DESALINATION BEHAVIORS FROM SEAWATER USING NATURAL ZEOLITE AND CALCINED CA-FE LAYERED DOUBLE HYDROXIDE FOR CULTIVATION

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ABSTRACT: In recent years, it has been considered to secure water and food using seawater desalination technology, and a new simple desalination material for reducing the high concentration of sodium chloride in seawater is required. In this study, desalting agents were prepared from natural zeolite, obtained from Kagoshima prefecture, Japan, with the addition of calcined Ca-Fe type layered double hydroxide (LDH), to desalinate seawater for cultivation. By treating with the mixture of natural zeolite and calcined Ca-Fe LDH at a mixing ratio of 5: 4, the salinity could be reduced from 3.61% in seawater to 0.97% by cation exchange reaction of natural zeolite and reconstruction reaction of calcined Ca-Fe LDH, and the pH of the obtained solution is 8.4-9.0. The desalination reaction using this mixture was almost the same regardless of temperature. Radish sprouts, bean sprouts, and mung beans could be harvested using seawater treated with a mixture of natural zeolite and calcined Ca-Fe LDH, while it was not possible to harvest using seawater.

Keywords: Natural zeolite, Calcined Ca-Fe LDH, Desalination, Growth test

1. INTRODUCTION

Water is not only an essential material for human beings (70% of our bodies by weight) but also an important resource for human activities, such as agriculture, commerce, industry, domestic use, and so on. In the 21st century, water scarcity is one of the most serious global challenges. Now, over one-third of the world's population lives in water-stressed countries, and, by 2025, this is predicted to rise to nearly two-thirds [1, 2]. The challenges of providing ample and safe water resources are further complicated by population industrialization, contamination growth, of available freshwater resources, and climate change.

For water resources on earth, 70 % of Earth's surface is covered with water, and the total amount of water resources on the earth is about 1.3 billion km³. In water resources, seawater is about 97.5%, freshwater is about 2.5%, and human beings can only use about 0.01% of the total water resources, due to the most freshwater present as a glacier, clouds, vapor, and so on. According to the Food and Agriculture Organization (FAO) of the United Nation (UN), the annual water withdrawal worldwide from natural water bodies is estimated to be around 4250 km³, of which agricultural use is the largest consumer at around 71.7% of the total withdrawal [3]. This portion is even higher in arid and semi-arid countries. Therefore, the supply of agricultural water as a freshwater resource is one of the important issues in the 21st century.

One way of supplying water resources is

seawater desalination. Commercial desalination technologies can be classified into two main categories. Thermal-based technologies, such as multi-stage flash (MSF) and multi-effect distillation (MED), and membrane-based technologies, such as reverse osmosis (RO), together constitute more than 80% of the global desalination capacity currently in operation worldwide [4]. However, these technologies are expensive for agricultural use because they produce high-quality fresh water for domestic or industrial use. Therefore, the development of technology for obtaining agricultural water from high-salinity water, such as seawater, is desired to secure food.

In previous studies, desalination behaviors of natural zeolite and synthetic zeolite in seawater were examined [4-7], and seawater was treated with calcined Mg-Al layered double hydroxide (LDH) and natural zeolite in two steps to obtain a solution that can be used for germination of radish sprouts from seawater [8-12]. Furthermore, we have succeeded in cultivating radish sprouts using seawater treated with a mixture of mordenite-type natural zeolite and calcined Ca-Fe LDH (calcination at 500 °C) (mixing ratio of zeolite to LDH =5: 4), which are cheap materials [13-16]. There is a possibility to develop a new desalination material that reduces the high salinity in seawater, causing salt damage to a level where crops can grow, using natural zeolite with calcined LDH. However, little information can be available on the desalination behaviors of natural zeolite with calcined Ca-Fe LDH.

In this study, the desalination behaviors of natural zeolite, calcined Ca-Fe LDH, and the mixture of these in seawater were examined to produce the solution for plant growth from seawater.

