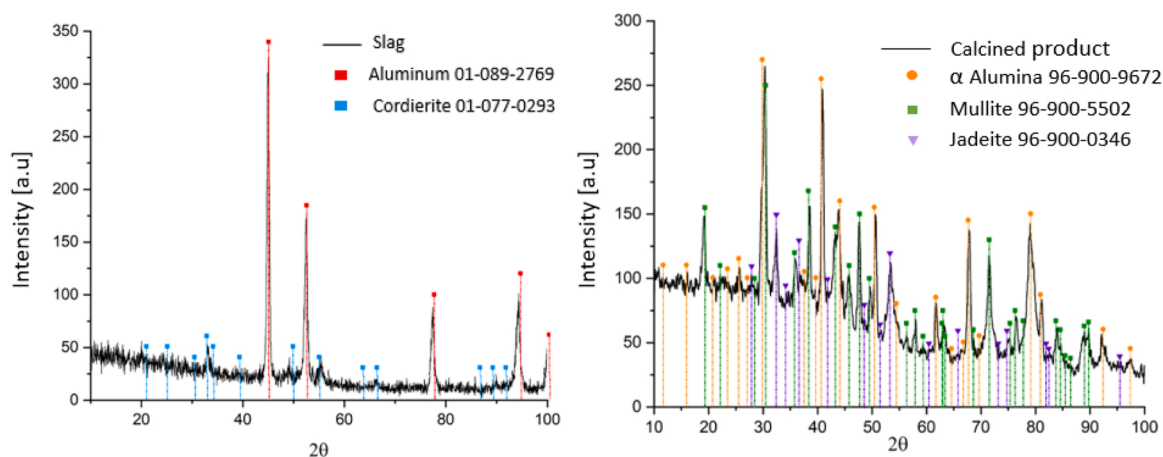

Fig. 7. Slag diffractogram showing the aluminum and cordierite phase (($Mg,Fe)_2Al_4Si_5O_{18}$) (left image). Diffractogram of the calcined product, where the phases α-alumina, mullite ($Al_6Si_2O_{13}$) and jadeite ($Na(Al,Fe^{3+})Si_2O_6$) are observed (right image) (Trejo-Trejo et al., 2021).
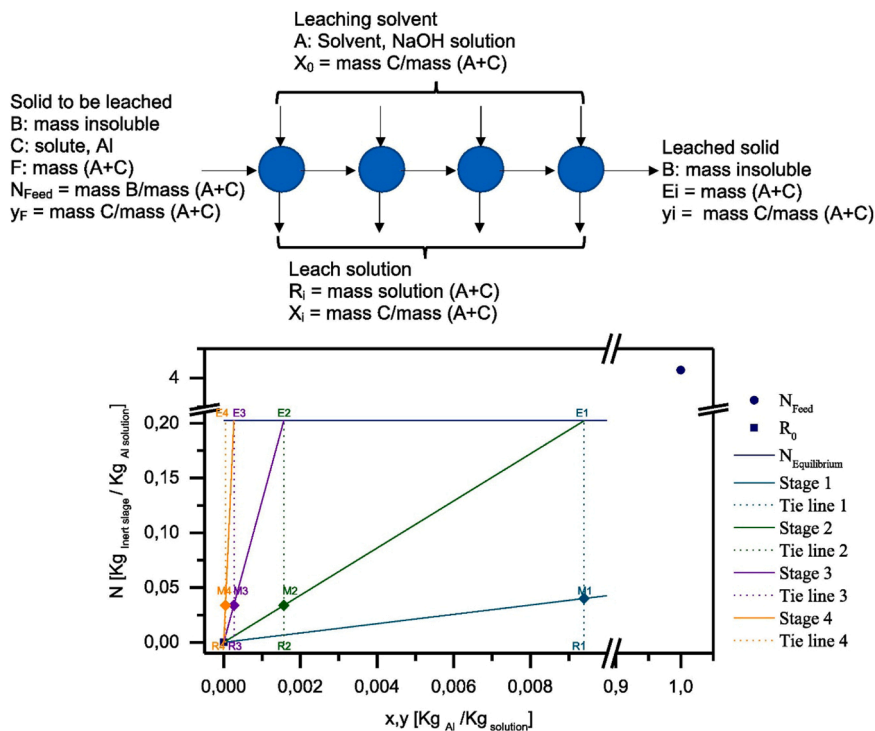
Fig. 8. Aluminum multistage crosscurrent leaching with NaOH (Yoldi et al., 2019).
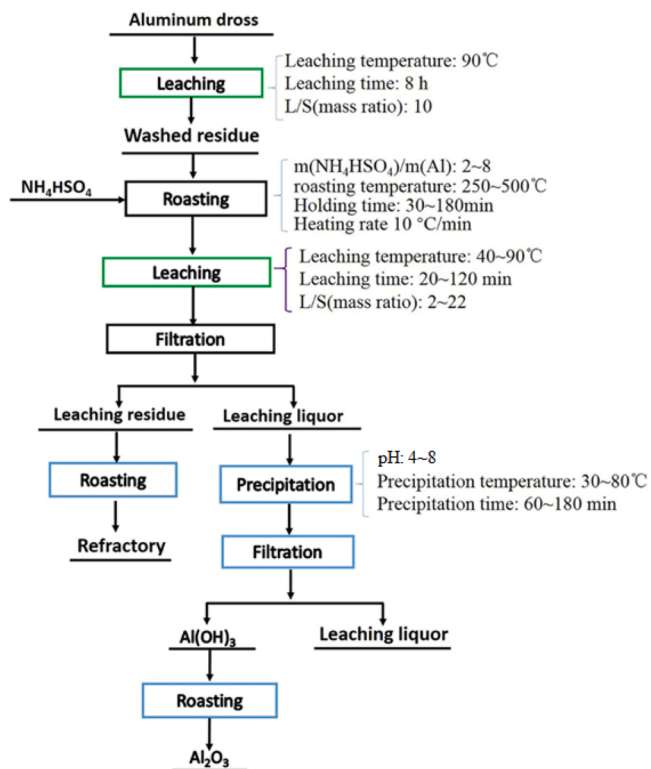

