Processing of Pond Liquor from an Alumina Refinery

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Abstract

Increasing alumina production in the Russian Federation leads to an increase in bauxite residue. An important parameter affecting bauxite residue behaviour during storage is the presence of a significant amount of aqueous pond liquor. The pond liquor, which is stored in the bauxite residue disposal areas (BRDAs), contains a significant amount of alumina that gradually precipitates from the pond liquor and increases the non-recoverable alumina production losses. Moreover, pond liquors have a high pH, increasing the environmental impact of the BRDAs. This paper examines how flue gas carbonation of pond liquors allows for the commercial extraction of valuable components, producing various aluminium hydroxide products − such as amorphous aluminium hydroxide, pseudoboehmite, and hydrated sodium carboaluminate − depending on the raw material. Furthermore, the study discusses processing options to produce these products from pond liquors. Besides the production of new value-added products, additional benefits of the process include reducing pH of liquid effluents and improving the carbon footprint due to the treatment and capture of carbon dioxide from the flue gases.

Keywords: Carbonation, Pond Liquor, Aluminium Hydroxide, Processing.

1. Introduction

Global consumption of aluminium is steadily growing, resulting in a corresponding increase in primary production. The Bayer process, the main method for processing bauxite, produces large amounts of bauxite residue − a challenging industrial waste to recycle. It is estimated that the refineries of the Urals region already have approximately 150–200 million tonnes of bauxite residue in storage, while global accumulation is estimated at 4.0–5.0 billion tonnes [1].

Specialized hydraulic storage facilities, known as Bauxite Residue Disposal Areas (BRDAs), are used for the accumulation and long-term storage of bauxite residue. These sites can serve as cascade pressure reservoirs reaching heights of 30 to 50 metres. The BRDA area varies from 50 to 100 hectares, with the total storage volume reaching tens of millions of tons of solid and liquid wastes [2]. Pond liquor, the alkaline liquid component in the lower part of the BRDA, represents a significant fraction of the internal volume of the BRDA and remains in the BRDA after the precipitation of the solid phase.

Pond liquor originates from several processes, including the thickening, settling and re-slurring of the bauxite residue [3]. The largest volumes of pond liquor originate from dilution of the bauxite residue prior to transportation or washing. Since the particle size distribution, composition and mechanical properties of the bauxite residue depend on the raw materials and the processing conditions, the re-slurring conditions vary among the refineries. In turn, this causes differences in the pond liquor composition and concentration. The content of aluminium oxide (Al_2O_3) in the pond liquor can be relatively high, which results from incomplete extraction of aluminates at previous stages of the process [4]. The disposal of such liquors to the BRDAs causes an accumulation of soluble alkaline phase, accompanied by irreversible losses of the target product, alumina, which reduces the overall refinery performance.

Large volumes of pond liquor make the operation of the BRDAs more challenging and significantly increases the total volume requiring storage in the BRDA. Therefore, new areas for BRDA site expansion are constantly needed. Currently, the total area of land occupied by such facilities exceeds 1 500 h, which has a negative impact on the environment, including soil degradation and disruption of natural landscapes. An additional environmental burden results from the need to engineer and construct protective structures, including dams, membranes, and compaction walls. The soil typically used for their construction is removed directly from adjacent territories, without any specialized quarry development, which increases the man-induced impacts on the ecological system near the production area [4].

In addition to the intensive land requirements, BRDAs represent increasing environmental risk. One of the key hazards is the possibility of alkaline pond liquor penetration into surface and underground water, both due to leakages during routine operation and in emergencies. The main component in such liquors is sodium, which can reach the environment in the form of soluble compounds, including hydroxides and carbonates [5]. Moreover, pond liquor often contains constituents from the refinery's surface runoff, containing heavy metals, fluorides, and other industry-related compounds. Such leaks can lead to large-scale pollution of water bodies and degradation of the soil cover, especially in the event of dam failures or other emergencies. In emergency scenarios, millions of cubic meters of highly alkaline liquid waste may be released into the environment, leading to catastrophic consequences for the ecological systems in the adjacent areas.

Despite the considerable experience of engineering protection and monitoring of BRDAs, the risk of emergencies related to spills of alkaline wastes remains high. Moreover, growing climate instability and occurrence of natural abnormalities (heavy precipitation, floods, and seismic activity) increase the probability of industry-related incidents [6]. In this regard, it becomes obvious that environmental risks can be reduced only by eliminating the traditional concept of long-term storage of bauxite residue and adopting the processing and utilization through the extraction of valuable components.

