

Development and Process Optimization of a Mini-Technology for Producing High-Purity Sodium Carbonate from Technical Soda

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ABSTRACT: A mini-technology for the production of high-purity sodium carbonate suitable for laboratory and semi-industrial applications has been developed based on technical grade soda ash. The proposed process includes dissolution, filtration, controlled carbonation with carbon dioxide, crystallization of sodium bicarbonate, thermal calcination, and final product conditioning. Process parameters such as CO₂ flow rate, temperature, and excess gas ratio were systematically optimized to ensure high yield and product purity. Optimal carbonation conditions were established at a CO₂ flow rate of 20 m³/h with a 7% excess over stoichiometric demand and a temperature range of 55–65 °C, resulting in a product yield of 78.8% and a sodium carbonate purity of 99.87%. The concentrations of iron and heavy metals were reduced to 10⁻⁵–10⁻⁶%, meeting the requirements for high-grade chemical reagents. Additionally, process modeling and simulation were performed using Aspen Plus software with ELECNRTL and Peng–Robinson thermodynamic models to validate mass and energy balances and to support process scalability. The developed mini-technology demonstrates technical feasibility, reproducibility, and potential applicability for local production of high-purity sodium carbonate in research laboratories and educational or pilot-scale facilities.

KEY WORDS: Sodium carbonate; process optimization; carbonation; mini-technology; chemical engineering; Aspen Plus simulation; high-purity reagents.

I. INTRODUCTION

Sodium carbonate (Na₂CO₃) is one of the most widely used inorganic chemicals and is extensively applied in the chemical, food, glass, pulp and paper, and related industries. In laboratory practice and analytical chemistry, sodium carbonate of high purity is particularly important, as it is used for the preparation of buffer and standard solutions, titrimetric analyses, and research procedures requiring a minimal content of inorganic and metallic impurities [1].

Despite the large-scale industrial production of sodium carbonate, the availability of high-purity grades suitable for analytical and research applications remains limited, especially in regions dependent on imported chemical reagents. This situation increases the cost of laboratory work and creates additional logistical constraints for educational and research institutions. Consequently, the development of compact and reproducible mini-technologies for producing high-purity Na₂CO₃ using standard laboratory or pilot-scale equipment is of significant practical interest [2].

Industrial sodium carbonate is mainly produced via the ammonia–soda (Solvay) process, processing of natural carbonate minerals, and subsequent manufacture of technical-grade soda ash. However, soda ash obtained by these routes typically contains chloride, sulfate, calcium, iron, and organic impurities. The presence of these

components restricts its direct use in analytical chemistry and reagent preparation without additional purification steps [3].

Previous studies have shown that carbonation-based routes involving bicarbonate and sesquicarbonate intermediates can be effectively applied to improve the purity of sodium carbonate and related products. In particular, the purification of sodium bicarbonate from mother liquors formed during sesquicarbonate production has been demonstrated as a viable approach for reducing impurity levels [1]. Patented technologies further emphasize the importance of controlled carbonation, solid–liquid separation, and thermal treatment as key stages for obtaining sodium carbonate of chemically pure and analytical-grade quality [2]. Experimental investigations of soda solution carbonation with carbon dioxide have also confirmed that parameters such as temperature, gas flow rate, and solution composition play a decisive role in phase formation, yield, and impurity distribution [3]. Based on these considerations, the objective of the present work was to develop and validate a mini-technology for upgrading technical soda ash to high-purity sodium carbonate suitable for chemically pure and analytical-grade applications. The proposed approach is designed for implementation under laboratory and semi-industrial conditions and focuses on process reproducibility, technological simplicity, and stable product quality.

II. SIGNIFICANCE OF THE SYSTEM

This article investigates the development of a mini-technology for producing high-purity sodium carbonate from technical soda ash. The materials and experimental methodology are described in Section IV, the experimental results and their discussion are presented in Section V, and Section VI summarizes the main conclusions and outlines directions for future research.

