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Adsorption of cesium from waste desalination brine through Dowex G26 resin and comparison with t-BAMBP/kerosene and t-BAMBP/C_2mimNTf_2 systems

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ABSTRACT

Cesium can be widely used in many industries now. However, the primary reservoir of cesium is often located in high mountains and deserts, so it is challenging to obtain cesium resources. To solve this problem, cesium was recovered from waste desalination brine through Dowex G26 resin in this study. Due to the lack of water resources, desalination technology was developed since the 20th century. Nevertheless, its by-product, brine, is harmful to the environment. To reduce the threat of waste brine, this experiment aims to provide a simple procedure to recover cesium resources from brine. The study could be divided into three parts. First, the adsorption isotherms and the thermodynamics parameters such as enthalpy, entropy, and Gibbs free energy of cesium adsorption were investigated. In the second part, the batch experiments would explore the optimal adsorptive period, pH value, and liquid-solid ratio (L/S ratio) of cesium adsorption through Dowex G26 resin. The results showed that the adsorption of cesium fit with the Langmuir model, and the reaction was spontaneous. On the other hand, cesium could be recovered efficiently under the conditions of 4 min of contacting time, pH 9, and L/S ratio of 1,000. After adsorbing, the cesium could be desorbed through nitric acid, and solid cesium nitrate would be obtained by drying the desorbed solutions. In the end, the Dowex G26 system would be compared with the t-BAMBP/kerosene system and t-BAMBP/C_2mimNTf_2 system to demonstrate its advantages and drawbacks.

Keywords: Cesium; Ion exchange; Dowex G26; Desalination; Brine; Recovery

1. Introduction

Cesium is an alkali element, and it has an atomic number of 55, an atomic weight of 132.90, a melting point of 28.5°C, and a boiling point of 705°C [1]. Cesium was discovered by Bunsen and Kirchhoff from mineral water with an optical spectroscope. They initially named it caesius in Aladdin due to its light blue line in the spectrum [2,3]. The average abundance of cesium is 7 ppm in the Earth's crust, and it was ranked 40th among all the elements. According to the U.S. Geological Survey (USGS) report, the cesium resources are all in the form of compounds and mainly from garnet, pollucite, and lepidolite [4]. Pollucite contains 5% to 32% cesium oxide, and the reserves of cesium are estimated based on the abundance of pollucite. The world's total amounts are 200,000 metric tons in 2021, and the major reservoirs are in Australia, Canada, China, Namibia, and Zimbabwe [4].

Cesium can be applied in many industries in the form of element and compounds from the past to now. For example,

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the iconoscope in the television was manufactured by cesium due to its excellent photoelectric properties [1]. Cesium atomic clock is widely used in communication, transportation, and the military due to a small margin of error [5]. The isotope of cesium, ^{137}Cs, can be used in agriculture, treatment of cancer, and food disinfection [6]. On the other hand, cesium compounds are commonly applied in the optoelectronics industry, organic manufacturing, molecular biology, catalyst, and pharmaceutical industry [7–10]. For example, cesium nitrate can be used as a light refraction regulator in the optical fiber industry and glass industry [11]. Due to the unlimited development of cesium and its compounds, the extraction of cesium was gradually crucial in many countries.

Nowadays, cesium is mainly extracted from saline lakes and ores [12,13]. However, the saline lakes and ores are often located in high mountains and deserts, so it is difficult to acquire cesium resources. In this case, cesium was recovered from waste desalination brine in our previous study [14]. Due to the extreme climate, the lack of water resources has become a big issue in many countries. To solve this problem, seawater desalination technology has developed since the beginning of the 20th century [15]. Although the water shortage has been solved through desalination technology, the other environmental problem, waste brine, has gradually affected our lives. Waste brine would change the composition and temperature of seawater which has side effects on the ecosystem. Even if the waste brine has many disadvantages, it still has one significant merit. Waste brine is a concentrated solution in which the concentrations of metals are higher than in seawater. It contains abundant lithium, rubidium, cesium, magnesium, and other metals. If waste brine can be treated correctly, it will be an essential resource of many metals [16]. Due to this reason, our group investigated different methods to recover cesium from waste brine to create its adding value, and the techniques would be applied and adjusted to other metals in the future.

