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A study of reverse osmosis-desalinated water quality adjustment with artificially treated dolomite

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ABSTRACT

Here, the influences of artificially treated dolomite on the quality of osmosis-desalinated water are explored. The scanning electron microscopy and X-ray diffraction analysis patterns of dolomite were used to measure the physical and chemical characteristics of the reverse osmosis-desalinated water, and its water quality effects were assessed by measuring pH, hardness, alkalinity, Mg^{2+}, and Ca^{2+} contents. The optimal calcinations condition of dolomite is at 750°C for 1 h. At a retention time of 3 min, Ca^{2+} and Mg^{2+} ions in the effluent reached 18.15 and 2.26 mg/L, respectively, which are 7 and 1.5 times the concentrations observed in raw dolomite. Hardness of the effluent reached 56.59 mg/L as $CaCO_3$ and alkalinity concentrations of the effluent reached 68.11 mg/L as $CaCO_3$, which are about 3.5 and 3.7 times the concentrations observed in raw dolomite, respectively. The resultant pH reached 11.12. When the dolomite was acidized at 50°C for 3 h, the effluent pH reduced down to 10.59, and the Mg^{2+} content increased to 20.42 mg/L. Proper aeration can achieve the requirement of pH in Israel criteria.

Keywords: Desalinated water adjustment; Dolomite; Calcinations; Acidify; Water quality improvement

1. Introduction

Desalinated water by reverse osmosis is a major component of the total freshwater supply currently in use. However, removal rates for various ions differ greatly: reverse osmosis tends to remove divalent ions more (e.g., Ca^{2+} and Mg^{2+}) than monovalent ions (e.g., Na^+ and Cl^-). As a result, desalinated water contains few Ca^{2+} and Mg^{2+} ions. Additionally, desalinated water typically has reduced stability, which is often measured by three indices: the buffering capacity, the propensity of $CaCO_3$ to precipitate, and the soluble Ca^{2+} ions [1]. The Langelier saturation index and calcium carbonate precipitation potential (CCPP) are commonly used as $CaCO_3$ stability indices. Mg^{2+} ions are also considered, because their effects on both human health [2] and agriculture are widely recognized. Because desalinated water is considered "over clean" except for certain elements such as boron, it is widely accepted that remineralizing is needed to stabilize water, minimize corrosion and human health disorders, and to reduce damage to agricultural crops [3,4].

There are many criteria for drinking water, but only few for desalinated water. A quality criterion for desalinated water was appointed in 2005 by Israeli Ministry of Health and accepted by the government later. The criteria are as follows [1,3]:

- Alkalinity > 80 mg/L as $CaCO_3$,
- $80 < [Ca^{2+}] < 120$ mg/L as $CaCO_3$,
- $3 < \text{CCPP} < 10$ mg/L as $CaCO_3$, and
- pH < 8.5.

The guidelines for drinking water quality proposed by the WHO are also applicable to desalinated water supply systems [5]. Several studies have also proposed recommendations for

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water quality parameters of desalinated water or brackish water [3,4].

Three types of processes are commonly used for remineralizing of desalinated water: (1) dosing of chemicals such as $CaCl_2$, $MgCl_2$ and $MgSO_4$ directly into water; (2) mixing desalinated water with other high-mineral-containing water sources; and (3) dissolving ores into desalinated water to provide increased Ca^{2+}, Mg^{2+}, and alkalinity. This last option is more practiced because ores are naturally occurring and are cost-effective. Calcite and limestone, in particular, are readily available and therefore used to remineralize desalinized water. Although dolomite has a slower dissolving rate compared with calcite, it can supply both Ca^{2+} and Mg^{2+} [6]. Birnhack et al. [6] tested three combined dolomite–calcite dissolution alternatives using acidified water and found that the most promising method is to dissolve dolomite first and then pass the effluent through a calcite filter. This yields water with the following properties:

- $[Mg^{2+}]$ = 12.4 mg/L,
- Alkalinity = 75 mg/L as $CaCO_3$,
- $[Ca^{2+}]$ = 120 mg/L as $CaCO_3$,
- pH = 8.17, and
- Total hardness = 170 mg/L as $CaCO_3$.

