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ORIGINAL RESEARCH ARTICLE

Sedimentation based process development for Li_2CO_3 and Mg–K–Ca–PO_4–SO_4 fertilizer recovery from Aral Sea brine

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ABSTRACT

The high salinity of Aral Sea water, as well as high amounts of accompanying magnesium, potassium, and sulfate ions, significantly limit the rational use of these waters for lithium extraction. The effective separation of these impurities is a complex scientific and technological task that requires selective and economically justified solutions. The current study evaluates the feasibility of Aral Sea water purification using a chemical precipitation method for the removal of magnesium and potassium compounds and sulfate ions. The method is based on the selective precipitation of magnesium and potassium in the form of double phosphate salt $KMgPO_4$ using sodium hydrogen phosphate as the precipitating agent, with the precipitation of sulfate ions as calcium sulfate dihydrate. The key process variables, including reagent proportions, pH, temperature, and precipitation time, were examined and optimized to achieve maximum purification efficiency. The removal efficiency under the established optimal conditions reached 97.1% for magnesium compounds, 96.3% for potassium, and 93.4% for sulfate ions. The precipitates obtained had stable phase composition and good filtration properties, thus improving the technological feasibility of the proposed method. The findings confirm the high efficiency of the proposed method and its great potential for deep purification of highly mineralized waters.

Keywords: Aral Sea saline water; sodium hydrogen phosphate; sodium carbonate; lithium chloride; precipitation

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1. Introduction

Lithium's unique properties render it highly attractive across various industrial sectors. The greatest demand is observed in the production of rechargeable batteries for electronics and electrical engineering, driven by the increasing consumption of lithium-ion batteries for computers, smartphones, and the development of large-capacity green energy storage systems[1-3]. In nature, lithium does not occur in its free state; it is a relatively rare element found in both liquid and solid forms. Liquid lithium resources include seawater, ocean water, brines of saline lakes, geothermal fluids, and produced waters associated with oil and gas extraction, whereas solid sources comprise mineral ore deposits and waste from lithium-ion batteries and accumulators[4].

Approximately 70-80% of global lithium reserves exist in liquid form as highly dilute solutions in oceanic and marine waters, which currently lack industrial applicability. Geothermal waters and saline lake brines contain higher lithium concentrations, yet their processing is complicated by various metallic impurities, particularly magnesium, which hinder efficient extraction[5,6]. Currently, the main lithium-based products are lithium carbonate, mineral concentrates, and lithium hydroxide, which collectively account for approximately 80% of the market. Lithium carbonate is produced by extracting and processing spodumene ores and saline lake brines[7]. Spodumene was the first raw material used for industrial lithium production and occurs in pegmatite rocks, where lithium content typically ranges from 1 to 4%, with extraction efficiencies reaching 60-70%[8,9].

Despite the low lithium concentrations in aqueous solutions, this avenue is considered highly promising due to the broad availability of resources and the relative simplicity of their processing[10,11]. Extensive scientific and technical literature addresses various methods for lithium extraction from aqueous solutions, which support industrial production of lithium compounds that are environmentally benign, highly selective, economically feasible, and technically straightforward. Production from aqueous solutions, particularly brines, is generally less costly and less energy-intensive than extraction from solid ores. The Republic of Uzbekistan has various water resources that carry valuable chemical components, such as lithium, magnesium, and potassium. However, among these resources, the Aral Sea is of particular importance due to its substantial lithium reserves. The Aral Sea region is considered a highly promising water resource for future development and economic opportunities due to its high and diverse composition. Lithium, a component of the Aral Sea brine, is highly strategic as a chemical element. Based on available data, the total reserves of lithium chloride in the Aral Sea's aquatic environment are estimated at 19.82 million tons. The Aral Sea had an average surface area of 68,000 square kilometres until the 1960s. At that time, it was ranked the fourth-largest inland sea in the world, after the Caspian Sea, Lake Superior in North America, and Lake Victoria in Africa. Moreover, it was ranked the second-largest inland sea in the world, after the Caspian Sea, on the Eurasian continent. Natural brines with lithium concentrations ranging from 0.32 to 1 g/L are typically processed by solar evaporation[12-16]. The solar evaporation process comprises sequential stages of precipitation and crystallization of Li_2CO_3[17], yet it is extremely time-consuming. Additionally, the resulting Li_2CO_3 is often contaminated with undesirable salts such as NaCl, $Mg(OH)_2$, and $MgCO_3$, necessitating further purification. Chemical precipitation involves forming lithium compounds, such as lithium aluminate or lithium carbonate, by adding aluminium chloride or soda ash, respectively[18,19]. However, this method is unsuitable for brines with high concentrations of interfering salts, particularly those with elevated Mg^{2+}/Li^{+} ratios. Moreover, it requires substantial reagent consumption and generates significant amounts of sludge[20].

