

Research Article

Swelling Characteristics of Fractal-Textured Bentonite Eroded by Alkaline Solution

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To study the swelling characteristics of bentonite eroded by alkaline solutions, swelling deformation (δ_s) tests on the bentonite subjected to immersion conditions of NaOH and KOH solutions were conducted. It is found that the δ_s of bentonite decreases with the increase in the concentration of alkaline solution. The swelling deformation δ_s of bentonite eroded by NaOH solution exceeds that in the KOH solution for the same level of concentration. Moreover, by performing XRD tests, it is revealed that the reduced swelling performance of bentonite in alkaline solutions is mainly attributed to the dissolution of the swelling mineral, i.e., montmorillonite (Mt.). The higher the concentration of alkali solution, the stronger the dissolution of Mt., thus leading to more significant attenuation of the swelling properties. Besides, the K^+ exhibits low hydration energies, which tends to fit into the silicon-oxygen tetrahedral cavity of smectite, forming a stronger K-linkage which leads to closely spaced crystal layers. The $e-p_e$ fractal relation can be calculated using the δ_s of bentonite eroded by alkaline solution. The $e-p_e$ fitting relation of the experimental data depicts that the dissolution of Mt. minerals in alkaline solution reduces the swelling coefficient κ , however, it increases the surface fractal dimension (D_s).

1. Introduction

Geo-environmental engineering is a major concept related to the human living environment and sustainable development. The related research aspects include pollutant migration, treatment measures, reuse of industrial waste, and comprehensive utilization. In recent years, many researchers have focused on the composition and scale of mineral particles in soil and pore water, ion concentration and chemical composition of pore water, microstructure characteristics, stress history, and thermal expansion performance, especially their coupling process [1–8]. The disposal of high-level radioactive waste (HLRW) is related to sustainable development, energy, and nuclear industry, and is related to the safety of the living environment of future generations, has attracted wide attention around the world. Up to now, deep geological disposal is considered the most feasible method for the disposal of HLRW. The disposal

repository is a complex multibarrier system that includes HLRW in sealed canisters, buffer/backfill materials, and host rocks. Because of its outstanding buffering and backfilling qualities (high swelling capacity, low permeability, and good adsorption properties, etc.), bentonite is considered the artificial barrier material for deep geological repository [9, 10]. During the operational phase of the repository, the concrete surrounding the rock lining deteriorates due to the infiltration of groundwater, thereby initially releasing Na^+ and K^+ to form a strong alkaline pore water solution (pH 13–13.5). In addition, Ca^{2+} and Na^+ are subsequently formed in the later stage, which exhibits a pH of 12–12.5, thus causing irreversible changes in bentonite properties and further threatening the safe use of the repository [11, 12]. Therefore, it is significant to study the swelling performance of bentonite under the action of an alkaline solution to efficaciously design, construct, and maintain the repository.

Montmorillonite (Mt) is the main swelling mineral in bentonite and it largely governs the buffering and backfilling characteristics of bentonite. Savage et al. [13] found that the pore solution formed as a result of cement degradation tends to reduce the swelling properties of bentonite. Chen et al. [14] analyzed the influence of NaOH solution with different concentrations on the Chinese GaoMiaoZi (GMZ) bentonite. The results showed that, due to the dissolution of Mt. in alkaline solution, the wing colloid formed depicted a variety of cracks. With increasing NaOH concentration, the Mt. content decreases whereas the degree of fragmentation increases, which leads to a decrease in the swelling properties. Fernández et al. [15] found that when the highly alkaline solution permeates into the bentonite-based buffer backfill material, the solution will dissolve the minerals present in the bentonite, which will eventually weaken the swelling deformation (δ_s) as well as the self-sealing properties of the backfill material. Furthermore, Xiang et al. [16] studied various changes in bentonite in alkaline solutions, and they revealed that the Mt. content decreased gradually while the specific surface area and adsorption capacity also decreased. The scanning electron microscopy (SEM) micrographs illustrated that the surface fractal dimension (D_s) increased. Sánchez et al. [17] studied the changes in mineral composition of bentonite under different temperature ranges and various concentration levels of alkaline solution with the help of X-ray diffraction (XRD). The results showed that as the concentration and temperature increase, the Mt. content, as opposed to the nonswelling minerals, gradually decreases. In another study, Kamland et al. [18] incorporated alkaline NaCl and saturated CaCl₂ (Ca(OH)₂) as soaking solutions, and they concluded that the swelling property of Wyoming bentonite in saline solution reduced due to the cation exchange reaction, while that in the alkaline solution also reduced mainly because of the Mt. dissolution. Bauer et al. [19] analyzed the effects of temperature and KOH solution concentration on Mt. content, and they found that the dissolution of Mt. was accompanied by a change in crystal shape as the reaction produced zeolite, quartz, and other minerals. Additionally, the reaction turned out to be more vigorous with increasing concentration and temperature. Similarly, Gaucher et al. [20] found that with the increase of the alkaline solution concentration, the dissolution rate of Mt. increased sharply at 60°C especially in highly alkaline solution (pH = 13). Ramirez et al. [21] studied the reaction of bentonite with highly alkaline solutions at different temperatures and observed the precipitation of nonexpansive minerals, i.e., zeolite, whereas the magnesium content in the Mt. increased, which led to the decreased swelling performance. It was noted that a further increase in temperature would intensify the reaction. Herbert et al. [22] analyzed the swelling performance of MX-80 bentonite by incorporating different solutions using transmission electron microscope (TEM), and they found that the change of Mt. and kaolinite content primarily weakens the swelling performance of bentonite. Fernández et al. [23] studied the effects of alkaline solution diffusion and found that brucite was formed on the surface of bentonite that dissolved the Mt. minerals, hence decreasing the swelling properties of the FEBEX bentonite.