Fig. 9. Flow chart of the process of extracting aluminum from secondary aluminum dross (Shi and Li, 2022).

consisted on the water leaching of the non-metallic part of the slag and the fraction that was not soluble in water was pelletized and calcined in an oxidizing atmosphere to produce a mixture of α-Al₂O₃ and MgO-·Al₂O₃. Plasma processing of slag has been carried out by some researchers, although full utilization of this process was not established

(Lavoie and Lachance, 1995). Yang et al. (2014) produced high purity aluminum oxide (Al₂O₃) fine powder from aluminum slag using a high temperature source based on a radio frequency (RF) plasma synthesis system, reporting a purity of 99.95% alumina and a particle size distribution (d_{50}) of 8 μm. Thermal plasma technology is being studied in areas with high population in order to eliminate waste (Mahinroosta and Allahverdi, 2018a). With this technology temperatures of 10,000 °C are achieved in which most of the material evaporates resulting in atoms, molecular cells, radicals and ions (Chabert and Braithwaite, 2012).

4. Synthesis methods of alumina

4.1. Precipitation method

The most efficient method for synthesizing alumina is precipitation, since it is an easy and simple process that can be carried out with low-cost raw materials, produces low contamination and allows obtaining a product of high purity, thermal stability, with almost homogeneous size nanoparticles and particle size control (Ziva et al., 2021). Zhang et al. (2016) prepared mesoporous γ-Al₂O₃ with a high specific surface from the calcination of NH₄Al(OH)₂CO₃ obtained by co-precipitation with NH₄HCO₃ and NH₄Al(SO₄)₂ as raw materials. In the experiment, 250 mL of 0.2 M NH₄Al(SO₄)₂ solution in 100 mL of 2 M NH₄HCO₃ were added at a speed of 10 mL/min with magnetic stirring and 60 °C temperature. The pH was kept constant at a certain value by adding amounts of ammonia. Kumar et al. (2013) reported the preparation of palladium nanoparticles on the surface of alumina by coprecipitation from the metal precursors: palladium (II) chloride (PdCl₂) and aluminum nitrate nonahydrate (Al(NO₃)₃·9 H₂O). In the experiment, the concentration of Al(NO₃)₃ 0.1 M was kept constant at various concentrations of PdCl₂. Ammonia solution was added to keep the pH at 9.0 and the suspension was stirred for 2 h in order to uniformly disperse the particles, the precipitate was dried at 120 °C and calcined at 600 °C for 2 h. Jbara et al. (2017) synthesized γ-alumina by the coprecipitation method starting from a solution of tris(dimethylamino) aluminum(III) (Al(N (CH₃)₂)₃) and 25% wt. ammonia. The reaction was kept under constant stirring for 3 h and the co-precipitated products were left overnight for

deposition, then the precipitate was filtered and dried at 70 °C for several hours to remove moisture, and finally, the powders were ground and calcined at several temperatures and heating ramps of 5 °C/min.