There is no research yet reporting the method of adsorbing cesium from waste desalination brine through Dowex G26 resin. This study aims to provide a simple method and optimal parameters to recover cesium resources. The experiment could be divided into three parts. In the first part, the adsorption isotherms, described by means of the Langmuir and Freundlich isotherms, were used to investigate the ion exchange behaviors of cesium. On the other hand, the parameters of thermodynamics such as enthalpy, entropy, and Gibbs free energy were investigated as well. In the second part, the optimal conditions of cesium adsorption through Dowex G26 resin were demonstrated. The adsorptive period, pH value, and liquid–solid ratio (L/S ratio) would be explored in the batch experiments, and the adsorptive capacity would be shown, too. After having the optimal parameters, Dowex G26 resin would be compared with the t-BAMBP/kerosene system and t-BAMBP/$C_2mimNTf_2$ system, and the results would be revealed in the last part. The parameters and comparison demonstrated that the adsorption efficiency of cesium through the Dowex G26 resin system is equal to or a little worse than the extraction system and can reduce the danger of experiment, expense, and energy usage. It is expected to recover valuable metals, reduce waste, and protect the environment.

2. Materials and methods

2.1. Reagents, chemicals, and apparatuses

Dowex G26 was purchased from Lenntech (Delfgauw, Netherlands) and used to adsorb cesium from simulated solution and waste desalination brine in this research. Dowex G26 resin is a strong acid exchange resin which can adsorb cation ions efficiently, and its adsorption mechanism is shown in Eq. (1) [17,18]. Its appearance and basic information are shown in Fig. 1 and Table 1 [18]. Cesium carbonate (99.9%) was acquired from Sigma-Aldrich (St. Louis, MO, USA) and dissolved in deionized water (resistivity 18.0 MΩ cm). The adsorption isotherms and thermodynamic parameters were investigated with Dowex G26 resin and cesium carbonate solutions. After obtaining the data, Dowex G26 resin was then contacted with waste desalination brine to conduct the experiment of the adsorptive period, pH value, and liquid–solid ratio (L/S ratio). The waste brine was prepared from the previous study, and the principal elements and the composition are shown in Table 2 [14]. On the other hand, ammonia (32%) and sulfuric acid (95%–98%) were both obtained from Sigma-Aldrich (St. Louis, MO, USA). They were used to adjust the pH value to get the optimal parameters. In the analysis part, ICP standard solutions were purchased from High-Purity Standards, Inc., (North Charleston, SC, USA). The nitric acid (65%–67%) was from Alfa Aesar (Haverhill, MA, USA) and was diluted to 1% to be the background value and thinner for ICP analysis.

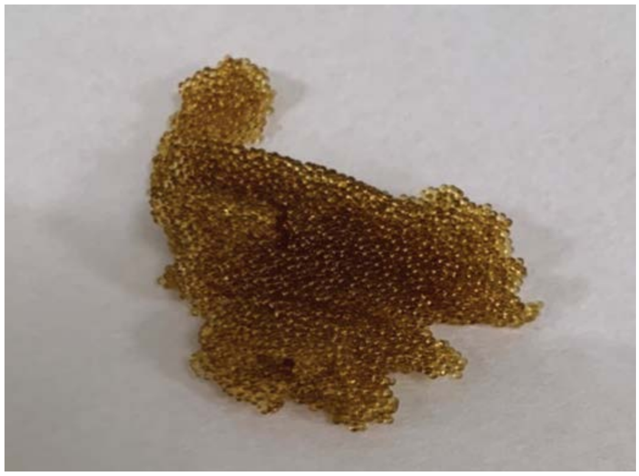

Fig. 1. The appearance of Dowex G26 resin.

Table 1
Basic information of Dowex G26 resin [18]

Matrix	Styrene-divinylbenzene (gel)
pH group	0–14
Functional group	Sulfuric acid
Moisture holding capacity	45%–52%
Particle size	0.6–0.7 mm
Total exchange capacity	2.0 meq/ml

Table 2
Main elements and composition of waste desalination brine [14]

Elements	Concentration (mg/L)
Li	167
Na	49,180
K	91
Ca	622
Mg	13,570
Rb	6.94
Cs	42.14

In this experiment, the main apparatuses are inductively coupled plasma optical emission spectrometry (ICP-OES, Varian, Vista-MPX, PerkinElmer, Waltham, Massachusetts State, America) and pH meter (SP-2300; SUNTEX; New Taipei City, Taiwan). ICP-OES was used to determine the concentration of cesium ion and other ions such as potassium and rubidium. The relative standard deviation (RSD) of it was below 3%. The pH meter in this experiment was used to determine the pH value, and the RSD was below 1%. Both were calibrated initially to ensure the accuracy of values.