The surface morphology of bentonite with regard to encountering moisture is complex in nature, and the δ_s refers to the process of uptake of water by the Mt. content of bentonite. Xu et al. [24] proposed a relationship between swelling force and adsorbed water volume $V_w/V_m = K_p D_s - 3$, where V_w is the final adsorbed water volume of bentonite, V_m is the volume of smectite in bentonite, and as mentioned earlier, D_s is the surface fractal dimension of bentonite. Li et al. [25] explored the calculation method of the osmotic suction coefficient and the osmotic suction of different types of solutions by introducing the Debye-Hückel formula. Moreover, Xu et al. [26] derived the effective stress calculation equation by considering the impact of osmotic suction in the salt solution.

Presently, the research on the mechanistic effect of alkaline solutions on the swelling properties of bentonite is mostly reflected in the properties and mineral content of bentonite. However, the numerical calculation of δ_s of bentonite is scarcely reported and needs research to cover the deficiencies in this particular direction. Therefore, in the current study, NaOH and KOH solutions are used as erosion solutions to study and analyze the attenuation mechanism of δ_s of bentonite, fitting calculations for bentonite δ_s , and explaining the influence of alkali metal cations on the performance of bentonite. Furthermore, this research study is aimed to provide guidance suggestions for the efficacious design and reliable construction of a deep geological repository.

2. Materials and Methods

2.1. Materials. The basic properties of bentonite used in this paper are summarized in Table 1. According to the standard test requirements, the original bentonite is placed inside an oven at 105°C for drying. After that, a 500 g soil sample is weighed each time such that 90 ml of distilled water is sprinkled on the soil sample at a designated water content of approximately 18%. Then the sample is stored by enclosing it in plastic wrapping. In addition, distilled water was used to prepare the NaOH and KOH solutions with prescribed concentrations of 0.3 and 1.0 mol/l (M), respectively.

2.2. Swelling Deformation Test. The moisture content of the saturated soil was remeasured for the sake of accuracy prior to sample preparation. The cutting ring was placed on the base of the prototype machine and was tightened with the help of a hoop. Then, about 58.5 g of wet soil was weighed each time, which was incorporated into the cutting ring in batches while applying a backing plate cover on it afterward. After that, a jack was used to statically press the plate into a cakelike sample with Φ 61.8 mm \times H 10 mm, and a density of 1.7 ± 0.5 g/cm³. This range of density has been widely applied in experimental studies on compacted bentonite [27]. After following the aforementioned requirements, the static pressure was applied and maintained for a period of time [28].

The operation of the δ_s in this study can be explained in seven major steps: (i) preparing three groups of samples (i.e.,

TABLE 1: Basic physical and chemical indices of tested bentonite.

Parameter	Value
Specific gravity	2.75
Liquid limit (%)	210.7
Plastic limit (%)	25.1
Plasticity index I_p	185.6
Total specific surface area (m^2/g)	570
Montmorillonite content (%)	84
Cation exchange capacity (cmol/kg)	60.1

distilled water, NaOH solution, and KOH solution) (ii) setting the loads at six different levels (i.e., 100, 200, 400, 800, 1200, and 1600 kPa), for a total of 30 number of samples, (iii) permeable stone, filter paper, test sample and permeable stone are sequentially placed layer-wise in the bottom of oedometer cell, (iv) installing a dial indicator, adjusting the dial gauge, and recording the initial reading, and then applying the required load at one time, (v) the soaking solution is injected into the oedometer cell and the sample is immersed, (vi) recording the readings of dial gauge (note that the reading interval is shorter in the early stage and longer in the later stage), and (vii) when the pointer of dial gauge remains unchanged for 24 hours, it is considered that the δ_s tests were completed.

2.3. XRD and SEM Test. The XRD tests were used to analyze the mineral composition of bentonite. Prior to testing, the bentonite samples after the δ_s test were washed with distilled water several times to remove the adsorbed salts. The samples were oven-dried at 105°C and pulverized. Thereafter, it was sieved using a 200-mesh sieve, and a small portion was taken for XRD tests. XRD tests were carried out in the Modern Analysis and Test Center of the Anhui University of Technology, China, by using a D-8 ADVANCE X-ray diffractometer by Brook Company, Germany. The target material was copper, while the power was kept at 3 kW. The scanning range of the goniometer was between 3° and 80°.