Depending on the applied technologies, existing approaches to the bauxite residue processing can be divided into three main groups: pyrometallurgical, hydrometallurgical and direct application technologies, which involve the use of bauxite residue as a component of construction materials, sorbents or catalysts [7]. Despite the active theoretical and applied research in this field, a single uniform solution that ensures a high degree of extraction of valuable components, environmental safety, and economic feasibility has not been developed yet. It significantly limits the large-scale integration of existing technologies into commercial production.

An alternative approach to reducing the volume of stored material in BRDAs considers the utilization of the liquid phase, i.e. pond liquor and associated solids. As noted above, this liquid contains a significant amount of alumina in form of soluble aluminate ions, which makes it a promising and available source of aluminium hydroxide. A major advantage of extracting alumina from the pond liquor is the possibility to implement this process without interference with the main process flows, as opposed to processing strong aluminate liquors used in the main production [2]. Thus, the use of pond liquors as a secondary raw material could facilitate the minimization of environmental risks associated with their accumulation, as well as provides an additional economic benefit due to the generation of value-added products.

2. Pond Liquor Carbonation Tests.

In the Russian Federation, alumina production mainly uses two processes: combined Bayer-sintering and sintering. The combined process is used at the RUSAL Kamensk-Uralsky refinery (UAZ refinery) and RUSAL Krasnoturyinsk refinery (BAZ refinery), while RUSAL Achinsk refinery (AGK refinery) applies the sintering process.

Regardless of the applied process, the water balance for alumina production comprises a significant contribution of recycled water used for filtration, washing of bauxite residue, and its hydraulic transport. As a result, the liquid phase is enriched with sodium and aluminate ions; therefore, a large amount of Na_2O and Al_2O_3 passes into the pond liquor. Based on the results of X-ray diffraction analysis (XRD) and chemical and physical tests, crystalline components of boehmite (γ-AlO(OH)), calcite ($CaCO_3$), tricalcium hydroaluminate ($3CaO \cdot Al_2O_3 \cdot 6H_2O$) and hematite ($\alpha$-$Fe_2O_3$) were identified in the pond liquor.

To extract valuable components from the pond liquor, predominantly aluminium hydroxide, the carbonation process was considered using the exhaust gases of sintering kilns. To evaluate the effectiveness of the proposed method, laboratory tests were conducted using the samples of pond liquor collected from the BAZ, UAZ and AGK refineries. Table 1 presents the chemical composition of the samples.

Table 1. Chemical composition of the pond water.

Refinery	**Pond water concentration, g/L**				**αk (molar Na_2O/Al_2O_3 ratio)**
	Al_2O_3	Na_2O_{total}	$Na_2O_{caustic}$	$Na_2O_{carbonate}$	
BAZ	7.2	10.7	10.6	0.1	2.4
UAZ	3.3	4.6	4.1	0.5	2.0
AGK	2.5	17.7	6.2	11.5	4.1

Each of the refineries studied in this paper utilise sintering kilns in their process. The exhaust gases from these kilns, enriched with carbon dioxide, are an accessible and technically and economically feasible neutralizing agent. Neutralisation using carbon dioxide allows for the precipitation of residual aluminium and sodium from the pond liquors by the carbonate mechanism.

The parameters of the experiment were selected taking into consideration the actual conditions at each of the refineries included in this study. All carbonation tests were carried out at a temperature of 40 ˚C using a pond liquor sample volume of 2 L and a carbon dioxide-containing gas-air mixture flow rate of 3 L/min. The reaction reached completion when a pH=8.5 was achieved, corresponding to the neutralization of the alkalinity and the precipitation of the target components. Table 2 presents the results of the experiments.

Table 2. Results from the carbonation of alumina refinery pond liquors.

Liquor	**CO_2 concentration, wt.%**	**Reaction time, m**	**Phase composition of the precipitate**	**Na_2O content in the precipitate, wt.%**
BAZ	5	70	PB	0.1
UAZ	10	44	AmHA	0.15
AGK	18	53	GCAS	8.5

PB – pseudoboehmite; AmHA – amorphous hydroxide aluminium; GCAS – hydrocarboaluminate sodium.

The filtrates collected at the end of the carbonation process of the pond liquors when the pH reached 8.5 did not contain soluble aluminium compounds, which indicates the almost complete precipitation of aluminium into the solid phase.

