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Application of sodium carbonate and sodium sulfate for removal of lithium and strontium from oilfield produced water

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Produced water is the largest byproduct of oil and gas production, which contains various environmental contaminants such as heavy metals, salts, and organic compounds. Among all cations present in produced water, lithium and strontium are of particular environmental concern. Lithium poses potential toxicity to aquatic organisms, while strontium contributes to scale formation and facilitates the co-precipitation of naturally occurring radioactive materials. Although selecting an appropriate treatment method remains a significant challenge, chemical precipitation has demonstrated promising potential for cation removal. This study investigates the effectiveness of chemical precipitation for removing lithium and strontium from highly saline produced water obtained from an oil field in Iran. Sodium carbonate and sodium sulfate were applied at concentrations of 0.12, 0.14, and 0.16 M. Experiments were conducted at two temperatures, 25 °C and 90 °C, to assess the influence of temperature and salt concentration on precipitation efficiency. The results revealed that Na_2SO_4 was particularly effective in strontium removal, achieving a maximum removal efficiency of 86% at 90 °C and 0.16 M. In contrast, Na_2CO_3 exhibited limited efficacy in lithium removal, with a maximum removal rate of only 10%. The results imply that although both salts are promising choices to remove strontium, more optimization is needed to enhance lithium extraction, potentially employing multi-step treatments or other pretreatment techniques.

Keywords Produced water treatment, Chemical precipitation, Lithium extraction, Strontium extraction

Produced water (PW), a byproduct of hydrocarbon production, is generated from both conventional oil reservoirs and unconventional sources such as tight sands, coal bed methane, and shale gas reservoirs[1]. The global rate of PW from oil and gas production operations is estimated to reach 600 million barrels per day[2,3]. Different options are available for the disposal of PW, including injection into disposal wells, surface discharge, and reinjection for enhanced oil recovery (EOR) or hydraulic fracturing operations. Among these, injection into disposal wells is the most widely employed method, with associated costs ranging from $0.40 to $1.75 per barrel 4. PW contains organic components, heavy metals, dissolved solids, and chemicals[5,6]. Cations such as Sodium (Na^+), Potassium (K^+), Lithium (ra), Calcium (Ca^{2+}), Magnesium (Mg^{2+}), Barium (Ba^{2+}), Strontium (Sr^{2+}), and heavy metals like Cadmium (Cd^{2+}), Lead (Pb^{2+}), Nickel (Ni^{2+}), and Silver (Ag^+) can be found at wide concentrations ranging from a few parts per million (ppm) to thousands of ppm, depending on pH, age, geology of the formation, and other conditions. Some of these species occur naturally, while others are introduced through chemical additives used in secondary and tertiary recovery processes. Due to their potential environmental hazards, the removal of such contaminants from PW is considered essential[7–10]. The primary environmental concern regarding PW in onshore fields is the presence of salt content. Some cations, such as Sr, exist at high concentrations and tend to remain in the ecosystem for long periods. However, in offshore fields, the main issue is the discharge of organic compounds into the sea and their toxic effects on aquatic organisms 5,10. In addition to their toxicity, the interactions of metals with anions in PW warrant careful consideration. For instance, the reaction of iron with dissolved oxygen can lead to the formation of precipitates that may clog reservoir pores during reinjection, potentially disrupting oil and gas production extraction. Improved recovery operations frequently rely on substantial volumes of water for processes such as EOR, water injection to maintain reservoir pressure, and hydraulic fracturing. Historically, freshwater sources were predominantly utilized for such purposes[10]. However, recent trends indicate that approximately 65% of PW is now treated and reinjected into reservoirs, while around

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30% is injected into disposal wells, depending on aquifer conditions. The remaining fraction is discharged as surface water[11,12]. Therefore, promoting PW treatment can help conserve freshwater resources, benefiting both the economy and the environment[13,14].