Raw dolomite requires pretreatment because it is hard to dissolve. Research has shown that the final solid decomposition products of dolomite are calcium and magnesium oxides, but there is a large disparity with respect to the decomposition mechanisms of these compounds [7]. One option is the one-stage process which can be conducted with nitrogen, ambient air, or CO_2 with a pressure below 150 mm Hg [7,8]. The thermal decomposition equation is as follows:

$$CaMg\left(CO_3\right)_2 \rightleftharpoons CaO + MgO + 2CO_2 \quad (1)$$

Another two-stage process is described as follows for a synthetic-ordered dolomite:

$$CaMg\left(CO_3\right)_2 \rightleftharpoons CaCO_3 + MgO + CO_2 \quad (2)$$

$$CaCO_3 \rightleftharpoons CaO + CO_2 \quad (3)$$

In the first stage, carbon dioxide is released from carbonate ions associated with the magnesium, and calcite and magnesium oxide are formed accordingly. Then, in the second stage, at a higher temperature, calcite is decomposed to carbon dioxide and calcium oxide [9]. However, the temperatures for the two stages are not unified. A more detailed mechanism is also proposed for the first stage such that dolomite is first decomposed into carbonates and then immediately dissociated to magnesium oxide [7,10].

$$CaMg\left(CO_3\right)_2 \rightleftharpoons CaCO_3 + MgCO_3 \quad (4)$$

$$MgCO_3 \rightleftharpoons MgO + CO_2 \quad (5)$$

Treated dolomite (half-calcined dolomite) has been reported to be more efficient in water remineralization for both real and demineralized water compared with typical dolomite [11,12]. However, to date, little is known about the ideal methods to use treated dolomite to remineralize desalinated water. Here, we test how treating dolomite with heat and acid influences its dissolution behavior and creates impact on water quality. We also used a commercial ore sample for comparison.

2. Materials and methods

The dolomite sample (identified here as DS) was purchased from Shijiazhuang (Hebei province, China). The commercially applied ore sample (identified here as DG) used here was from Germany. DS was calcined and acidified to attain better dissolution behavior. DG dolomite was used for comparison. The water quality parameters of the reverse osmosis-desalinated water used here are listed in Table 1.

The DS ore was calcined with a muffle furnace. The calcined DS ore was then acidified with a tube furnace with a different flow of vapor and carbon dioxide. After acidization treatment, carbon dioxide remained on for 1 h at 100°C to stabilize the crystallized product. The dissolution process was conducted using a conical flask and thermostatic oscillator. The sample was then placed in a conical flask with a water/ore ratio of about 5:4 (250 mL of desalinated water and 200 mL of sample). Immediately, the conical flask was placed in a thermostatic oscillator. The retention time for dissolving was controlled at levels of 1, 2, 3, 5, and 10 min with a stroke speed of about 120 rpm. The pH, Ca^{2+}, and Mg^{2+} contents, hardness, and alkalinity of the effluent were measured and analyzed after ore dissolved, and each experiment was replicated three times.

Thermogravimetry-differential thermal analysis/differential scanning calorimetry (TG-DTA/DSC) curves were obtained with continuous heating from room temperature to 1,000°C at a warming rate of 10°C/min. X-ray diffraction analysis (XRD) analysis was performed with a X pert Powder Diffractometer with CuKα radiation of λ = 0.154 µm, 40 kV power, and 20 mA current. The Supra 55 microscope was used as the scanning electron microscopy (SEM).

3. Results

3.1. Characterizations

Dolomite is typical sedimentary rock associated with calcite. The XRD results show that the main phase in the DS

Table 1
Water quality parameters of RO-desalinated water

Parameters	Values
pH	6.9
TDS (mg/L)	222
Ca^{2+} (mg/L)	0.22
Mg^{2+} (mg/L)	0.38
Hardness (mg/L as $CaCO_3$)	4.2
Alkalinity (mg/L as $CaCO_3$)	4.45
Turbidity (NTU)	0.2

TDS — Total dissolved solids.

ore was $CaMg(CO_3)_2$ and with an accompanying peak of $Ca(Mg, Fe)(CO_3)_2$ [13]. DG ore is a half-burnt ore containing not only $CaMg(CO_3)_2$ but also $CaCO_3$ and an MgO phase. The element analysis is in accordance with the study by Chen et al. [13]. The main undesirable impurities in dolomite are silica, iron, and alumina. The DS ore has a higher iron oxide content of 6.12%, and the DG ore has magnesium content higher than DS (24.58% vs. 21.42% magnesium oxide). Both of them have a similar amount of calcium oxide, 31.02% and 30.58%, respectively. Detailed composition analysis of the sample is presented by Chen et al. [13].