III. METHODOLOGY

A. Raw Material

The object of the study was technical-grade soda ash (sodium carbonate, Na_2CO_3) produced by the Kungrad Soda Plant (Republic of Karakalpakstan, Uzbekistan). This material was used as the starting raw material for the development of a mini-technology aimed at producing sodium carbonate of chemically pure and analytical-grade quality.

B. Reagents and Analytical Control

Analytical-grade hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) were used for titrimetric determination of sodium carbonate content and neutralization of residual impurities. Ethanol ($\text{C}_2\text{H}_5\text{OH}$, chemically pure) was applied during recrystallization stages to promote impurity precipitation. Methyl orange was employed as an acid–base indicator. Aqueous ammonia and a freshly prepared 0.1% aluminum solution were used for qualitative control of aluminum traces and nitrogen-containing impurities. *p*-Nitrophenol served as an indicator for the determination of silicate impurities, while bromine water was applied for the detection of sulfur-containing species. Ammonium salts (ammonium carbonate and ammonium acetate) were used as auxiliary reagents in photometric analyses.

All aqueous solutions were prepared using distilled water, and insoluble impurities were separated using ash-free filter papers. All reagents were of chemically pure or analytical-grade quality.

C. Quality Assessment Procedures

The quality of the obtained sodium carbonate was evaluated using standard titrimetric and gravimetric methods to confirm product identity and purity. The mass fraction of Na_2CO_3 was determined by acid–base titration after thermal pretreatment of the sample to constant mass. The loss on ignition was measured by calcination at elevated temperature until a constant mass was achieved. Insoluble matter was determined by filtration of an aqueous solution followed by drying and weighing of the residue.

Trace impurities, including nitrogen-containing compounds, silicates, sulfates, phosphates, chlorides, and aluminum, were assessed using photometric and turbidity-based methods. The obtained values were compared with reference standards to confirm compliance with the requirements for chemically pure and analytical-grade sodium carbonate.

D. Process Modeling and Simulation

Process modeling and simulation were performed using Aspen Plus software to support technological validation and assess scalability. The system components included H_2O , CO_2 , Na_2CO_3 , and NaHCO_3 , for which physical and thermodynamic properties such as density, heat capacity, enthalpy, and phase behavior were defined. Thermodynamic calculations were carried out using the ELECNRTL and Peng–Robinson models, which provide reliable descriptions of electrolyte solutions and gas–liquid equilibrium.

The process flowsheet was constructed using standard Aspen Plus unit operation blocks, including MIXER for stream combination, HEATER for temperature control, FLASH2 for phase separation, SEPARATOR for solid–

liquid separation, and RSTOIC or RGIBBS blocks for reaction modeling. Operating parameters such as temperature, pressure, flow rate, and component concentrations were specified for each unit, and material connections were established to reflect the process sequence.

The main reaction considered in the model was the carbonation of sodium carbonate according to the stoichiometric equation:



The reaction was defined either by a specified conversion in the RSTOIC block or by equilibrium calculations in the RGIBBS block based on Gibbs free energy minimization. Simulation results were analyzed in terms of stream composition, temperature and pressure profiles, phase distribution, and energy demand, and were used to evaluate process efficiency, energy consumption, and final product purity.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A mini-technology for producing chemically pure and analytical-grade sodium carbonate from technical soda ash was developed and experimentally validated. The proposed process was successfully adapted for laboratory operation and small-scale semi-industrial implementation. The technological approach is based on a sequence of conventional unit operations, including dissolution, filtration, controlled carbonation, recrystallization, drying, and calcination.

The use of standard laboratory and pilot-scale equipment at each stage ensures process simplicity, reproducibility, and operational reliability. As a result, sodium carbonate with the required purity level can be obtained without the need for complex industrial installations or specialized purification systems. The integrated combination of solution purification, solid–liquid separation, and thermal treatment proved to be effective for upgrading technical-grade soda ash to high-purity sodium carbonate suitable for reagent and analytical applications.

