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Research paper

# Effects of pH and temperature on the swelling pressure and hydraulic conductivity of compacted GMZ01 bentonite

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ABSTRACT

Gaomiaozi (GMZ) bentonite has been recognized as the first choice for using as buffer/backfill materials in deep geological repository for the disposal of high-level nuclear waste (HLW) in China. Groundwater in Beishan area, which has been considered as a potential site for the construction of Chinese deep geological repository, may reach a high pH value because of its chemical background and possible cement degradation during the operation of the repository. Meanwhile, temperature may increase with decay heat released from the waste in the canister. Investigation of pH value of alkaline-solutions and temperature effects on the behavior of compacted GMZ01 bentonite is of great importance for the Chinese deep geological repository program. For this purpose, a series of swelling pressure and hydraulic conductivity tests with various pHs of NaOH solutions were conducted at different temperatures. The X-ray diffraction (XRD) exploration was performed on the GMZ01 bentonite specimens before and after experiencing the swelling pressure and hydraulic conductivity tests, in order to find out the influences of temperature and pHs on the mineralogy of GMZ01 bentonite. Results show that the swelling pressure of GMZ01 bentonite decreases as the pH value of NaOH solutions increases, while the decreasing rate significantly depends on temperature. The swelling pressure evolution curve was "double-peak" structured, which faded with the increases of pH and temperature. The hydraulic conductivity of GMZ01 bentonite increases with the increase of the pH value of NaOH solutions and the rise of temperature. All these observations were consistent with the XRD test results: the dissolution of montmorillonite in GMZ01 bentonite increases with the pH increases. This process was accelerated by the temperature rise.



## 1. Introduction

Under the ideal concept of a multi-barrier repository for the disposal of high level radioactive nuclear waste (HLW), it is necessary to build a concrete ring, located between the geological formation and the bentonite barrier that serves as a structural support for the galleries (Glasser, 2001). During the long-term operation of the repository, the concrete will degenerate and act as a source of alkaline fluids when the pore water of the formation saturates the system (Fernández et al., 2009) and the pH of the leached pore water may be higher than 13 due to the release of sodium and potassium hydroxides (Taylor and Harry, 1987). Meanwhile, the high pH porewater solutions will continue to interact with the bentonite barrier during their direct contact (Fernández et al., 2009; Ramırez et al., 2002), significantly altering the in-situ pH, solution composition and mineralogy of the surrounding clay barrier (Bauer and Velde, 1999). This kind of alteration of clays due to the interaction with alkaline fluids has been confirmed by numerous researchers in literature (Adler et al., 1999; Alexander et al., 1992; Bauer and Velde, 1999; Fernández et al., 2006; Gaucher and Blanc, 2006; Melkior et al., 2004; Milodowski et al., 1992; Nakayama et al., 2004; Pusch et al., 2003; Smellie et al., 2001). Moreover, heat released from radioactive decay in the canister will accelerate this cement degradation, speeding up the rise of the pH value of the pore fluid. Once these hydroxides infiltrate into the buffer/backfill materials, the interlayer cation of montmorillonite will exchange with alkali metal ions in groundwater. Consequently, some montmorillonite will be dissolved and non-expansive minerals will be generated, resulting in the decrease of the swelling properties and the increase of the hydraulic conductivity of the buffer/backfill materials. Nakayama et al. (2004) have confirmed an increase of porosity and permeability of a compacted bentonite in contact with a highly alkaline (NaOH, pH = 14) solution tank, due to montmorillonite dissolution.