Li is one of the critical metals with rapidly increasing demand due to its widespread use in rechargeable batteries[15]. Recovering Li from alternative sources, such as oilfield PW, offers both economic benefits and a more sustainable approach compared to conventional mining. Li is a non-essential element for organisms and may cause ecological concerns in aquatic environments, such as including oxidative stress, inhibition of growth and reproduction, hepatotoxicity, and neurological disorders[16]. Indeed, its extraction from PW not only contributes to resource recovery but also helps reduce its potential environmental impact. Sr is another metal of concern in PW, typically occurring at higher concentrations than in seawater[17,18]. It poses environmental risks due to its tendency to form strontium sulfate ($SrSO_4$) scales during subsurface injection, which can cause formation damage and operational challenges[19]. Additionally, $SrSO_4$ may co-precipitate naturally occurring radioactive materials (NORM), such as radium, contributing to radioactive waste generation and complicating environmental management[20]. Regarding the necessity of removing these metals from PW, methods such as membrane filtration[6,11,21], electrochemical techniques[22–24], adsorption[25,26], solvent extraction[27–29], and chemical precipitation[20,30,31] have been employed to achieve selective extraction of the metals from PW. Membrane technology requires a high budget and pressure in addition to low removal efficiency due to complicated brine matrices and fouling[3,17]. Although electrochemical techniques are cost-effective technologies with zero pollutant discharge, they have only been tested on a laboratory scale[3]. Adsorption is an environmentally friendly method; however, its dependence on pH and competition between cations has limited its application on an industrial scale[17,32]. High recovery rates are achieved by solvent extraction in a short operational time, but organic extractants make it harmful to the environment[17,33].

Chemical precipitation is one of the most widely used treatment methods for removing metals from PW due to its high efficiency and low cost. It has established a significant position on an industrial scale[34,35]. The reduction in the solubility of dissolved particles causes ions to convert into solid precipitates during the precipitation process, allowing for their easy separation from water[36]. Various polymers and inorganic salts have been used to precipitate the selected metals. Shafer-Peltier et al.[14] examined four polyelectrolytes for Ba and Sr removal from Kansas oil field PW. Approximately 60% of Sr was removed using 4-styrenesulfonic acid-co-maleic acid. The choice of precipitating agent depends on water quality: sodium hydroxide (NaOH) is usually used for low concentrations of temporary hardness, calcium hydroxide ($Ca(OH)_2$) for low concentrations of permanent hardness, and sodium carbonate (Na_2CO_3) for high concentrations of permanent hardness[37]. In addition, sodium sulfate (Na_2SO_4) has recovered metals such as Sr and Ba[38]. The presence of oil, grease, and organic acids, especially carboxylic acids, in water significantly reduces the precipitation rate of $SrSO_4$[36,39]. Acid mine drainage (AMD) as a source of sulfate has been used to precipitate more than 70% of Sr in PW[40,41]. On the industrial scale, most Li obtained from brines is in the form of lithium carbonate (Li_2CO_3), produced by adding Na_2CO_3[42]. The Mg/Li concentration ratio in most PW exceeds 20. The co-precipitation of Mg is the reason why Li_2CO_3 precipitation has not been employed as a single-step method[43]. An increase in total dissolved solids (TDS) concentration also reduces the selectivity of the precipitating agent for Li[44].

Various methods have been employed for the removal of metals from PW; however, chemical precipitation offers precise control over reaction parameters such as temperature, pressure, ionic ratios, pH, and solute concentration. A thorough understanding of these parameters is crucial for the efficient treatment of PW. Gusa et al.[45] analyzed the effect of different pH and ionic ratios (Sr/Ba and SO_4/Ba) on $SrSO_4$ precipitation in PW from Marcellus Shale. In the present study, the effects of temperature and salt concentration on the precipitation of Li and Sr were investigated using PW obtained from a carbonated reservoir in Iran. Notably, this work was conducted without the integration of any additional treatment methods or pretreatment steps. To date, no comparative study has systematically evaluated the efficiency of Na_2CO_3 and Na_2SO_4 in precipitating Li and Sr from highly saline PW. This research addresses this knowledge gap by assessing and comparing the performance of these two salts in metal recovery, thereby offering new insights for optimizing the chemical precipitation process.