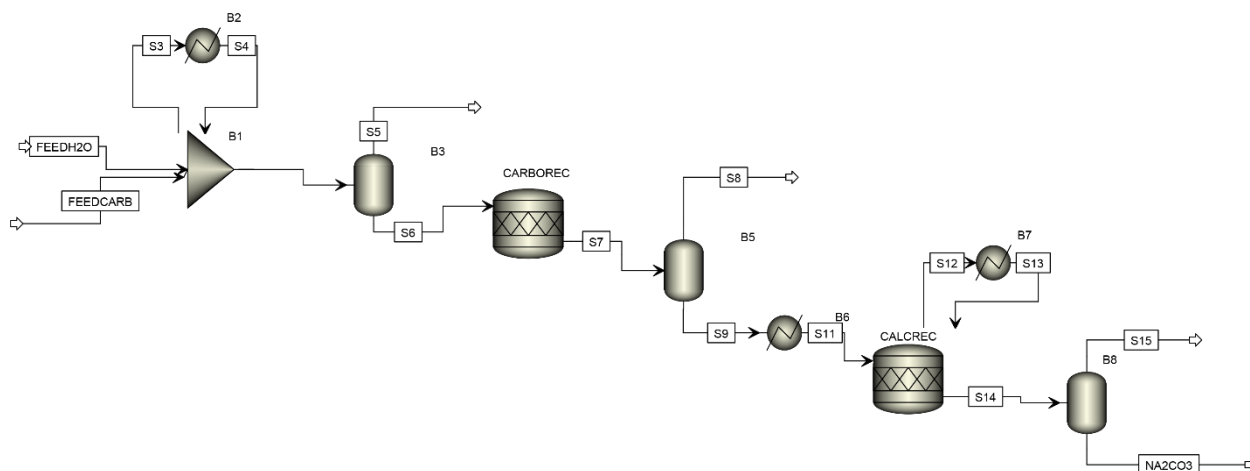


Figure 1. Process flow diagram of the proposed mini-technology.

[B1 – reactor; B2 (HEATER) – solution heating unit; B3 (SEPARATOR) – nutsche filter for solid–liquid separation; CARBOREC (RSTOIC) – carbonation reactor; B5 (SEPARATOR) – nutsche filter for sodium bicarbonate separation; B6 (HEATER) – drying unit; CALCREC (RSTOIC) – calcination furnace; B8 (SEPARATOR) – grinding and sieving unit.]

As shown in Figure 1, the proposed process includes dissolution of technical-grade Na_2CO_3 in demineralized water, solution filtration, controlled carbonation of the clarified liquor with the formation of sodium bicarbonate, separation and washing of crystals, drying and calcination to regenerate Na_2CO_3 , followed by grinding, sieving, packaging, labeling, and storage. The mini-technology is based on sequential purification and continuous control of key operating parameters at each processing stage.

In reactor B1, 700.0 L of demineralized water with a total dissolved solids content below 0.1 mg/L were charged, and 300.0 kg of technical soda ash were added under mechanical stirring at 60–120 rpm. The temperature was maintained at 40 °C using the heating unit B2. Under these conditions, a 30 wt.% sodium carbonate solution was obtained with a density of 1.318 g/cm³ and a pH value above 11.5. The solution was then directed to the nutsche filter B3 to remove insoluble impurities. The clarified filtrate was monitored for density and pH and subsequently fed to the carbonation reactor CARBOREC.

Carbonation was carried out by supplying carbon dioxide at a flow rate of 20.0 m³/h under a pressure of 5.5 kg/cm², corresponding to an excess of approximately 7% relative to stoichiometric demand. The process was conducted at 55–65 °C with a CO₂ overpressure of about 10 kPa until the pH decreased to approximately 8.5, resulting in the formation of a sodium bicarbonate suspension. The suspension was filtered to separate NaHCO₃ crystals, while the mother liquor was withdrawn via stream S10 and either recycled to reactor B1 or used for auxiliary technical purposes. The separated crystals were washed with demineralized water to remove soluble impurities, and the wash waters were also discharged through stream S10.