2. Materials and methods

For the study, water from the Aral Sea was used, with its composition presented in **Table 1**.

Table 1. Chemical composition of the Aral Sea water.

	TDS (g/l)	pH	Ionic composition, (g/l)							
			Li^+	Na^+	K^+	Ca^{2+}	Mg^{2+}	B_2O_3	Cl^-	SO_4^{2-}
Aral Sea water	130.9	8.0	0.11	54.92	3.67	0.80	10.25	0.03	70.90	22.11
	TDS (g/l)	pH	Salt composition, (g/l)							
			LiCl	NaCl	KCl	$CaCl_2$	$MgCl_2$	Na_2SO_4		
	130.9	8.0	0.66	126.20	7.01	2.22	40.57	32.70		

The water contained lithium at 0.11 g/L, sodium 54.92 g/L, potassium 3.67 g/L, magnesium 10.25 g/L, chloride 70.90 g/L, and sulfate 22.11 g/L, present as chlorides of lithium, sodium, potassium, calcium, magnesium, and sodium sulfate. The extraction of Mg^{2+}, K^+, and SO_4^{2-} ions for the production of a complex fertiliser from Aral Sea brines was conducted via precipitation in a stirred reactor. The target was achieved by removing magnesium, potassium, and sulfate ions using sodium hydrogen phosphate, calcium chloride, and sodium hydroxide. Mixing was performed at 100 rpm and a solution pH of 10. Sodium hydrogen phosphate was employed to precipitate magnesium and potassium ions, $CaCl_2$ to precipitate sulfate ions, and sodium hydroxide to regulate the pH of the mixed solutions.

3. Results and discussion

The saline water of the Aral Sea has an average salinity of approximately 130.9 g/kg. When salts dissolve in water, they dissociate into ions, which are either negatively charged anions or positively charged cations. The primary components of Aral Sea water are the cations sodium, potassium, magnesium, and calcium, and the anions chloride and sulfate, together accounting for 99.99% of the dissolved substances. Sodium chloride (NaCl) alone constitutes 56% of the total dissolved ions. The molar concentration of magnesium in the Aral Sea water is approximately five times that of calcium. Both magnesium and calcium exist in equilibrium, and minor changes in alkalinity or the presence of carbon dioxide can trigger precipitation. Magnesium ions are less likely to form solid precipitates compared to other cations, such as calcium.

The nucleation rate of magnesium phosphates is slightly lower than that of calcium phosphates at equivalent pH values and total phosphate concentrations. In contrast, the precipitation rate of calcium exceeds that of magnesium between pH 8.0 and 9.0. Within the pH range of 8 to 10, phosphate precipitation is substantial; however, at pH 10 to 12, it gradually decreases, likely due to competition between phosphate and hydroxide ions (OH^-).

A method was developed to precipitate magnesium and potassium ions from saline water using sodium hydrogen phosphate, according to the reaction:

$$MgCl_2+KCl+LiCl+Na_2HPO_4+NaOH \rightarrow MgKPO_4\downarrow+3NaCl+LiCl+H_2O \quad ^{(1)}$$

For sulfate ion removal, calcium chloride was used, forming gypsum via the reaction:

$$Na_2SO_4+CaCl_2+2H_2O \rightarrow CaSO_4\cdot 2H_2O+2NaCl \quad ^{(2)}$$

During solvation and ionization of cations, these chemical processes are exothermic. The solubility of calcium sulfate in water is only 0.2 g/100 g H_2O. Sulfate removal experiments were performed in a reactor at a stirring speed of 100 rpm and a temperature of 25 °C, with a 15% $CaCl_2$ solution and suspension mixing times of 10, 20, and 30 minutes.

The precipitation process was conducted in two stages. In the first stage, the effect of sodium hydrogen phosphate on magnesium removal was investigated. A 100 mL sample of Aral Sea water was placed in the reactor, incremental amounts of sodium hydrogen phosphate were added, mixed for 10 minutes, and the pH was adjusted to 10 using a 2N sodium hydroxide solution. In the second variant, after adding sodium hydrogen phosphate and mixing for 10 minutes, increasing amounts of calcium chloride were introduced, followed by 10 minutes of stirring and pH adjustment to 10 with sodium hydroxide.