The SEM tests were used to analyze and determine the overall effect of the micromorphology of bentonite minerals. SEM analysis was also performed using a NANO SEM 430 field emission scanning electron type microscope produced by the FEI company.

3. Test Results

Figure 1 shows the swelling rate of bentonite at a pressure of 100 kPa. It can be seen that the varying trends of bentonite samples soaked in solutions of different concentrations are identical and hence depict three main stages: first gradually increasing, then followed by a relatively sharp increase, and finally reaching a stable state [29, 30]. It can be seen from Figures 1(a) and 1(b) that the swelling rate is the highest in distilled water as it approaches 61%. Hence, it can be inferred that the swelling performance of bentonite is greatly reduced after the action of the alkaline solutions. In addition, it is noteworthy to mention that with the increase in the alkaline concentration, the swelling ratios of bentonite also tend to

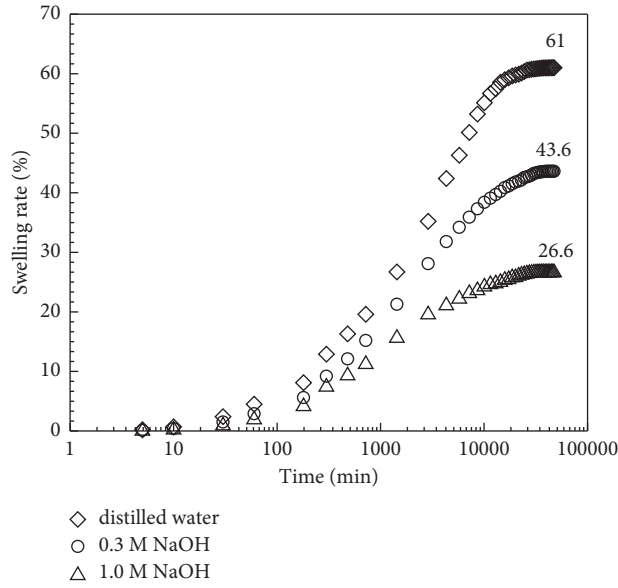
decrease. At 1.0 M, the aforementioned three stages of swelling ratio of bentonite vary with time, but not significantly. It is because the Mt. mineral in bentonite gets dissolved in the alkaline environment. Therefore, with the increase of solution concentration, the reaction is intensified, which reduces the swelling performance.

Figure 2 illustrates the influence of distilled water, 0.3 M NaOH, and KOH solutions on the swelling characteristics of bentonite subjected to an overlying pressure of 100 kPa. It can be seen that the swelling rates of the three solutions increase as distilled water > NaOH > KOH, with the maximum swelling rates recorded to be 61%, 43.6%, and 39.7%, respectively. In an alkaline solution, the δ_s of bentonite experiences a much larger large decrease than in the case of bentonite soaked in distilled water. In the case of KOH solution, the hydration ability of K^+ is low, and it can easily fit inside the cavity of the silicon-oxygen tetrahedral grid, which produces strong K linkage. As a result, the crystal layer contacts become more closely spaced and the expansion deformation is observed to be the minimum [31, 32].

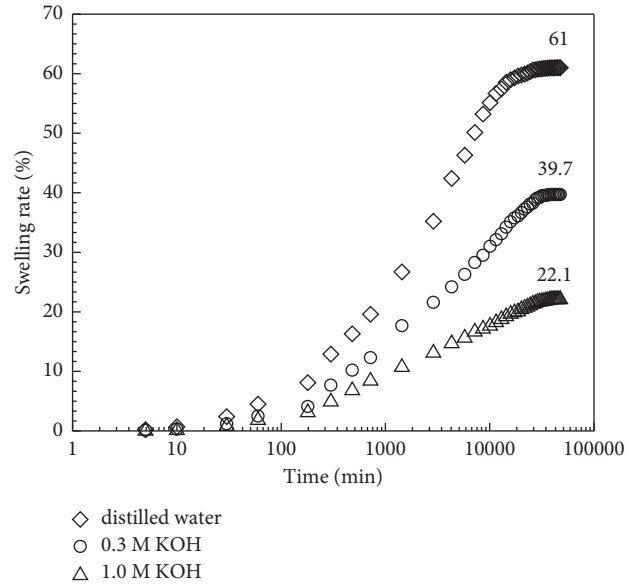
Figure 3 shows the relationship between the maximum δ_s of bentonite and the overlying compressive stress. It can be seen that at the same dry density, the maximum δ_s of bentonite decreases with the load increase. When the overlying pressure equals 100 kPa, the maximum swelling rate is, respectively, distilled water (61%), 0.3 M NaOH solution (43.6%), 1.0 M NaOH solution (26.6%), 0.3 M KOH solution (39.7%), and 1.0 M KOH solution (22.1%). On the contrary, when the overlying pressure is 1600 kPa, the maximum swelling rate is distilled water (10.4%), 0.3 M NaOH solution (8.7%), 1.0 M NaOH solution (4.4%), 0.3 M KOH solution (7.6%), and 1.0 M KOH solution (2.1%), respectively. The two cases were observed to witness reduction by almost 82.95%, 80.05%, 83.46%, 80.86%, and 90.50%, respectively.