The washed sodium bicarbonate crystals were dried in unit B6 at 75–80 °C to a residual moisture content below 0.5% and then transferred to the calcination furnace CALCREC, where thermal treatment was carried out at 300 °C. The resulting sodium carbonate was subsequently ground and sieved in unit B8, packaged, and sent to storage. Fine dust particles generated during handling were captured by cyclone separators and recycled to reactor B1 via stream S2.

Under the optimized operating conditions, 236.4 kg of chemically pure sodium carbonate were obtained. The overall yield reached 78.8% of the theoretical value, while the mass fraction of the main component was 99.87%. The concentrations of iron and heavy metals in the final product were reduced to the level of 10⁻⁵–10⁻⁶%, confirming compliance with the requirements for chemically pure and analytical-grade sodium carbonate.

Table 1.
Effect of carbonation parameters on yield and quality of sodium carbonate

CO ₂ flow rate (m ³ /h)	CO ₂ excess (%)	Yield (%)	Purity (%)	Iron content (%)	Heavy metals content (%)	Process temperature
20	7	78,8	99,87	10 ⁻⁵	10 ⁻⁶	60

Table 1 summarizes the key parameters of the carbonation stage and their relationship with the yield and quality of the final sodium carbonate product. At a CO₂ flow rate of 20 m³/h and a gas excess of 7% relative to stoichiometric requirements, the yield of sodium carbonate reached 78.8% of the theoretical value. This yield indicates efficient progression of the carbonation step; however, a portion of the supplied CO₂ is unavoidably consumed by dissolution and lost during gas–liquid mass transfer, which limits the attainment of a 100% theoretical yield.

In terms of quality, the obtained product meets the requirements for chemically pure sodium carbonate. The mass fraction of the main component reached 99.87%, while impurity levels remained at trace concentrations, with iron content on the order of 10⁻⁵% and heavy metals at approximately 10⁻⁶%. These values are well below the permissible limits for chemically pure and analytical-grade reagents, confirming the effectiveness of the filtration, washing of NaHCO₃ crystals, and subsequent calcination stages.

The carbonation temperature range of 55–65 °C, with an average value of about 60 °C, was found to be technologically justified. Within this interval, sufficient carbonation kinetics and stable crystallization of sodium bicarbonate are ensured. Lower temperatures are expected to slow down the process and deteriorate crystallization conditions, whereas operation above the optimal range increases the risk of partial bicarbonate decomposition, resulting in reduced yield.

Overall, the combined effect of controlled CO₂ flow rate, moderate gas excess, and maintenance of the carbonation temperature within 55–65 °C provides a balanced compromise between product yield and purity. The obtained results confirm the applicability of the proposed mini-technology for the production of high-purity sodium carbonate under laboratory and semi-industrial conditions.

V. CONCLUSION AND FUTURE WORK

A mini-technology for the production of chemically pure and analytical-grade sodium carbonate from technical soda ash was developed and experimentally validated. The proposed process includes dissolution of the raw material, filtration, controlled carbonation with the formation of NaHCO₃, separation and washing of crystals, drying, calcination to regenerate Na₂CO₃, followed by grinding and sieving of the final product.

It was established that a CO₂ flow rate of 20 m³/h with a 7% excess relative to stoichiometric requirements at a carbonation temperature of 55–65 °C provides a product yield of 78.8% of the theoretical value and a sodium carbonate purity of 99.87%. The concentrations of iron and heavy metals in the final product were reduced to the level of 10⁻⁵–10⁻⁶%, which meets the regulatory requirements for high-purity chemical reagents.



The developed mini-technology can be implemented using standard laboratory and pilot-scale equipment and is suitable for laboratory and small-scale semi-industrial applications. Future work will focus on further optimization of carbonation, washing, and calcination parameters, as well as assessment of process scalability and energy efficiency to improve overall yield and industrial applicability.

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