$$\text{R-SO}_3^-\text{H}^+ + \text{Cs}^+ \rightarrow \text{R-SO}_3^-\text{Cs}^+ + \text{H}^+ \quad (1)$$

2.2. Ion exchange process

In this part, Dowex G26 resin was contacted with cesium carbonate solution first to investigate the adsorption isotherms model and the parameters of thermodynamics such as enthalpy, entropy, and Gibbs free energy. To get the isotherm model, the Dowex G26 resin was added into different initial concentrations of Cs^+ (10, 20, 200, 400, 800, 1,100, 1,300, 1,400, and 1,500 ppm) for 24 h and the relationship between C_e (concentration of adsorbate in the liquid when adsorption is in equilibrium) with q_e (equilibrium adsorption capacity of the adsorbent) would be shown in Section 3. On the other hand, to get the high preciseness of the adsorption model, Langmuir and Freundlich's equations were used to create the figures. Eqs. (2) and (3) are the Langmuir equation and Freundlich equation, separately [19,20]. In the Langmuir model, the maximum adsorption capacity q_m and adsorption equilibrium constant K_L could be obtained by the relationship between C_e and C_e/q_e. In the Freundlich model, empirical constant n and the adsorption equilibrium constant K_F could be calculated from the relationship between lnC_e and lnq_e. R^2 in the figures then could demonstrate which adsorption behavior of cesium through Dowex G26 resin was.

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} \quad (2)$$

$$\ln q_e = \ln K_F + \frac{1}{n}\ln C_e \quad (3)$$

To obtain the thermal dynamics parameters, 0.1 g Dowex G26 was contacted with 100 mL cesium carbon solutions under the conditions of 298 to 348 K for 24 h. According to the results, the slope and intercept of the figure could be obtained. Enthalpy, entropy, and Gibbs free energy then could be calculated by Eqs. (4)–(8). R in Eqs. (4)–(6), and (8) is the ideal gas constant, 8.314. K_d in Eq. (7) is the partition coefficient when the adsorption equilibrium. C_i and C_f mean the concentration of cesium in the solution before adsorption and after adsorption. V and M are the volume of solution and the molarity of resin, separately.

$$\Delta H = \text{Slope} \times R \quad (4)$$

$$\Delta S = \text{Intercept} \times R \quad (5)$$

$$\Delta G = -RT \ln K_d \quad (6)$$

$$K_d = \frac{C_i - C_f}{C_i} \times \frac{V}{M} \quad (7)$$

$$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (8)$$

After getting the isotherm model and thermal dynamics parameters of cesium adsorption through Dowex G26 resin, the resin was then contacted with waste desalination brine to recover cesium resources. The adsorptive period (2–1,024 min), pH values (5–11), and solid-liquid ratio (100–1,000) were set up in the ion exchange experiment. After recovering the cesium, the optimal parameters would be demonstrated and compared with the t-BAMBP/kerosene and t-BAMBP/C_2mimNTf_2 system.

3. Results and discussion

3.1. Adsorption isotherm model

Before adsorbing cesium from waste desalination brine, the saturated adsorption capacity and isotherm model should be known first. To get the isotherm model, 0.1 g Dowex G26 resin was added into 100 mL different initial concentrations of Cs^+ (10, 20, 200, 400, 800, 1,100, 1,300, 1,400 and 1,500 ppm) for 24 h. Fig. 2 shows the relationship between C_e (concentration of adsorbate in the liquid when adsorption is in equilibrium) and q_e (equilibrium adsorption capacity of the adsorbent). According to the figure, the saturated adsorption capacity of cesium through Dowex G26 resin was about 450 mg/g.