The resulting precipitates were dried at 100 °C to constant mass. Sodium, potassium, calcium, and magnesium concentrations were analyzed by X-ray fluorescence, while chloride, sulfate, and phosphate ions were measured spectrophotometrically. **Figure 1** shows the effect of sodium hydrogen phosphate dosage and process duration on magnesium precipitation. A high degree of magnesium removal, reaching 97.1%, was achieved with 1.72 g of sodium hydrogen phosphate over a 30-minute process. Increasing the amount of sodium hydrogen phosphate resulted in greater magnesium ion precipitation until the maximum removal was attained.

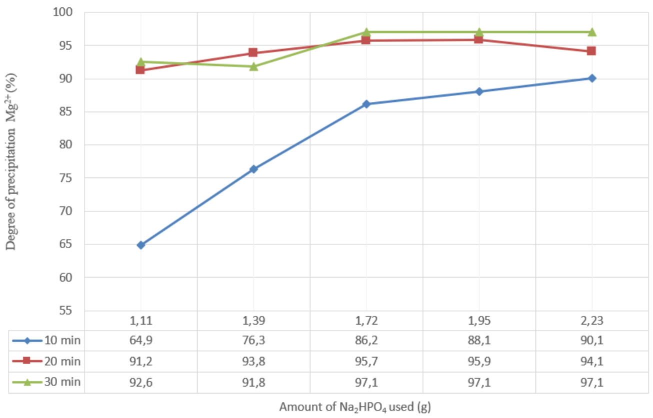

Figure 1. Effect of sodium hydrogen phosphate (Na_2HPO_4) dosage on the extent of magnesium ion precipitation.

Figure 2 presents the effect of Na_2HPO_4 dosage and process duration on the removal of K^+ ions from the solution. A similar trend is observed as with magnesium ions: increasing both the amount of Na_2HPO_4 and the stirring time leads to higher potassium ion precipitation. Under optimal conditions, using the same Na_2HPO_4 dosage and mixing duration, the removal efficiency of K^+ ions reached 96.3%.

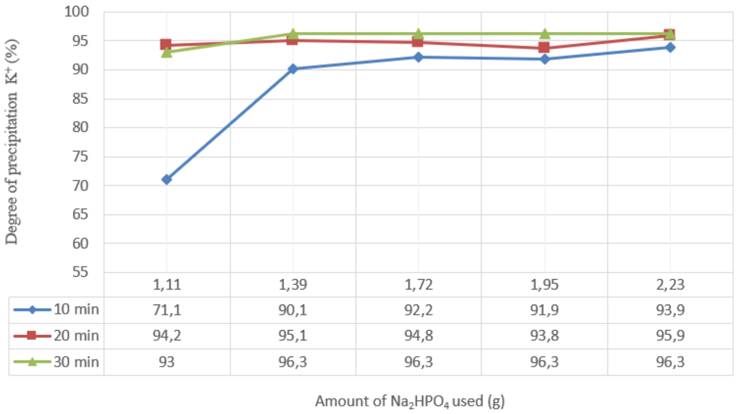

Figure 2. Effect of sodium hydrogen phosphate (Na_2HPO_4) dosage on the extent of potassium ion precipitation.

Figure 3 presents the effect of $CaCl_2$ dosage and process duration on the removal efficiency of SO_4^{2-} ions.

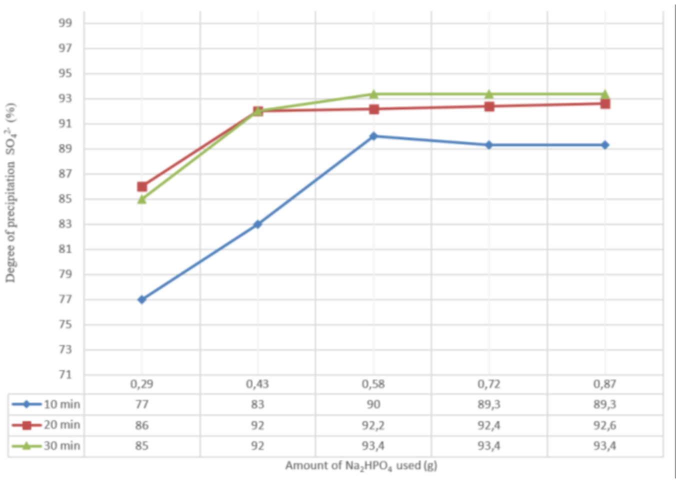

Figure 3. Effect of calcium chloride dosage on the extent of sulfate ion precipitation.

The results demonstrated that increasing the dosage of calcium chloride enhances the precipitation of SO_4^{2-} ions as calcium sulfate dihydrate. It is evident from the data that the maximum removal efficiency of sulfate ions, reaching 93.4%, was achieved with the addition of 0.58 g of $CaCl_2$ and 30 minutes of suspension mixing. **Figure 4** presents a block diagram of the Aral Sea water purification process from associated impurities.

