The Effects of Different Nanoadditives on the Physical and Mechanical Properties of Similar Silty Mudstone Materials

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1. Introduction

In the experimental study on the ratio of similar materials in silty mudstone, gypsum is used as the binder, and barite powder is used as the aggregate to make similar materials. Because the physical and mechanical parameters of similar materials such as density, natural water absorption, uniaxial compressive strength, softening coefficient, and swelling are quite different from those of the silty Original rock, they cannot represent the physical and mechanical characteristics of the natural rock and affect the accuracy of model tests. The existing related research shows that choosing appropriate additives to incorporate into similar materials can improve the related physical and mechanical properties of similar materials of silty mudstone [1, 2]. In recent years, with the rapid development of nanotechnology, the production cost...
of nanomaterials has been decreasing year by year. At the same time, nanomaterials have shown physical effects that some macroscopic materials do not have, such as small size, large specific surface area, and high surface energy, and have been widely concerned [3]. The nanomaterials, often used as inorganic additives, are being widely used in housing, water conservancy, bridge, municipal, and other projects, which has achieved good application results [4–8].

At present, the appropriate amount of nanoadditives in the existing materials can effectively improve the relevant physical and mechanical parameters of the materials, formulate mixed materials suitable for different needs, and increase the scope of the original materials [9–14]. In terms of improving the cement substrate, Wang et al. found that adding nano-CaCO₃ into Portland cement can significantly improve the mechanical properties of cement-based materials and make the cement substrate more uniform. In improving the mechanical properties of cement-based materials, the nanoadditives mentioned above can effectively control the loss of liquid in cement when used separately, among which 3% nano-SiO₂ can reduce the amount of loss by 72%, with the most obvious effect. In terms of improving the performance of mixed materials, Anuj [21] found that, in the study of the properties of mixed materials, adding appropriate nanometer additives can not only promote hydration reaction but also play a role in filling the original micropores of the materials, thus improving the internal pore structure and significantly improving the physical and mechanical properties of the materials [22, 23]. In summary, the current research on the improvement of the physical and mechanical properties of related materials by different types of nanoadditives has obtained certain research results [24]. The incorporation of nanoadditives is mainly to improve the physical and mechanical properties of the materials, thereby increasing the range of materials used; however, about incorporating suitable nanoadditives, to improve the physical and mechanical properties of silty mudstone similar materials, studies on the effects of different types of nanoadditives on the physical and mechanical properties of similar silty mudstone materials are rarely involved [25, 26].

In order to deeply analyze the effects of different types of nanoadditives on the physical and mechanical properties of similar silty mudstone materials, based on the existing research results of similar silty mudstone materials [27, 28] on the basis of research results, using common NTi and NAl and NBe additive for silty mudstone similar to the analysis of the physical and mechanical properties of related materials, and reveal its micromechanism, the following are carried out. Firstly, the scheme of mixing ratio of similar materials with different dosage of nanoadditives was designed. The effects of nanometer additives on the density, natural water absorption, uniaxial compressive strength, tensile strength, softening coefficient, and expansibility of similar materials were analyzed, and the effects of different nanometer additives on similar materials were analyzed. Finally, the influence mechanism of nanoadditives on the microstructure of silty mudstone was revealed by SEM. It provides some guidance for the application of nanometer additives in similar materials.

## 2. Test Content

### 2.1. Sample Preparation

#### 2.1.1. Raw Materials. Similar to the materials of silty mudstone, barite powder is used as aggregate and gypsum is used as cementing material. Among them, the barite powder is produced by Hebei Lingshou, and its particle size is between 2 and 32 μm. And the chemical composition is shown in Table 1. The cementing material uses ultrahigh-strength gypsum with a particle size between 100 and 600 μm, and its chemical composition is shown in Table 2. The experiment used tap water for the laboratory.

#### 2.1.2. Nanometer Additive Material. The additives mainly include NTi, NAI, and NBe. NTi is a kind of white loose powder material with hydrophilicity, good dispersion, and weather resistance. The particle diameter is between 25±5 nm, the relative molecular weight is 79.9, the appearance is white, the specific surface area is 80–85 m²/g, and the purity is greater than 99.9%. NAI is a new type of ultrafine white crystal powder material with a particle size between 30 and 60 nm, a relative molecular weight of 101.96 nm, an average particle size of 50 nm, a specific surface area of 5–10 m²/g, and a purity greater than 99.99%. The particle size of the nanobentonite is between 40–80 nm, with an average particle size of 60 nm. It is a white and light yellow powder with a good ability to bind water. The main composition of the NBe is more than 90% montmorillonite and a small amount of illite, kaolinite, and other minerals.