TG-DTA/DSC curves of selected DS are presented in Fig. 1. The curve only shows one endothermic peak here, at about 810°C. The performance of mass loss shows a V-shaped curve that differs from prior research [14,15]. The decomposition temperature and decomposition rate of dolomite may vary depending on the proportion of $CaCO_3$ to $MgCO_3$ in the sample and are therefore difficult to predict [7]. The decomposition temperatures of calcium and magnesium may overlap due to various impacts (e.g., due to the texture, impurities, size, etc.) and so a typical S-shaped curves may not form. According to Fig. 1, when the temperature is below 700°C, the rate of mass loss is slow, only up to about 5%. This first mass loss may be attributed to volatilization of moisture content and other easily decomposed components. The slope of the curve becomes larger as the temperature increases. XRD analysis presented in Fig. 2 illustrates similar results. When heated at 700°C for less than 3 h, no MgO phase is observed. At this stage, dolomite may not start to decompose, or the decomposed phases are too weak to be observed. When heated at 750°C for 1 h, MgO and $CaCO_3$ are observed and the dolomite phase hardly appears. When heated for 3 h, a CaO phase appears, which means the selected dolomite decomposed at about 750°C, and the intensity of diffraction peak of $CaCO_3$ and MgO enhanced with increasing calcining time. $CaCO_3$ alone usually decomposes at a higher temperature than MgO, but when in the form of $CaMg(CO_3)_2$, the decomposition of $MgCO_3$ and other impurities may affect the stability of $CaCO_3$, leading to a lower decomposition temperature that is close to $MgCO_3$.

The mass loss at the end of the plateau is around 46%, close to the theoretical weight loss of 47.8% and the TG-DTA/DSC result of 48.2% (Figs. 1(a) and (b)). At or below 700°C, dolomite is not fully decomposed because the decomposition rate is less than 20% even at 4 h of heating.

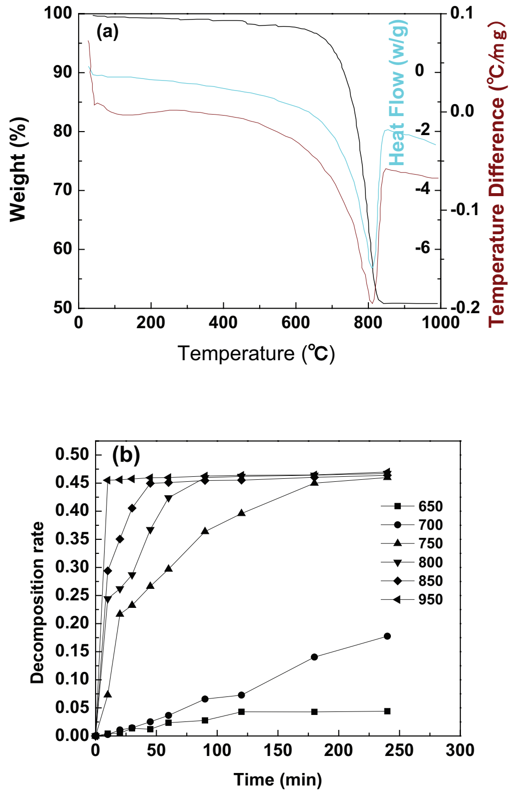

Fig. 1. (a) TG-DTA/DSC curve and (b) decomposing rate of DS.

