The Influence of γ-Rays Irradiation and Thermal Sequence Aging on the Mineral Composition of GMZ Sodium Bentonite

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Abstract. This paper aims to determine the impacts of γirradiation and thermal sequence aging on the mineral composition of Gaomiaozi natural sodium bentonite with Beishan groundwater and to broaden the assessment and scope of buffer materials for geological disposal of high-level radioactive waste (HLW). FTIR, XRD and Mössbauer spectra of the bentonite were studied that under accumulative radiation with doses at 3.0 MGy, hereafter heated at 90 ℃, 150 ℃, 170 ℃, 190 ℃ and 210 ℃ respectively continue 2 monthes, 4 monthes, 6 monthes, 8 monthes, 10 monthes and 12 months. In addition, the solubility of silicon in groundwater from Beishan area have been tested. The results indicate that, the content of montmorillonite decreased significantly with the increment of the thermal temperature and time. The concentration of SiO$_3^{2-}$ in groundwater increased obviously as the thermal temperature and time increased. There have the tendency that Fe (Ⅱtransformed to Fe(Ⅲ) in the bentonite after aged, however, the content of Fe(II) increased and the content of Fe(Ⅲ) decreased aged by γ irradiation. But the types of mineral and functional groups remain the same. All in all, the mineral composition of GMZ natural sodium bentonite have been varied after thermal aged, and further research is needed to comprehend specific sitiations.

1. Introduction
At present, it is suitable that the buffer/backfill material for deep geological disposal of high-level radioactive waste (HLW). Gaomiaozi bentonite deposit in Inner Mongolia have been selected as the preferred mineral deposit for buffer/backfill material for geological disposal of HLW in china[1]. As the last artificial barrier, the function of bentonite is supported and fixed the container of waste, homogenized the surrounding rock stress, proteced the seepage groundwater and migration of nuclide[2]. Due to the radioactivity and decay heat of HLW, radiation and heat effects have been existed in the near field of the repository for long-term. Bentonite with montmorillonite as the main mineral component may undergo mineral component transformation, the ability of swelling and ion exchange gradually
losing. Therefore its barrier properties will be weakened and affected the long-term safety of the repository 
[3-5].

In the aspect of irradiation aging, scholars R. Pusch [6], B. X. Gu [7], M. PIÖtze [8] and so on have 
carried out research on the influence of γ ray and electron beam on the properties of bentonite/montmorillonite. Due to the constraints of conditions in China, there are few studies on the effects of irradiation on bentonite. The research group carried out some exploratory research in the early stage [9-13].

Satoru Suzuki, S. Kaufhold, W. P. Gates et al. [14-16] conducted a related study on the temperature properties of bentonite components. In China, only Chen Bao and Ye Weimin from Tongji University [17, 18] have carried out the effects of alkali solution on the swelling, permeability and microstructure of GMZ bentonite. The results show that alkaline pore water will gradually dissolve the montmorillonite in the bentonite, which leads to an increase in the porosity of the bentonite and a decrease in the expansion force. However, little research has been reported on the effects of gamma irradiation-thermal sequential aging on the mineral composition of bentonite.

In this study, Gaomiaozi bentonite with a water content of about 17 wt. % was subjected to γ 
irradiation-thermal sequential aging under sealed conditions, and then the effect of irradiation-heat sequential aging on the mineral composition of Gaomiaozi bentonite was studied by XRD, FTIR and Mössbauer spectroscopy.

2. Experimental

2.1. Experimental materials

The research object of this experiment is the natural sodium bentonite (bentonite) of Gaomiaozi Mine in Xinghe County, Inner Mongolia, which is formed by alteration of tuff. The bentonite used in the experiment is gray-white powder with a particle size of less than 200 mesh. The main components and properties are listed in Table 1. The Beishan groundwater is the original groundwater sample obtained from exploration drilling in the Beishan area of Gansu Province. The main components are shown in Table 2.