To obtain the precise value and the isotherm models, Eqs. (2) and (3) were used to create the Langmuir model and Freundlich model, respectively. Fig. 3 demonstrates that R^2 was 0.99309 in the Langmuir model, and the q_m (saturated adsorption) and K_L (adsorption equilibrium constant) could be acquired through the slope and intercept of the figure. Fig. 4 reveals that R^2 was 0.98242 in the Freundlich model, and the n (empirical constant) and K_F (adsorption equilibrium constant) could be obtained from the slope

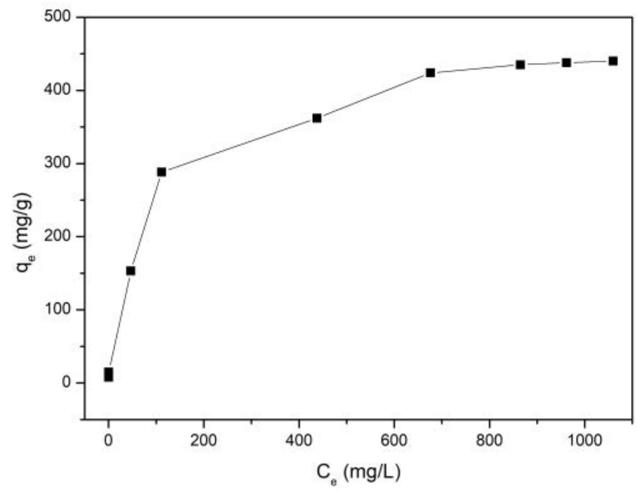

Fig. 2. The adsorption capacity of cesium through Dowex G26 resin.

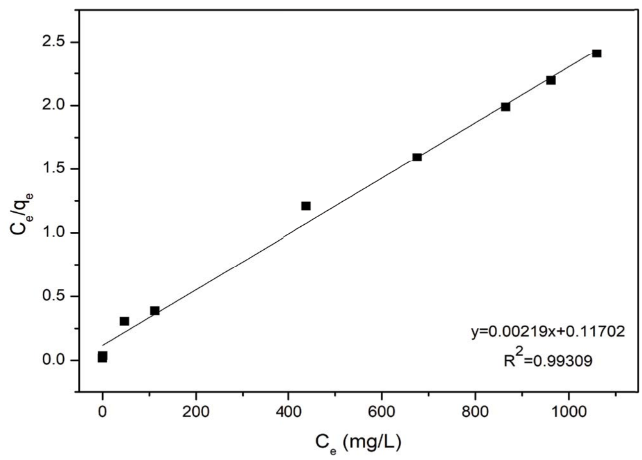

Fig. 3. The adsorption capacity of cesium through Dowex G26 resin – Langmuir model.

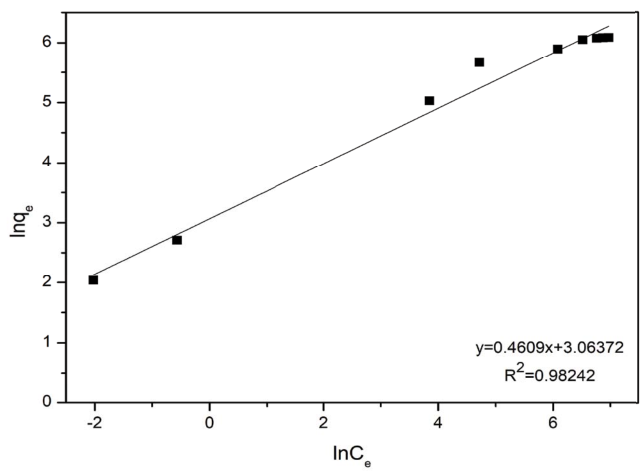

Fig. 4. The adsorption capacity of cesium through Dowex G26 resin – Freundlich model.

and intercept as well. The details about the two models are shown in Table 3. The results indicated that the cesium adsorption through Dowex G26 resin fit with the Langmuir model because its R^2 was larger than the Freundlich model. It also represented that the Dowex G26 resin has a uniform adsorption position on the surface and the accurate saturated adsorption capacity was 456.6 mg/g.

3.2. Thermodynamics of cesium adsorption through Dowex G26

800 ppm of cesium carbonate solution was prepared in this part, and the temperatures were set up from 298 to 348 K. The other fixed parameters were contacting time of 24 h, pH 7, and L/S ratio of 1,000 (100 mL to 0.1 g Dowex G26 resin). Fig. 5 reveals that the partition coefficient would increase when the temperature increased. It means that the adsorption of cesium through Dowex G26 resin was an endothermic reaction. According to the slope and intercept of the figure, enthalpy, entropy, and Gibbs free energy could be calculated by Eqs. (4)–(8). The detailed data are shown in Table 4. The ΔGs in this experiment were all negative, and both ΔS and ΔH were positive, demonstrating that the cesium adsorption through Dowex G26 resin was spontaneous and could react efficiently at a higher temperature. Although a higher temperature could reach a higher adsorption capacity, 298 K would be the operational temperature in the continuous procedures due to energy saving.

