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Effects of Aqua-Dispersing Nano-Binder on Clay Conductivity at Different Temperatures

Cuiying Zhou [1,2,3], Xingxing Ge [1,2,3], Wei Huang [1,2,3], Dexian Li [1,2,3] and Zhen Liu [1,2,3,*]

[1] School of Civil Engineering, Sun Yat-sen University, 135 XinGangXiLu, Guangzhou 510275, China
[2] Guangdong Engineering Research Centre for Major Infrastructure Safety, School of Civil Engineering, SunYat-sen University, Guangzhou 510275, China
[3] Research Center for Geotechnical Engineering and Information Technology, Sun Yat-sen University, 135 XinGangXiLu, Guangzhou 510275, China
* Correspondence: liuzh8@mail.sysu.edu.cn

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Abstract: Soil nutrients are the basis of ecological remediation. Soil amendments can form a reticular membrane structure on the soil surface to increase nutrient storage and alleviate nutrient imbalances, and are affected by the environmental temperature. At present, the qualitative evaluation of the effect of soil amendment is mainly based on vegetative growth. However, with the increasing use of soil amendments, how to conveniently and quantitatively evaluate the impact of soil amendments on ecological restoration under different temperature conditions from the perspective of soil urgently needs to be solved. Therefore, a new soil amendment named aqua-dispersing nano-binder (ADNB) and silty clay that is commonly used for ecological restoration in South China were used as research subjects, and the important soil nutrient storage capacity—soil conductivity index—was used as the starting point to find solutions to the above problems. We independently developed a multifunctional instrument to measure the soil amendment concentration. Clay conductivity measurements were used by adding different concentrations of ADNB within the range of 0 to 50 °C, and the mechanism by which temperature and ADNB affect the conductivity of clay was revealed. In addition, the quantitative relationship between the clay conductivity, ambient temperature and concentration of ADNB was elucidated. According to the growth conditions of *melinis minutiflora* and *pigeon pea* under different concentrations of ADNB, the optimal ADNB concentration needed to improve ecological restoration was obtained, which provided a new way to evaluate the effects of the large-scale use of soil modifiers on ecological restoration.

Keywords: temperature; aqua-dispersing nano-binder; clay; soil amendment; optimal concentration

1. Introduction

Soil nutrients are the basis of ecological remediation. Soil amendments can form a reticular membrane structure on the soil surface to solidify the soil, which can increase nutrient storage and alleviate nutrient imbalance, so these amendments play an important role in improving ecological remediation [1,2]. Soil nutrients are made up of a variety of salts in the form of ions. In describing the soil salinity, soil conductivity is commonly used. When serious problems such as soil pollution, heavy metal accumulation and salinization are not considered, soil conductivity represents the status of inorganic salt nutrients such as N, P and K in soil. Thus, soil conductivity is the main index reflecting the storage of nutrients in the soil [3–6]. In fact, pH is also an important parameter to evaluate soil nutrient storage, but the influence law of pH on soil is not obvious due to comprehensive factors such as discrete, soil particles and so on. So, the emphasis is on conductivity in this study.

At present, soil amendments consist of three main types: artificial types using polymers, natural types using organic and inorganic substances, and biological types using mycorrhizae [7–11]. Due to the good water absorption and water release characteristics of soil amendments, the proportions of water, heat and gas in the soil are effectively optimized and adjusted to affect the moisture status, bulk density, aggregate structure and strength, especially soil nutrients [12–19]. Researchers have used many methods to study the effect of soil amendment on soil ecology. Liu et al. carried out an unconfined compression test, direct shear test, water stability test and surface erosion test to explore the impact of new organic polymer soil stabilizer named STW amendment on sloped soil in the laboratory [20,21]; Zhang et al. observed the growth of Yang chai during the growing season for 4 months to explore the effects of a soil amendment named darkadolithon on the physicochemical properties of sandy soil [1]. These methods had obvious effects but due to the various experimental processes and long times required, we cannot use past results to evaluate the effects of soil amendments and make improvements to decrease test costs and difficulty. Thus, we need to find a simple and rapid method for these evaluations, and the soil electrical conductivity test is an important breakthrough to this end, as it can reflect the degree to which soil amendment influences soil nutrient sand it employs simple measurements. Some scholars have previously performed tests to measure soil conductivity. For example, Liu et al. determined the change in soil conductivity under different salinities with respect to the relationship between conductivity and soil salinity test results [3], but the influence of temperature on soil conductivity was ignored. This result did not reflect the reality that temperature also changes soil conductivity. Moreover, the test was performed with soil only, and soil amendment was not taken into account. Therefore, finding the trends and the mechanisms of soil conductivity changes under different temperature conditions is a simple, effective and low-cost way to study the effect of soil amendment on ecological restoration and improve the performance of a soil amendment. Ni et al. integrated a field monitoring and computational modelling approach, then found the effects of soil water content and temperature on electrical conductivity in vegetated soils and built a model that could predict nonlinear relationships between electrical conductivity and soil temperature and water content [22]. Our model was based on the foundation.