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It is recognized that the behavior of clayey materials is strongly dependent on the physico-chemical interactions between clay particles and pore fluid chemistry (Castellanos et al., 2008). The influences of alkaline solutions and temperature on the mineral composition, microstructure, swelling property and hydraulic conductivity of bentonite have been widely investigated. Bauer and Velde (1999) found that, at elevated temperatures (20–150 °C) and in the presence of alkaline water, smectite alteration is highly probable. Using different materials, Barrer (1982) experimentally confirmed that, over a certain range of temperatures, pHs and solution compositions, zeolites, K-feldspar and clays can be produced. Ramırez et al. (2002) investigated the mineralogical changes in bentonite in contact with alkaline solutions (pH = 10–13.5) at 35, 60 and 90 °C. Results indicated that, when pH > 12.6, the zeolite crystallization and the increase of magnesium in a non-exchangeable form in the smectitic clay fraction are the main mineralogical changes. Furthermore, both processes are enhanced by the duration time and temperature of reaction. Fernández et al. (2006) found that NaOH fluids produced minor changes in the mineralogy of compacted bentonite at 60–25 °C. Based on an investigation of the reaction of synthetic alkaline solutions (K–Na–OH and Ca–$(OH)_2$, pH = 8–8.5) with a Mg-saturated compacted FEBEX-bentonite at 60 °C, Fernández et al. (2009) found that montmorillonite is partially dissolved. Cuisinier et al. (2008) reported that saturated portlandite water (pH = 12.4), which circulated through the compacted MX-80 and argillite mixture samples at 60 °C, caused an increase of the macrospore void ratio by almost 1.5 times.

Researches also indicated that high pH of the pore space solutions may cause changes in the swelling capacity of bentonite, possibly affecting the long term performance of the engineering barrier system. Based on a 3-year study on the changes of MX-80 bentonite properties after the reaction with solutions of different ionic strengths and pHs, Herbert et al. (2008) confirmed that the swelling pressure of bentonite was the highest (>4 MPa) when in contact with water, significantly lower (about 2 MPa) when in contact with low cation solutions and the lowest (mostly below 1 MPa) when in contact with high saline brines. This phenomenon is mainly due to the alteration and partial dissolution of montmorillonite (Karnland et al., 2007).

Kinsela et al. (2010) found that the addition of calcium (50 mM) greatly decreased the size of the structural network, resulting in the increase of hydraulic conductivity by approximately 65-fold, especially in the case of high pH values. Additionally, the changes of fabric and porosity caused by temperature changes can also affect the hydraulic conductivity (Romero et al., 2001). Based on the saturated infiltration tests on compacted bentonite with different dry densities and at different temperatures, Towhata et al. (1993) and Cho et al. (1999, 2000) found that the hydraulic conductivity increases with temperature rise; the value at 80 °C is three times that at 20 °C. This temperature effect can be explained by the changes of water viscosity (Constantz and Murphy, 1991; Hopmans and Dane, 1986), water density, and to some extent the intrinsic hydraulic conductivity. The water viscosity has been found to be the most significant factor (Delage et al., 2000).

As for the research in China in the field of deep geological disposal for high-level radioactive waste, the program was launched in the middle of 1980s. Beishan in Gansu province has been selected as one of the potential sites for the Chinese geological repository. Field investigation results show that groundwater in Yemaquan area at Beishan, was rich in $K^+$, $Na^+$, $Ca^{2+}$ and $Mg^{2+}$ with a pH value of 7.1–8.8 (Guo et al., 2001, 2004; Yang and Guo, 1999).

Gaomiaozi bentonite originates from the Inner Mongolia Autonomous Region, 300 km northwest from Beijing, China (Ye et al., 2009). Thanks to its favorable physical and mineralogical properties and large volume in deposit, it has been recognized as a potential buffer/backfill material in the Chinese deep geological disposal program for HLW (Liu et al., 2001). Investigations on the mineral composition, expansive property, soil–water characteristic and hydraulic conductivity of GMZ bentonite have been receiving more and more attentions (Cui et al., 2011; Liu et al., 2001; Ye et al., 2009, 2010a,b, 2012; Zhu et al., 2013). For the compacted GMZ01 bentonite specimens, it was found that the swelling pressure decreases and the hydraulic conductivity increases with the increasing concentration of infiltrating salt solutions. Furthermore, the impact of NaCl solutions on the swelling pressure and hydraulic conductivity is higher than that of $CaCl_2$ solutions at same concentrations (Zhu et al., 2013).

However, the influences of alkaline solution and temperature on the mineral composition, microstructure, expansive property and hydraulic conductivity of GMZ bentonite have not been reported.

In this study, swelling pressure and saturated hydraulic conductivity tests on the compacted GMZ01 bentonite in contact with NaOH solutions with different pH values were conducted at different temperatures. X-ray diffraction (XRD) tests were performed for determining the variation of minerals' nature and content in the compacted GMZ01 bentonite after experiencing the swelling pressure and saturated hydraulic conductivity tests. Based on the test results, the effects of temperature and pH on the swelling pressure, the saturated hydraulic conductivity and the variation of minerals were analyzed.