#### 2.1.3. Preparation of Similar Materials. In order to study the physical and mechanical effects of nanomaterial additive (including NTi, NAI, and NBe) on the physical and mechanical effects of similar silty mudstone, the dosage of different types of nanometer additives was set as 0 (control group), 2%, 4%, 6%, and 8% in the sample preparation process, respectively. The mixing scheme of similar materials is shown in Table 3.

#### 2.2. Test Plan. In the process of testing the physical and mechanical properties of similar materials with different nanometer additives, the density, natural water absorption rate, uniaxial compressive strength, tensile strength, softening coefficient, and expansibility of the samples were mainly measured. The specific test plan is shown in Table 4 below. In order to analyze the influence mechanism of nanometer additives on the physical and mechanical properties of similar materials, uniaxial compression tests were carried out on the samples with different dosage of nanometer additives, and SEM tests were carried out on the typical damaged samples. Each group of samples is cured to a specified age in a standard curing box and tested according to the test regulations recommended by the international society for rock mechanics (IRSM) [29]. There are 5 parallel
samples in each group, and the average values of the 3 samples closest to each other are taken as the experimental results. Due to the large number of samples and batch production, only parts of the finished samples are shown (Figure 1).

2.3. Test Equipment. According to the test scheme in Table 4, different groups of the standard age samples with different types and dosage of nanoadditives were tested. During the immersion process, the temperature is controlled at 25 ± 5°C by using a three-use water tank (as shown in Figure 2(a)). After immersing the sample for 48 h, it is fully saturated. The device can better control the immersion time and water pressure; during the drying process in the experiment, place the cured standard test in an electric thermostatic drying oven at 60 ± 5°C (as shown in Figure 2(b)) for 48 h, then move it to a glass dryer to cool for 48 h (as shown in Figure 2(c)). The uniaxial compression test in this paper adopts the MTS-810 electrohydraulic servo system testing machine in the highway engineering experimental center of Changsha University of Science and Technology (as shown in Figure 2(d)) to load the sample. According to the engineering rock mass test method standard, the loading speed of 0.8 MPa/s is set to apply axial pressure to the sample.
Figure 1: Standard samples of similar materials with different contents of nanometer.

Figure 2: Test equipment: (a) three tanks; (b) electric constant temperature drying oven; (c) glass dryer; (d) MTS-810 electrohydraulic servo system testing machine.
3. Similar Material Optimization Experiment

3.1. Effects of Different Nanoadditives on Physical Properties of Similar Materials. Test the density of similar material samples with different nanoadditive dosages. The test results are shown in Figure 3. (1) With the addition of three nanoadditives in the same amount, compared with the control group, the effects of NAl on the density of similar materials are the most significant, followed by MBe and the minimum is NTi. (2) When the dosage of different nanoadditives increases from 0% to 6%, the density of similar materials increases with the continuous increase of the dosage of nanoadditives. (3) When the dosage is between 6% and 8%, similar materials are gradually increasing trend in density tends to 0. When incorporated into 8%, compared with the control group, NTi, NAl, and NBe, respectively, from 2.01 g/cm³ it increased to 2.16 g/cm³, 2.30 g/cm³, and 2.26 g/cm³. When 6% NBe is added, the density of similar materials is closest to the average density of silty original rocks [30]. It shows that all the three kinds of nanoadditives can effectively fill the pores between barite powder and gypsum particles so that the structure of similar material samples is more compact, and nanoadditives fully fill the pores of similar material samples, increasing the density.

3.2. Effect of Different Nanoadditives on Natural Water Absorption of Similar Materials. The natural water absorption rate reflects the water absorption capacity of dry similar material samples, which can effectively reflect the development degree of the material’s microcracks. According to the test requirements of “Road Engineering Rock Test Regulations” (JTG E41-2005) [26], the free water absorption method was used to determine the water absorption of similar materials with different types of nanoadditives. The test results are shown in Figure 3.

