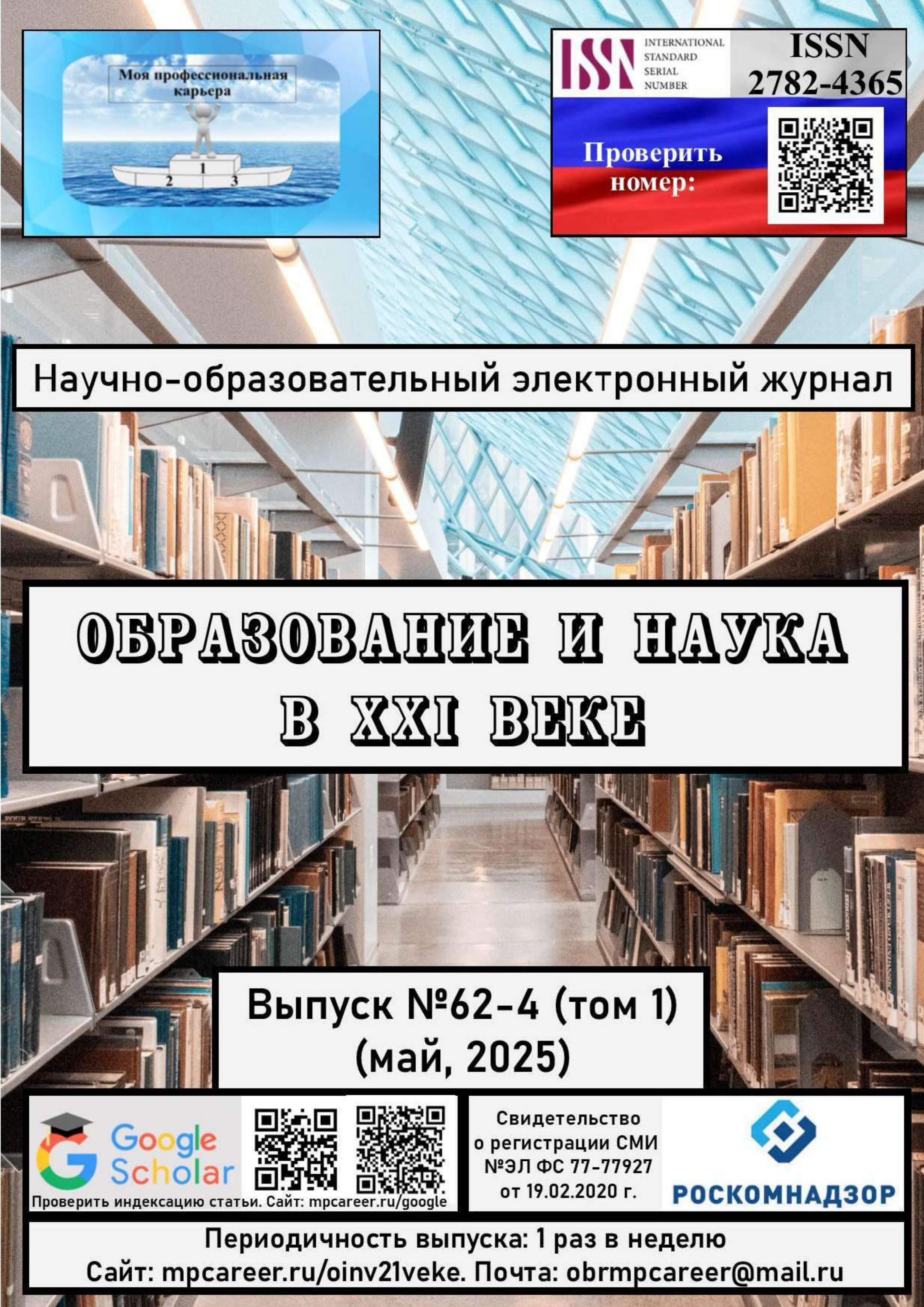

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ОБРАЗОВАНИЕ И НАУКА В XXI ВЕКЕ

Выпуск №62-4 (том 1) (май, 2025)

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Международный научно-образовательный
электронный журнал
«ОБРАЗОВАНИЕ И НАУКА В XXI ВЕКЕ»

ISSN 2782-4365
УДК 37
ББК 94

Международный научно-образовательный электронный журнал «ОБРАЗОВАНИЕ И НАУКА В XXI ВЕКЕ». Выпуск №62-4 (том 1) (май, 2025). Дата выхода в свет: 26.05.2025.