2. RESEARCH SIGNIFICANCE

The research significance of this study would be a great contribution to solve the global problems, such as water and food shortage, carbon dioxide reduction, and natural restoration, by desalination using two simple clay minerals, natural zeolite and layered double hydroxide. While each desalination behaviors of natural zeolite and layered double hydroxide are not enough to desalinate seawater for cultivation, the combination reaction of natural zeolite and layered double hydroxide can desalinate seawater for cultivation. These results in this paper indicate new knowledge from both scientific and practical points of view.

3. EXPERIMENTAL

3.1 Samples

Mordenite-type natural zeolite obtained from Koriyama mine, Kagoshima Prefecture, Japan was used as a natural zeolite sample. The exchangeable cations (ECs) and cation exchange capacity (CEC) of natural zeolite were shown in Table 1. The sample was ground and sieved under 250 μ m, and dried to be used for the experiment.

Table 1 ECs and CEC of natural zeolite sample

EC (mmol/g)				CEC
Na^+	\mathbf{K}^+	Mg^{2+}	Ca^{2+}	(mmol/g)
0.52	0.25	0.01	0.18	1.95

The calcined Ca-Fe LDH was prepared as follows. A mixed solution of Ca^{2+} and Fe^{3+} (Ca/Fe = 2) (200 mL) was prepared from 0.2 M CaCl₂ solution and 0.1 M FeCl₃ solution, and dropwise to 0.3 M NaCl solution (100 mL) at 4 mL/min. The stirring was carried out for 1 h while bubbling nitrogen gas, and the pH of the NaCl solution was maintained at 12.5 by dropping the NaOH solution during the stirring. After stirring, the mixture was filtered, dried, and calcined in an electric furnace at 500°C for 1 h to obtain calcined Ca-Fe LDH.

Seawater used in this study was collected from the surface layer of Imari Bay, Saga prefecture, Japan. The chemical composition, salinity, and pH of seawater were shown in Table 2.

3.2 Desalination Treatment

The desalting abilities of natural zeolite,

calcined Ca-Fe LDH, and the mixture of natural zeolite and calcined Ca-Fe LDH were evaluated as follows.

Natural zeolite (15 g), calcined Ca-Fe LDH (12 g) or the mixture of natural zeolite (15 g) and calcined Ca-Fe LDH (12 g) was added to seawater (40 mL) heated at 20, 50 and 80 °C, and stirred. After stirring for 0 - 60 min, the solution was filtered, the pH of the filtrate was measured by pH meter (Horiba, F-72), salinity was measured by salinity meter (LAQUA, ES-71), total dissolved solids (TDS) was measured by electrical conductivity meter (LAQUA, F72), the concentrations of SO42-, Cl-, Na+, K+, Mg2+ and Ca2+ in the filtrate were measured with an ion chromatograph (Tosho, IC-2010) and those of Fe³⁺, Si4+ and Al3+ were determined using an atomic absorption spectrophotometer (Perkin Elmer, AAnalyst 200). The residue was dried, and the mineralogical structure of the natural zeolite, Ca-Fe LDH, and the residue obtained at each temperature were confirmed by a powder X-ray diffraction apparatus (Rigaku, MiniFlex 600).

The amount of solute adsorbed on the sample at any time, q_t (mg/g), was determined by Eq. (1).

$$q_t = \frac{(C_0 - C_t) \cdot V}{w} \tag{1}$$

with C_0 (g/L) is the initial TDS, C_t (g/L) is the TDS at any time, V is the volume of the solution (L) and w is the mass of the sample (g).

3.3 Growth Test

Radish sprouts, bean sprouts, and mung beans were used as test plants for 10 days growth test. Tap water, seawater or seawater treated with the mixtures of natural zeolite and calcined Ca-Fe LDH was given by a spray every day, and germinations of test plants were observed.

4. RESULTS AND DISCUSSION

Figure 1 shows the salinity and pH of seawater treated with natural zeolite during the treatment at 20, 50, and 80 °C. Regardless of temperatures, the salinity rapidly decreased to be almost constant at about 2.4 %. With increasing the temperature, the time for salinity to decrease to about 2.4 % was faster. At 20 °C and 50 °C, the pH of seawater (8.0) decreased to about 7.5, while the pH of seawater decreased to 5.8 at 80 °C. It is noted that TDS of seawater decreased from 32.4 g/L to about 21.5 g/L.