From the effect of different nanoadditive dosages on the natural water absorption of similar materials, as shown in Figure 4 (1) as the nanoadditive dosage increases, the natural water content of samples of similar materials tends to decrease first and then increase; (2) when the dosage is 4%, the natural water absorption of the similar material sample doped with NTi reaches the minimum, followed by NAl, and the maximum is NBe, which is reduced by 0.23%, 0.12%, and 0.08% compared with the control group; (3) when the doping amount is 8%, the natural water absorption of similar material samples doped with NTi, NAl, and NBe increases from 6.16% to 5.93%, 6.04%, and 6.15% respectively. However, when the doping amount is 6%, the natural water absorption rate of similar material samples is close to the average value of silty raw rocks. It shows that the incorporation of nanoadditives can effectively fill the pores of samples of similar materials and reduce the natural water absorption rate of samples. However, with the increase of nanoadditives, excess nanoadditive materials increase the natural water absorption rate of samples.

3.3. Effect of Different Nanoadditives on Expansion Rate of Similar Materials. The specific changes of the expansion rate of similar materials under different nanoadditive dosages are shown in Figure 4. It can be seen from Figure 5 that (1) the expansion rate of samples of similar materials is increasing with the increase of three nanoadditives, in which the addition of NBe has the greatest influence on the expansion rate of the sample, followed by the effect of NAl, and the effect of NTi is the least; (2) compared with the control group, when 8% NTi, NAl, and NBe are added, the expansion rates of similar material samples increase by 0.028%, 0.035%, and 0.078% in sequence; (3) the expansion rate of silty primary rock is about 0.16%, while the similar material after incorporation of 6% NBe is 0.16% close to the original rock. It shows that the incorporation of nano-NBe has the most significant effect on increasing the water absorption expansion rate of similar materials mainly because the NBe itself has strong water absorption expansion characteristics.

3.4. Effect of Different Nanoadditives on Mechanical Properties of Similar Materials

3.4.1. Effect of Different Nanoadditives on the Uniaxial Compressive Strength of Similar Materials. The MTS-810 electrohydraulic servo system test machine was used to perform uniaxial compressive test on similar materials with different amounts of nanoadditives. Then, select the typical stress-strain curve, the average peak stress, and the typical sample failure form from each group for analysis.

It can be seen from Figures 6(a)–6(c) the curves under the three nanoadditives. Under the condition of incorporation of nanoadditives, the stress-strain curves of all the samples are roughly similar, and all of them undergo the compaction stage; in the elastic stage, plastic deformation stage, and strain softening stage, and when the dosage is 4%, the uniaxial stress of the sample is the largest. When the same nanoadditive is incorporated, for example, when NTi is incorporated into 0%, 2%, 4%, 6%, and 8%, the peak corresponding strains are 1.13%, 1.76%, 1.33%, 1.81%, and 1.31%, respectively. The maximum peak stress is 4.93 MPa, 6.14 MPa, 9.50 MPa, 9.70 MPa, and 6.80 MPa. Compared with the control group, no matter which kind of nanoadditive is added in an appropriate amount, it can improve the uniaxial compressive strength of similar materials to a certain extent.

It can be seen from Figure 6(d) that, under the condition of doping the same nanoadditives, with the increase of the amount of nanoadditives, the uniaxial compressive strength of similar materials showed a trend of first increasing and then decreasing. When it is 4%, the average value of uniaxial compressive strength of similar materials reaches the maximum. It shows that the proper addition of nanoadditives has obvious improvement effect on the uniaxial compressive strength of similar materials, but not as much as possible. There is an optimal dosage of about 4%. The maximum values of NTi, NAl, and NBe are, respectively, 9.70 MPa, 10.58 MPa, and 9.43 MPa.

According to the above stress-strain curve of the sample and the test after the sample is mixed with different nanoadditives, combined with the failure morphology of multiple sets of nanoadditives during the test, it is found that
the damage morphology of the sample is mainly affected by nanoadditives which vary considerably and affect the dosage, and the different types of nanoadditives incorporated shape of specimen destruction of substantially identical, and the sample was mostly dominated shear failure. Taking into account the limited space, the following will take Nb as the analysis object; typical damage photos are shown in Figure 7.