## 2. Experimental investigation

### 2.1. Apparatus

The experimental setup developed by Zhu et al. (2013), which was improved by introducing a water/alkaline-solution converter, was employed for swelling pressure and saturated infiltration tests with alkaline-solutions under temperature control (Fig. 1). It consists of five parts, a stainless steel cell, a digital oven, a data logging system, a set of pressure–volume controller and a water/alkaline-solution converter. The stainless steel cell is made up of a basement, a specimen ring for the emplacement of specimen sandwiched between two porous stones and a stainless piston with a load transducer on its top. All these parts were fixed by a screw cap. A digital oven having an accuracy of ±0.1 °C was employed for controlling temperature in the cell. Temperatures 25 °C, 60 °C and 80 °C were applied in this test. A data logging system was employed for recording the evolution of swelling pressure. A pressure–volume controller (0–1.5 MPa to an accuracy of ±1 kPa; 200 $cm^3$ to an accuracy of ±1 $mm^3$) was used for imposing water pressure and measuring the volume of an alkaline solution infiltrated into the specimen during the test. As the alkaline solution cannot be directly stored in the pressure–volume controller, a water/alkaline-solution converter was designed (Fig. 1). It composes two parts: one end was connected to the pressure–volume controller and the other end was connected to the stainless steel cell. De-ionized water and alkaline solution were filled from the two ends respectively and some silicone oil was kept between them. Three pH values (8, 10 and 12) of NaOH solutions were considered for the hydration tests.

### 2.2. Materials and specimen preparation

GMZ01 Na-bentonite tested here is white gray powder. Some basic properties are listed in Table 1 (Wen, 2006; Ye et al., 2009). A high cation exchange capacity and adsorption ability can be identified.

For the specimen preparation, the GMZ01 bentonite powder was firstly hydrated to a given suction by the vapor equilibrium technique. The saturated solution of $K_2CO_3$ was employed for applying 113 MPa suction (Tang and Cui, 2005). After the equilibrium, the hydrated powder was statically compacted at a constant loading rate of 0.375 kN/min to a designed maximum compaction pressure of 45 kN, which corresponds to a target dry density of 1.70 $Mg/m^3$. A total of 9 as-compacted cylindrical specimens with dimensions of 10 mm in height and 50 mm in diameter were prepared.

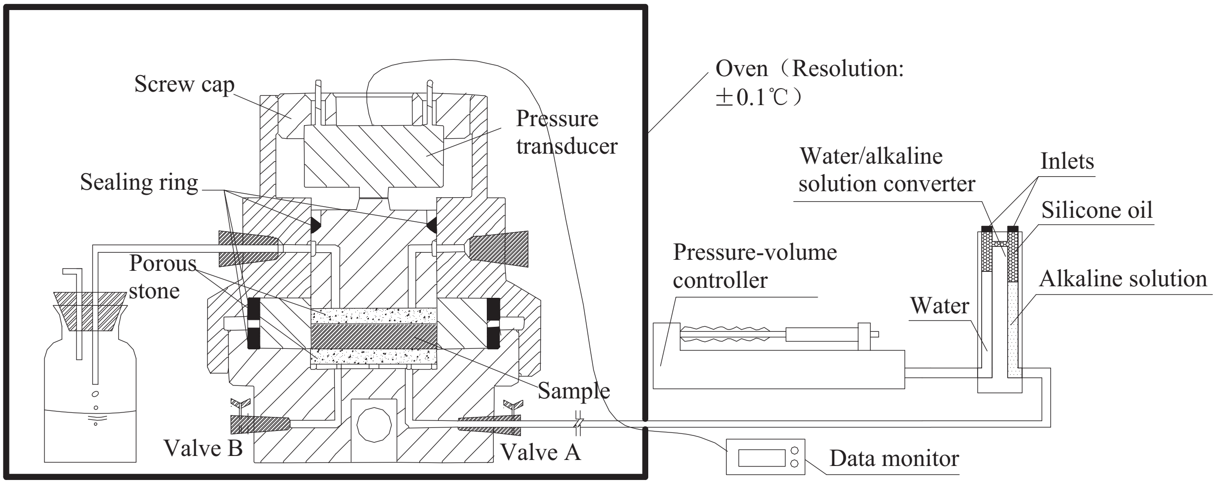


**Fig. 1.** Setup for swelling pressure and saturated hydraulic conductivity tests with temperature control.

### 2.3. Test procedure

Firstly, tests at ambient temperature 25 °C were conducted. At this stage, infiltration tests were conducted on three as-compacted specimens using three NaOH solutions with pH = 8, 10 and 12, respectively.