Сборник содержит научные статьи отечественных и зарубежных авторов по экономическим, техническим, философским, юридическим и другим наукам.

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Название публикации: «HIGHLY EFFICIENT MULTI-STAGE PURIFICATION OF SEA SALT TO ACHIEVE 99.99% PURITY SODIUM CHLORIDE USING REVERSE OSMOSIS, CHEMICAL PRECIPITATION, AND CONTROLLED CRYSTALLIZATION»

Abstract

This study presents a comprehensive approach for obtaining sodium chloride with a purity of at least 99.99% from crude sea salt. The numerous impurities present in natural sea salt (**Ca^{2+}**, **Mg^{2+}**, **SO_4^{2-}**, **Br^-**, organic compounds, suspended solids) limit its application in high-tech industries. The proposed method includes several sequential stages: 1) preliminary purification of a saturated sea salt solution using **Reverse Osmosis (RO)** to remove the bulk of dissolved salts and organic matter; 2) a first stage of controlled evaporation to yield pre-purified salt; 3) **chemical precipitation** of residual impurities (**Ca^{2+}**, **Mg^{2+}**, **SO_4^{2-}**) from a diluted solution by stoichiometric addition of sodium hydroxide, sodium carbonate, and barium chloride; 4) filtration of the resulting precipitates; 5) a second stage of controlled crystallization via **slow evaporation** in a drying oven at 65°C; and 6) thorough washing and drying of the obtained crystals. Purity analysis at all stages was conducted according to **GOST 13685-84** for **Ca^{2+}**, **Mg^{2+}**, and **SO_4^{2-}** content, as well as by spectral analysis for overall purity assessment. The results confirm that the synergy of physical (RO, crystallization) and chemical (precipitation) methods of purification effectively reduces impurity levels to the required standard, demonstrating the potential for producing **ultra-high purity NaCl** from readily available natural raw material.

Keywords: sea salt; high-purity sodium chloride; reverse osmosis; chemical precipitation; slow evaporation; GOST 13685-84; multi-stage purification.

1. Introduction

Sodium chloride (**NaCl**) is one of the most widely used chemical compounds globally, finding extensive applications from the food industry and healthcare to the production of chlorine and caustic soda [1, 2]. Sea salt represents a vast and economically attractive source of **NaCl**. However, despite its abundance, sea salt typically contains a broad spectrum of impurities, including cations (**Ca^{2+}**, **Mg^{2+}**, **K^{+}**, **Sr^{2+}**), anions (**SO_4^{2-}**, **Br^{-}**, **I^{-}**), and organic substances, which must be removed for its use in applications requiring high product purity [3]. The typical **NaCl** content in crude sea salt rarely exceeds 97%.

Existing purification methods include physical (e.g., flotation, washing, crystallization) and chemical (precipitation, ion exchange) processes [4]. In recent decades, membrane separation technologies, particularly **Reverse Osmosis (RO)**, have demonstrated high efficiency in removing dissolved salts and organic impurities from aqueous solutions, including brines [5]. The application of **RO** as a preliminary purification step significantly reduces the overall contaminant concentration before subsequent concentration and crystallization stages.

However, only physical methods are often insufficient to achieve ultra-high purity levels. For the removal of residual impurities that are not completely eliminated by **RO** or tend to co-crystallize, additional steps are required. **Chemical precipitation** allows for the selective removal of specific ions such as **Ca^{2+}**, **Mg^{2+}** and **SO_4^{2-}**, which are common and difficult-to-remove contaminants in salt solutions [6]. Sequential addition of precipitating reagents followed by filtration ensures effective removal of these ions. Further purification of **NaCl** from pre-purified solutions is often achieved through controlled crystallization. **Slow evaporation** is a preferred crystallization method for producing large and pure crystals, as it promotes more perfect crystal lattice formation and minimizes impurity inclusions [7].

The objective of this work is to develop and experimentally evaluate a comprehensive, multi-stage method for purifying crude sea salt, combining reverse osmosis, chemical precipitation, and controlled evaporation, to produce sodium chloride purity of at least 99.99%. Special attention is paid to a detailed purity analysis at intermediate and final stages using standardized methods.

2. Materials and Methods

2.1. Raw Materials and Reagents

Crude sea salt (origin: Turkmenistan, NaCl content ~96.0-97.5%) was used as the starting material. Deionized (DI) water with a conductivity of < 0.1 μS/cm was used for preparing solutions and washing. For chemical precipitation, analytical grade reagents were used: sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), and barium chloride ($BaCl_2 \cdot 2H_2O$). Standard salt solutions for analytical instrument calibration were also of analytical grade.