According to the failure morphology and stress-strain curve of similar material samples after uniaxial compression failure under different Nb contents, as the Nb content increases, the sample cracks crack from the stressed end of the sample and continue to extend to the bottom. The more obvious the sudden stress drop when the specimen fails, the more obvious the shear failure characteristics. When the dosage is 6%, the destruction characteristics of similar materials in Figure 7(e) are basically the same as those in Figure 7(a).

3.4.2. Effects of Different Nanoadditives on the Tensile Strength of Similar Materials. Figure 8 shows the effect of different nanoadditives on the tensile strength of similar materials. It can be seen from Figure 7 that, as the amount of nanoadditives increases, the tensile strength of similar materials increases first and then decreases. When the amount of nanoadditives is 4%, the tensile strength of similar materials reaches the maximum, and NTi, NA1, and Nb are added the maximum value, respectively, is 2.14 MPa, 2.31 MPa, and 2.41 MPa. Later, as the amount of nanoadditives continued to increase, the tensile strength of similar materials decreased significantly. It shows that the incorporation of nanoadditives has a very significant effect on the tensile strength of similar materials; when 6% Nb is incorporated, the tensile strength of similar materials is taken to the average of silty raw rocks.

3.4.3. Effects of Different Nanoadditives on the Softening Coefficient of Similar Materials. Figure 9 shows the effect of blending different nanoadditives on the softening coefficient of similar materials. It can be seen from Figure 8 that the incorporation of different nanoadditives reduces the softening coefficient of similar materials. As the amount of nanoadditives increases, the softening coefficient of similar materials continues to decline, but the decline becomes slow. In the process of increasing the additive content from 0% to 4%, the softening coefficient of similar materials is significantly affected; from 4% to 8%, the softening coefficient
gradually stabilizes, and finally the softening coefficient of similar materials after NTi, NAl, and NBe decreased by from 0.70 to 0.51, 0.52, and 0.54; when incorporated NBe is 6%, softening coefficient similar material is closest to the average value of the original rock silty.

In summary, the test results of the density, natural water absorption rate, swelling rate, uniaxial compressive strength, tensile strength, and softening coefficient of similar materials after mixing different nanoadditives can be seen; with the addition of nanoadditives, silt physical and mechanical properties of mudstone similar materials change significantly. When doped with 4% nanoadditives, it can effectively improve the related physical and mechanical properties of similar material samples can reach or be closer to the silty raw rock except uniaxial compressive strength. The failure form of the axial compression specimen is also consistent with that of the original rock and can be used as the best choice. When the 8% nanometer is added immediately, the physical and mechanical properties of the silty mudstone similar materials will change obviously, and it is quite different from the parameters of the silty raw rock.

4. Microstructure and Analysis of Similar Material Samples

The nanometer additive added has high surface energy, and the high surface energy effect of nanometer additive in similar materials can be brought into play when added in an appropriate amount. The nanoparticle materials can play the following three effects [31–35]: (1) Size effect: selected raw materials of various particle sizes are above 2 μm, while additive nanometer average particle size of additive is between 10 and 100 nm. Because of the smaller geometric size
of nanosized particles, they have stronger adsorption. Adding proper amount of nanometer material into the mixture showed better dispersion. Because the size of nanoparticles is small and the specific surface area is larger, when mixing too much, the particles will adsorb each other and tend to aggregate. (2) Filling effect: the appropriate amount of nanoadditives can optimize the particle gradation of the mixed material, and the nanoparticles have the function of filling the pores, displacing the water in the voids of samples of similar materials, reducing the porosity, and making the maximum pore size and average of the similar materials. The pore size is significantly reduced, improving the internal pore structure of similar materials, resulting in more uniform and dense similar materials. (3) Surface effect: it can be seen from the hydration of gypsum that the barite powder particles in similar materials are wrapped by the gypsum hydration product. The nanoadditive particles are extremely fine and the specific surface area is extremely large, which increases the contact surface with water. The surface of the nanoadditive has high activity and can adsorb a large amount of fine particles, which promotes the hydration reaction of gypsum. As shown in Figure 10, the microscopic structure of similar materials is now observed under 300 times electron microscopy at 0%, 2%, 4%, and 8% of NBe, and the microstructure of similar materials is revealed when nanoadditives are added an influence mechanism.