XRD was used to determine the phase composition of the solid products. BAZ pond liquors treated with flue gas formed pseudoboehmite (AlOOH), as evidenced by the characteristic broadened diffraction peaks typical for this phase (Figure 1). Carbonation of pond liquors from UAZ generated more amorphous phase of aluminium hydroxide in the precipitate (Figure 2), which may be due to the specific ionic composition of the liquor and the low crystallinity of the product. XRD results for the AGK carbonated pond liquors showed that the main phase of the precipitate was amorphous sodium hydrocarboaluminate, which reflects the interaction of aluminate and sodium ions under carbonation conditions (Figure 3).

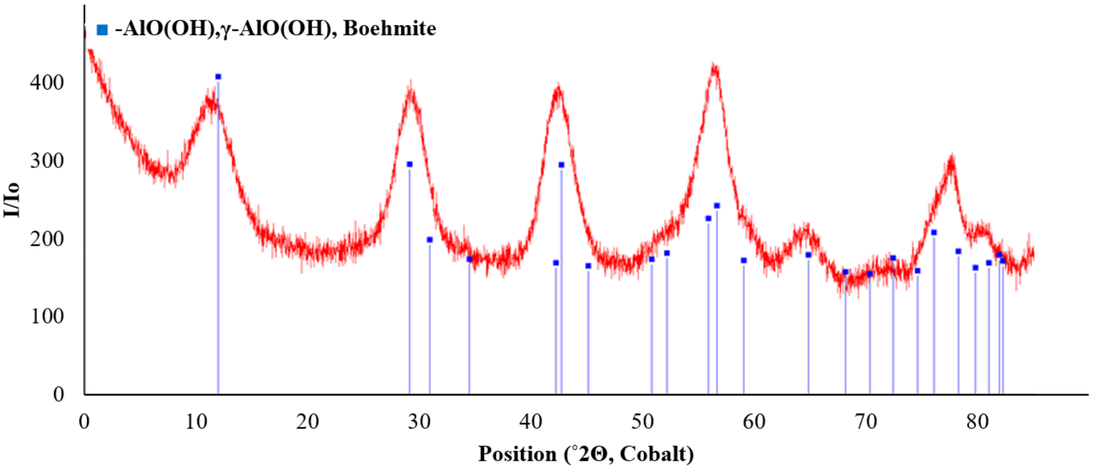

Figure 1. XRD of the solid phase after carbonation of pond liquor from the BAZ refinery.

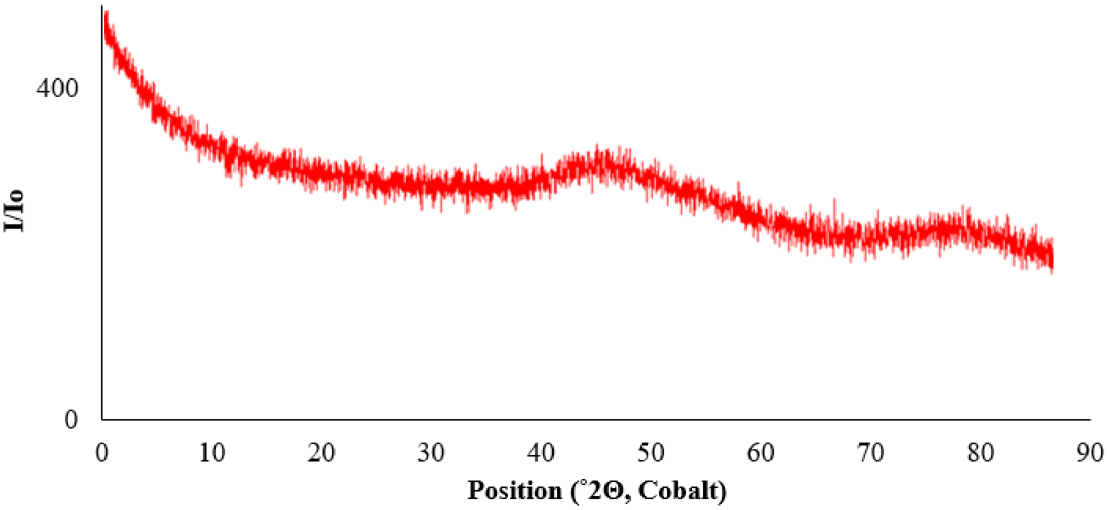

Figure 2. XRD of the solid phase after carbonation of pond liquor from the UAZ refinery.