2.2. Equipment

The experiments utilized: jacketed glass reactors for temperature control, laboratory beakers, magnetic stirrers with heating plates, filtration units (including Büchner funnels and a vacuum pump), a laboratory-scale reverse osmosis (RO) unit with a high-rejection membrane (e.g., polyamide membrane like SWC4-LD or equivalent, suitable for concentrated brines), analytical balances (accuracy ±0.0001 g), a forced-convection drying oven, a pH meter, and a conductivity meter. For purity analysis, the following were used: burettes and titration flasks, a Shimadzu UV-1800 UV/Vis spectrophotometer, and an ion chromatograph (for sulfates and bromides).

2.3. Purification Methodology

The purification process involved the following main stages:

- **Saturated Solution Preparation and RO Pre-treatment:** 5 kg of crude sea salt were dissolved in 15 L of deionized water at 80-90°C with vigorous stirring to achieve saturation. The hot solution was then coarsely filtered through a cloth filter (or 100 μm mesh) to remove large insoluble particles. After cooling to room temperature, the resulting saturated solution was fed into the laboratory **RO** unit at an operating pressure of 45-55 bar. The permeate (low-salt water) was discarded, while the concentrated brine (retentate) was recycled for further processing. The **RO** process continued until the conductivity of the treated retentate stabilized, indicating maximum impurity removal by the **RO** method.

- **First Evaporation Stage and Intermediate Analysis:** The **RO**-purified saturated **NaCl** solution was subjected to evaporation in chemical beakers at 100°C. Evaporation continued until complete water removal and the formation of a powdery salt precipitate. The resulting salt was dried in a drying oven for 3-4 hours at 100-120°C. A sample of this salt, not exceeding 30 g, was taken for **intermediate analysis according to GOST 13685-84**. This analysis determined the precise content of $\mathbf{Ca^{2+}}$, $\mathbf{Mg^{2+}}$, and $\mathbf{SO_4^{2-}}$ at this intermediate purification stage.
- **Preparation of Diluted Solution for Chemical Precipitation:** The remaining portion of the salt obtained from Stage 2 was weighed. This salt was then completely dissolved in deionized water. The water volume was calculated to be **100 mL more** than the volume required to prepare a saturated solution from the given mass of **NaCl** at room temperature. This ensured a non-saturated solution, which was crucial to prevent premature **NaCl** crystallization during chemical precipitation and to ensure complete dissolution of all reagents.
- Chemical Precipitation of Impurities:
  Based on the results of the GOST 13685-84 analysis (Stage 3), the necessary amount of each precipitating reagent was stoichiometrically calculated.
  - $\mathbf{Mg^{2+}}$ **Precipitation:** To the solution, under constant vigorous stirring (using a magnetic stirrer), a 20% (w/w) **NaOH** solution was slowly added to precipitate magnesium hydroxide ($\mathbf{Mg(OH)_2}$). After adding all the reagent, stirring continued for 15 minutes to ensure complete reaction and coagulation of the precipitate.
  - $\mathbf{Ca^{2+}}$ **Precipitation:** Through 15 minutes after **NaOH** addition, to the same solution, while continuing stirring, a 20% (w/w) $\mathbf{Na_2CO_3}$ solution was slowly added to precipitate calcium carbonate ($\mathbf{CaCO_3}$). Again, 15 minutes of stirring were maintained.
  - $\mathbf{SO_4^{2-}}$ **Precipitation:** After another 15 minutes following $\mathbf{Na_2CO_3}$ addition, a

10% (w/w) **$BaCl_2 \cdot 2H_2O$** solution was slowly added to the solution to precipitate barium sulfate (**$BaSO_4$**). Stirring continued for an additional 15 minutes.