To address the above mentioned issues, a new soil amendment named aqua-dispersing nano-binder (ADNB) and silty clay, which are commonly used for ecological restoration in South China, were used as research subjects, and the important nutrient storage capacity—the soil conductivity index—was used as the starting point to do related experiment. Although we independently developed a multifunctional instrument to measure the soil amendment concentration, clay conductivity measurements were used by adding different concentrations of ADNB within the range of 0 to 5 °C to study the effects of ADNB amendment on ecological restoration.

2. Materials and Methods

2.1. Test Materials

In a previous study on soil amendment, it was found that the ADNB used on building plates had the same effect as a soil amendment [23]. The material is a white emulsion at room temperature, as shown in Figure 1. In addition, we conducted experiments on many side slopes in South China and found that ADNB has an improvement effect by comparing the growth conditions of plants and soil conditions on the side slopes. Meanwhile, ADNB can reduce the compactness of the soil and form a reticular membrane structure on the soil surface to increase nutrient storage, which can accelerate the growth of the root length of the plants, and it can promote the activity of soil microflora from the current research. The degradation time of ADNB is approximately 24 months, and the final products are water and carbon dioxide. There are no contaminants in the final products [24,25]. So, it can be used for ecological remediation. The major component of ADNB is polyvinyl acetate with a pH value of 6 to 7, a density of 1.01 g/cm^3, and a molar mass of 6×10^6 g/mol, and its solid content is 41%. ADNB is insoluble but has good dispersity in water and can be configured into an aqueous solution.

Figure 1. Aqua-dispersing nano-binder (ADNB).

The soil used in this study is Quaternary silty clay in South China. It is mainly composed of kaolinite and illite, but also contains small amounts of chlorite, montmorillonite, calcium feldspar and quartz as found by X-Ray Diffraction (XRD) test, as shown in Figure 2, and Table 1 summarizes the basic physical properties of the soil.

Figure 2. Silty clay used in this study (scale bar 1:4).

Table 1. Physical properties of silty clay.

Proportion	Plastic Limit %	Liquid Limit %	Maximum Dry Density g/m^3	Optimum Moisture Content %
2.70	14.10	25.76	1.864	15.10

2.2. Research Equipment

To study the effects of soil amendment on ecological restoration, we developed a multifunctional instrument to measure the soil amendment concentration according to the relationship between soil conductivity and the concentration of the soil amendment, as shown in Figure 3. The soil conductivity can be measured automatically, and the change in the concentration of the soil amendment in the soil can be displayed by the programmable system according to the relationship between the soil conductivity and the concentration of the soil amendment. Its conductivity sensor is a kind of dielectric type sensor, which determines the dielectric constant or permittivity of the medium by measuring the variation of the capacitance on the sensor. The sensor has four electrodes. When the probe of the conductivity sensor is inserted into the soil, two external electrodes provide a current, and then the voltage between the two internal electrodes is measured to obtain the soil conductivity according to Ohm's law. The operating parameter of the conductivity sensor is as follows: the measuring range is 0 to 10,000 µs/cm, and the measuring accuracy is 10 µs/cm. It can quantitatively analyze the change in the concentration of the soil amendment in soil within a certain range to reflect the effect of the soil amendment on ecological restoration.