Figure 4 shows the XRD diffraction pattern of the bentonite. Figure 4(a) shows the diffraction pattern of bentonite soaked in distilled water such that the diffraction peak intensity of Mt. is 523 CPS. Figures 4(b)–4(e) show the diffraction pattern of bentonite eroded by NaOH and KOH solutions. In addition, the peak values of Mt. correspond to 408, 302, 354, and 215 CPS for samples immersed in 0.3 M NaOH, 1.0 M NaOH, 0.3 M KOH, and 1.0 M KOH, respectively. It can be found that under the erosion of alkaline solutions, the peak diffraction intensity of Mt. gradually decreases, whereas the phenomenon is more significant with increasing concentration. Thus, it is indicated that the dissolution of Mt. minerals is the main reaction process that results in a significant reduction in the δ_s .

Figure 5 is a schematic diagram of the SEM performed on a variety of bentonite samples. The sample (a) soaked in distilled water exhibits compact particle structural integrity, witnessing no fragmentation phenomenon. In addition, the wing-shaped colloids are produced by the hydration of Mt. that are present on the surface of bentonite particles [14]. For samples (b)–(e) in the alkaline solution, the bentonite particles gradually split into small pieces and the wing-shaped



(a)



(b)

FIGURE 1: The relationship curve of bentonite swelling rate and time under 100 kPa.

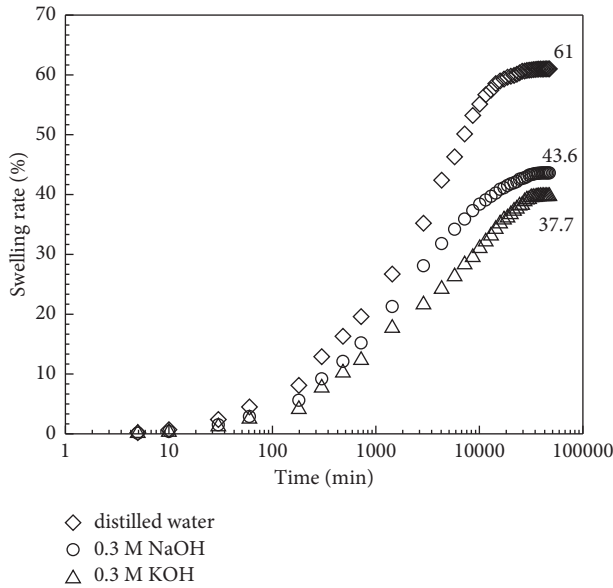


FIGURE 2: The swelling rate of different solutions varies with time at 100 kPa.

colloid gradually gets dissolved with increasing concentration. In the case of 1.0 mol/L NaOH and KOH solutions, the winged colloids were found to disappear completely, while the degree of particle breakage was also high. Comparing the two groups of samples (b) and (d), it can be seen that the bentonite eroded by KOH solution has comparatively larger particles and fewer cracks, which is attributed to the stronger bonding of Mt. crystal layers under the action of K^+ .

4. $e-p_e$ Relationship for Bentonite in Alkaline Solution

The δ_s of bentonite decreases with the increase of effective stress p_e . Based on the fractal theory, Debye-Hückel (D-H) formula can be introduced to obtain the osmotic suction coefficient for the determination of osmotic suction. Then, using the value of osmotic suction, the theoretical calculation method between effective stress and δ_s can be obtained. The $e-p_e$ fitting curve was analyzed to further explore the erosion mechanism of alkaline solutions on bentonite.

According to the fractal theory, Xu et al. [26] deduced the relationship between δ_s of bentonite and modified effective stress p_e in the form of the following equations:

$$e_m = \frac{V_w}{V_m} = K p_e^{D_s-3}, \quad (1)$$

$$e_m = \frac{e}{\alpha}, \quad (2)$$

$$e = \alpha K p_e^{D_s-3} = \kappa p_e^{D_s-3}, \quad (3)$$

where e_m is the void ratio of Mt, V_w is the water absorption volume of the Mt, and V_m is the volume of Mt. In bentonite, K is the swelling coefficient of Mt, p_e is the modified effective stress, e is the total void ratio of the sample, which can be calculated from δ_s , α is the mass fraction of Mt, and κ is swelling coefficient. Based on the fractal theory, p_e can be calculated from the overlying pressure and osmotic stress, and the calculation formula is as given as follows [26]:

$$p_e = \sigma + \pi \left(\frac{\sigma}{\pi} \right)^{D_s-2}, \quad (4)$$

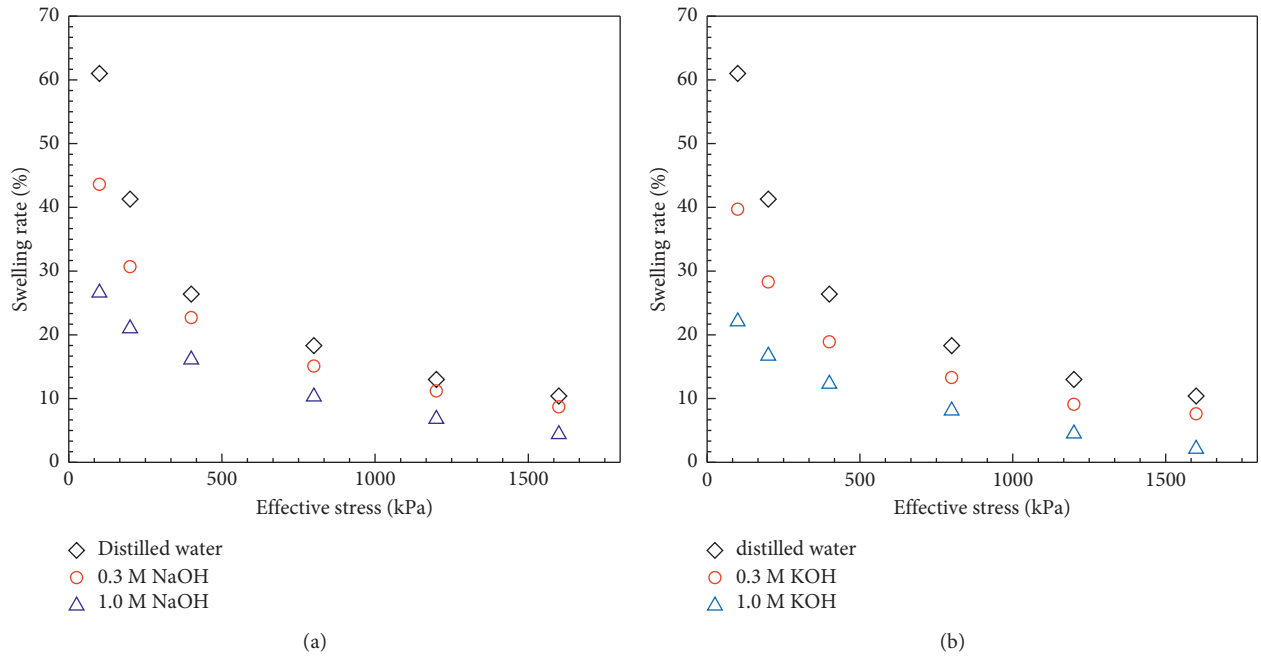


FIGURE 3: The relationship curve of maximum swelling deformation with load.