For tests with NaOH solution of pH = 8, the following steps were followed.

Step 1 An as-compacted GMZ01 bentonite specimen was introduced in the testing apparatus as shown in Fig. 1. After air-bubbles in the test system were exhausted, NaOH solution (pH = 8) was injected into the specimen through a water-alkali solution converter at a pressure of 100 kPa by the pressure–volume controller. Evolution of the volume of NaOH solution injected was recorded through the measurement of the volume of water supplied by the pressure–volume controller. The evolution of swelling pressure over time was also recorded. When the specimen was saturated (which was characterized by the stabilization of the water intake and the swelling pressure; Villar and Lloret, 2004), the swelling pressure test was considered as being completed, and step 2 started.

Step 2 The constant hydraulic head method for the determination of saturated hydraulic conductivity was employed. The infiltration pressure was increased to 1 MPa and then maintained constant throughout the whole test. NaOH solution infiltration was monitored through the corresponding volume of water that the pressure–volume controller provided. Based on the test results, the hydraulic conductivity was determined using Darcy's law. When the saturated hydraulic conductivity reaches the stable state (Ye et al., 2012), the test was considered as being finished.

Step 3 The test setup was dismantled. Some soil was cut from the tested specimen and freeze-dried. Then, it was ground and sized with a 1 mm sieve for further XRD investigations.

A new as-compacted specimen was assembled and the former steps 1 to 3 were repeated for the tests with NaOH solutions of pH = 10 and 12, respectively.

Then, temperatures 60 °C and 80 °C were applied successively on new as-compacted specimens using the digital oven. The same test procedure as that at temperature 25 °C was followed. Correspondingly, evolutions of swelling pressures with time were recorded and the hydraulic conductivities were determined using Darcy's law as at temperature 25 °C.

**Table 1**
Some basic properties of the GMZ01 bentonite (Wen, 2006).

| Property | Description |
|---|---|
| Specific gravity of soil grain | 2.66 |
| pH | 8.68–9.86 |
| Liquid limit (%) | 276 |
| Plastic limit (%) | 37 |
| Total specific surface area/($m^2 \cdot g^{-1}$) | 570 |
| Cation exchange capacity/($mmol \cdot g^{-1}$) | 0.773 |
| Main exchanged cation/($mmol \cdot g^{-1}$) | $Na^+$ (0.4336), $Ca^{2+}$ (0.2914), $Mg^{2+}$ (0.1233), $K^+$ (0.0251) |
| Main minerals[a] | Montmorillonite (75.4%), quartz (11.7%), feldspar (4.3%), cristobalite (7.3%) |

[a] Results of XRD tests.

### 2.4. XRD test

A $D_{max}$ 12 kW rotating anode X-ray diffractometer, which operates at 100 mA and 40 kV, was employed in this investigation. The X-ray powder diffraction (XRD) pattern was recorded for the as-compacted GMZ01 bentonite specimen and other specimens (pH8T60, pH8T80, pH10T25, pH10T60, pH12T60, pH12T80, where, numbers following "pH" stands for pH value and those following "T" stands for temperature) that experienced swelling pressure and hydraulic conductivity tests. XRD analysis was performed with CuKα radiation (λ = 0.15418 nm) and the 2θ-scanning rate was 2°$min^{-1}$. Patterns were identified by comparison to the PDF2 standards (ICDD, 2013).

## 3. Experimental results

### 3.1. Effects of pH value and temperature on swelling pressure

Evolution curves of the swelling pressure of compacted GMZ01 bentonite in contact with different pH values of NaOH solutions at temperatures 25 °C and 80 °C were presented in Fig. 2.