Experimental

Produced water characterization

The PW sample was collected from an oil field located in western Iran, specifically from the water outflow of the first-stage separator (Fig. 1). This separator represents the initial phase of the oil–water separation process, in which a mixture of oil, gas, and PW is separated. Sampling was conducted under controlled conditions to ensure consistency and representativeness of the water composition for subsequent analyses. The samples remained unfrozen during transportation, and no pretreatment was applied prior to analysis. The pH of the solution was measured using a Sentek pH meter, and it was reported to be 6.5 at ambient temperature. The inductively coupled plasma-optical emission spectrometer (ICP-OES, VISTA-PRO, Varian Inc., USA) was used to measure the metals as soon as they arrived in the laboratory. The initial characteristics of the PW are shown in Table 1. The concentration of TDS refers to the Salinity of water. Since ions dissolved in water conduct electricity, and measuring TDS directly is costly, it is commonly measured by electrical conductivity (EC). The EC of the PW sample was directly measured using an Endress + Hauser Smartec CLD130 portable conductivity meter (detection range: up to 1000 mS/cm), capable of measuring high salinity levels without sample dilution. The measured EC value was approximately 277,864 µS/cm, reflecting the high ionic strength of the sample. To estimate the TDS, a conversion factor of 0.65 was applied, resulting in an approximate TDS value of 180,612 ppm. The conversion factor of 0.65 was chosen based on the dominance of Na^+ and chloride Cl^- ions, which are the primary contributors to the conductivity of the sample. Other ions, such as Mg^{2+}, Ca^{2+}, and SO_4^{2-}, while present, are in lower concentrations and have a minimal effect on the overall conductivity.

Fig. 1. The PW sample.

Na^+	57,489 ppm	Sr^{2+}	629.4 ppm
K^+	4752 ppm	Cl^-	109,386 ppm
Li^+	20.02 ppm	SO_4^{2-}	798 ppm
Ba^+	2.13 ppm	CO_3^{2-}	0
Ca^{2+}	6220 ppm	HCO^{3-}	620 ppm
Mg^{2+}	1342 ppm	TDS	180,612 ppm
Fe^{2+}	5 ppm	pH	6.2

Table 1. Ion concentration and other water quality data for the collected PW.

Sample	Temperature (°C)	Concentration(M)	Employed salt	Required salt weight
1	90	0.12	Na_2CO_3	0.25
2		0.14		0.30
3		0.16		0.35
4	25	0.12		0.25
5		0.14		0.30
6		0.16		0.35
7	90	0.12	Na_2SO_4	0.34
8		0.14		0.40
9		0.16		0.45
10	25	0.12		0.34
11		0.14		0.40
12		0.16		0.45

Table 2. Properties of the samples.

Method

Three different concentrations of Na_2CO_3 (98%, Fisher Scientific) and Na_2SO_4 (Merck) with 20 mL of PW were prepared (0.12, 0.14, and 0.16 M). The samples were homogenized using a vortex mixer and then incubated for 24 h at two different temperatures: 25 °C and 90 °C, using a Binder oven (Model FED 115). The properties of the 12 prepared samples are presented in Table 2. Visual appearances of the PW samples following the addition of Na_2CO_3 and Na_2SO_4 are shown in Fig. 2. To investigate the effect of temperature on the reaction rate, the volume of precipitates formed after 1 h of reaction was compared between the samples maintained at 25 °C and those at 90 °C. After 24 h, the resulting precipitates, formed as insoluble salts, had settled at the bottom of the containers. The concentrations of Sr and Li remaining in the supernatant were measured using ICP-OES analysis.

Results and discussion

After 24 h of adding the precipitating agents, a noticeable amount of precipitate was observed in the samples incubated at both 25 °C and 90 °C (Fig. 3). The concentrations of Li and Sr remaining in the supernatant were quantified using ICP-OES analysis. As presented in Table 3, both temperature and salt concentration had a significant impact on metal removal efficiency, with the highest removal observed at 90 °C and a concentration of 0.16 M. The maximum Li removal, achieved using Na_2CO_3, was approximately 10%, highlighting the inherent difficulty of extracting Li from high-salinity PW. In contrast, Sr removal reached up to 86% with the addition of Na_2SO_4 under the same temperature and concentration conditions.