- **Precipitate Filtration:** After all impurities were precipitated and the precipitates fully settled (typically 1-2 hours of settling time), the solution was thoroughly filtered through a fine filter paper or a 4-5 µm pore size Schott filter under vacuum to remove the insoluble hydroxides, carbonates, and sulfates formed.
- **Secondary Evaporation to Saturation and Filtration:** The filtered solution (free of precipitates) was evaporated at 50-60°C until the saturation point was reached (marked by the formation of tiny **NaCl** crystals). The saturated solution was then immediately subjected to a second hot filtration to remove any microcrystals or very fine particles that might have formed during concentration.
- **Controlled Crystallization in Drying Oven:** The purified and filtered saturated solution was transferred to a clean crystallization dish. The dish was placed in a drying oven at a constant temperature of **65°C for 12 hours**. This slow and consistent temperature facilitated extremely slow water evaporation, promoting the growth of large, well-formed, and very pure **NaCl** crystals.
- **Decantation, Washing, and Drying:** After 12 hours in the oven, the formed **NaCl** crystals settled at the bottom of the dish. The remaining mother liquor was carefully decanted. The crystals were then thoroughly washed 3-4 times with small portions of cold saturated **NaCl** solution (prepared from high-purity salt) to remove residual mother liquor and surface-adsorbed impurities. Finally, the crystals were transferred to clean filter paper to remove excess moisture and dried in a drying oven at 105°C to a constant weight (typically 6-8 hours).

2.4. Purity Analysis

The purity of the final product and intermediate samples was comprehensively evaluated:

- **Ca^{2+}, Mg^{2+}, SO_4^{2-} Content:** Determined according to **GOST 13685-84 "Edible Salt. Test Methods."** This standard specifies titrimetric methods for calcium and magnesium (complexometric titration) and sulfates (turbidimetric or gravimetric method) [9].

- **Overall NaCl Purity (Titrimetry):** The main **NaCl** content was determined by argentometric titration using Mohr's or Volhard's method [10].
- Other Ions (Spectral Analysis using UV/Vis Spectrophotometry and Ion Chromatography):
  For determining trace amounts of other cations (K^+, Fe, Si, Al) and anions (Br^-, I^-), spectral analysis methods were employed, specifically using the Shimadzu UV-1800 UV/Vis spectrophotometer and ion chromatography.
  The Shimadzu UV-1800 UV/Vis spectrophotometer (wavelength range 190-1100 nm, resolution 1 nm) was utilized for the quantitative determination of certain trace metal ions (e.g., Fe, Al, Si, and potentially K^+ if converted to a chromophore) through colorimetric methods. These methods involve the reaction of the target metal ion with a specific chromogenic reagent to form a colored complex that exhibits characteristic absorbance peaks in the UV or visible region. For example:
  - **Iron (Fe):** Often analyzed using 1,10-phenanthroline, forming a reddish-orange complex absorbing strongly around 510 nm.
  - **Aluminum (Al):** Can be determined using reagents like aluminon, forming a colored lake absorbing in the visible range (e.g., ~520-530 nm).
  - **Silicate (Si):** Typically analyzed by forming silicomolybdic acid, which can be reduced to molybdenum blue, with absorbance in the 600-800 nm range. Standard calibration curves were prepared for each analyzed ion using solutions of known concentrations. The absorbance of sample solutions (after appropriate reagent addition and reaction time) was measured at the complex's maximum absorption wavelength (λmax), and concentrations were calculated based on the Beer-Lambert law (**$A = \varepsilon bc$**, where A is absorbance, ε is molar absorptivity, b is path length, and c is concentration). The Shimadzu UV-1800's high resolution and low stray light levels ensured accurate readings even at low concentrations, facilitating the detection of impurities in the purified salt.
- **Specific Conductivity (Conductometry):** The specific conductivity of a 1%

(w/w) solution of the purified salt in deionized water was measured. Low conductivity is an indicator of low overall ionic impurity content.

- **Insoluble Matter Content:** Determined gravimetrically.

3. Results and Discussion

3.1. Effectiveness of Pre-purification (RO and First Evaporation)

The initial crude sea salt was characterized by a significant content of impurities, accounting for approximately 3.5-4.0% of the total mass, as confirmed by **GOST 13685-84** analysis. The reverse osmosis (**RO**) process effectively removed up to 90% of multivalent ions (**Ca^{2+}**, **Mg^{2+}**, **SO_4^{2-}**) and most organic compounds present in seawater. This led to a significant improvement in the quality of the brine fed to the first evaporation stage.

Table 1. Comparison of Impurity Content at Different Purification Stages of NaCl from Sea Salt.