3.3. Effect of adsorptive period

After investigating the adsorption isotherm model and thermodynamics parameters of cesium adsorption through

Table 3
Data of Langmuir model and Freundlich model

Langmuir model (R^2 = 0.99309)	Freundlich model (R^2 = 0.98242)
q_m = 1/slope	n = 1/slope
1/0.00219 = 456.6 mg	1/0.4609 = 2.17
$K_L = 1/(q_m \times \text{intercept})$	$K_F = e^{\text{intercept}}$
1/(456.6 × 0.11702) = 0.0187	$e^{3.06372}$ = 21.4

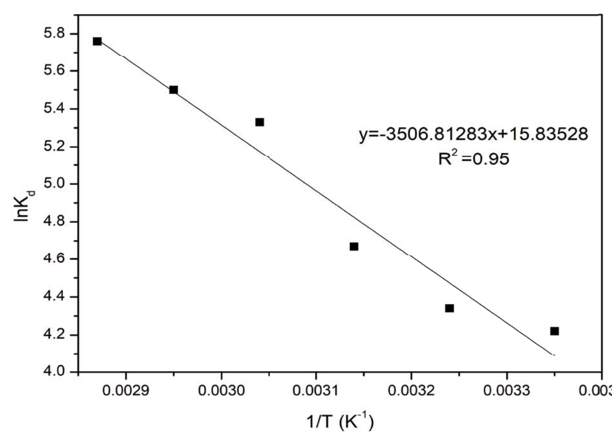

Fig. 5. The partition coefficient (lnK_d) of cesium at different temperatures (1/T).

Dowex G26 resin, the resin was then applied to recover cesium from waste desalination brine. The concentration of cesium in the brine was 42.14 ppm, so the theoretical adsorption capacity was 37.1 mg/g by calculating through Eq. (2). On the other hand, due to the characteristics of Dowex G26 resin and our experiment, the main impurities which could influence the cesium adsorption were rubidium and potassium. Therefore, the adsorption capacities of rubidium and potassium would be investigated as well. The adsorptive periods were set up from 2 to 1,024 min, and other fixed parameters were pH 7 and the L/S ratio of 1000. Fig. 6 illustrates that the adsorption behavior of cesium and rubidium began from 4 min and got equilibrium immediately. Their adsorption capacities were about 14.4 and 2.4 mg/g, respectively. However, potassium would be adsorbed from 32 min and could increase gradually to 16 mg/g when the contacting time was 1,024 min. To reduce the side effect of potassium and save time, contacting time of 4 min was chosen as the optimal parameter in this process.

3.4. Effect of pH value

The pH values were set up from pH 5 to pH 11 to investigate the adsorption behavior of cesium through Dowex G26 resin under acidic, neutral, and alkaline situations. Other fixed parameters were contacting time of 4 min and the L/S ratio of 1,000. Fig. 7 shows that the adsorption capacity of cesium increased from 8.6 to 36.5 mg/g when

Table 4
lnK_d, enthalpy, entropy and Gibbs free energy of cesium adsorption

K	lnK_d	ΔG (kJ/mol)	ΔS (J/mol K)	ΔH (kJ/mol)
298	4.22	–10.81		
308	4.34	–11.11		
318	4.67	–12.35	131.65 J/mol K	29.16 kJ/mol
328	5.33	–14.53		
338	5.50	–15.46		
348	5.76	–16.67		

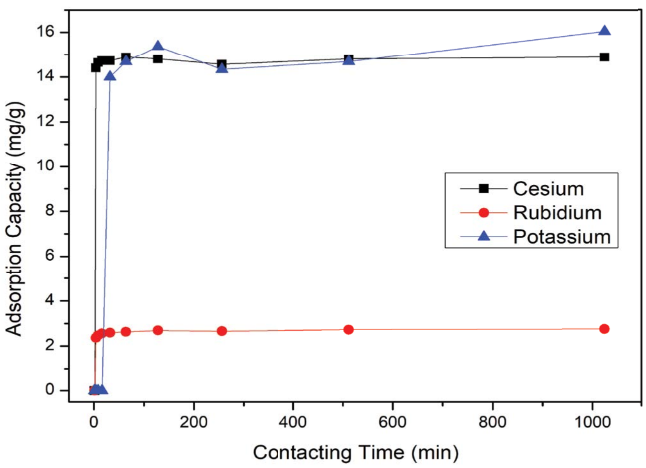

Fig. 6. The effect of contacting time of adsorption capacity.