Adans et al. (2016) proposed an alternative leaching method to produce γ-alumina from aluminum cans. In this case, they carried out two consecutive precipitation reactions, the first with hydrochloric acid and the second with sodium hydroxide. 4 g of aluminum powder and 100 mL of water were added to a beaker. 100 mL of 6 M hydrochloric acid were added dropwise under constant pressure, producing $AlCl_3$. Aluminum chloride was then reacted with dropwise addition of 6 M sodium hydroxide to become $Al(OH)_3$ at room temperature. After constant stirring for 24 h, the mixture was centrifuged and the solid was washed with ammonia, dried at 110 °C for 24 h and calcined at 800 °C for 4 h. With this method they obtained 10 g of dry sample and 6 g of calcined sample of alumina. On the other hand, another synthesis of alumina was carried out, in this case replacing NaOH with ammonia as precipitating agent. This precipitating agent is interesting since, ammonia and HCl are eliminated during the calcination by decomposition of ammonium chloride and therefore do not contaminate the final solid sample although, as a disadvantage, these gases are harmful to health and the environment. Comparing the two alkaline precipitating agents used, NaOH presented more advantages than ammonia because it did not need to eliminate products that are harmful to the environment and gamma-alumina presented a surface area of 204 m^2/g, close to that of commercial alumina Cyanamid Ketjen (180 m^2/g) or that prepared by conventional precipitation (203 m^2/g).

4.2. Sol-gel method

The sol-gel method is applied in the development of new materials for catalysis (Aegerter, 2001; Øye et al., 2006), membranes (Brinker et al., 1995; Pakizeh et al., 2007; Park et al., 2008), photochromic applications (Volkan et al., 2005), chemical sensors (Carmona et al., 2007; Maduraiveeran and Ramaraj, 2007), and the nuclear and electronic industries, among others (Akpan and Hameed, 2010). Sol-gel method is based on the hydrolysis of precursors such as alkoxides, acetylacetonates, carboxylates, chlorides and nitrates to generate a homogeneous solution and produce a gel by subsequent densification or heat treatment (Torrez-Herrera et al., 2020). Machida et al., (1989, 1988) carried out the synthesis of Ba-hexaaluminates using barium and aluminum isopropoxides dissolved in 2-propanol as raw materials. Sol-gel is a technology used to synthesize powder and thin film catalysts with the additional advantage that the complex oxides can be treated at low temperature due to the homogeneity of the components at the molecular level (Doležal et al., 2019). Three approaches are used to fabricate sol-gel monoliths: Method I (colloidal powder solution gelation); Method II (hydrolysis and polycondensation of alkoxide or nitrate precursors followed by hypercritical drying of gels); Method III (hydrolysis and polycondensation of alkoxide precursors followed by aging and drying under ambient atmospheres) (Hench and West, 1990). Sols are colloidal particles dispersed in a liquid, while colloids are solid particles with diameters varying between 1 and 100 nm. A gel is a rigid red that is interconnected with pores whose average length is greater than one micrometer. A silica gel can be formed by growth of a network from a matrix of colloids (Method I) or by formation of an interconnected three-dimensional network by simultaneous hydrolysis and polycondensation of a precursor (Method II and Method III) (Saupe, 1965). When the liquid in the pores is removed at or near ambient pressure by thermal evaporation, it is called a xerogel (Hench and West, 1990).