It is observed that swelling pressure increases rapidly at the beginning of infiltration and then reaches the first peak followed by an intermediate phase where the swelling pressure decreases, well before the soil reaches its full saturation. When the specimen reaches its complete saturation under constant volume conditions, swelling pressure increases up to a constant value, which was defined as the equilibrium swelling pressure of the tested specimen under the considered test conditions. The swelling pressure evolution curve presents a "double-peak" structure. This observation was in accordance with the results obtained from swelling pressure tests on the compacted FEBEX bentonite with a dry density of 1.50 $Mg/m^3$ at room temperature (Villar and Lloret, 2008), on compacted GMZ01 bentonite with an initial dry density of

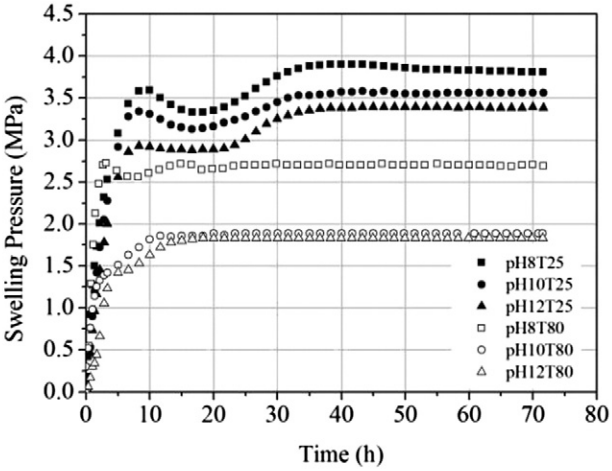


**Fig. 2.** Evolution of the swelling pressure of GMZ01 bentonite.

1.70 $Mg/m^3$ using de-ionized water (Ye et al., 2013) and using saline water (Zhu et al., 2013). However, this "double-peak" feature faded with the increase of pH and temperature, which was accompanied by a decrease of swelling pressure.

The relationships between the measured swelling pressure and pH value of NaOH solutions at temperatures 25 °C, 60 °C and 80 °C are shown in Fig. 3.

It can be observed that, for a given temperature, the measured swelling pressure decreases with the pH increase of NaOH solution. The decreasing rate significantly depends on pH for higher temperatures. This observation is consistent with the results of an expansive Azraq Green clay reported by Abdullah et al. (1999) (Fig. 4) and those of MX-80 reported by Herbert et al. (2008).

It can also be identified from Fig. 3 that, for a given pH value of the infiltration solution, the measured swelling pressure decreases as the temperature increases. Furthermore, Lingnau et al. (1996) also observed a reduction in swelling pressure with temperature rise for a sand/bentonite mixture.

### 3.2. Effects of temperature and pH value on hydraulic conductivity

Evolutions of NaOH solutions infiltrated in densely compacted GMZ01 bentonite at 25 °C and 60 °C are presented in Fig. 5.

It appears that from the beginning of the infiltration test, the measured flux decreases gradually and then turns to a stable state after 150 h hydration. Using Darcy's law, the saturated hydraulic conductivity was obtained and the relationship between the saturated hydraulic conductivity and the pH value of NaOH solutions at different temperatures was determined (see Fig. 6). It is observed that, for a given temperature, the measured hydraulic conductivity of GMZ01 bentonite increases as the pH value of NaOH solutions infiltrated increases.