Fig.1 Salinity and pH of seawater treated with the zeolite during the treatment at 20, 50, and 80 °C

Figure 2 shows the salinity and pH of seawater treated with calcined Ca-Fe LDH during the treatment at 20, 50, and 80 °C. Regardless of temperatures, the behaviors of salinity decrease are almost the same, and the salinity of seawater (3.61%) rapidly decreased to be almost constant to about 3.3 %. At 20 °C, the pH of the seawater increased to about 10.5, while the pH of seawater increased to about 11 at 50 °C and 80 °C. It is noted that TDS of seawater decreased from 32.4 g/L to about 29.5 g/L.



Fig.2 Salinity and pH of seawater treated with calcined Ca-Fe LDH during the treatment at 20, 50, and 80 $^{\circ}$ C

From these results for natural zeolite and calcined Ca-Fe LDH, with increasing the temperature, the change of solution pH was larger and the desalination reaction was faster.

Figure 3 shows the salinity and pH of seawater treated with the mixture of natural zeolite and calcined Ca-Fe LDH during the treatment at 20, 50, and 80 °C. Regardless of temperatures, the behaviors of salinity decrease are almost the same, and the salinity of seawater gradually decreased to 0.95 - 0.99 %, while the pH of seawater was almost constant (8 - 9). It is noted that TDS of seawater decreased from 32.4 g/L to about 8.7 g/L.



Fig.3 Salinity and pH of seawater treated with the mixture of natural zeolite and calcined Ca-Fe LDH during the treatment at 20, 50, and 80 °C

Table 2 shows the salinities of seawater and the seawater treated with natural zeolite, calcined Ca-Fe LDH, and the mixture of natural zeolite and calcined Ca-Fe LDH at 20, 50, and 80 °C after 60 min treatment. Natural zeolite can reduce the salinity due to the cation exchange reaction, but the reaction causes a pH decrease to increase H⁺, which is a competitor for the cation exchange reaction, therefore the decrease of salinity stopped to about 2.4% of salinity. Calcined Ca-Fe LDH can reduce the salinity due to the reconstruction reaction, but the reaction causes pH increase to increase OH⁻, which is a competitor for removal anions by reconstruction reaction of calcined Ca-Fe LDH, therefore the decrease of salinity stopped to be about 3.3 % of salinity. The mixture of natural zeolite and calcined Ca-Fe LDH can reduce the salinity due to the reactions of both cation exchange of natural zeolite and reconstruction of calcined Ca-Fe LDH, these reactions simultaneously supply H⁺ from natural zeolite and OH⁻ from calcined Ca-Fe LDH to be neutralized, and smoothly occurs to decrease the salinity of seawater.

Table 2 Salinities of seawater and the seawater treated with natural zeolite (NZ), calcined Ca-Fe LDH (CFL), and the mixture of natural zeolite and calcined Ca-Fe LDH (Mixture) at 20, 50, and 80 °C after 60 min treatment

Salinity (%)	Seawater	20°C	50°C	80°C
NZ		2.35	2.44	2.42
CFL	3.61	3.29	3.26	3.28
Mixture		0.97	0.99	0.95

Table 3 shows the chemical compositions, salinities and pHs of seawater and the seawater treated with the mixture of natural zeolite and calcined Ca-Fe LDH at 20, 50, and 80 °C after 60 min treatment. Seawater used in this study indicates that chemical composition, salinity and pH are typical values of seawater [17, 18]. After treatment

of seawater with the mixture of natural zeolite and calcined Ca-Fe LDH at 20, 50, and 80 °C, regardless of temperatures, the salinity decreased to lower than 1 % (0.95 - 0.99 %) and pH is 8.0 - 9.1, which can be used for plant cultivation. The contents of $SO_4^{2^-}$, Cl⁻, Na⁺, K⁺ and Mg²⁺ decreased, while that of Ca²⁺ increased.