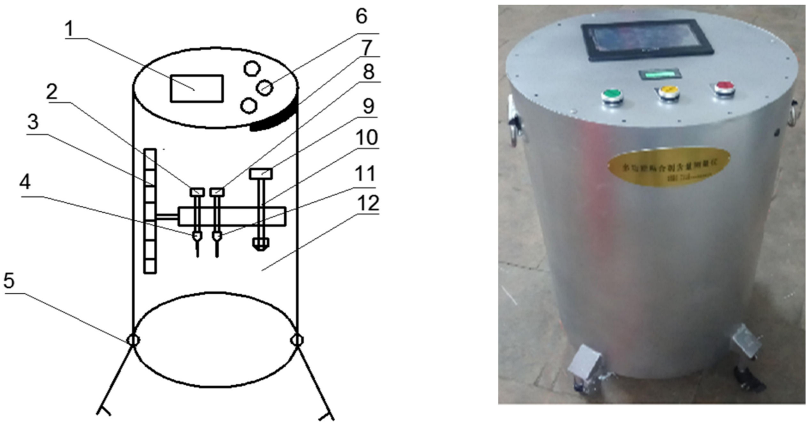


Figure 3. The multifunctional instrument developed to measure the soil amendment concentration. (1—data display screen; 2, 8, 9—servo motor; 3—up and down feed distance controller; 4—soil conductivity sensor; 5—movable support; 6—control panel; 7—programmable system; 10—up and down motion controller; 11—pH sensor; 12—cylindrical body).

2.3. Test Methods and Process

To study the influence of ADNB on the clay conductivity under different temperature conditions, according to the annual temperatures in South China, which range from 0 to 50 °C, temperatures of 0, 10, 20, 30, 40 and 50 °C were set to carry out the clay conductivity measurement with different concentrations of ADNB. At the same time, considering that the clay conductivity at different temperatures may be related to pure water, ADNB and pure clay itself, we also measured the conductivity of pure water, ADNB and pure clay to study the relationship among them.

For the pure water conductivity test, pure water (the conductivity is 7.4 μs/cm at room temperature) was subjected to the temperature gradient for four hours, and then its conductivity was measured.

For the ADNB conductivity test, pure water (the conductivity is 7.4 μs/cm, and it can be ignored within the range of error tolerance and instrument measurement accuracy, and interference ions can be eliminated) was used as a disperse carrier; the ADNB and pure water were fully mixed by stirring with a SY-5LM1S agitator. When the ADNB was evenly dispersed in the water and the mixture looked transparent, it was poured into a mold, oscillated slightly to remove bubbles and then sealed to prevent water evaporation. Then, 7 L water and 30 g ADNB were used to configure seven groups of test samples, i.e., 1, 2, 3, 4, 5, 6 and 7 g/L, as shown in Figure 4, and they were placed in a temperature-controlled incubator under a set temperature gradient, as shown in Figure 5. The constant-temperature incubator simulates the actual engineering environment well, making the experiment more realistic.

Figure 4. Different concentrations of ADNB.

Figure 5. ADNB was placed in a constant-temperature incubator to conduct the experiment.

For the pure clay conductivity test, silty clay samples were poured into an oven and baked to constant weight (weighed every 30 min until the weight error did not exceed 0.1 g). To ensure the uniformity of the clay samples, the samples were screened to obtain clay particles with diameters smaller than 0.25 mm through a 0.25 mm sieve. Then 30.2 g pure water and 200 g selected clay particles were mixed to prepare slurry with a water content of 15% (the optimal water content), and the slurry conductivity was measured at different temperatures. The same moisture content was adopted for the soil samples in this test, eliminating the influence of the difference in the water content of the soil sample on the conductivity measurement. For the clay with ADNB conductivity test, the aforesaid soil paste and ADNB were stirred well by a glass rod and were placed in the constant-temperature incubator under a set temperature gradient to conduct the constant-temperature experiment. The conductivity was measured immediately at the corresponding temperature. To ensure reproducibility, each of the above experiments was performed three times and standard deviations were calculated.

3. Results

The Influence of ADNB on the Conductivity of Clay at Different Temperatures

When the temperature increased from 0 to 50 °C, the conductivity of pure water increased, but this increasing trend was not obvious, and the water conductivity was low; the maximum water conductivity was just 0.00753 ms/cm, so the effect on clay conductivity can be ignored. However, when the temperature increased in the range of 0 to 30 °C and 40 to 50 °C, the conductivity of ADNB at different concentrations, pure clay, and clay with ADNB all showed an increasing trend, which decreased to some extent when the temperature was between 30 and 40 °C, as shown in Figure 6 (the average of the three experiments). According to Figure 7 (the average of the three experiments), the conductivity of the clay with ADNB increased with the increase in the concentration of the ADNB. This indicated that the conductivity of clay with ADNB was related to the ADNB and clay itself. Those experiments were repeated three times. The standard deviation ranges of the conductivity of pure soil, pure ADNB, and clay with ADNB were 0.0004 to 0.0021 ms/cm, 0.0004 to 0.0021 ms/cm, and 0 to 0.002 ms/cm, respectively. Each of them is negligible compared to that of the samples, which shows that the reproducibility of the experiment and the experimental data are reliable.