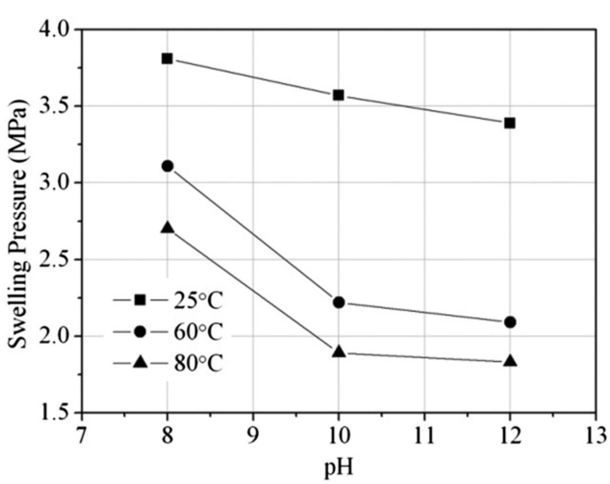


**Fig. 3.** Relationship between swelling pressure and pH value.

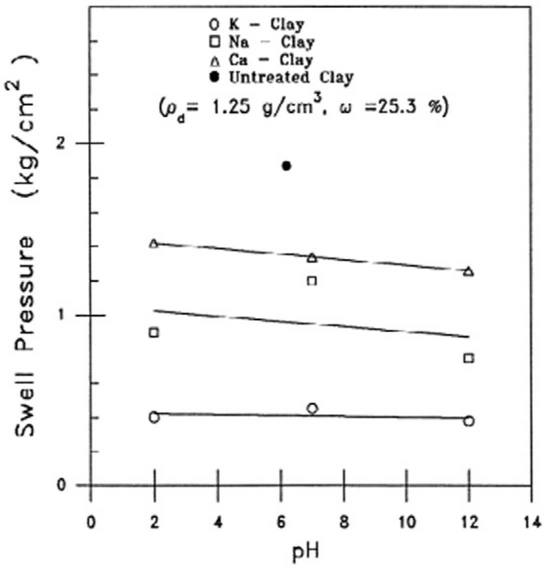


**Fig. 4.** Influence of pH on swelling pressure (Abdullah et al., 1999).

It can also be observed that, for a given pH value, the hydraulic conductivity increases significantly as the temperature increases. Moreover, the increasing rate depends on temperature. The hydraulic conductivities at 80 °C are almost three times those at 25 °C for a given pH value. This observation is consistent with the results of a geosynthetic clay liners (GCLs) reported by Bouazza et al. (2008). Cho et al. (1999) also found that the hydraulic conductivities in water-saturated Kyungju bentonite increase with increasing temperature.

### 3.3. Effects of temperature and pH value on mineralogy

The X-ray powder diffractograms of the intact GMZ01 bentonite specimen and those that experienced the swelling pressure and hydraulic conductivity tests are presented in Figs. 7 and 8, respectively.

Results in Fig. 7 show that, for a given temperature, the peak intensity of montmorillonite in GMZ01 bentonite that experienced the infiltration of NaOH solution is lower than that of intact bentonite specimen, suggesting a decrease of the montmorillonite content of GMZ01 bentonite after the contact with NaOH solutions. Furthermore, it continues to decrease as the pH value of NaOH solutions increases. This