Figure 4 shows the concentrations of (a) SO_4^{2-} . (b) Cl^{-} , (c) Na^{+} , (d) K^{+} , (e) Mg^{2+} , (f) Ca^{2+} , (g) Fe^{3+} , (h) Si^{4+} and (i) Al^{3+} during the treatment with the mixture of natural zeolite and calcined Ca-Fe LDH at 20 °C, 50 °C and 80 °C. Regardless of temperatures, concentrations of Cl⁻, SO₄²⁻, Na⁺, K⁺, and Mg²⁺ decreased and those of Ca²⁺, Fe^{3+,} and Si⁴⁺ increased with the addition of the mixture. It was considered that the decreases of Cl⁻ and SO₄²⁻ were caused by calcined Ca-Fe LDH, those of Na⁺ and K⁺ and increase of Ca²⁺ by natural zeolite, and the increase of Fe^{3+} and Si^{4+} by the dissolution from calcined Ca-Fe LDH and natural zeolite, respectively. It is noted that the Al concentration is almost zero without the dissolution from the mixture.

Table 3 Chemical compositions, salinities and pHs of seawater and the seawater treated with the mixture of natural zeolite and calcined Ca-Fe LDH at 20, 50, and 80 °C after 60 min treatment

	Seawater	20°C	50°C	80°C
Content				
(mmol/L)				
SO_4^{2-}	42	5	4	7
Cl	904	222	219	221
Na^+	594	67	70	72
\mathbf{K}^+	14	3	3	3
Mg^{2+}	75	2	0	0
Ca^{2+}	17	656	650	674
Salinity (%)	3.61	0.97	0.99	0.95
pН	8.0	8.44	9.08	8.88

Figure 5 shows the XRD patterns of (a) Ca-Fe LDH, (b) calcined Ca-Fe LDH, (c) the mixture of natural zeolite and calcined Ca-Fe LDH, and the residue of the mixture of natural zeolite and calcined Ca-Fe LDH after 60 min treatment at (d) 20 °C, (e) 50 °C and (f) 80 °C. Ca-Fe LDH phase was transformed into an amorphous and calcite phase (Fig. 5 (a), (b)). After desalination treatment, all residues have natural zeolite mineral phases, mordenite, and calcite, and Ca-Fe LDH phase which were formed from calinced Ca-Fe LDH by reconstruction reaction (Fig. 5 (c)-(f)). These results indicate that the desalination of seawater by the mixture of natural zeolite and calcined Ca-Fe

LDH was caused by the cation exchange of zeolite and the reconstruction of calcined Ca-Fe LDH.



Fig.5 XRD patterns of (a) Ca-Fe LDH, (b) calcined Ca-Fe LDH, (c) the mixture of natural zeolite and calcined Ca-Fe LDH, and the residue of the mixture of natural zeolite and calcined Ca-Fe LDH after 60 min treatment at (d) 20 °C, (e) 50 °C and (f) 80 °C.

Figure 6 shows the adsorbed amounts for (a) NZ, (b) CFL and (c) mixture during the stirring at 20, 50 and 80 °C. With increasing the reaction time, the adsorbed amounts for NZ and CFL increase within 15 minutes, and then almost constant, while that for mixture gradually increases above 60 minutes.

The kinetics results obtained from batch experiments were analyzed using different kinetics models such as pseudo-first-order-kinetics models [19-21] and pseudo-second-order-kinetics models [22-24]. Pseudo-first-order kinetic model is given by Eq. (2), while it linear form is presented in Eq. (3).

$$q_t = q_e \left(1 - e^{-k_1 \cdot t} \right)$$
(2)

$$\ln(q_e - q_t) = \ln q_e - k_1 \cdot t \tag{3}$$

with q_t , q_e and k_1 are the amount of salt ion adsorbed on sample at any time t (mg/g), and the rate constant of adsorption kinetics (1/s), respectively.

Pseudo-second-order kinetic model is given by Eq. (4), while it linear form is presented in Eq. (5).

$$q_{t} = \frac{q_{e}^{2}k_{2}t}{1 + q_{e}k_{2}t}$$
(4)

$$\frac{t}{q_t} = \frac{1}{q_e^2 k_2} + \frac{t}{q_e}$$
(5)

with k_2 is the rate constant of pseudo-second-order kinetics (g/(mg \cdot s)).