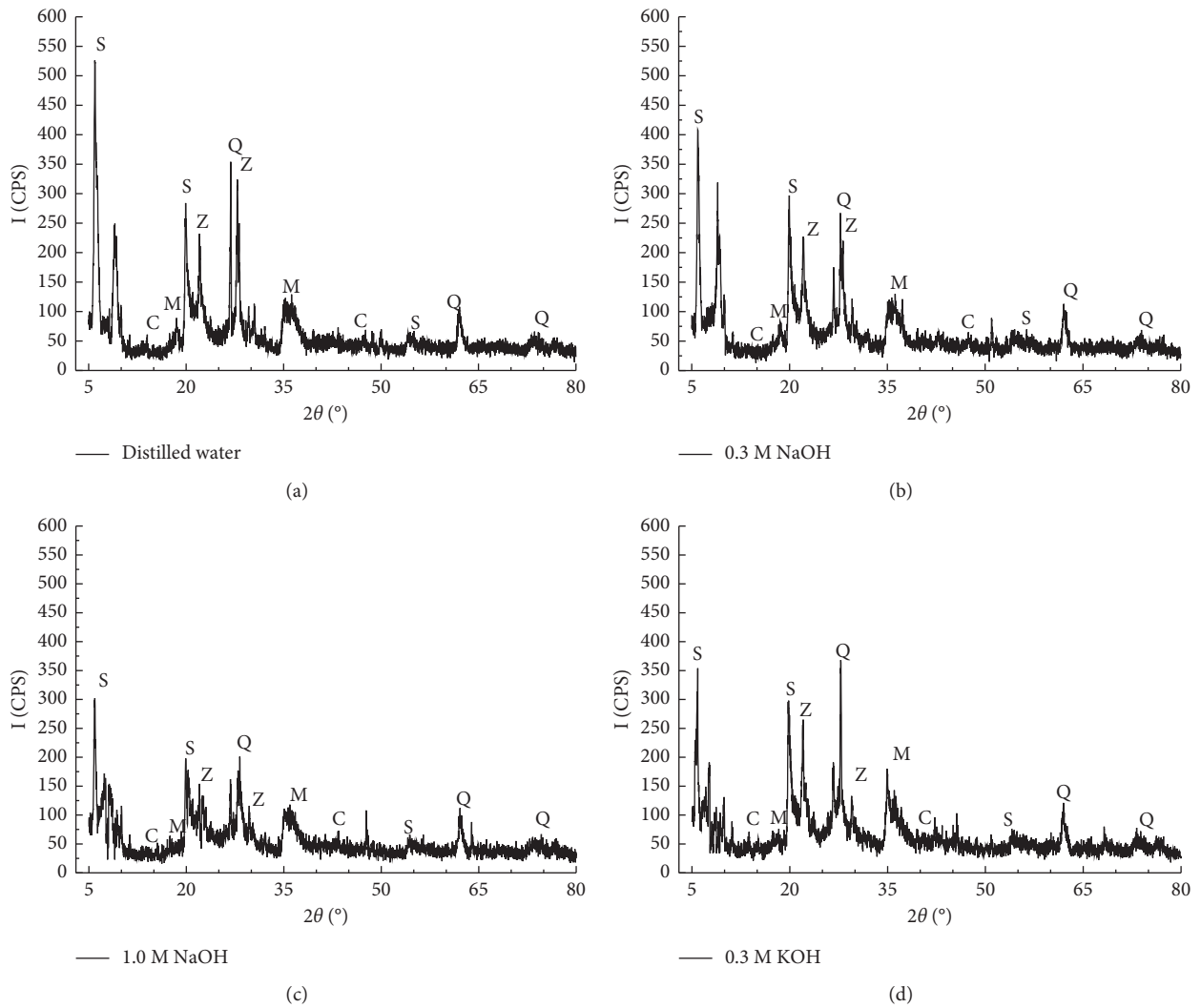


FIGURE 4: Continued.

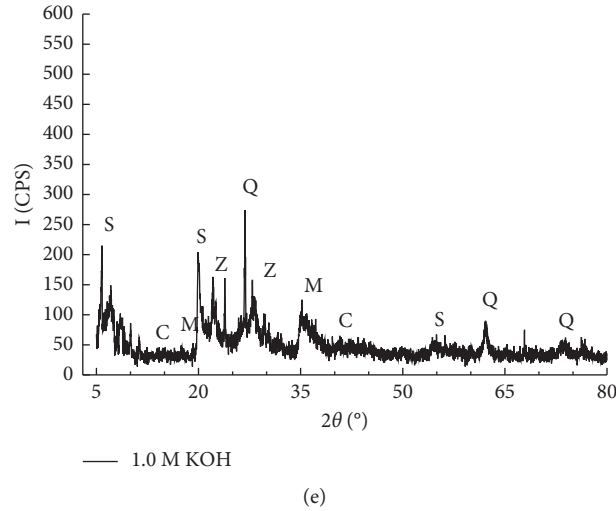


FIGURE 4: XRD diffraction pattern of bentonite sample: (a) distilled water, (b) 0.3 M NaOH, (c) 1.0 M NaOH, (d) 0.3 M KOH, and (e) 1.0 M KOH.

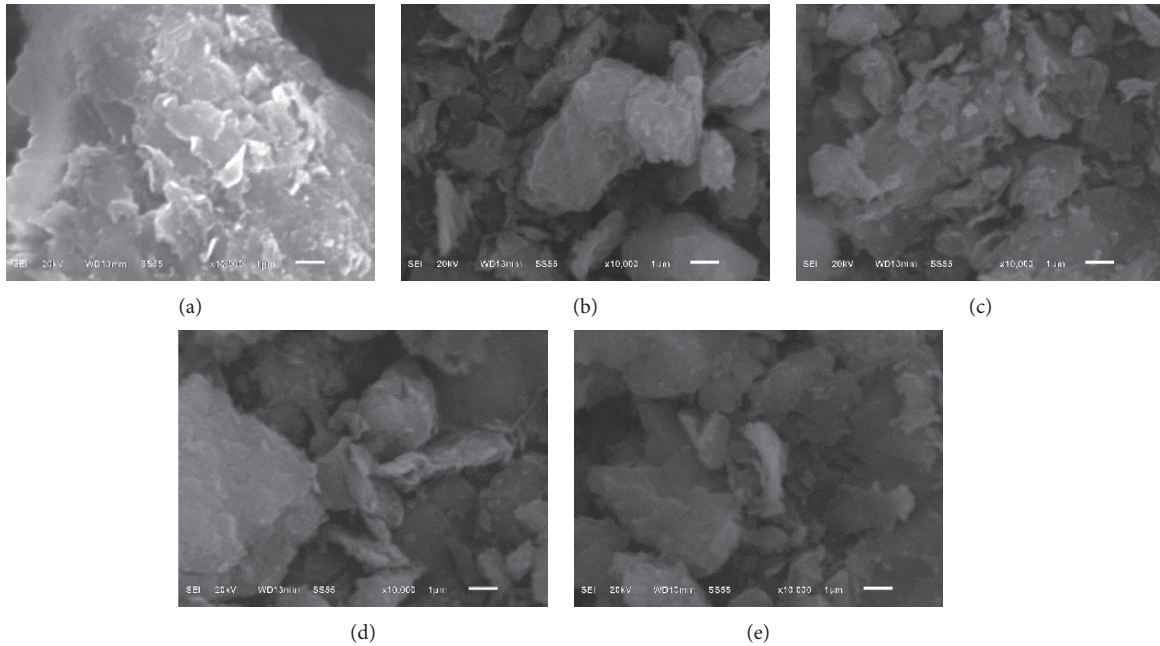


FIGURE 5: Scanning electron microscope (SEM) images of bentonite treated with a variety of alkaline solution concentrations: (a) distilled water, (b) 0.3 M NaOH, (c) 1.0 M NaOH, (d) 0.3 M KOH, and (e) 1.0 M KOH.

where σ is the overlying pressure and π is the osmotic suction of the alkaline solution.