According to Figure 10, it can be seen that, under the scanning electron microscope at 300 times, the mesostructure structure of similar materials is in silty mudstone under different NBe dosages. As shown in Figure 10(a), when it is 0%, a network structure is formed inside the similar material, and the mesostructure is loose, containing a large number of holes and cracks. From Figure 10(b), when the NBe content is 2%, the internal network structure of similar materials is gradually dense, and the pores of the mesostructure are filled in a large amount, but there are still a lot of pores and cracks. From Figure 10(c), a similar material doped with 4% NBe has the best effect of improving the structure interface and the structure is more dense. The gap between the barite powder and the gypsum hydration product can hardly be seen in the interface transition area. According to Figure 10(d), when the content of NBe is 6%, the structure interface has the best improvement effect, and the structure is more uniform and dense, and the interface transition area is even and smooth, with only a few holes. It shows that the appropriate amount of nanoadditives can promote the hydration reaction of internal gypsum and interweave with the hydration products, thereby forming a dense whole, making the mesostructure of similar materials more uniform and dense. As shown in Figure 10(e), 8% NBe similar material is blended. Because a large amount of nanoparticles will agglomerate, in similar materials, gypsum hydration products will wrap the barite powder particles and nanoagglomerates, and the internal structure will generate a large number of uneven pores. Destroying the dense structure inside similar materials leads to the deterioration of the mechanical properties of similar materials and the decrease of the softening coefficient.

The above analysis revealed the influence mechanism of nanoadditive dosage on the microstructure of similar

**Figure 7:** Shear failure patterns of similar material samples with different NBe contents (water content are controlled at 10% to 12%).

**Figure 8:** Effects of nanoadditives on uniaxial compressive strength of similar materials.
materials, and the following conclusions were drawn: (1) in the control group, only gypsum and water hydration reaction occurred, and a large number of gypsum crystals were generated and interlaced into a network structure. After cementation, there was a certain strength, and the cementation product would have a large number of pores. The mixture of gypsum and barite powder was even. After hydration reaction of gypsum, the barite powder particles were wrapped by cementation, which could improve the compactness, but there would still be a large number of pores and the sample density was small. In this process, the macroscopic mechanical properties of similar materials are mainly affected by cementation. (2) When 2%–6% content is added, on the one hand, nanoparticles can promote the hydration

Figure 9: Effect of nanoadditives on softening coefficient of similar materials.

Figure 10: SEM photos of similar material samples with different NBe contents: (a) the structure is loose and contains many holes; (b) the structure gradually compacted and a large number of holes became smaller; (c) the surface is denser and the structure contains more pores; (d) the interface of the structure is smooth and dense with only a few holes; (e) the structure interface is agglomerated and there are many holes (×300 times).
reaction of gypsum and accelerate the production of cement. On the other hand, the right amount of nanoparticles can fully disperse and fill the pores in similar materials, reduce the water in the pores, and make the materials more dense, and the sample density gradually increases. The macroscopic mechanical strength of similar materials corresponding to this process is further improved. (3) When 6%–8% nanoadditive is added, the nanoparticle agglomeration occurs. The strength of the sample after the nanoparticle agglomeration decreases, and it becomes a primary defect in the similar materials after cementation. Besides, there is a large gap between the nanoadditive particles and the surrounding hydration products, with a poor interface. In this process, the macro-mechanics of similar material samples weakened to some extent, but too many nanoadditives can still further improve the density, expansion rate, and natural water absorption of similar material samples.

5. Conclusions

(1) Compared with the control group, with the addition of nanoadditives, the physical and mechanical properties of similar materials of silty mudstone changed significantly. When 4% nanoadditive is added, the physical and mechanical properties of similar materials can be improved effectively. When 8% nanoadditive is added, the mechanical properties of silty mudstone similar materials deteriorate obviously. This is because adding nanometer additive can effectively promote the hydration reaction of gypsum, accelerate the production of cement, fill the pores of similar materials, reduce internal defects, and make similar materials more compact. When too much is added, the nanoadditive will aggregate and degrade, which will reduce the mechanical properties of similar materials.

(2) When the content of NBe is 6%, all the physical and mechanical parameters of similar materials, except the uniaxial compressive strength, can reach or be closer to the silty original rock, and the failure morphology of uniaxial compression samples is consistent with that of the original rock, which can be used as the best choice. Therefore, appropriate incorporation of NBe can effectively improve the similarity between similar materials and silty rock.

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 they have no conflicts of interest.

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