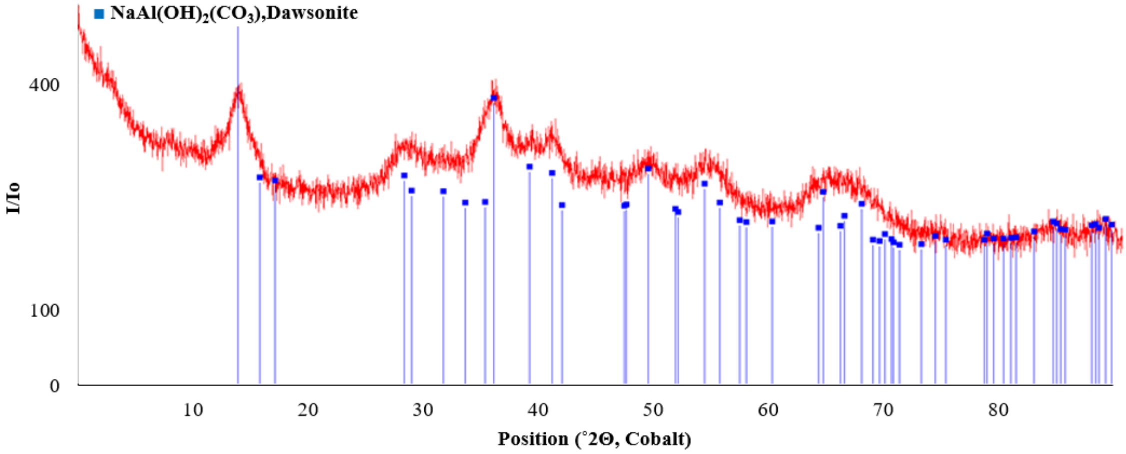

Figure 3. XRD of the solid phase after carbonation of pond liquor from the AGK refinery.

To determine the effect of the temperature on the kinetics of the aluminium hydroxide precipitation, laboratory tests using BAZ pond liquor were conducted at different temperatures: 20 °C, 40 °C and 60 °C. The chemical properties of the initial solution were as follows:

- Concentration of Al_2O_3: 7.3 g/L,
- Concentration of $Na_2O_{caustic}$: 10.4 g/L,
- pH: 12.

Figure 4 illustrates the laboratory setup used for these experiments. For each test, 1 litre of pond liquor was placed into the beaker and the liquor was heated to a specified temperature. A gas-air mixture containing carbon dioxide was injected into the pond liquor while being stirred using a two-blade mixer to ensure uniform interaction of the phases. Samples were collected at each specified carbonation time.

The samples were filtered using a Büchner vacuum flask and funnel . The precipitate was washed with hot distilled water at a liquid-to-solid ratio of 2:1 to remove residual alkalinity and impurities.

At each temperature examined during the carbonation process, crystallization began within five minutes of the gas-air mixture being introduced, demonstrating a short induction period.

Figures 5 and 6 show the kinetics of pH change and aluminum precipitation for each test. Figures 5 & 6 both illustrate typical curves characterizing the carbonation process of pond liquors under different temperature conditions. The curve has four discrete sections corresponding to the successive stages of chemical and phase transformations:

- Section 1: neutralization of free (caustic) alkali, accompanied by a decrease in pH due to the interaction of hydroxide ions with carbon dioxide;
- Section 2: initiation of aluminate ion breakdown followed by the formation of amorphous aluminium hydroxide;
- Section 3: crystallization of aluminium oxyhydroxide, mainly in the form of boehmite;
- Section 4: formation of bicarbonate, in which the maximum degree of precipitation of aluminium hydroxide and stabilization of the pH of the system are achieved.

Figure 4. Laboratory setup for carbonation of pond liquor at different temperatures.