Saiz et al. obtained pseudoboehmite (AlOOH) through the reaction between aluminum and isopropyl alcohol to produce aluminum isopropoxide using mercury iodide as a catalyst at a temperature of 80 and 85 ºC for 3 h. Once the alkoxide was obtained, it was hydrolyzed carrying out a condensation reaction. The next stage was peptization and required an acid as a peptizing agent, in this case nitric acid. Peptization consisted of taking a 2 M solution of pseudobohemite and heating it to 80 ºC, slowly adding the acid, with constant stirring and reflux (Saiz et al., 2011). Yoldas et al. (Yoldas, 1975) synthesized a transparent alumina sol by hydrolyzing aluminum trisopropoxide with water. After heating a mixture of the alkoxide and water at 80 °C for 1 h with stirring, acetic acid was added as a peptizing agent. After 8 h of reaction, the solution became clear. Li et al. (2006) used $Al(NO_3)_3 \cdot 9 H_2O$ as starting material and citric acid ($C_6H_8O_7 \cdot H_2O$) to prepare α-Al_2O_3. The molar ratios of citric acid to metal nitrate were 0.5, 1.0 and 2.0. The solution was stirred for several hours at a temperature of 60 °C until it turned into a yellowish gel; subsequently, the nitrate-citrate sol was rapidly heated to 80 °C with constant stirring. Finally, a thermal treatment at 200 °C for 2 h was carried out, generating a spongy polymeric citrate precursor. In order to obtain fine alumina powder, it was calcined and ground at various temperatures in a muffle furnace.

Rajaeiyan and Bagheri-Mohagheghi (2013a) performed the comparison between the sol-gel and coprecipitation methods regarding the structural properties and the phase transformation of nanoparticles of γ and α alumina. The results showed that both the γ and α phases were reached at lower temperatures with the coprecipitation method. Regarding the size of the Al_2O_3 particles synthesized by sol-gel, it was between 10 and 15 nm and that of the particles synthesized by coprecipitation showed values of 10–50 nm. At 750 °C, the coprecipitation powder presented a higher specific surface area (206 m^2/g) compared to the sol-gel (31 m^2/g). The same authors (2013b) used the sol-gel method based on the Pechini process to synthesize various phases of alumina nanoparticles starting from aluminum nitrate. Pechini's process dates back to the 1967 patent where a method for the fabrication of thin capacitor films by using multifunctional organic acids capable of chelating metal ions into stable complexes and a diol that served as a solvent during the formation step of the complex was reported (Estrada-Moreno et al., 2017). Rajaeiyan and Bagheri-Mohagheghi (2013b) studied the effect of two complexing agents: urea and citric acid. The size of alpha-alumina synthesized with urea was reported to be 10–15 nm, and that produced with citric acid had a particle size of 200 nm. In addition, using urea a specific surface area of 84 m^2/g was obtained, while it was 40 m^2/g when using citric acid.

4.3. Hydrothermal synthesis

Hydrothermal synthesis procedure has great environmental advantages and also consumes low energy as it is carried out in a single step without the need for subsequent high-temperature calcination or extensive grinding (Ghanizadeh et al., 2014). Among other advantages, this process allows excellent control of particle morphology together with high chemical purity, low level of aggregation and a narrow distribution of crystallite sizes in the product (Suchanek et al., 2010).

The phase relationships in the Al_2O_3-H_2O system have been extensively studied (Ervin and Osborn, 1951; Ghanizadeh et al., 2014; Laubengayer and Weisz, 1943; Kennedy, 1962; Suchanek et al., 2010; Su and Li, 2011). The presence of four phases in the temperature range 0–500 °C and pressure range 1–100 MPa has been demonstrated. These phases include $Al(OH)_3$ (gibbsite), γ-AlOOH (boehmite), α-AlOOH (diaspore), and α-alumina. When the temperature is increased under hydrothermal conditions, boehmite converts directly to α-alumina at 380 °C and pressures between 1 and 15 MPa without forming transition alumina (see Fig. 10).

For the synthesis of α-alumina, Datta et al. (2014) used the generated aluminum slag residue as raw material in a two-step procedure. In the first step, almost 90% of the aluminum in the waste was recovered as nanocrystalline boehmite, γ-AlOOH, by hydrothermal treatment. In the second stage, alumina was obtained by calcination of boehmite at 1400 °C. The final composition of the alumina produced was 95% Al_2O_3, 3.3% Fe_2O_3, 0.8% SiO_2 and traces of other oxides.