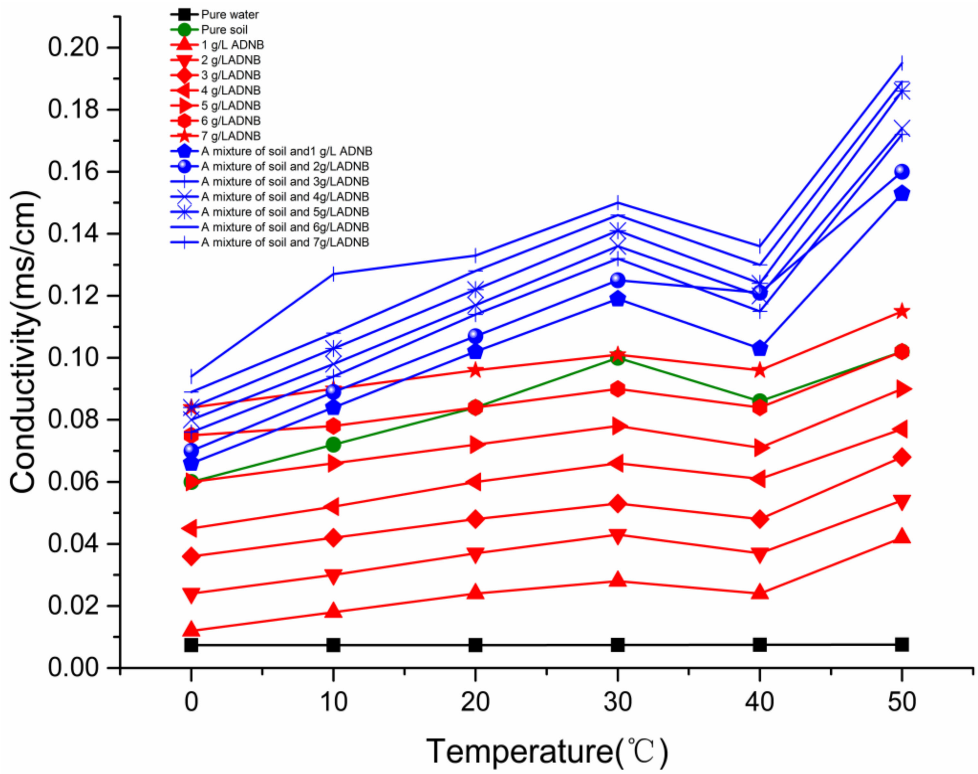


Figure 6. Comparison of various conductivity changes with temperature under different concentrations of ADNB.