It can be seen from (4) that p_e is related to osmotic suction. Li et al. [25] derived the osmotic suction coefficient and the calculation method of osmotic suction under different solutions based on the D-H formula in the form of

$$\pi = \varepsilon RTc\Phi, \quad (5)$$

where ε is the ion number of solute decomposition (for NaOH or KOH, $\varepsilon = 2$), R is the generalized gas constant (8.314 J/mol/k), T is the absolute temperature, c is the molar concentration of solute, and Φ is the osmotic suction

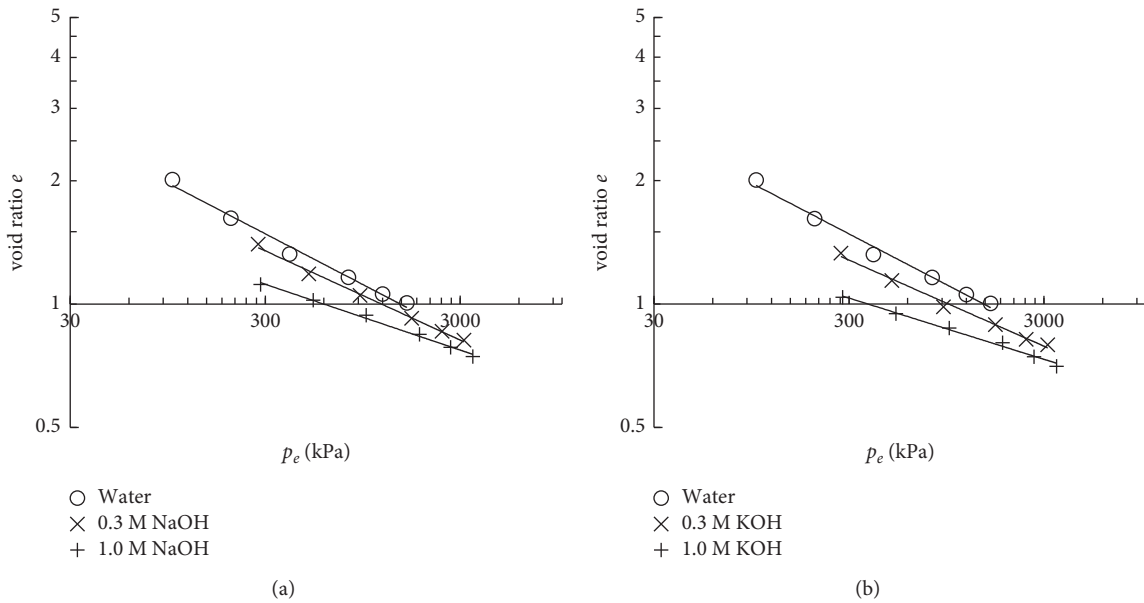
coefficient. The calculation formula of Φ in solution containing monovalent ion electrolyte is given by the equation as follows [33]:

$$\varphi = |z_M z_X| f + m \left(\frac{2v_M v_X}{v} \right) B_{MX} + m^2 \frac{(v_M v_X)^{3/2}}{v} C_{MX} + 1, \quad (6)$$

where z_M, z_X are the chemical valence that correspond to anions and cations, respectively, v_M, v_X correspond to the number of negative cations in the chemical formula, m is the molar number, C_{MX} is the empirical coefficient, and for strong electrolytes the formula of B_{MX} is in the form of following equation:

TABLE 2: Calculation results of osmotic suction in alkali solution.

Concentration	A	NaOH				KOH			
		f	B_{MX}	Φ	π	f	B_{MX}	Φ	π
0.01	0.447	-0.0399	0.294	0.963	46.710	-0.0399	0.392	0.964	46.758
0.05	0.447	-0.0788	0.248	0.934	226.424	-0.0788	0.334	0.938	227.469
0.1	0.447	-0.1024	0.221	0.920	446.083	-0.1024	0.300	0.928	449.913
0.2	0.447	-0.1300	0.190	0.908	880.921	-0.1300	0.261	0.922	894.644
0.3	0.447	-0.1477	0.171	0.904	1315.443	-0.1477	0.237	0.924	1344.129
0.4	0.447	-0.1607	0.158	0.903	1752.266	-0.1607	0.220	0.928	1800.530
0.5	0.447	-0.1709	0.148	0.904	2192.677	-0.1709	0.208	0.934	2264.872
0.6	0.447	-0.1794	0.140	0.906	2637.500	-0.1794	0.198	0.941	2737.817
0.7	0.447	-0.1865	0.134	0.909	3087.348	-0.1865	0.190	0.948	3219.872
0.8	0.447	-0.1928	0.129	0.913	3542.721	-0.1928	0.183	0.957	3711.462
0.9	0.447	-0.1982	0.124	0.917	4004.049	-0.1982	0.178	0.965	4212.970
1.0	0.447	-0.2031	0.121	0.922	4471.718	-0.2031	0.173	0.974	4724.746