| Mineral | Quartz | Montmorillonite | Albite | Feldspar | Zeolite | Mica |
|---------|--------|----------------|--------|----------|---------|------|
| composition/\% | ~16.0 | ~56.0 | ~14.0 | ~1.0 | ~13.0 | ~0.2 |
| SiO2 | ~70.0 | Al2O3 | ~13.5 | Fe2O3 | TiO2 | CaO | MgO | K2O | Na2O |
| EK | ~0.7 | ENa | ~3.5 | E1/2Ca2+ | E1/2Mg2+ | CEC | ~0.7 | ~31.5 | ~15.5 | ~5.5 | ~60.0 |
| True density /g/m3 | ~2.5 | ~19.0 | ~88.0 | ~80.0 | ~33.0 | ~220.0 |
| Methylene blue index/g/100g | ~19.0 | ~88.0 | ~80.0 | ~33.0 | ~220.0 |

Table 2 Composition of Beishan Groundwater (pH≈7.56)

| Ion species | Na+ | NH4+ | K+ | Ca2+ | Mg2+ | Fe2+ | Fe3+ | Al3+ | Li+ |
|-------------|-----|------|----|------|------|------|------|------|-----|
| Concentrations (mg/L) | 913.30 | 0.12 | 19.54 | 179.49 | 42.17 | 0.022 | 0.033 | 0.06 | 0.011 |
| Ion species | Sr2+ | Cu2+ | F- | Cl- | NO3- | SO42- | HCO3- | Br- | SiO32- |
| Concentrations (mg/L) | 0.715 | 0.0001 | 2.55 | 969.98 | 32.60 | 1364.15 | 130.9 | 0.0001 | 13.82 |

2.2. Instruments

60Co irradiation facility with dose rate is about 3.0 kGy/h; Suying PH-250 precision oven; Rigaku D/max2500 automatic X-ray diffractometer is manufactured in Japan; PE Lambda 750 UV-Vis-NIR spectrophotometer is fabricated in USA; Thermo S50 Fourier Transform Infrared Spectrometer is made in USA; Kuhner ISF4-X Temperature Controlled Oscillating Shaker is created in Swiss; WSS-10 Mössbauer Spectrometer is manufactured in Germany.
2.3. Experimental methods

Depending on the waste, the thickness of the container, and the spatial arrangement, the initial exposure dose rate of the outer surface of the container when the repository closed is about 0.2 to 2 Gy/h, and it is reduced by an order of magnitude in 100 years [19-20]. The maximum cumulative absorbed dose on the surface for 1000 years is about 0.7 MGy; the long-term cumulative absorbed dose is estimated at several MGy.

Referring to the Swedish and Finnish conceptual design models, the external surface temperature of the high-level waste container in the repository is not more than 90 ℃, and the thermal period of the repository is thousands of years [21]. However, the change of performance and structure of bentonite are not obvious under the condition of heat aging at 90 ℃[22]. In order to obtain the affected results as soon as possible, accelerated aging is needed. According to Arrhenius's law, a conservative 1000-fold acceleration is performed for 90 ℃ aging, and the basic accelerated heat aging simulation temperature is selected as 190 ℃, while retaining 90 ℃. The conditions were appropriately extended to the high and low temperature ranges according to the estimation results, and the heat aging temperatures of the test were finally determined to be 90 ℃, 150 ℃, 170 ℃, 190 ℃ and 210 ℃.

With the frozen mixing method, the water content of bentonite was adjusted to about 17 % with Beishan groundwater. The sample was evenly placed in a stainless steel aging container (φ120 mm×360 mm, wall thickness 2 mm). The aging container was placed in a large commercial 60Co irradiation facility. When the cumulative irradiation dose is taken up after 3 MGy, the aging container is placed in a precision oven at the corresponding temperature for heating aging, and the cumulative heating time is 2 months, 4 months, 6 months, 8 months, 10 months and 12 months respectively. Then the aging container was disassembled in a hypoxic glove box, and the sample was taken out and stored immediately.

3. Results and discussion

3.1. FTIR analysis results

Fig. 1 shows the infrared spectrum of the bentonite samples before and after aging. In the figure, about 3600 cm⁻¹ is the Al-OH stretching vibration peak; about 3400 cm⁻¹ is the H-O-H stretching vibration peak of the interlayer water molecule; about 2800-3000 cm⁻¹ is the asymmetric and symmetric CH₃, CH₂ and CH stretching vibration peak; about 1640 cm⁻¹ is the H-O-H bending vibration peak of interlayer water molecules; about 1040 cm⁻¹ is the interlayer Si-O-Si stretching vibration peak; about 840-920 cm⁻¹ is Al-Al-OH/Al-Fe-OH/Al-Mg-OH bending vibration peak; Si-O-Si stretching vibration peak at about 790 cm⁻¹; quartz peak at about 690 cm⁻¹; The bending vibrational peak of Si-O-Al in the lattice at 525 cm⁻¹ and the bending vibrational peak of Si-O-Si in the lattice at about 470 cm⁻¹.