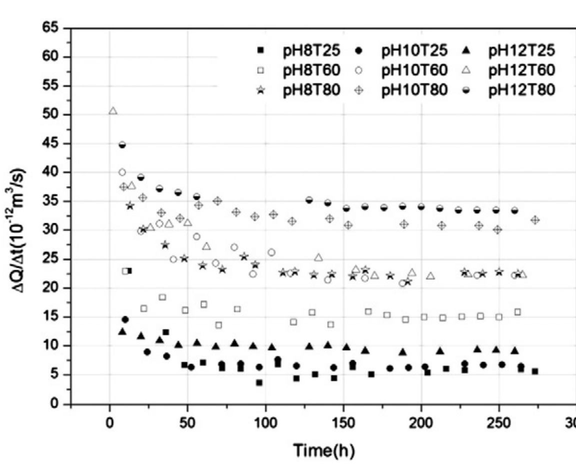


**Fig. 5.** Evolution of fluxes of NaOH solutions measured at different temperatures.

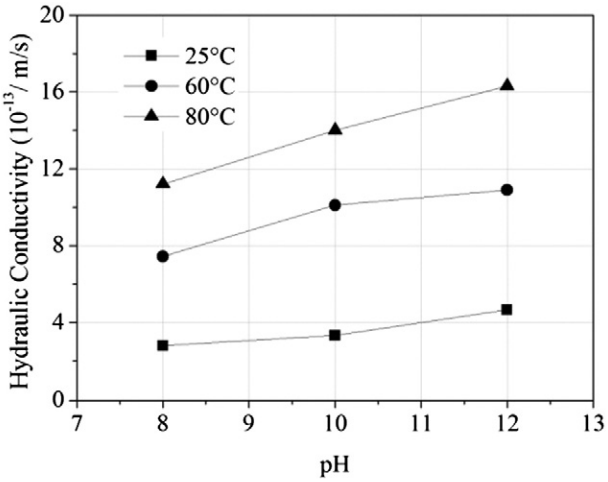


**Fig. 6.** Relationships between the hydraulic conductivity and pH value of NaOH solutions at different temperatures.

observation is consistent with the results reported by Bauer and Velde (1999) — the peak intensity of Ibeco beidellitic smectites was greatly diminished after the contact with KOH solution. It can also be observed from Fig. 7 that, with increasing pH value, the peak intensity of montmorillonite decreases slightly at 25 °C (Fig. 7a), magnificently decreased at 60 °C (Fig. 7b) and reduced to almost half at 80 °C (Fig. 7c). This observation indicates that the montmorillonite content decreases with the increase of pH value and the decreasing rate depends on temperature.

Results in Fig. 8 show that, for a given pH value, the peak intensity of montmorillonite decreases as the temperature increases. This observation is consistent with the results reported by Sánchez et al. (2006) who confirmed that the smectite is significantly transformed with temperature rise. This observation also agreed with the results reported by Fernández et al. (2006) who observed that the montmorillonite content in the "La Serrata" bentonite decreased with temperature rise (from 20 °C to 120 °C) and by Nakayama et al. (2004) who observed that the dissolution of montmorillonite followed a linear relationship with time under the considered test conditions with pH 13 to 14 and temperatures 90 °C to 170 °C, respectively.

Fig. 8 also indicates that the peak intensity of montmorillonite significantly decreases with the increase of temperature under the conditions of pH = 10 and 12 (Figs. 8b and c). However, for pH = 8, no obvious decrease is observed (Fig. 8a). Therefore, the reduction of the montmorillonite content with increasing temperature clearly depends on pH values.

## 4. Discussion

The swelling pressure decreases with the increase of pH of NaOH solutions. This phenomenon can be explained by the observations made at XRD test: the amount of montmorillonite dissolved increases with the increase of pH, resulting in the reduction of the swelling potential of GMZ01 bentonite. This phenomenon was enhanced by the increase of temperature (Fig. 7). This explanation is supported by the results reported by Pusch et al. (2003): some slight dissolution of the montmorillonite occurred when in contact with the low pH cement solutions at low (room) temperatures. It is also consistent with the observation made by Sánchez et al. (2006) that the smectite content in the Serrata de Níjar bentonite decreased with the rise of pH (12.9, 13.26 and 13.52) of NaOH solutions.

As the temperature and pH value increase, the evolution curves of swelling pressure gradually faded from a typical "double peak" structure. This phenomenon can also be explained with the XRD test results that with the increasing pH value or rising temperature, more and more montmorillonite was dissolved, resulting in the decrease of swelling pressure (Figs. 7 and 8). As the original structure of GMZ01 bentonite cannot be broken by the swelling pressure reduction, the typical "double peak" curve cannot be produced. This explanation is supported by several former works (Bauer and Velde, 1999; Fernández et al., 2006; Ramırez et al., 2002; Sánchez et al., 2006; Zhu et al., 2013).

The hydraulic conductivity increases as the pH value and temperature increase. This was also consistent with the observations from the XRD test results — the amount of dissolved montmorillonite increases with the increasing pH value of NaOH solution (Fig. 7). This dissolution results in the decrease of effective montmorillonite dry density. This explanation is supported by the results reported by Yamaguchi et al. (2007): that hydraulic conductivity of a sand–bentonite mixture increased with the decreasing effective montmorillonite dry density. The effect of temperature on the hydraulic conductivity may result from the evolution of viscosity of the permeating fluid (Abuel-Naga et al., 2006; Bouazza et al., 2008), or the increase of effective cross-section area of pore space for permeation (Cuisinier et al., 2008; Fernández et al., 2009; Nakayama et al., 2004) with temperature rise.

## 5. Conclusions

Gaomiaozi bentonite has been recognized as the first choice of buffer/backfill material in the construction of the Chinese deep geological repository for the disposal of high-level nuclear waste (HLW). Groundwater in Beishan area that has been considered as a potential site for the construction of the Chinese deep geological repository may reach a high pH value because of its chemical background and possible cement degradation during the operation of the repository. Thus, the investigation of the swelling pressure and saturated hydraulic conductivity of compacted GMZ01 bentonite in contact with NaOH solution with different values of pH and at different temperatures is of great importance.