FIGURE 6: The e - p_e relationship curve of bentonite in NaOH and KOH solutions.

$$B_{MX} = \beta_{MX}^{(0)} + \beta_{MX}^{(1)} e^{-\alpha I^{1/2}}, \quad (7)$$

where $\beta_{MX}^{(0)}, \beta_{MX}^{(1)}$ can be obtained by looking up the code. f can be calculated by the following formula:

$$f = -A \frac{I^{1/2}}{1 + bI^{1/2}}, \quad (8)$$

where I is the ionic strength, b is an empirical parameter equaling 1.2, and A is the D-H coefficient, which can be calculated by the following equation:

$$A = \frac{1}{3} (2\pi N_A \rho_{sl})^{1/2} (e^2 / (0.4\pi\epsilon_r\epsilon_0 kT))^{3/2}, \quad (9)$$

where N_A is Avogadro constant ($6.023 \times 10^{23}/\text{mol}$), ρ_{sl} is solvent density (0.998 g/cm^3), e is the charge of electrons ($1.60 \times 10^{-19} \text{ C}$), k is Boltzmann constant ($1.381 \times 10^{-23} \text{ J/K}$), and ϵ_r, ϵ_0 are relative dielectric constant (78.36) and vacuum

dielectric constant ($8.8542 \times 10^{-12} \text{ F/m}$), respectively. The calculation results are shown in Table 2.

Figure 6 depicts a fitting relationship curve between the p_e of bentonite and the void ratio e , drawn based on experimental data. It is found that the δ_s of bentonite in an identical solution is in accordance with the unified e - p_e relationship, and as the concentration of solution increases, the curve lowers down. According to the fitted curve equation, the D_s and swelling coefficient κ of bentonite in various solutions can be obtained, as shown in Table 3. It is also important to mention that the D_s of bentonite surface after erosion in alkaline solution is greater than the one observed in the case of distilled water immersion, and it increases with the increase of alkaline solution concentration. Combined with SEM tests, the main reason is that bentonite particles are broken apart under the action of an alkaline solution, thereby producing smaller fragments. Moreover, the surface of flaky Mt. is extremely irregular and corrosion trace extension leads to a rough bentonite surface and increased D_s value [14].

TABLE 3: e - p_e fitting curve of bentonite in different solutions.

Solutions	e - p_e	κ	D_s	R^2
Distilled water	$e = 6.084p_e^{-0.247}$	6.08	2.75	0.986
0.3 NaOH	$e = 4.677p_e^{-0.218}$	4.68	2.78	0.994
1.0 NaOH	$e = 2.791p_e^{-0.161}$	2.79	2.84	0.991
0.3 KOH	$e = 4.241p_e^{-0.21}$	4.24	2.79	0.988
1.0 KOH	$e = 2.423p_e^{-0.149}$	2.42	2.85	0.992

In addition, it can be found that the swelling coefficient κ of bentonite in the case of distilled water is the highest, whereas it decreases with the increase in alkali concentration. At the same concentration of alkali solution, the κ of bentonite eroded by NaOH solution is greater than that of KOH solution. On the one hand, the main swelling mineral Mt. in bentonite is dissolved in the alkaline solution and the dissolution rate increases with the increase of alkaline concentration, while on the other hand, K^+ can enter the cavity of the Mt. structure to form K-linkage and the crystal layer is more tightly bonded, which vilifies the swelling performance.

5. Conclusion

The swelling deformation (δ_s) tests of bentonite in alkaline solution, combined with XRD, SEM tests, and analysis, were conducted and the following main conclusions are drawn.

- (1) The δ_s curve of bentonite exhibits the characteristics in the form of three distinct stages. With the increase in the concentration of alkaline solution, the δ_s of bentonite gradually decreases, while the dissolution of swelling mineral Mt. is the main reaction process. K^+ has low hydration ability and easily fits into the silicon-oxygen tetrahedral cavity of Mt, producing stronger K-linkage, which leads to closely spaced crystal layers, thus resulting in lesser δ_s of the bentonite in KOH solution as compared with that in NaOH solution.
- (2) The δ_s of bentonite eroded by alkaline solutions can be calculated by a unified e - p_e fractal relationship. On the double logarithmic coordinate axis, the higher the concentration, the lower the δ_s curve. As the concentration of alkaline solution increases, the dissolution rate of Mt. also increases, hence reducing the swelling coefficient κ . Simultaneously, when Mt. dissolves, the clay particles disintegrate and the surface becomes coarser, which gradually increases the surface fractal dimension (D_s) of bentonite.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Authors' Contributions

Guosheng Xiang contributed to the conceptualization and methodology. Weimin Ye contributed to the conceptualization. Contributors to the investigation included Zhijie Hu, Lei Ge, and Yinkang Zhou.

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