3.2. Adjustment effects of RO desalinated water

3.2.1. Hardness and Ca^{2+} and Mg^{2+} contents of calcined dolomite

Several kinds of ores including DG ore, DS ore (raw dolomite), and calcined DS ore were tested here. Desalinated water is not acidified by H_2SO_4 or CO_2. As shown in Fig. 3, hardness contents in effluents of dolomites have a large increase at the beginning of the retention time and remain stable thereafter. This may be caused by the reduction of mass transfer force from the dissolving process with increasing retention time, and the decreasing contact area due to the covering of carbon dioxide on the surface of reaction interface. The DG ore, noted as half-burnt dolomite, shows a higher mineral content than the raw DS ore. Artificially processed dolomite has an increased porosity and therefore larger contact area. And also, calcined dolomite has a chemical composition different from dolomite. XRD results show that the main composition is $CaCO_3$. The solubility product

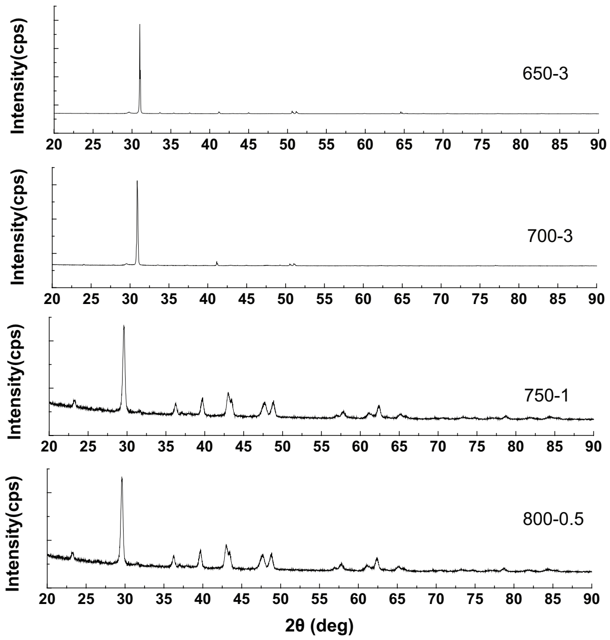

Fig. 2. XRD analysis of calcined dolomite.

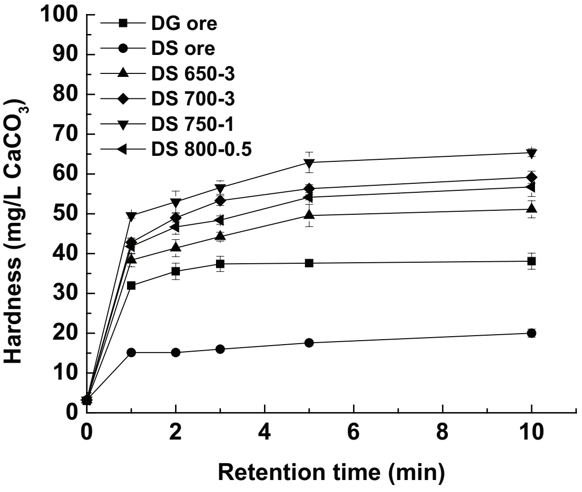

Fig. 3. Hardness in the effluents of different calcined dolomite.

constant (K_{sp}) of $CaCO_3$ is about 0.3–1.2 × 10^{-9} higher than the K_{sp} of dolomite which is 0.8–2.9 × 10^{-17} [16,17]. It was reported that the porous $MgO \bullet CaCO_3$ mixture reacts about three times faster than marble with carbon dioxide [12,18]. And, higher equilibrium concentration values (1.2 mmol/L) and dissolution coefficient (0.009 min^{-1}) for Ca^{2+} and Mg^{2+} ions were obtained with calcined dolomite than dolomite [12]. Thus, the hardness contents of calcined dolomite effluents are much higher than that of raw DS. Dolomite heated at 750°C for 1 h yields the highest effluent hardness content, followed by calcined dolomites at 700°C for 0.5 h, 800°C for 0.5 h, and 650°C for 3 h. Porous oxides are formed over the course of the dolomite's decomposition, and after calcinations, the resultant oxides have larger surface areas, lower molar volumes, and greater porosities than the raw ore. SED results at different temperatures across time shown in Fig. 4 again suggest that the dolomite calcined at 750°C for 1 h has a relatively clearer crystal grain boundary and a more complete crystal structure compared with dolomite heated at 650°C and 700°C for 3 h. The grain growth of the surface crystal is small when heated at 800°C for 0.5 h. When exposed to desalinated water, the larger solid–liquid interface and formation of more easily dissolved oxides make the mineral elements dissolve more easily. The hardness content of calcined dolomite heated at 750°C for 1 h yields a concentration of 56.59 mg/L as $CaCO_3$ with a retention time of 3 min, which is about 3.5 times that of raw DS and much higher than that of raw dolomite with a retention time of 10 min.