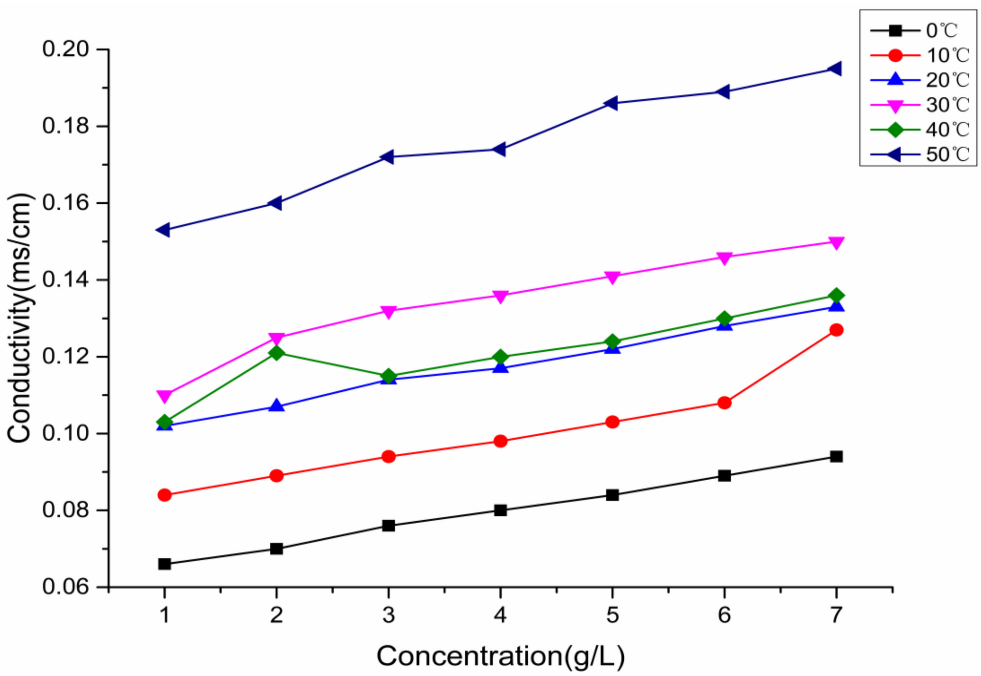


Figure 7. The relationship between the conductivity of clay with ADNB and the concentration of ADNB at different temperatures.

4. Discussion

4.1. The Relationship Between the Conductivity of Clay with ADNB, Temperature and the Concentration of ADNB

The conductivity of clay with ADNB increased with the increase in the concentration of ADNB and the temperature in the range of 0 to 30 °C and 40 to 50 °C, but it decreased in the range of 30 to 40 °C, as shown in Figure 8 (the average of the three experiments). However, there was a quantitative relationship among the three factors.

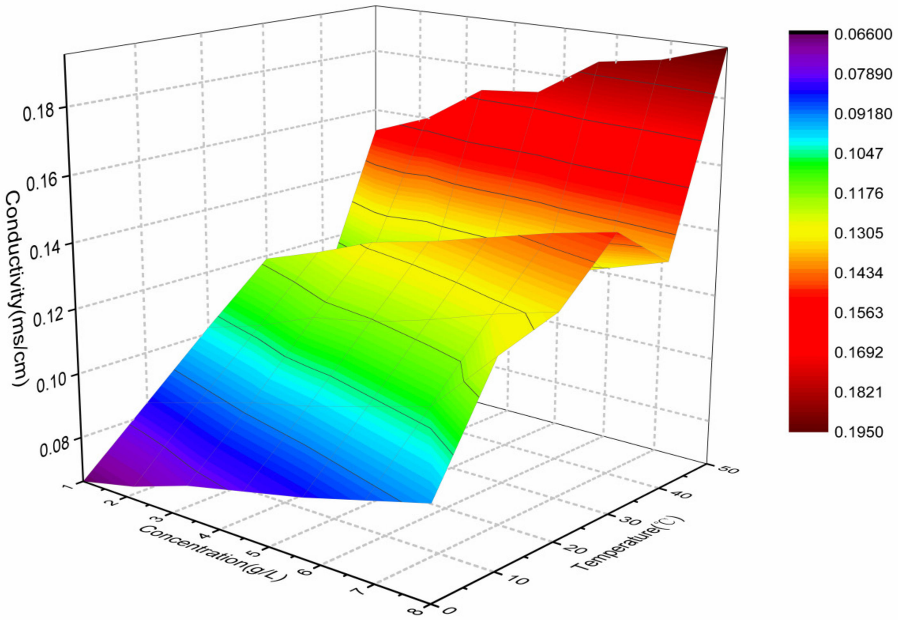


Figure 8. The three-dimensional relationship of the conductivity of clay with ADNB, the temperature and the concentration of ADNB.

According to the statistical modelling theory proposed by scholars when studying the effect of plant roots on soil conductivity to determine the magnitude of soil conductivity [22], the following relational model was derived:

$$EC = a_{00} + a_{01}T + a_{02}M + a_{11}T^2 + a_{22}M^2 + a_{12}TM \tag{1}$$

Here, EC represents the output response of clay conductivity, the temperature T and concentration M are the design variables, and a_{00}, a_{01}, a_{02}, a_{11}, a_{22} and a_{12} are the model coefficients. According to the analysis and calculation of the three-dimensional relationship, the model is as follows:

(1) When $T \in [0°C, 30°C) \cup (40°C, 50°C]$:

$$EC = 0.61 + \left(T, M, T^2, M^2, TM\right)\begin{pmatrix} 1.78 \times 10^{-3} \\ 4.6 \times 10^{-3} \\ 2.6 \times 10^{-6} \\ 0 \\ 1.7 \times 10^{-5} \end{pmatrix} \tag{2}$$

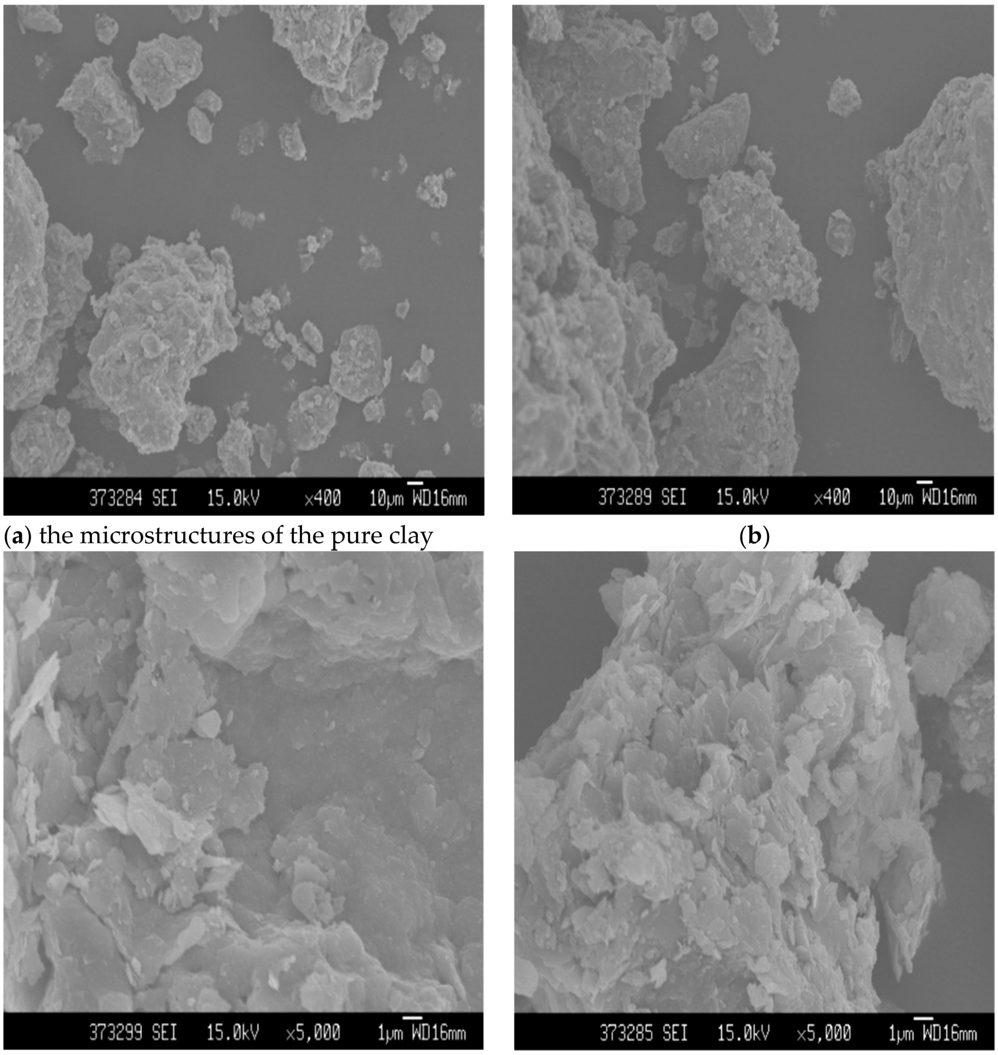
(2) When $T \in [30°C, 40°C]$:

$$EC = 0.18 + \left(T, M, T^2, M^2, TM\right)\begin{pmatrix} -2.6\times10^{-3} \\ 4.7\times10^{-3} \\ 1.4\times10^{-5} \\ 0 \\ 1.7\times10^{-5} \end{pmatrix} \quad (3)$$

Due to the limitation of research object and environmental factor, this model is applicable in South China, where silty clay is dominant in the temperature range of 0 to 50 °C, and the concentration of ADNB in the range of 0 to 7 g/L is appropriate according to the requirements of field construction. We will continue to explore other soil and temperature ranges to provide a method reference for exploring ecological restoration means in the Gobi region, in the northwest of the Loess region.