pH values were from 5 to 11. This is because that Dowex G26 resin is a strong-acid ion exchange resin, it can easily exchange H^+ with other metal ions under alkaline conditions. Although the adsorption efficiencies of cesium at pH 10 and 11 were higher, the resin would begin to adsorb a great amount of potassium as well. The adsorption capacities of potassium were 17 and 28.3 mg/g at pH 10 and pH 11. To prevent more impurities from adsorbing by Dowex G26, the optimal pH value was chosen as pH 9 in this process.

3.5. Effect of liquid–solid ratio (L/S ratio)

In this procedure, the L/S ratios were set up from 100 to 1,000 to explore the adsorption behavior. Other fixed parameters were contacting time of 4 min and pH 9. Fig. 8 illustrates that the L/S ratio did not influence the adsorption capacities of cesium and rubidium. The adsorption capacities of cesium were 31.67 and 28.31 mg/g when the L/S ratio was 100 and 1,000. However, the adsorption capacity of potassium would change dramatically due to the L/S ratio. The speculated reason is that the concentration of potassium in the waste desalination brine was much higher than cesium

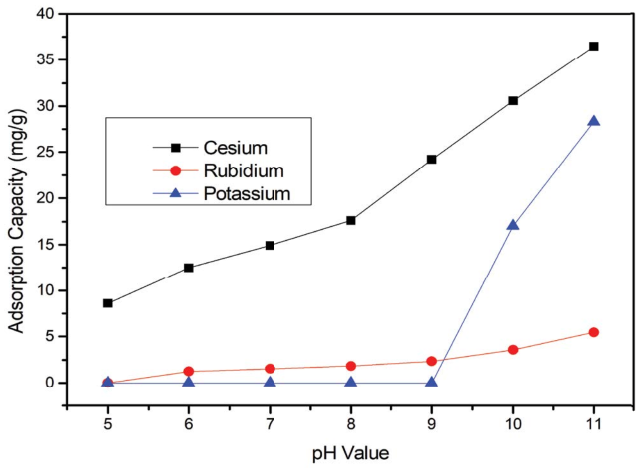

Fig. 7. The effect of pH value of adsorption capacity.

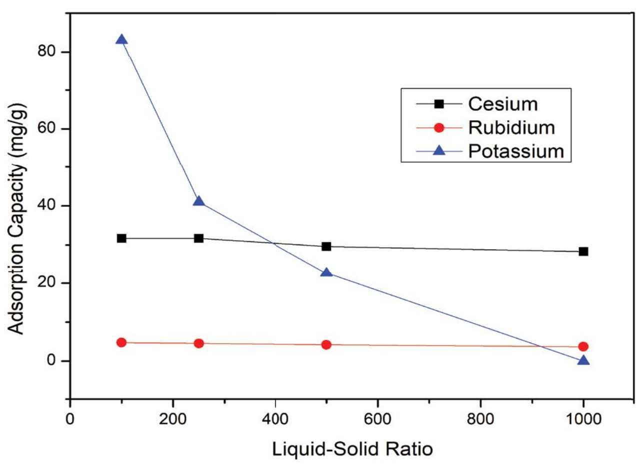

Fig. 8. The effect of L/S ratio of adsorption capacity.

and rubidium, so the increase of Dowex G26 resin would adsorb potassium priorly. To decrease the concentration of potassium in this part, the L/S ratio of 1,000 was chosen as the optimal parameter. After recovering cesium through Dowex G26 resin under the conditions of contacting time of 4 min, pH 9, and L/S ratio of 1,000, the adsorption capacities of cesium, rubidium, and potassium are shown in Table 5.

Two-stage desorption could be used to separate cesium and rubidium after adsorbing the cesium and rubidium through Dowex G26 resin. 1% HNO_3 could desorb all the rubidium under the conditions of contacting time of 30 min and L/S ratio of 1,000 and keep cesium in Dowex G26 resin. Cesium then could desorb individually under the situation of 10% HNO_3, contacting time of 24 h and L/S ratio of 1,000. After the desorbing procedure, cesium nitrate solutions could be dried and turned into solid cesium nitrate. In this experiment, the cesium resources could be recovered from waste desalination brine and reused in other industries by adsorbing and desorbing processes.