Ca^{2+} and Mg^{2+} content with retention time is shown in Fig. 5. Among all dolomites, effluent of calcined dolomite contains higher Ca^{2+} and Mg^{2+} content than the raw DS ore. Ca^{2+} contents in the effluent rank is as follows: calcined dolomite (750°C, 1 h) > calcined dolomite (700°C, 3 h) > calcined dolomite (800°C, 0.5 h) > calcined dolomite (650°C, 3 h) > the DG ore > the DS ore. Mg^{2+} contents rank is as follows: the DG ore > calcined dolomite (750°C, 1 h) > calcined dolomite (800°C, 0.5 h) > calcined dolomite (700°C, 3 h) > calcined dolomite (650°C, 3 h) > the DS ore. Ca^{2+} and Mg^{2+} content of calcined dolomite at 750°C for 1 h reaches 18.15 and 2.26 mg/L at retention time of 3 min, which is about 7 and 1.5 times of that of the DS ore, respectively.

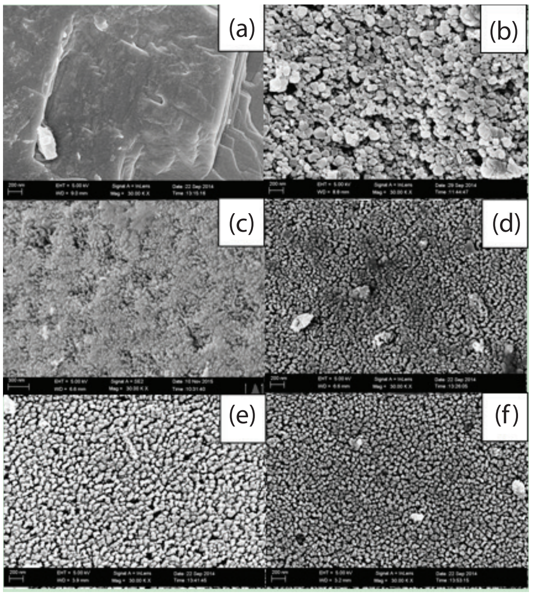

Fig. 4. SEM patterns of dolomite: (a) raw dolomite, (b) DG dolomite, and calcined DS of (c) 650°C 3 h, (d) 700°C 3 h, (e) 750°C 1 h, and (f) 800°C 0.5 h).

Investigations of tap water in China show that Ca^{2+} contents range from 10 to 50 mg/L, mostly between 20 and 30 mg/L, and Mg^{2+} contents are from 2 to 20 mg/L, mostly between 5 to 10 mg/L [13,15]. Although calcinations improve mineral contents in effluent, there is still a gap of hardness and Mg^{2+} contents comparing with tap water. Ca^{2+} concentrations in the effluent are similar to that of tap water, but still do not meet the quality standards in Israel.

3.2.2. pH values and alkalinity contents of calcined dolomite

As demonstrated in Fig. 6, both pH and alkalinity increase with calcination. Alkalinity in drinking water is typically defined as the proton-accepting capacity of the water with respect to H_2CO_3 as reference species.

$$\text{Alkalinity} = 2\left[CO_3^{\,2-}\right] + \left[HCO_3^{\,-}\right] + \left[OH^-\right] - \left[H^+\right] \tag{6}$$

Buffering capacity is defined as the ability of water to withstand the changes of pH caused by a strong base or a strong acid dosage. For a given pH, a higher total inorganic carbon (TIC) concentration results in a higher buffering capacity. When the pH is close to the pK of the system (in the carbontate system at pH of 6.3 and 10.3), the liquid attained a higher buffering capacity for a given TIC (or alkalinity) [19]. pH values of calcined dolomite are around or above 10, which is higher than the DS and the quality criteria of 8.5 in Israel. Alkalinity reached 68.11 mg/L and can be around 80 mg/L as $CaCO_3$ when the dolomite is heated at 750°C for 1 h with a retention time of 3 and 5–10 min. In our experiment, the pH is too high and CCPP of the effluent water is too positive referring to Rossum and Merrill's [20] model calculation. CCPP was calculated according to Eqs. (7)–(13), and the details are presented in the study by Rossum and Merrill [20]. When the water pH is 11.12,