4.2. Comparative Analysis of the Microstructure of Pure Clay and Clay with ADNB

To observe the change in clay microstructure, the clay was poured into a circular vessel to be formed, and then it was cut into pieces. Finally, the clay slices were dried for Scanning Electron Microscope(SEM)experiments. Figure 9a shows the microstructures of the pure clay with SEM under a magnification of 400 times, and we could observe that the soil particles randomly disperse and their approximate diameters were in the range of 0.01 to 0.25 mm. Figure 9b shows the microstructures of the clay with ADNB under a magnification of 400 times, and we could observe that the number of fine soil particles (0.01~0.1 mm) decreased, but the number of coarse soil particles (>0.2 mm) increased. Comparing Figure 9a,b, the loose, fine soil particles were combined into larger-sized particles by the cohesive effect of ADNB, which made the structure compacted and tighter. Figure 9c shows the microstructures of the pure clay with SEM under a magnification of 5000 times, and we could observe that overall structure of the clay supported the formation of heterogeneous pore structures on the surface. Figure 9d shows the microstructures of the clay with ADNB with SEM under a magnification of 5000 times, and we could observe that the overall structure of the clay with ADNB was a mosaic structure, and there was a large amount of fine cement content on the clay surface. Comparing Figure 9c,d, the ADNB increased the content of cement that binds fine particles in the clay and formed a type of film to change the structure and increase the strength of the clay. By comparing the microstructures, we could speculate that the ADNB penetrated into the clay and attached to the surfaces of skeleton particles and the contact positions of particles to form an elastic and viscous film that could store nutrients and reduce the degree of nutrient loss.



(**a**) the microstructures of the pure clay (**b**)

(**c**) (**d**)

Figure 9. SEM images of the microstructural changes of clay with or without the addition of ADNB:(**a**) the pure clay under a magnification of 400 times;(**b**) the clay with ADNB under a magnification of 400 times;(**c**) the pure clay under a magnification of 5000 times;(**d**) the clay with ADNB under a magnification of 5000 times.

4.3. Effect Mechanism of ADNB on Soil Conductivity

ADNB exists in the form of latex granules and is evenly dispersed in water; the latex particles contain polymer chains [24]. When ADNB was mixed into the clay, on the one hand, the surfaces of the clay particles were wrapped by the polymer chains of the latex particles and were connected with each other to form elastic and sticky films, as shown in Figure 10. However, there were carboxyl groups (COOH) and hydroxyl groups (OH) on the polymer chain, which reacted with the positive ions on the clay particles and generated physical and chemical bonds (hydrogen bonds or van der Waals forces) to improve clay cohesion [26]. Meanwhile, the cohesion effect is also due to the electrostatic forces by the charges from clay surface and ADNB. At the same time, the increase in temperature accelerated the evaporation of soil water to enhance the interaction between clay particles and accelerate the diffusion of the polymer chains of the latex particles. Therefore, the protective film on the clay surface was thicker and the cohesion was greater due to the ADNB and the environmental temperature, which is good for soil nutrient storage, so soil conductivity increased. However, the clay conductivity did not always increase with increasing temperature. The conductivity decreased to some extent when the temperature ranged from 30 to 40 °C. The reason for this decrease may be that the ADNB was affected by the reduced physical and chemical reactivity of soil within this temperature range.

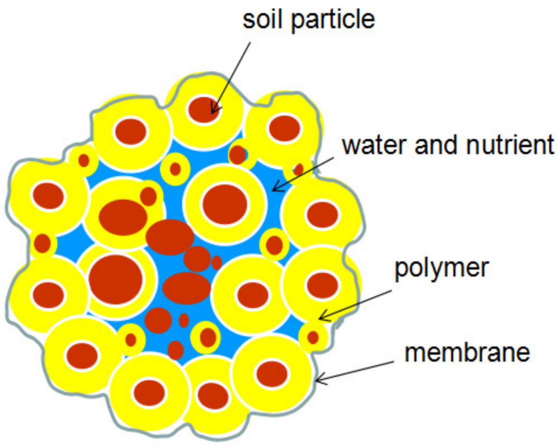


Figure 10. Diagram of the action mechanism of ADNB in clay.