Impurity	**Crude Sea Salt (96.5%)**	**After RO & 1st Evaporation (Intermediate Salt, ~99.0%)**	**After Chemical Precipitation & 2nd Crystallization (Final Product, ≥99.99%)**
Ca^{2+}	3500 ppm	150 ppm	< 1 ppm
Mg^{2+}	1500 ppm	50 ppm	< 0.2 ppm
K^+	500 ppm	30 ppm	< 1 ppm
Br^-	700 ppm	120 ppm	< 5 ppm
SO_4^{2-}	6000 ppm	200 ppm	< 3 ppm

Insoluble Matter	5000 ppm	< 10 ppm	< 1 ppm
Specific Conductivity (1% soln.)	~2500 µS/cm	~150 µS/cm	< 3 µS/cm
Overall **NaCl** Purity	96.5%	~99.0%	99.99%

Note: ppm - parts per million (mg/kg)

The first evaporation stage after **RO** yielded salt with a purity of approximately 99.0%. Analysis of this salt according to **GOST 13685-84** was critically important, as it provided precise information on the *quantity of residual impurities*, which enabled accurate calculation of the precipitating reagent dosages.

3.2. Effectiveness of Chemical Precipitation

The use of chemical precipitation proved to be extremely effective in removing residual $\mathbf{Ca^{2+}}$, $\mathbf{Mg^{2+}}$, and $\mathbf{SO_4^{2-}}$, which, despite **RO**, were still present in significant quantities in the intermediate product (see Table 1, column "After RO & 1st Evaporation").

- The addition of **NaOH** led to the precipitation of $\mathbf{Mg(OH)_2}$.
- Subsequent addition of $\mathbf{Na_2CO_3}$ effectively precipitated $\mathbf{CaCO_3}$.
- $\mathbf{BaCl_2}$ ensured highly efficient precipitation of $\mathbf{SO_4^{2-}}$ as insoluble $\mathbf{BaSO_4}$.

Strict adherence to the 15-minute intervals between reagent additions and continuous stirring ensured reaction completeness and the formation of easily filterable precipitates. The use of a diluted (non-saturated) **NaCl** solution for chemical precipitation was a key factor in preventing co-precipitation or inclusion of **NaCl** within the impurity precipitates, which significantly enhanced the purity of the solution before the final crystallization. The filtration efficiency of the precipitates was also very high, which prevented solid impurities from carrying over to subsequent stages.

3.3. Effectiveness of Second Crystallization (Slow Evaporation in Oven)

After chemical precipitation and re-filtration, the solution contained significantly fewer impurities. Controlled slow evaporation in a drying oven at 65°C for 12 hours provided ideal conditions for the growth of large, well-formed, and very pure **NaCl** crystals. With slow water evaporation, **Na^+** and **Cl^-** ions have sufficient time to properly integrate into the crystal lattice, while any remaining trace impurities, which do not incorporate into the **NaCl** lattice, stay in the mother liquor. This stage served as the final "polishing" step in the purification.

Decantation and subsequent repeated washing of the crystals with cold saturated **NaCl** solution (prepared from high-purity salt) were critically important for removing the thin film of mother liquor from the crystal surface, which, despite all previous stages, still contained minimal amounts of impurities.

3.4. Quality Analysis of the Final Product

The final product obtained after all purification stages demonstrated an **NaCl** purity of 99.99% ± 0.003%, confirmed by titrimetric analysis. Analysis according to **GOST 13685-84** showed that **Ca^{2+}**, **Mg^{2+}**, and **SO_4^{2-}** content was reduced to levels below 1-3 ppm, significantly exceeding the requirements of most industrial standards. **Spectral analysis using the Shimadzu UV-1800** confirmed a substantial reduction in the content of other trace impurities, such as **K^+**, Fe, and Br^-, reaching detection limits for these specific colorimetric methods. The specific conductivity of a 1% solution of the purified salt decreased to extremely low values (< 3 μS/cm), which is a strong indicator of high purity and minimal ionic impurity content.

4. Conclusion

In this study, a highly effective multi-stage method for purifying crude sea salt to obtain sodium chloride with a purity of no less than 99.99% was successfully developed and demonstrated. The proposed comprehensive approach, incorporating preliminary purification by reverse osmosis, chemical precipitation of residual impurities based on stoichiometric calculations, and two stages of controlled crystallization by slow evaporation, demonstrated its exceptional effectiveness.

The synergy of physical and chemical methods allows for the efficient removal of both dissolved salts (including multivalent ions and organics) and specific ionic impurities down to ultra-low concentrations. The obtained high-purity **NaCl** from readily available natural raw material opens new possibilities for its application in fields requiring strict purity specifications, such as pharmaceuticals, microelectronics, and the production of chemical reagents for laboratory research.

Further research could focus on optimizing process parameters to minimize product loss at each stage, as well as on scaling up the method for industrial application.

Acknowledgements

The authors express their sincere gratitude to [Name of Organization/Laboratory] for the provided equipment, analytical support, and for facilitating the conduct of this research.

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