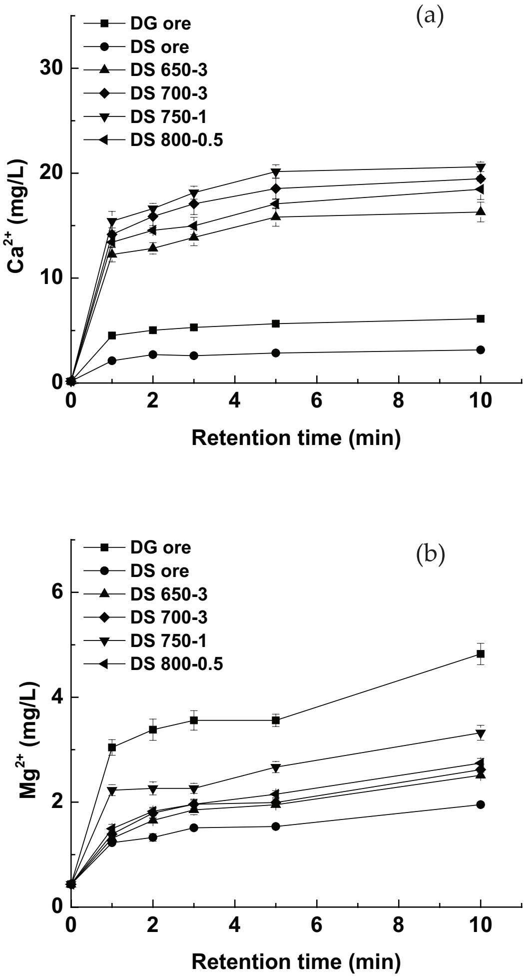

Fig. 5. (a) Ca^{2+} and (b) Mg^{2+} content in the effluents of different calcined dolomite.

alkalinity is 68.11 mg/L as $CaCO_3$, Ca^{2+} is 18.15 mg/L, total dissolved solids is 250 mg/L, and temperature is 25°C, the calculated CCPP is 115.8 mg/L as $CaCO_3$. After 1 h of aeration at 20 L/min, pH can be adjusted to 7.99.

$$\mathrm{Acy}_i = \frac{\mathrm{Alk}_i + [\mathrm{H}^+] - \dfrac{K_w^{'}}{[\mathrm{H}^+]}}{\dfrac{2K_2^{'} + [\mathrm{H}^+]}{[\mathrm{H}^+]}} \times \left(\frac{2[\mathrm{H}^+] + K_1^{'}}{K_1^{'}}\right) + [\mathrm{H}^+] - \frac{K_w^{'}}{[\mathrm{H}^+]} \quad (7)$$

$$2[\mathrm{Ca}^{2+}]_i - \mathrm{Alk}_i = \frac{2K_s^{'} r_{\mathrm{eq}} p_{\mathrm{eq}}}{t_{\mathrm{eq}}(\mathrm{Acy}_i - s_{\mathrm{eq}})} - \frac{t_{\mathrm{eq}}(\mathrm{Acy}_i - s_{\mathrm{eq}})}{p_{\mathrm{eq}}} + s_{\mathrm{eq}} \quad (8)$$

$$t_{\mathrm{eq}} = \frac{[\mathrm{H}^+]_{\mathrm{eq}} + 2K_2^{'}}{[\mathrm{H}^+]_{\mathrm{eq}}} \quad (9)$$

$$p_{\mathrm{eq}} = \frac{2[\mathrm{H}^+]_{\mathrm{eq}} + K_1^{'}}{K_1^{'}} \quad (10)$$