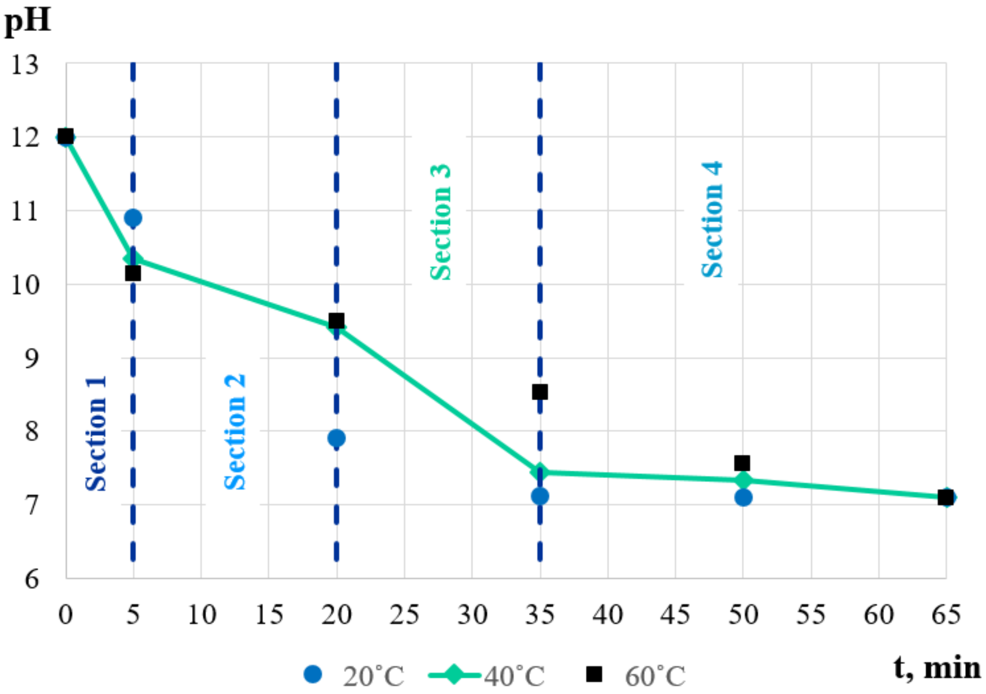

Figure 5. pH changes in pond liquor during carbonation at different process temperatures.

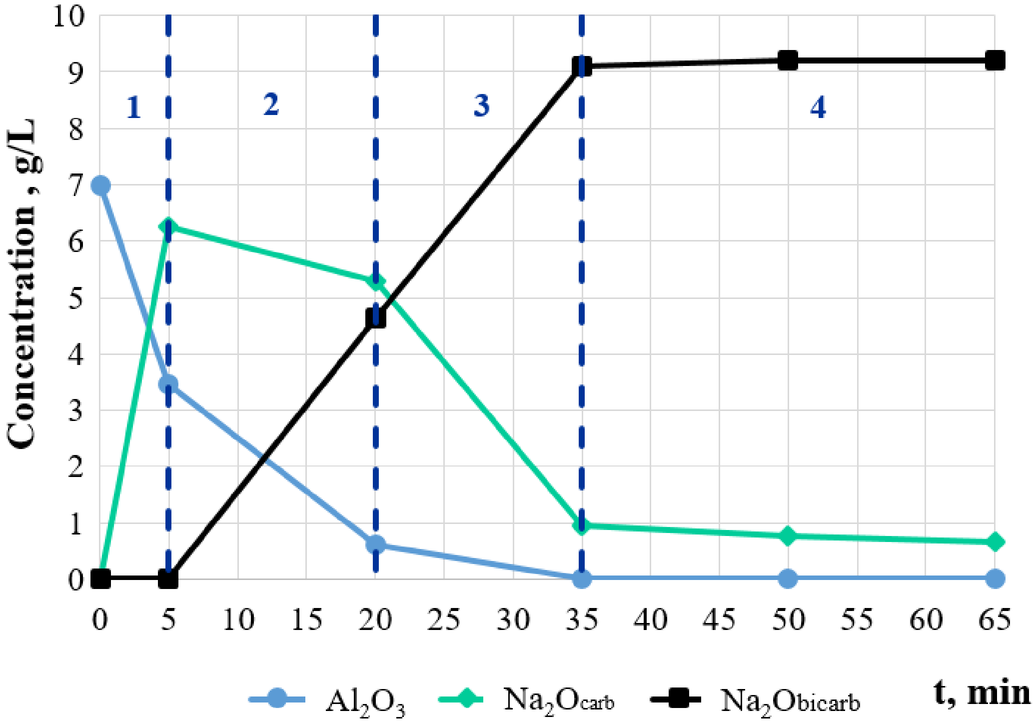

Figure 6. Concentration of Al_2O_3, $Na_2O_{carbonate}$ and $Na_2O_{bicarbonate}$ in pond liquor during carbonation at 40 °C.