After the cumulative dose of bentonite reached 3.0 MGy, even after aging at 210 ℃ for 12 months, the infrared spectrum was similar to the reference infrared spectrum, and no new characteristic peaks were generated in the spectrum, and no original features were found. The peak disappears, but the absorbance of some functional groups changes. In general, the Al-OH stretching vibration peak, the Si-O-Al bending vibration peak and the Si-O-Si stretching and bending vibration peaks in the lattice are more obvious, and the H-O-H stretching and bending vibration peaks are also reduced to some extent. However, the peaks of quartz and the bending vibration peaks of Al-Al-OH/Al-Fe-OH/Al-Mg-OH were not significant. It indicated that the Si-O structure and Al-O structure of the bentonite-related mineral components decreased after irradiation, while the quartz minerals were not significantly affected. At the same time, the hydroxyl groups also decreased slightly, and no significant changes were found in other structures.
In view of D. Gournis et al. [23], it was found that gamma radiation induced the formation of paramagnetic defects in montmorillonite structure; R. Pushkareva et al. [24] found that the irradiated clay minerals produced two defect centers, namely Si-O center and Al-O-Al center. L. M. Wang, S. X. Wang, B. X. Gu et al. [7, 25-27] found that mineral materials are prone to amorphization under the action of radiation, while C. Fourdrin et al. [28] found that the amorphous form makes montmorillonite lose large-scale crystal structure, and accompanied by the process of dehydroxylation, Stephanie Sorieul et al. [29] believed that the dehydroxylation process promoted the amorphous structure of the crystal structure. R. Pusch [30] found that bentonite at 125 °C and 150 °C will be converted into a bound silicate; J. J. Howard [31] and other studies have shown that at 250 °C, Al-rich salts will be formed in bentonite; R. M. Johnston [32], D. D. Carstea [33], P. Wersin [34] and M. Ochs [35] believe that Al-OH complexes are formed under high temperature conditions. Therefore, the mineralization and point defects caused by gamma radiation and the formation of substances related to the heat aging process are potential causes of changes in the absorbance of the relevant functional groups. In summary, from the results of infrared spectroscopy, irradiation-thermal sequential aging did not result in changes in the molecular functional groups of bentonite, but the content of functional groups changed.

3.2. XRD analysis results
As shown in Fig. 2 and Fig. 3, the radiation-thermal sequential aging causes a certain degree of change in the mineral component content in Gaomiaozi bentonite. In general, as the aging temperature increases, especially at the aging temperature ≥150 °C, the montmorillonite content decreases, the content of mica, quartz and albite increases slightly, and the zeolite and potassium feldspar decrease slightly; Prolonged, the content of montmorillonite decreased more obviously, while the content of other mineral components did not change significantly. At the higher aging temperature, the content of zeolite and quartz increased slightly.
Fig. 2 Curves of content for mineral component with temperature of GMZ Na-bentonite

Fig. 3 Curves of content for mineral component with time of GMZ Na-bentonite
Related literature [7,23-28] found that γ-irradiation induced defects and amorphous formation of montmorillonite structure, and the stability was poor. The possible causes of defects and amorphization are ionization and atomic displacement, and the doses of different minerals causing amorphous are different with temperature. A. Inoue et al. [36-38] found that montmorillonite is converted to stable silicates such as mica and zeolite under high temperature conditions, and the time effect of the process is significant. This is in agreement with the results of this study, so the change in the content of the above mineral components is most likely the result of the point defect caused by γ-irradiation induction and the superposition of montmorillonite in the process of amorphization and heat aging.

In view of the fact that the test object itself is a natural mineral, there is a certain degree of heterogeneity, that is to say, the content of mineral components of Gaomiaozi bentonite in different sampling lots and locations is not uniform, and there are some results in the Rietveld full-spectrum fitting results. Therefore, it is necessary to provide a clear result after comprehensive analysis with other test results.