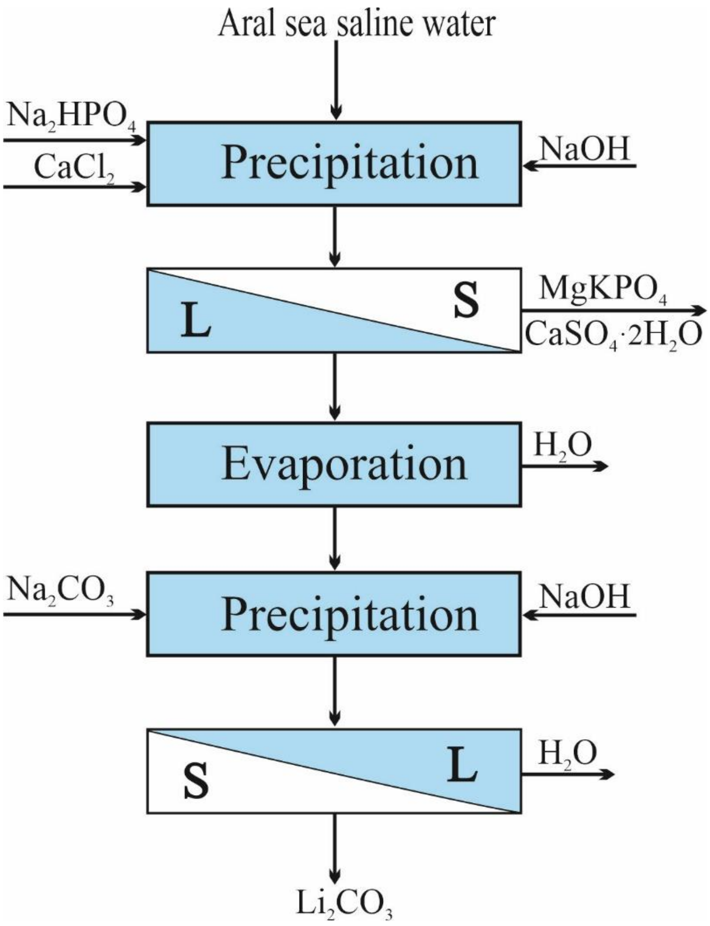

Figure 4. Block diagram of the Aral Sea water purification process.

Figure 5 presents the X-ray diffraction pattern of calcium sulfate dihydrate precipitated from Aral Sea saline water using $CaCl_2$, while **Figure 6** shows the pattern of the complex fertilizer obtained by treating the same water with Na_2HPO_4. The second diffraction pattern indicates that the product is largely amorphous with a minor fraction of crystalline material. The solid precipitate prepared with $CaCl_2$ contains several types of ions.

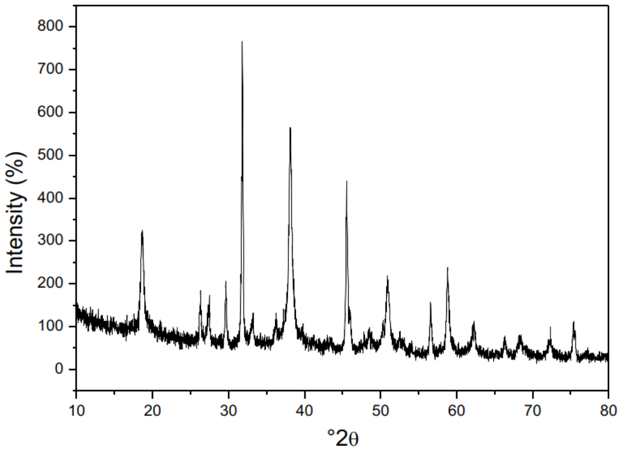

Figure 5. X-ray diffraction (XRD) pattern of $CaSO_4 \cdot 2H_2O$ precipitated from Aral Sea saline water using $CaCl_2$.

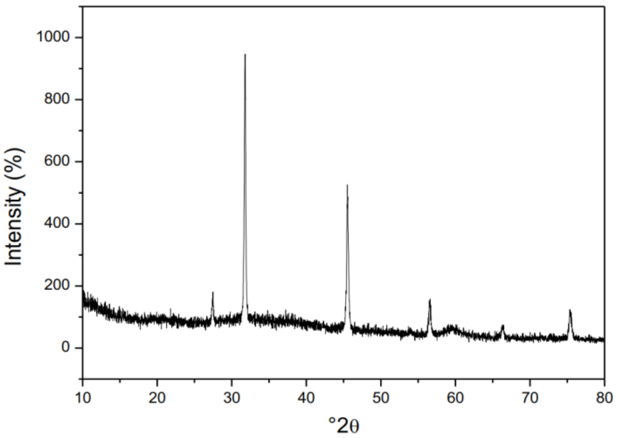

Figure 6. X-ray diffraction (XRD) pattern of the complex fertilizer ($MgKPO_4$) precipitated from Aral Sea saline water using Na_2HPO_4.