Figure 5 shows that the highest rate of pH decrease was observed at a temperature of 20 °C, especially in the initial phase of the reaction (alkali neutralization). The slowest pH decrease was recorded at a temperature of 60 °C, which may be due to the thermodynamic stability of aluminate complexes and lower CO_2 solubility at elevated temperatures. Comparative analysis of the data shows that it took 20 min to achieve the target pH = 8 at a temperature of 20 °C, 30 min at 40 °C, and 43 min at 60 °C. Thus, a decrease in temperature promotes acceleration of neutralization and transition of aluminium to the solid phase at the early stages of carbonation.

Figure 6 illustrates the changes in concentration of Al_2O_3, Na_2CO_3 and $NaHCO_3$ (in terms of Na_2O) during carbonation of pond liquor at 40 °C, highlighting the degree of aluminium oxide precipitation with pH at different stages of the process. The data indicates that ~ 30 % of the aluminium hydroxide precipitates as the pH decreases to 11, with a further ~ 35 % precipitating when the pH is decreased to 10. This reached ~ 85 % precipitation when the pH decreased to 9.5. Almost complete precipitation (close to 100 %) is observed at pH 7.5, which corresponds to the bicarbonate buffer zone favourable for completing the carbonation reaction.

At the initial stage of the carbonation process, sodium carbonate (Na_2CO_3) forms exclusively due to the interaction of carbon dioxide with the hydroxide-ion alkaline component. Complete neutralization of the active alkali ($Na_2O_{caustic}$) at a temperature of 40 °C is completed within the first 7 minutes of the process, which corresponds to Section 1 of the curve.

At subsequent stages of the carbonation process (with a decrease in pH to the range of 9.5–8.5), decomposition of previously formed Na_2CO_3 is observed to form sodium bicarbonate ($NaHCO_3$). The results show a decrease in the concentration of Na_2O ($Na_2O_{carbonate}$) to below 1 g/L after 33 minutes of carbonation, which then stabilizes until the end of the carbonation process.

3. Carbonation Process of Pond Liquor for Production Conditions

Drawing on the results of the conducted tests, technological methodologies have been established for producing amorphous aluminium hydroxide and pseudoboehmite from alumina production pond liquor through carbonation with carbon dioxide-containing gases.