The supercritical water method is one of the hydrothermal synthesis methods. The use of this method reduces losses in the materials although it causes agglomeration and large size in secondary particles (Noguchi
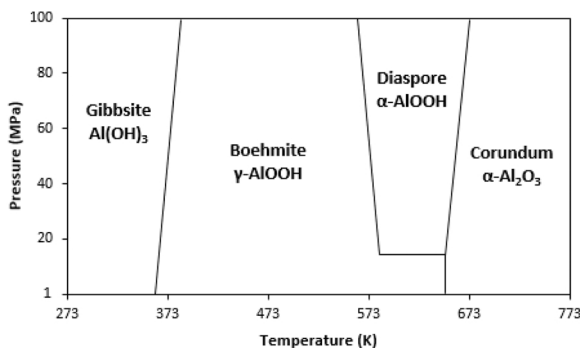
Fig. 10. Transition phases of alumina under hydrotreatment (Suchanek et al., 2010).

et al., 2008). These authors reported the rapid synthesis of γ-alumina nanoparticles using supercritical water by hydrothermal continuous flow reaction system. The raw materials used were aluminum nitrate (Al(NO$_3$)$_3$·9 H$_2$O) and distilled water (18 ohm). The water was placed in a container and withdrawn through a high pressure hose (25–35 MPa) entering an electric heater at 585 ºC. Aluminum nitrate was flowed through another hose at the same high pressure. The hydrothermal reaction took place when the two reactants entered the reactor tube at the correct temperature producing γ-alumina and AlOOH.

4.4. Combustion method

The combustion method is a complex, autonomous, chemical process that uses a homogeneous precursor solution. The initial stage of the process requires dehydration and deficiency of the precursor solution and involves several exothermic reactions that produce at least one solid and a significant volume of gas (Varma et al., 2016).

Syarif et al. (2019) successfully synthesized Al$_2$O$_3$ nanoparticles by self-combustion using Al(OH)$_3$ as precursor. The Al(OH)$_3$ powder extracted from the local bauxite was placed in a beaker with water and commercial sugar (mostly sucrose) was poured with continuous mixing (Al(OH)$_3$ and sugar ratio was 1:1). The mixture formed a sol which was heated to 150 °C whereupon a gel was formed which was heated to 600 °C and self-ignited at 450 °C. The material produced in the self-combustion was calcined at 1200 °C for 1 h to obtain Al$_2$O$_3$ nanoparticles. Karasev et al. (2004) generated alumina nanoparticles by burning aluminum droplets in air at atmospheric pressure. In the initial stage of alumina synthesis, liquid oxide particles were formed in the reaction zone located around the surface of the burning aluminum droplet. At relatively low temperatures, solidification and grouping of solid Al$_2$O$_3$ particles occurred. The solid rocket fuel sample was burned in a 20-liter combustion chamber previously filled with air filtered with a high-efficiency Petrianov aerosol filter. The sample burned within 5 s. The size of the aggregates varied in the range of 0.1 to a few micrometers. Aggregates were reported to be positively or negatively charged and the typical chart was a few elemental units. Afruz and Tafreshi (2014) successfully synthesized γ-alumina nanoparticles by various combustion methods using ammonium carbonate and aluminum nitrate as precursors. For the gel combustion mode, ammonium carbonate solution was added dropwise to the maintained aluminum nitrate solution. Gel formed was aged at room temperature for several days and placed in a preheated oven at 400 °C. The material underwent foaming followed by high volume detection of CO$_2$, N$_2$, NO$_2$ and H$_2$O, causing spontaneous ignition.

4.5. Microwave method

A powerful and fast heat source in the synthesis of nanoparticles from liquid phase is microwave radiation. This form of heating requires less energy than conventional methods since in a microwave oven the heat is generated within the sample itself by the interaction of the microwave waves with the material (Hasanpoor et al., 2017; Kozakova et al., 2015; Kumar et al., 2020; Sharifi et al., 2013). Hasanpoor et al. (2017) produced alumina nanoparticles from aluminum nitrate using five green plants for microwave-assisted synthesis. The solution was irradiated with a frequency of 2.5 GHz as a heat source for 10 min at 610 W. Subsequently, the irradiated solution was centrifuged and washed with ethanol and deionized water. The dissolved powder was treated with ultrasound at 150 W to avoid agglomeration.

4.6. Wet chemical method

Chemical synthesis is the most convenient route to produce ceramic particles (López-Juárez et al., 2018). The wet chemical method is based on letting a precipitate fall into a solution at a specific rate, the pH of the solution being one of the crucial parameters (Ziva et al., 2021). Lu et al. (2005) synthesized α-Al$_2$O$_3$ using Al(NO$_3$)$_3$·9 H$_2$O and ammonia as precursors. As an efficient additive for the formation of alpha-alumina particles, 2% mol of aluminum nanoparticles were introduced under strong agitation to obtain a complete dispersion. Aluminum nitrate was dissolved in distilled water and aluminum powder was added. Ammonia was dropped into the mixture at 100 drops/min with dilute HCl added to prevent aggregation.