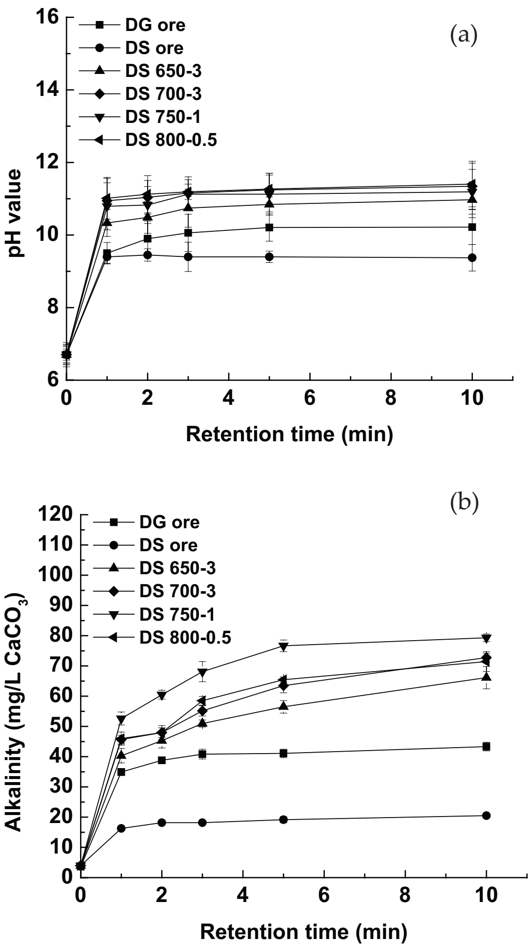

Fig. 6. (a) pH values and (b) alkalinity in the effluents of different calcined dolomite.

$$s_{\mathrm{eq}} = [\mathrm{H}^+]_{\mathrm{eq}} - \frac{K_w^{'}}{[\mathrm{H}^+]_{\mathrm{eq}}} \quad (11)$$

$$\mathrm{Alk}_{\mathrm{eq}} = \frac{t_{\mathrm{eq}}}{p_{\mathrm{eq}}}(\mathrm{Acy}_i - s_{\mathrm{eq}}) - s_{\mathrm{eq}} \quad (12)$$

$$\mathrm{CCPP} = 50000(\mathrm{Alk}_i - \mathrm{Alk}_{\mathrm{eq}}) \quad (13)$$

3.2.3. Adjustment effects of treated dolomite

Calcined dolomite was then acidified and the adjustment effects are shown in Figs. 7 and 8.

As with calcined dolomite, treated dolomite reached higher Ca^{2+} and Mg^{2+} concentrations than the DS. When compared with calcined dolomite, the treated dolomite had a higher Mg^{2+} concentration, similar alkalinity, and a lower Ca^{2+} concentration and pH value. The Ca^{2+} and Mg^{2+} contents of the effluent of treated dolomite (carbonated and acidified at 50°C for 3 h) are 1.49 and 20.42 mg/L, respectively. The pH

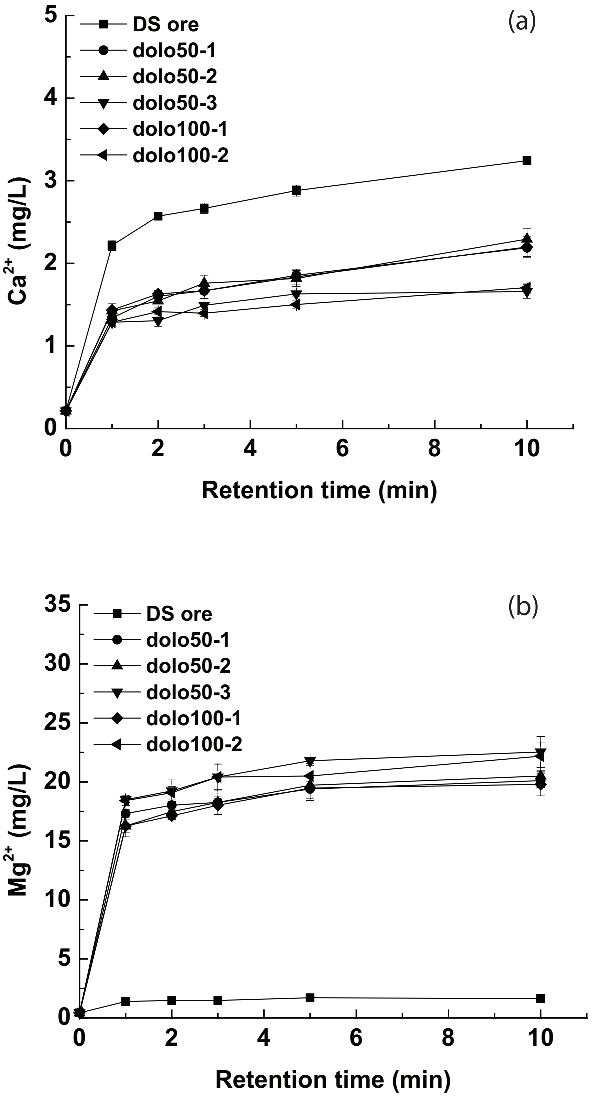

Fig. 7. (a) Ca^{2+} and (b) Mg^{2+} content in the effluents of different modified dolomite.