EDAX analysis revealed the distribution of ions in the solid precipitate obtained from the simultaneous treatment of Aral Sea saline water with calcium chloride and sodium phosphate (**Figure 7**). The results indicate that the precipitate comprises the following components: PO_4^{3-} – 10.84%, Mg^{2+} – 12.7%, SO_4^{2-} – 27.4%, Ca^{2+} – 23.6%, and K^+ – 2.5%. At pH 10, the removal efficiency reached 95% for Mg^{2+} and 93% for SO_4^{2-}. In the absence of Na_2HPO_4, the precipitate predominantly consisted of Mg^{2+} (9.3%), SO_4^{2-} (38.8%), and Ca^{2+} (37.9%).

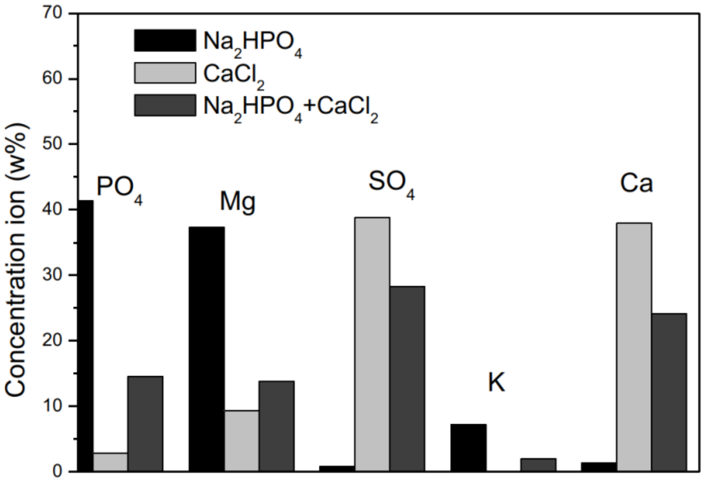

Figure 7. The distribution of ions in the dry product is based on ion precipitation efficiency.

The results demonstrated that ions Mg^{2+}, K^+, and SO_4^{2-} can be effectively removed from Aral Sea brine using precipitation with Na_2HPO_4 and $CaCl_2$. The removal efficiencies for Mg^{2+}, K^+, and SO_4^{2-} from the seawater were 97.1%, 96.3%, and 93.4%, respectively.

4. Conclusion

This study reveals an efficient approach to purify Aral Sea water samples through the selective precipitation of magnesium, potassium, and sulfate ions in the form of potassium magnesium phosphate and calcium sulfate dihydrate, utilizing sodium hydrogen phosphate, calcium chloride, and sodium hydroxide chemicals. This method was found to be efficient in achieving a high percentage of removal of interfering ions, with 97.1% removal of magnesium, 96.3% removal of potassium, and 93.4% removal of sulfate ions. These results confirm the effectiveness of this method in eliminating the major interfering ions, which are otherwise obstacles to the subsequent recovery of lithium from various resources.

The scientific novelty of this study lies in the application of the integrated approach to the precipitation method, which allows the concurrent removal of various interfering ions, thereby increasing the suitability of

Aral Sea brine resources as a potential lithium resource. Theoretically, this study contributes to the advancement of the understanding of the ion-selective precipitation process in complex systems, while the practical application of this method provides a potential approach to utilizing Aral Sea resources in the process of recovering lithium resources.

Author contributions

Bakhodir Abdullayev: Conceptualization, methodology, investigation, writing- original draft; Bakhodir Abdullayev: Visualization, project administration, writing- reviewing and editing; Murodjon Samadiy: Supervision, visualization, project administration, writing- reviewing and editing, reformatting, grammar editing; Khojakbar Egamberdiyev; Jasur Makhmayorov; Anvar Khudaykulov; Muslimbek Tuxliyev; Luiza Turdiqulova; Fayzulla Rakhmatullayev; Murodullo Rakhimov; Samugjon Nigmadjonov; Ozoda Sheralieva; Khusniddin Botirov; Dilafruz Gulboyeva; Tulkin Skakarov; Khusankhon Pulatov; Durbek Abdurashidov: Investigation and resource. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

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