4.7. Mechanochemical method

The mechanochemical method uses chemical processes combined with the use of pressure tools (Bodaghi et al., 2009). The most widely used procedure is ball milling to grind the powder to a desired particle size (Thamizharasan and Saravanan, 2017; Ziva et al., 2021). Using AlCl$_3$·6 H$_2$O and NH$_4$HCO$_3$ as precursors, α-alumina nanoparticles were prepared by both wet chemical and mechanochemical methods by Gao et al. (2018). In the wet chemical method, AlCl$_3$·6 H$_2$O and NH$_4$HCO$_3$ were dissolved in deionized water and filtered. NH$_4$AlO(OH)HCO$_3$ precipitate, abbreviated AACH, was synthesized by adding AlCl$_3$ solution dropwise to NH$_4$HCO$_3$ solution with vigorous stirring. The pH value was adjusted with ammonia to 9–10. After the completion of precipitation, the precursors were collected by vacuum filtration and washed. The resulting mixture was sonicated for 30 min, filtered and dried at 60 °C. Calcination of dry AACH was carried out at 1150 °C for 1.5 h. In the mechanochemical method, AlCl$_3$·6 H$_2$O and NH$_4$HCO$_3$ were mixed and ground for 15 min at room temperature. Then 20 mL of ammonia was added and the mixture was milled for further 5 min. The pH was maintained at 9–10. The precursor was filtered and washed with deionized water and ethanol, respectively. The calcination was carried out at 1100 °C for 1.5 h. Comparing the two "green" processes, the best prospects for commercial production were presented by the mechanochemical method.

4.8. Hydrolysis method

This method is based on the hydrolysis reaction of the raw materials used to synthesize Al$_2$O$_3$ nanoparticles with a large surface area and high thermal stability. Wang et al. (2009) used stoichiometric ratios of aluminum phosphide and deionized water as raw materials. First, the deionized water was added to the aluminum phosphide (powder) in the reactor (constant reaction temperature at 90 ºC). When the reaction ended, a precipitate of aluminum hydroxide was obtained, which was filtered and washed with deionized water. The Al$_2$O$_3$ particles were obtained by calcination at 500 °C for 3 h.

4.9. Cellulose template method

In the cellulose template method, a porous polymer is used as a template to prepare oxide materials with porous structure. Using this

method, Shigapov et al. (2001) have formed Al_2O_3 doped with lanthanum with a specific surface area of 141 m^2/g after calcination at 1050 °C for 12 h. Malgras et al. (2015) used a multilayer fused film of ammonium amphiphiles as a molecular template to obtain multilayer Al_2O_3 thin films with crystalline anisotropy and a large surface area.

5. Summary and conclusions

Globally, some 4,000,000 tons of aluminum saline slags are produced worldwide each year. This residue mainly contains Al_2O_3, SiO_2, $MgAl_2O_4$, Fe_2O_3, CaO, MgO, salts and elemental aluminum, among others. In most cases, this hazardous waste is deposited in landfills, producing serious consequences because it is toxic, flammable and irritating. An effective way to avoid this negative impact is by reusing aluminum slags in the production of materials such as alumina. Alumina (Al_2O_3) is a widely used material in industry, presenting itself as one of the most important materials in the world for future development as it has very attractive characteristics such as its large surface area, high chemical and thermal stability, and high mechanical resistance. It presents numerous crystalline phases although only two of them are commercially available, γ- and α-forms, being α-Al_2O_3 (corundum) the only thermodynamically stable phase. The most used way of recovering the aluminum present in the slag is by leaching the residue, either by acid or alkaline route. Numerous researchers have reported the synthesis of alumina from aluminum residues using both pathways, although technically the acid process is more attractive than the alkaline process due to the low iron content and high silica content of the aluminum ash. Finally, several methods have been reported that are used in the synthesis of alumina including precipitation, sol-gel, hydrothermal synthesis, combustion, microwave, wet chemical, mechanochemical, hydrolysis and cellulose template. Precipitation method is the most effective and widely used since it is an easy and simple process that can be carried out with low-cost raw materials, produces a relatively low level of contamination and also achieves thermal stability, a high purity in the product and nanoparticles of practically homogeneous size.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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