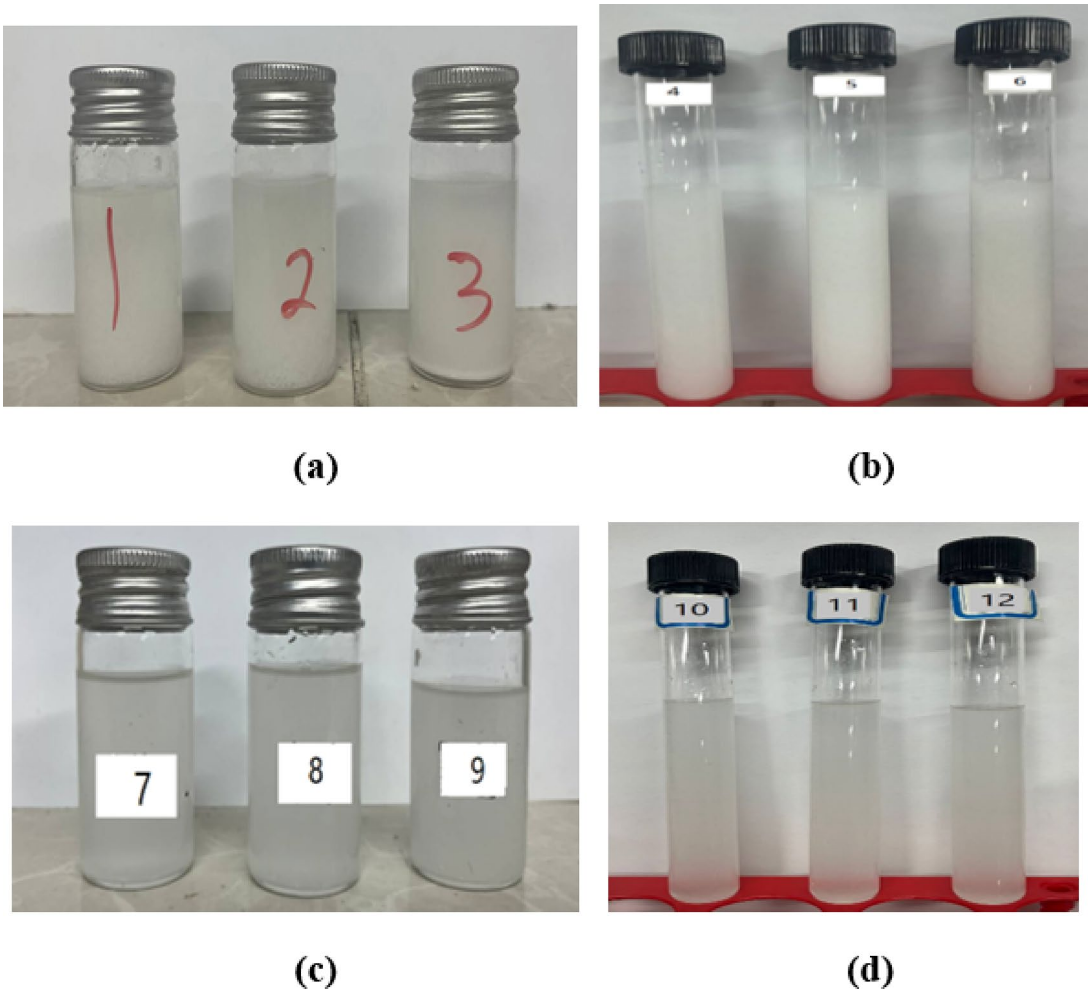

Fig. 2. PW Samples immediately after mixing with salts. (**a**) using Na_2CO_3 at 90 °C, (**b**) using Na_2CO_3 at 25 °C, (**c**) using Na_2SO_4 at 90 °C, (**d**) using Na_2SO_4 at 25 °C.

Strontium separation

The solubility of $SrCO_3$ and $SrSO_4$ has been reported as 0.0034 g/L at 20 °C and 0.135 g/L at 25 °C, respectively[46]. Without pre-treatment, promising results were obtained for Sr removal using both carbonate and sulfate salts due to the very low solubility of Strontium carbonate ($SrCO_3$) and $SrSO_4$, although Na_2SO_4 exhibited superior performance. More Sr removal could be achieved with precipitation and removal of Ca^{2+} as $CaCO_3$, for which a higher amount of chemical is needed[14].