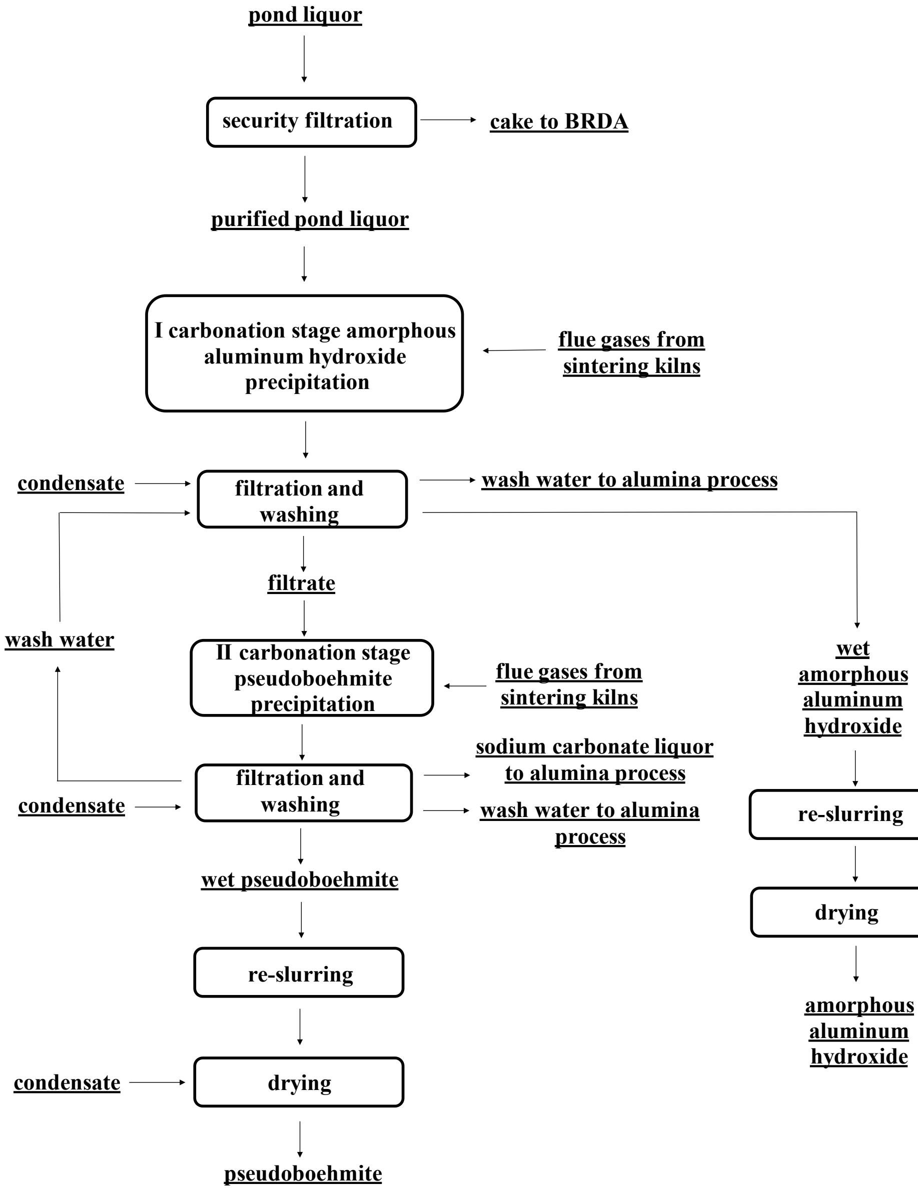

Figure 8. Process for carbonation of pond liquor from alumina production.

The developed process using BAZ alumina refinery pond water liquid phase includes a two-stage carbonation, allowing for the gradual precipitation of various phases of aluminium hydroxides from the pond liquors with subsequent production of target products in form of powders (Figure 8).

The main stages of the process are as follows:

1. The pond liquor having pH = 12 coming from the BRDA is supplied to the preliminary (security) filtration stage. The separated solid fraction (residue) is transported to the BRDA, while the filtrate is fed to the clarified pond liquor receiving tank.
2. From the receiving tank, the pond liquor is fed to the first stage of carbonation in the carbonation reactor. At the same time, flue gases cooled to 40–50 ºC and pre-treated in a bag filter are supplied into the reactor.
3. The carbonation process is carried out until the pH of 11–10.5 is reached. In this range, amorphous aluminium hydroxide precipitates. The resulting slurry is filtered in a filter press, and the resulting filtrate is accumulated in a collecting mixer.
4. The cake from the filter press is washed using filtrate from the pseudoboehmite filtration and washing unit, followed by washing and reslurry with process condensate from the alumina refinery circuit. The resulting slurry is fed to the spray drying.
5. Dried amorphous aluminium hydroxide powder is fed to the product packaging unit via a screw conveyor.
6. The filtrate obtained after the first carbonation stage is sent to the secondary carbonation unit, where the liquor is further saturated with carbon dioxide a pH of 9.0–8.0. At this stage, pseudoboehmite is precipitated. The resulting slurry is sent for secondary filtration to the filter press.
7. The filter cake is washed with condensate at 80–90˚C to improve the phase crystallization of pseudoboehmite and remove residual alkalinity.
8. The final filtrate is mixed with part of the wash water in the auxiliary mixer and returned to the alumina refinery circuit. The remaining part of the wash water is used to wash the amorphous aluminium hydroxide precipitate from the previous stage.
9. The filtered precipitate enters the agitated re-slurry tank, where it is mixed with hot condensate, forming a homogeneous slurry suitable for drying.
10. The obtained pseudoboehmite powder is also transported to the product packaging unit.

The implementation of the proposed process allows for obtaining of two products:

- Amorphous aluminium hydroxide for use in the construction industry as an accelerator of concrete hardening and is also a preferred raw material for the production of coagulants with better reactivity compared to ordinary aluminium hydroxide.
- Aluminium hydroxide in form of pseudoboehmite. The resulting product is in high demand in the market as a raw material for catalyst carriers. Figure 9 and Table 3 present the physical and chemical properties of the product.