3.6. Comparison with t-BAMBP/kerosene and t-BAMBP/C_2mimNTf_2 system

After getting the optimal parameters of the Dowex G26 system, it was compared with our previous work [14]. Table 6 demonstrates that cesium could be recovered in a short time and from the weak base solution through the Dowex G26 system and t-BAMBP/kerosene system. t-BAMBP/C_2mimNTf_2 system, however, required a longer reaction time due to its high viscosity, and it also extracted cesium efficiently at a higher pH value. Compared with other systems, the merits of Dowex G26 is that it could adsorb cesium soon, under weak base condition and at room temperature. On the other hand, the pollution of the ion exchange method was less than the solvent extraction method. The waste brine could be treated after recovering cesium, and there was no organic solvent in the whole process. The waste Dowex G26 resin could be turned into carbon through the thermal process and reduce the environmental burden. Besides, the t-BAMBP/kerosene system was volatile, and difficult to recycle the extractant. It was dangerous in the operating procedure because the kerosene was flammable as well. The viscosity of C_2mimNTf_2 was higher than common diluents, making the system need more time to extract and stripping cesium. (The viscosities of common diluents are in Table 7.) The price of C_2mimNTf_2 is much higher than diluents and Dowex G26 resin, too. C_2mimNTf_2 is 415 USD/100 g, and Dowex G26 resin is 43 USD/100 g, separately. Due to these reasons, Dowex G26 resin for cesium adsorption is deemed to have practical application value and could be used in the industry. Nevertheless, the efficiency of Dowex G26 resin is the lowest on account of its lower selectivity of rubidium and potassium. This situation led to Dowex G26 adsorb rubidium and potassium concurrently, and multi-stage adsorption would be needed to recover cesium completely. In a nutshell, the cesium could be recovered through Dowex G26 resin without inputting too much energy and chemicals and creating less pollution than the t-BAMBP/kerosene system and t-BAMBP/C_2mimNTf_2 system. Nonetheless, its disadvantages are that many times of adsorption process to recover all cesium resources from waste desalination brine would be required.

4. Conclusion

This study aims to provide a useful ion exchange system to recover cesium resources from waste desalination brine. The results showed that the saturated adsorption capacity of cesium through Dowex G26 resin was 456.6 mg/g and fit with the Langmuir model with spontaneous reaction. On the other hand, cesium could be adsorbed through Dowex G26 resin in the experiment under the conditions of contacting time of 4 min, pH 9, and L/S ratio of 1,000. The adsorption capacity and efficiency of cesium were 28.31 and 76.3%, respectively. After adsorbing, cesium resources could then be desorbed by nitric acid, and solid cesium nitrate would be obtained by drying the desorbed solutions. Compared with the t-BAMBP/kerosene system and t-BAMBP/C_2mimNTf_2 system, the Dowex G26 system could be a method without pollution, additional energy, and chemicals than the

Table 5
Adsorption capacities of cesium, rubidium, and potassium

Elements	Adsorption capacities (mg/g)
Cs	28.31
Rb	3.68
K	0

Table 7
Dynamic viscosities of C_2mimNTf_2, kerosene, cyclohexane, and xylene (293 K)

Diluents	Viscosity (mPa·s)
C_2mimNTf_2	43.1
Kerosene	1.71
Xylene	0.81
p-diethyl benzene	0.67

Table 6
Dowex G26 resin system compared with t-BAMBP/kerosene and t-BAMBP/C_2mimNTf_2 system [14]

System	Concentration (M)	Contacting time (min)	pH value of aqueous phase	L/S ratio (O/A ratio)	Reaction temperature (K)	Extraction efficiency of cesium (%)
Dowex G26 resin	X	4	9.0	1000	298	76.3
t-BAMBP/kerosene	0.1	3	8.0	0.1	308	99.8
t-BAMBP/C_2mimNTf_2	0.015	15	11.0	1	308	96

t-BAMBP/kerosene system and t-BAMBP/$C_2mimNTf_2$ system. If the problem of adsorption efficiency could be improved in the future, Dowex G26 resin would be applied to other metals and reach the goal of resource circulation.

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