Lithium separation

Metals at lower concentrations, such as Li, are usually difficult to extract and have higher separation costs[47]. By adding Na_2CO_3, a significant percentage of Li did not precipitate due to the high solubility of Li_2CO_3 (13 g/L at 25 °C[46]. Typically, single-step precipitation cannot produce high-purity Li_2CO_3 with a very high Li recovery rate, particularly from a PW containing low concentrations of Li[48]. On the other hand, an increase in water salinity reduces the selectivity of the precipitant for Li ions[44]. The co-precipitation of Mg is the main challenge in the Li precipitation process. In most PWs, the Mg/Li concentration ratio exceeds 20[44]. The ionic radii of Li^+ and Mg^{2+} are approximately similar, and their chemistry exhibits some parallels. However, it should be noted that lithium carbonate (Li_2CO_3) is significantly more soluble than magnesium carbonate ($MgCO_3$), primarily due to differences in their hydration energies and lattice energies[49,50]. Similar studies suggest that multi-step separation processes may be required to improve Li recovery efficiency in high-salinity environments[51]. Furthermore, Li ions do not directly precipitate with sulfate ions due to the relatively high solubility of Lithium sulfate (Li_2SO_4), which is 342 g/L at 25°C[46]. The presence of Na_2SO_4 in solution can alter the behavior of other constituents, including carbonates and chlorides. The addition of Na_2SO_4 increases the ionic strength of the solution, thereby decreasing the ionic activity of Li. This reduction in activity promotes the interaction of Li^+ ions with available anions, potentially resulting in the formation of a limited amount of lithium-containing precipitate[52]. Overall, the removal efficiency of Li is limited due to the presence of divalent cations, which exhibit higher reactivity compared to Li^+. Therefore, it is recommended that this method be applied subsequent to the separation of other divalent cations, using techniques such as solvent extraction, to improve Li removal efficiency[27,28].