The evolution curves of swelling pressures of GMZ01 bentonite specimens are "double-peak" structured, which faded with the increase of pH value and temperature. For a given temperature, the swelling pressure decreases as the pH value of NaOH solution increases. The

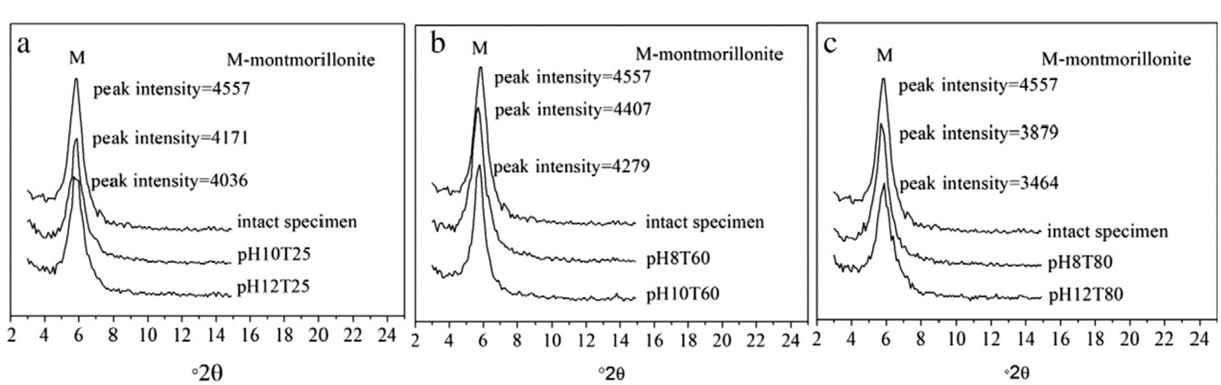


**Fig. 7.** X-ray diffraction profiles of compacted GMZ01 bentonite specimens.

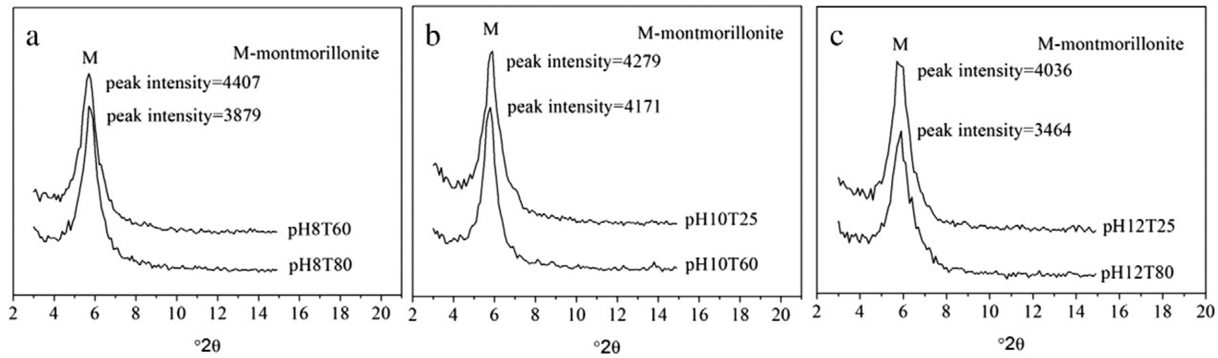


**Fig. 8.** Influence of temperature on the montmorillonite content in GMZ01 bentonite.

decreasing rate significantly depends on the pH value for higher temperatures. For a given pH, the swelling pressure decreases as temperature increases.

For a given temperature, the hydraulic conductivity of GMZ01 bentonite increases with the increase of pH value, and for a given pH value, a significant increase in hydraulic conductivity with the rise of temperature was observed. Moreover, the increasing rate depends on temperature.

XRD test results indicate that some montmorillonite of GMZ01 bentonite was dissolved after the contact with NaOH solutions and continued to decrease as the pH value increased, while the decreasing rate depending on temperature. These observations can well explain the test results of in terms of swelling pressure and hydraulic conductivity at different pH values of NaOH solutions and at different temperatures.

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