Table 4 shows the obtained values of adsorption kinetics parameters for salt ion onto sample according to pseudo-first-order model and pseudosecond-order model. It can be seen that the correlation coefficients (R^2) of pseudo-secondorder model is higher than pseudo-first-order, indicating that pseudo-second-order model better describe the adsorption kinetics of salt ions onto sample than pseudo-first-order model. Regardless of temperature, q_e for NZ were 28-34 mg/g and that for CFL were 10-11 mg/g, while that for mixture were 58-60 mg/g, which means that the adsorbed amount of mixture is 2 times and 6 times higher than NZ and CFL, respectively. With increasing the temperature, the adsorption rate constant for NZ increase, and that for CFL decrease, while that for mixture is almost constant regardless of temperature. The adsorption rate constant for mixture is lower than those for NZ and CFL. It is noted that the adsorbed amount of Indonesian natural zeolite, clinoptilolite, for salt ions in seawater is 48.57 mg/g, which is the best desalination ability among many papers for desalination adsorbent. and decreases with increasing the temperature of seawater [25, 26]. These results indicate that the mixture of NZ and CFL are superior desalination adsorbent to others.

Figure 7 shows the observation of radish sprouts, bean sprouts, and mung beans after 10 days' growth using (a) tap water, (b) seawater, and (c) the seawater treated with the mixture of natural zeolite and calcined Ca-Fe LDH. The germination of radish sprouts, bean sprouts, and mung beans could be confirmed in tap water and the seawater treated with a mixture of natural zeolite and calcined Ca-Fe LDH (Fig. 6 (a), (c)), while these plants cannot be germinated in seawater (Fig. 6 (b)).



Fig.6 The adsorbed amounts for (a) NZ, (b) CFL and (c) mixture during stirring at 20, 50 and 80 $^{\circ}$ C.

Table 4 Fitting kinetics parameters of adsorption of salt ions onto samples according to pseudo-first order model and pseudo-second-order model

Comm10	Temperature (°C)	Pseudo-first-order model			Pseudo-second-order model		
Sample		$q_e ({ m mg/g})$	k_{l} (1/s)	R^2	$q_e (\mathrm{mg/g})$	$k_2 \times 10^3 (g/(mg \cdot s))$	R^2
NZ	20	13.0	0.026	0.473	33.8	5.0	0.983
	50	2.6	0.030	0.286	28.7	30.9	0.998
	80	4.2	0.030	0.939	29.2	38.3	0.998
CFL	20	2.2	0.023	0.228	10.3	25.4	0.990
	50	4.0	0.034	0.995	11.0	28.1	0.998
	80	5.1	0.032	0.993	10.6	19.2	0.995
Mixture	20	51.9	0.015	0.927	58.1	0.5	0.958
	50	53.8	0.014	0.995	59.2	0.4	0.971
	80	52.6	0.015	0.923	58.5	0.5	0.994



Radish sprouts



Fig.7 Observation of radish sprouts, bean sprouts, and mung beans after 10 days of growth using (a) tap water, (b) seawater, and (c) the seawater treated with the mixture of natural zeolite and calcined Ca-Fe LDH.

5. CONCLUSION

For the development of desalination material to obtain agriculturally usable freshwater from seawater for a stable supply of water resources, desalination behaviors of the mixture of natural zeolite and calcined Ca-Fe LDH in seawater were examined for producing cultivation water from seawater.

Using natural zeolite, the salinity decreased from 3.5 % to about 2.5 %, and desalination speed was faster and pH was lower with increasing the temperature. Using calcined Ca-Fe LDH, salinity was a little low (about 3.3%), and pH rises to about 10.5 - 11.0 from 8.0. Regardless of temperatures, when seawater was treated with the mixture of natural zeolite and calcined Ca-Fe LDH, the salinity decreased to 0.95 - 0.99% below the available salinity for agriculture (about 1.0%) and pH is neutral (8 - 9). When using the seawater treated with a mixture of natural zeolite and calcined Ca-Fe LDH, the germination of radish sprouts, bean sprouts, and mung beans was confirmed.

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