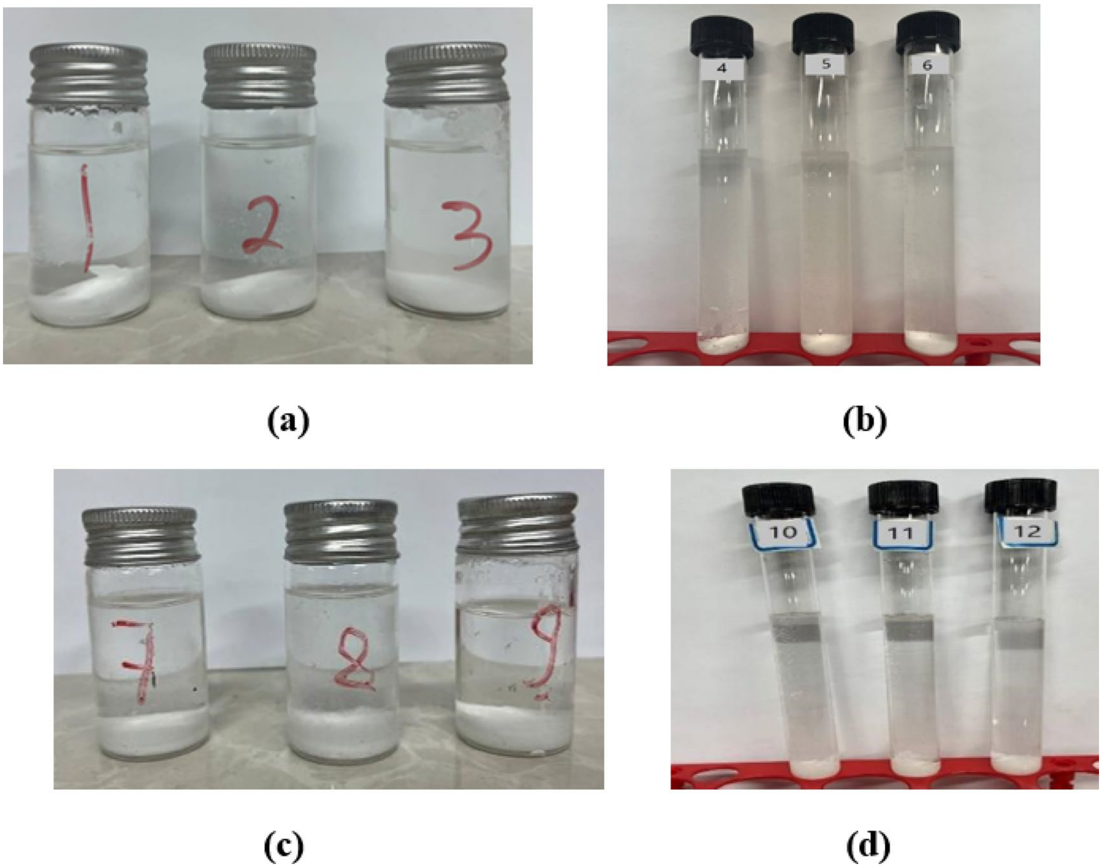

Fig. 3. Amount of precipitates after 24 h. (**a**) using Na_2CO_3 at 90 °C, (**b**) using Na_2CO_3 at 25 °C, (**c**) using Na_2SO_4 at 90 °C, (**d**) using Na_2SO_4 at 25 °C.

Sample	Temperature (C°)	Final Li^+ Concentration (ppm)	Removed (%)	Final Sr^{2+} Concentration (ppm)	Removed (%)
1	90	18.27	8.6	404	35.5
2		18.12	9.4	362	12
3		17.96	10.2	350	12
4	25	18.66	6.7	420.5	33.2
5		18.35	8.2	370.7	42.5
6		18.28	8.6	364.17	44
7	90	18.73	6.35	203	67.7
8		18.98	5	122.35	80.5
9		18.4	8	89	86
10	25	19.5	2.5	503	20
11		18.98	5	471.1	30
12		18.5	7.5	401.14	36.5

Table 3. Li and Sr concentration in the supernatant.

Effect of the Na_2CO_3 and Na_2SO_4 concentration and temperature on the precipitation process

The effect of temperature and precipitating agent concentration on the removal of Li and Sr is shown in Fig. 4. The observed increase in precipitation rate with higher concentration can be explained by Le Chatelier's principle, which dictates that a system in equilibrium will adjust to counteract changes. Specifically, when the concentration of a reactant is increased, the system will shift the equilibrium position to reduce the concentration of the reactant, leading to the formation of more product[53].

The experimental results indicate that increasing the temperature enhances the precipitation rate for both Li and Sr. The decreased solubility of Li and Sr compounds at higher temperatures contributed to the greater