3.3. Mössbauer analysis results

From the results of the Mössbauer spectrum analysis, it can be found that the content of Fe(II) and Fe(III) in Gaomiaozi bentonite has changed significantly after radiation-thermal sequential aging (Table 3). The content of Fe(II) increased slightly after γ irradiation, and the content of Fe(III) decreased slightly. The subsequent heat aging, with the increase of aging temperature and aging time, the content of Fe(II) decreased rapidly and the content of Fe(III) was rapid increase.

The iron present in bentonite can be divided into structural iron ions formed by replacing Al3+ in montmorillonite octahedron, iron oxides present on the surface of the structure, and iron ions coordinated with OH-coordinates at the edge of montmorillonite layer [8]. D. Gournis, M. Plotze and M. Holmboe et al. [8, 39, 40] showed that radiation under hypoxic conditions caused Fe3+ to Fe2+ conversion in montmorillonite structures. This is consistent with the results of this study, indicating that γ-ray irradiation leads to the conversion of Fe(III) to Fe(II) in bentonite; the subsequent heat aging process may be the residual oxygen in the container and some oxidizing substances in the bentonite to oxidize Fe(II) into Fe(III) leads to the conversion of Fe(II) to Fe(III) with the increase of aging temperature and aging time; in addition, some of the containers in this study have obvious corrosion and sealing failure, so the influence of corrosion products is also Possible factors for these changes occur, and potential outside air entry accelerates this change.

Table 3 The content of Fe(II) and Fe(III) in GMZ Na-bentonite after aged

| Cumulative dose/MGy | Time/month | Temperature/℃ | Content/% | Cumulative dose/MGy | Time/month | Temperature/℃ | Content/% |
|---------------------|------------|----------------|-----------|---------------------|------------|----------------|-----------|
| Reference           |            |                | 21.0      | 79.0                | Reference  |                | 21.0      |
|                     | -          |                | 21.4      | 78.6                | -          |                | 21.4      |
| 3.0                 | 90         | 8              | 20.7      | 79.3                | 4          | 210            | 14.2      |
|                     |             | 0              | 100.0     | 3.0                 |            |                | 210       |
| 210                 | 8          | 0              | 100.0     | 8                   | 210        | 0              | 100       |

3.4. Partial silicate solubility test results

The SiO32-concentration in the liquid phase was determined after the aging sample and the Beishan groundwater were fully applied.
It was found that the SiO32-concentration ratio in the Beishan groundwater after the aging sample was higher than that in the Beishan groundwater, and with the aging. As the temperature and aging time increase, the concentration of SiO32- in the liquid phase continues to increase (as shown in Fig. 4). The concentration of SiO32- in the Beishan groundwater with the cumulative exposure dose of 3.0 MGy increased by 8.4 %, which is consistent with the results reported in the literature, among which R. Pushkareva et al. [24] found that Si in montmorillonite after gamma irradiation. The solubility increases and the solubility of Al decreases. C. Fourdrin et al. [28] showed that the amount of Si dissolved in bentonite after irradiation was twice that of the blank sample, and the related literature [30, 41, 42] pointed out thermal aging. It will cause an increase in the solubility of Si in bentonite. It is indicated that gamma radiation-thermal sequential aging causes structural changes in mineral components in bentonite, resulting in increased solubility of certain Si-containing groups, showing significant aging temperature and aging time effects.

4. Conclusions
The cumulative dose of gamma radiation of Gaomiaozi sodium bentonite reached 3.0 MGy, followed by heat aging up to 210 °C for up to 12 months. The mineral components of bentonite changed significantly. With the increase of aging temperature and aging time, the montmorillonite content decreased, showing obvious aging temperature effect and aging time effect. After aging samples fully acted with Beishan groundwater, the concentration of SiO32- in the groundwater of Beishan is obviously increased, and it increases with the increase of aging temperature and aging time, indicating that aging causes structural changes of mineral components in bentonite, resulting in increased solubility of some Si-containing groups; The results of spectral analysis showed that the number of functional groups of bentonite changed after aging; Mössbauer analysis showed that γ-irradiation may lead to the transformation of Fe(III) to Fe(II) in bentonite, while thermal aging may cause mineral structure. The Fe(II) is converted to Fe(III).

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