4.4. Analysis of the Optimum Ratio of ADNB Suitable for Growing Plants

From the previous analysis, it could be seen that the higher concentration of ADNB was relatively more beneficial to the clay in terms of nutrient storage and improvement in the ecological restoration performance. However, higher concentrations are not more conducive to plant growth. To determine the best concentration of ADNB, we used *melinis minutiflora* and *pigeon pea*, which are common as research subjects in South China. We cultivated the plants and observed their growth trends (the plant growth height was the indicator used) under different concentrations of ADNB (the soil used for the plant growth experiment was all modified clay) at a constant temperature of 20 °C (the growing season of plants in South China is spring, during which time the average temperature is approximately 20 °C). It can be seen from Figure 11 (the average growth heights of the two plants) that *melinis minutiflora* and *pigeon pea* showed increasing growth with ADNB application in the range of 0 to 5 g/L, but growth gradually decreased to the range of 5 to 7 g/L, which suggested that the optimal concentration of ADNB for plant growth was 5 g/L. The reason for this is that soil amendments can increase the amount of soil aggregate, and the soil aggregate can improve soil porosity, permeability, water retention, aeration and erosion resistance. However, when the amount of soil amendments exceeds the limit, soil will harden and inhibit the breathing of the roots. We planted five seeds of *melinis minutiflora* and *pigeon pea in* clay with different concentrations of ADNB, then measured their growth heights and obtained the standard deviations. They are in the range of 0 to 0.13 mm and 0.02 to 0.15 mm. The small values of the standard deviation indicate that the measurements are reliable.

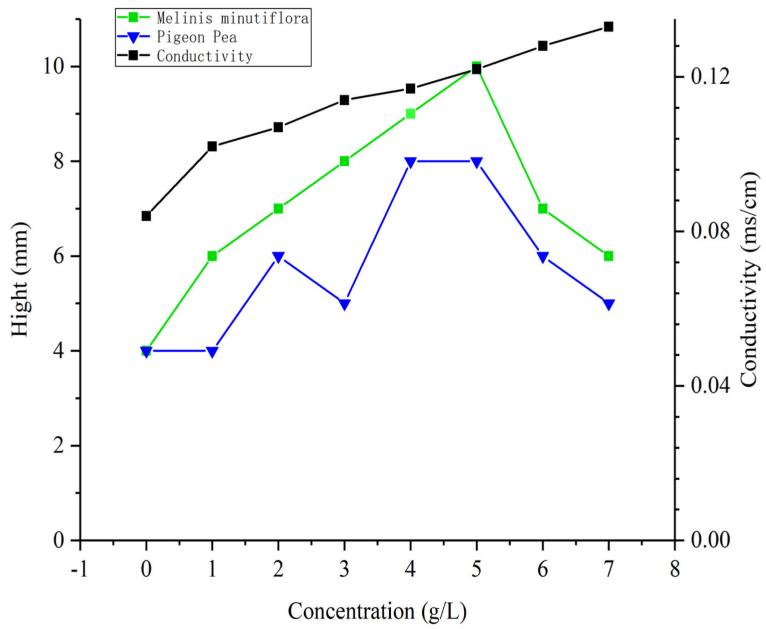


Figure 11. The heights of *melinis minutiflora* and *pigeon pea* under different concentrations of ADNB at 20 °C.

5. Conclusions

Based on research concerning ADNB and silty clay, clay conductivity was measured by adding different concentrations of ADNB at different temperatures. The results showed that conductivity measurements provided a reasonable and reliable way to quantitatively and conveniently evaluate the effect of amendment on ecological restoration. In the range of 0 to 30 °C and 40 to 50 °C, with the higher concentration of ADNB, the clay conductivity was higher, the nutrient storage capacity of clay was greater, and the ecological restoration performance was better. Thus, this study provided a new way to improve the evaluation of the effects of the large-scale use of soil modifiers on ecological restoration. At the same time, according to the growth conditions of *melinis minutiflora* and pigeon pea under different concentrations of ADNB, the optimal concentration was obtained to improve ecological restoration, thus providing supporting evidence for the large-scale use of ADNB, especially in South China. Furthermore, this practice may serve as a reference to explore means of ecological restoration in the Gobi region, in the northwest of the Loess region.

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