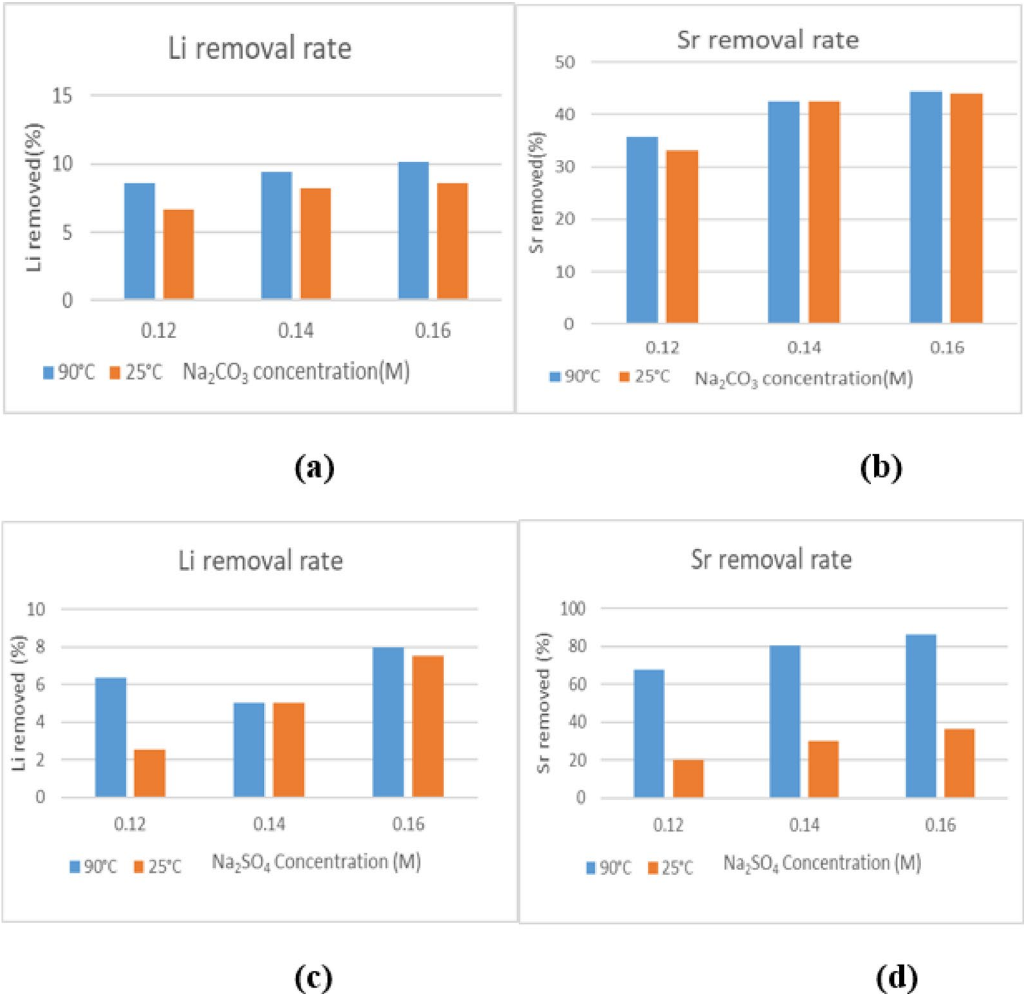

Fig. 4. Effect of the temperature and concentration on removal efficiency. (**a**) using Na_2CO_3 for Li, (**b**) using Na_2CO_3 for Sr, (**c**) using Na_2SO_4 for Li, (**d**) using Na_2SO_4 for Sr.

amount of precipitate formed. Although the solubility of $SrCO_3$ exhibits a negligible rise (< 0.7 g/L change) across the temperature range of 25–90 °C[54], the increased rate of crystal growth at higher temperatures might be the reason for more precipitation. Solute molecules or ions are accelerated with temperature, causing faster attachment and diffusion to growth sites per unit of time. As a result, the growing rate of crystals increased[55,56]. According to the Arrhenius equation, an increase in temperature results in an increase in the reaction rate constant (k). It means that the number of effective collisions between molecules and ions becomes greater. The temperature rise also increases the kinetic energy of particles, allowing more of them to have enough energy to overcome the activation energy barrier; as a result, the precipitation process occurs more rapidly. One hour after the reaction started, precipitation at 90 °C occurred significantly faster. Samples at 25 °C remained in suspension (similar to when salt is added to PW), while the samples at 90 °C precipitated rapidly. The precipitation status at two different temperatures (90 °C and 25 °C) after 1 h of reaction is illustrated in Fig. 5.

Conclusion

The presence of Li and Sr in produced water raises environmental concerns, as Li can be toxic to aquatic life, and Sr contributes to scale formation and the co-precipitation of radioactive materials. This research investigated the potential of chemical precipitation as an efficient method for removing Li and Sr from the PW collected from an oilfield in Iran. The experimental results underscore the significant influence of both temperature and the concentration of Na_2CO_3 and Na_2SO_4 on the precipitation rates of Li and Sr. While Sr removal yielded promising results, Li extraction remained relatively limited. Na_2SO_4 demonstrated high efficacy in Sr removal, achieving a maximum efficiency of 86%, whereas the maximum Li removal was 10.2% with Na_2CO_3. The limited efficiency of Li removal is primarily attributed to its chemical behavior and, more critically, the competitive interaction with divalent cations, which represent the main obstacle in the removal process. Maximum efficiency for both Na_2CO_3 and Na_2SO_4 was achieved at an elevated temperature of 90 °C and a concentration of 0.16 M for each salt. Higher temperatures may enhance crystal growth and increase the reaction rate constant, potentially leading to more efficient precipitation. Furthermore, increasing the concentration of Na_2CO_3 and Na_2SO_4 shifted