and alkalinity are 10.59 and 67.02 mg/L as $CaCO_3$, respectively. These results show that the pH value reduces and the Mg^{2+} contents increases. But, the CCPP is still too positive given the desired criteria. When heated, CO_2 overflows when CO_3^{2-} reached the surface of the dolomite accompanied by Mg^{2+}. Thus, MgO is more abundant at the surface of the ore. After acidification by CO_2 and H_2O, more dissolvable carbonate might form and thus lead to a higher Mg^{2+} contents in the effluent. Even so, there remains a large gap between our results and the desired endpoints of the Israel criteria.

3.2.4. Cost estimation

Using artificially treated dolomite may increase the cost by 186.3 USD/ton for calcined dolomite, which includes the cost of purchasing (146.1 USD/ton) and processing (40.2 USD/ton) the material. The dosage of the calcined dolomite is about 93.3g/m^3 water, and thus the cost of product water increased by 0.017 USD/m^3.

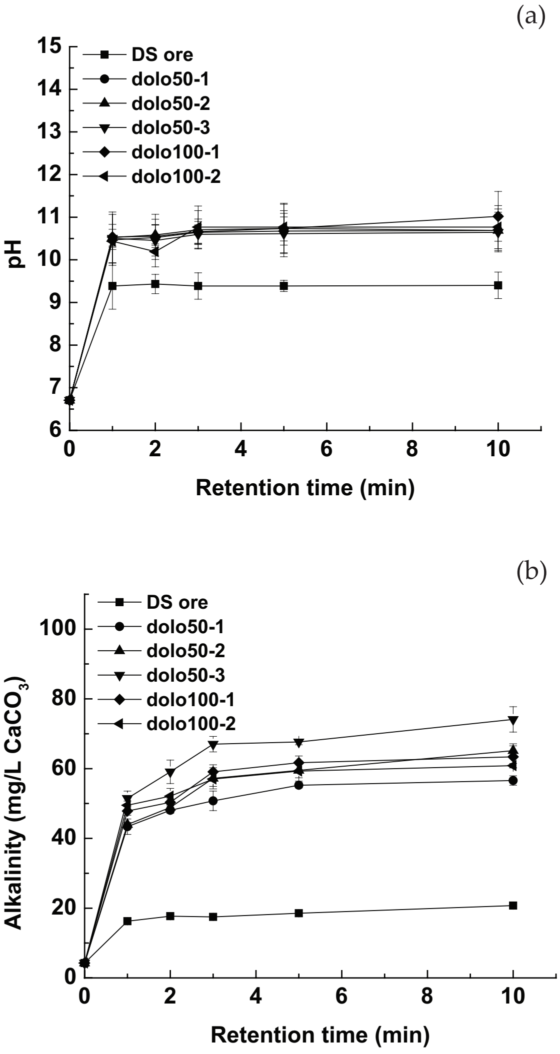

Fig. 8. (a) pH values and (b) alkalinity in the effluents of different modified dolomite.

4. Conclusions

DS ore, DG ore, and calcined and treated DSs are studied for adjusting the RO desalinated water without acidifying the influent. The optimal calcinations condition is at 750°C for 1 h. The Ca^{2+}, Mg^{2+}, hardness, and alkalinity contents of the RO effluent are 18.15 mg/L, 2.26 mg/L, 56.59 mg/L as $CaCO_3$ and 68.11 mg/L as $CaCO_3$ with a retention time of 3 min, respectively. The hardness, Ca^{2+} and Mg^{2+} contents, pH values, and alkalinity of the effluent are 89.8 mg/L as $CaCO_3$, 1.49 mg/L and 20.42 mg/L, 10.59, and 67.02 mg/L as $CaCO_3$, respectively, for acidized dolomite (at 50°C for 3 h). Proper aeration can achieve the requirement of pH in Israel criteria. More research work is needed before quality is on par with tap water and the Israel criteria.

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