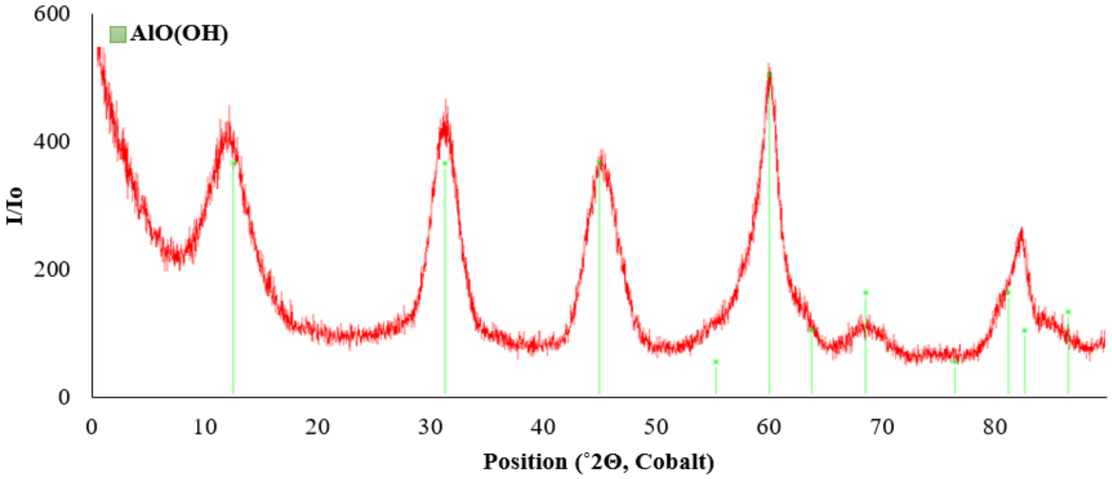

Figure 9. Pseudoboehmite XRD.

Figure 9 shows broad, diffuse peaks characteristic of amorphous substances. The absence of clear diffraction reflections confirms the amorphous structure of the obtained aluminium hydroxide, which corresponds to the pseudoboehmite phase.

Table 3. Chemical composition of pseudoboehmite (PB) and amorphous aluminium hydroxide (AmHA)

Name	**LOI**	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**TiO_2**	**P_2O_5**	**CaO**
PB	29	0,05	70,8	0,01	0,01	0,01	0,01
AmHA	40.48	0.45	57.3	0.02	0.05	0.12	0.05
Name	**MgO**	**Na_2O**	**K_2O**	**V_2O_5**	**Cr_2O_3**	**MnO**	**SO_3**
PB	0,01	0,01	0,01	0,01	0,01	0,01	0,1
AmHA	0.2	0.8	0.3	0.08	0.11	0.01	0.03

Table 4 presents the main target product specifications for aluminium hydroxide raw materials used as catalyst carriers with the product quality of the product obtained from the pond liquor carbonation process.

Table 4. Comparison of the pseudoboehmite sample quality and product specifications.

Description	**Value**	
	Target specification	**Sample #1**
Phase composition	**pseudoboehmite**	**pseudoboehmite**
Specific surface area (BET), m^2/g	>250	290 - 310
BJH Desorption cumulative volume of pores, cm^3/g	>0,8	1
BJH Desorption average pore diameter (4V/A)), nm	>10	13
Particle size (d_{50}), µm	8.5±0.5	5
Crystallite size, nm	1-5	6
Bayerite content	no	no
Na_2O, ppm	<100	100

Fe_2O_3, ppm	<100	100
SiO_2, ppm	<100	500

An analysis of the specifications for consumers of aluminium hydroxide raw materials to produce oil refining catalysts showed that the quality of the obtained product met most of the specified parameters indicators.

4. Conclusion

In the context of steady growth of global demand for aluminium hydroxide, there is a growing need to improve its production while also minimizing waste generation. The advancement of alternative methods, such as extracting aluminium hydroxide from alumina refinery pond liquor, represents a promising strategy for enhancing the alumina industry. The proposed carbonation process allows for the efficient use of secondary alkaline liquors containing dissolved aluminium, while also delivering a reduction in the volume of industry-related waste requiring storage in bauxite residue disposal areas. It delivers both an environmental benefit due to the reduction in the volume of disposed residue, and an economic benefit due to increasing the yield of commercial aluminium hydroxide without the need for additional resources.

This paper introduces a reliable, reproducible process flow diagram for pond liquor carbonation. Experiments show that aluminium hydroxide can be produced as pseudoboehmite, meeting commercial quality standards.

The results demonstrate the practical applicability of this process at operating alumina refineries. Tests performed on samples of the pond liquor from RUSAL Kamensk-Uralsky (UAZ), JSC RUSAL Krasnoturinsk (BAZ) and JSC RUSAL Achinsk (AGK) confirmed the flexibility of the process, both for alumina refineries using the sintering process and for alumina refineries using the Bayer-sintering method.

5. References

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