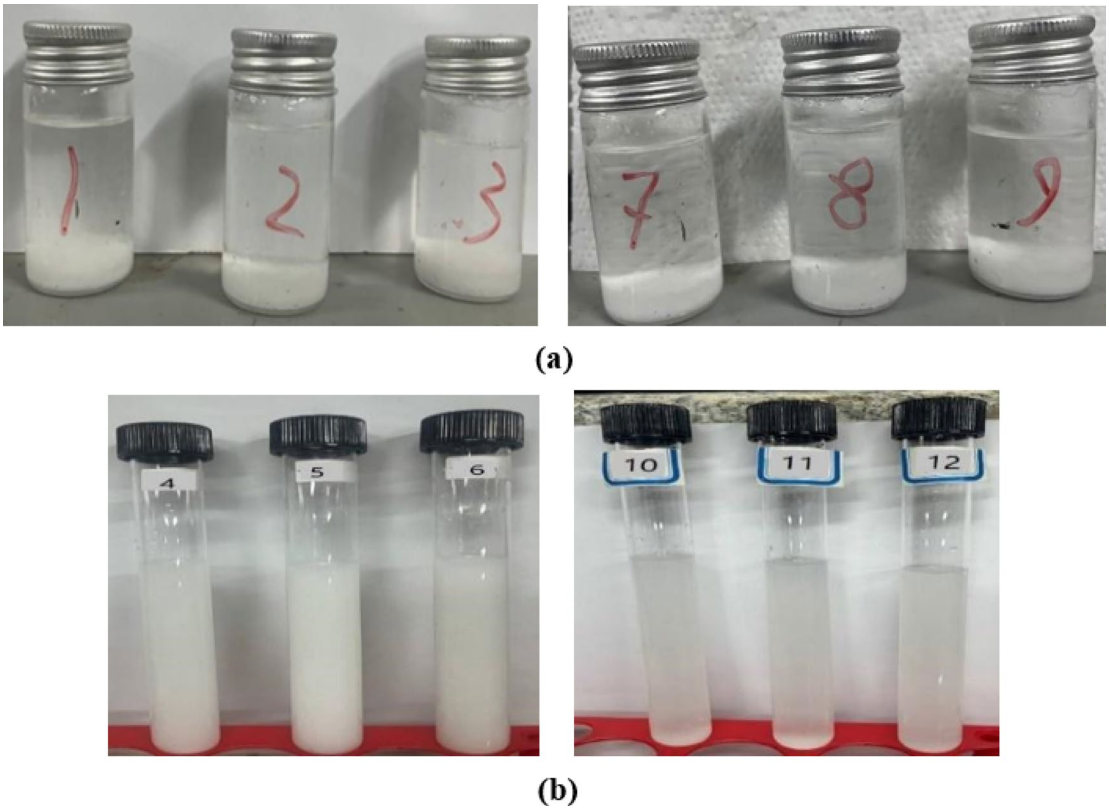

Fig. 5. Status of precipitation after 1 h (**a**) 90 °C (**b**) 25 °C.

the system's equilibrium, favoring precipitation. These findings offer valuable insights for optimizing chemical precipitation, contributing to environmental sustainability by minimizing contaminants in PW, and promoting water resource conservation.

Data availability

The data used and analyzed in this study are available at Amirkabir University of Technology and Tarbiat Modares University. Access to the data can be granted upon reasonable request to the corresponding authors.

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Author contributions

M.T was responsible for Methodology, Data analysis, Writing, and Editing. A.R as responsible for Methodology, Conceptualization, Data analysis, and Resources Y.R Supervised the research, contributed to conceptualization , and provided critical revisions to improve the manuscript.

Declarations

Competing interests

The authors declare no competing interests.

Additional information

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