

# Distribution and stability of water-stable aggregates as affected by long-term cattle manure application to saline-sodic soil in the black soil region of northeastern China

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**Abstract:** Saline-sodic soil has a poor structure, low nutrient content, and excessive sodium in the western Heilongjiang Province, resulting in low crop productivity. Experimental treatments were established by applying manure to the soil for 5 years, 12 years, and 16 years and soil without manure application was used as a control treatment (CK). The results indicate that the application of manure significantly increased soil macroaggregates, the mean weight diameter (MWD) and the geometric mean diameter (GMD) compared to those for the CK treatment. The soil organic matter (SOM) concentration increased from 17.8 to 47.9 g/kg, the soil pH decreased from 10.18 to 7.89, and the electrical conductivity (EC) decreased from 4.92 to 0.19 dS/m. The soil exchangeable  $\text{Na}^+$  was decreased and exchangeable  $\text{Ca}^{2+}$  was increased in the treatments with manure application compared with the CK treatment. And a decrease in the  $\text{CaCO}_3$  content was observed in the treatment with manure. Water-stable aggregates (WSAs) of greater than 2.0 mm were the dominant factor driving the changes in the MWD, and WSAs of 1.0-2.0 mm were the dominant factor driving the changes in the GMD. The correlation matrix showed that the SOM and soil exchangeable  $\text{Ca}^{2+}$  concentration was positively correlated with the stability of the WSAs, while the pH, EC, and soil exchangeable  $\text{Na}^+$  were negatively and significantly correlated. We conclude that the long-term application of manure to saline-sodic soil can increase the proportion of soil macroaggregates and thus increase the stability of WSAs, as a result of the formation of soil macroaggregates mainly caused by the increase in the organic colloidal matter and soil exchangeable  $\text{Ca}^{2+}$ , and by the decrease in soil exchangeable  $\text{Na}^+$ .

**Keywords:** cattle manure, geometric mean diameter, macroaggregate, mean weight diameter, solonet

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## 1 Introduction

Soil aggregate stability is considered to be a good indicator of soil structure, which is a keystone factor in sustaining soil function<sup>[1-3]</sup>. The existence of soil aggregates significantly affects soil physical characteristics, the ability of soil to support plant growth, and the maintenance of soil fertility, which is associated with crop productivity<sup>[4,5]</sup>. Moreover, the amount and quality of soil aggregates directly determine the stability of soil aggregates and have an indirect influence on crops. The maintenance of optimum soil physical conditions is an essential component of soil fertility management<sup>[6]</sup>. The existence of soil aggregates could affect the structure and physical properties of soil. An increase in water-stable aggregates (WSAs) reduces the pH and electrical conductivity (EC) through cation exchange and the subsequent leaching of toxic ions from the exchange sites of aggregates in saline-sodic soil<sup>[7]</sup>.

Soil that is affected by salinity has a poor structure and physical properties due to a lack of aggregates, which negatively affects the germination and growth of plants<sup>[8]</sup>. The Songnen Plain, which is characterized by saline-sodic soil, is rich in soluble

sodium ( $\text{Na}^+$ ) salts, such as  $\text{Na}_2\text{CO}_3$  and  $\text{NaHCO}_3$ , which degrade the soil structure<sup>[9]</sup>. Saline-sodic soil has excessive amounts of exchangeable  $\text{Na}^+$ , which cause soil dispersion that leads to poor soil physical properties such as infiltration, aggregation, and porosity<sup>[10]</sup>.  $\text{Na}^+$  is a monovalent ion that can disperse soil particles<sup>[11]</sup>. Thus,  $\text{Na}^+$  is a highly dispersive agent that can reduce the formation of aggregates and worsen stability<sup>[12]</sup>. Decreases in the exchangeable and/or soluble  $\text{Na}^+$  in soil are important for sustaining desirable soil properties and encouraging water to leach the salt out of the root zone<sup>[13]</sup>. Saline-sodic soil is not conducive to soil structure stabilization because of its negative impact on aggregate-forming processes, which makes it difficult to maintain soil fertility.

The addition of organic matter is a commonly used method to improve saline-sodic soil. Simultaneously, as a by-product of the livestock industry, a large amount of cattle manure was produced in our experimental area every year. The application of cattle manure to saline-sodic soil has been reported to decrease the soil pH and EC and increase the soil organic matter (SOM) and nutrient status as well as the density of soil organic carbon, which has a positive impact on soil structure amendments<sup>[14,15]</sup>. Meng et al.<sup>[16]</sup> found that an increase in the soil quality was characterized by the amendment of soil physical properties on the Songnen Plain.

Aggregates are considered to be the smallest functioning units in the soil structure<sup>[4]</sup>, and soil with an optimal amount of aggregates has a good structure, which affects soil physical properties such as infiltration and porosity. The application of organic matter to soil effectively improves the stability of

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aggregates and increases soil aggregation<sup>[17,18]</sup>. We hypothesize that improvements in the structural stability of WSAs are likely related to the formation of water-stable macro aggregates and the decrease in soil exchangeable  $\text{Na}^+$  caused by the application of cattle manure. Thus, a field experiment was performed in clay saline-sodic soil under the long-term application of cattle manure. The objective of this study was to evaluate the effect of the long-term application of cattle manure on the distribution and stability of WSAs through the change of ions in saline-sodic soil. This study also investigated the relationship between the distribution and stability of WSAs in saline-sodic soil under the long-term application of cattle manure.

## 2 Materials and methods

### 2.1 Study site

This study was conducted in a long-term experimental field with saline-sodic soil amelioration, which was established on the Songnen Plain in Zhaozhou County, Heilongjiang Province, China (longitude 125.06°E, latitude 45.48°N and altitude 149 m). The area has a continental monsoon climate and is in a temperate zone, with an annual average temperature of 3.7°C, annual average evaporation of 1800 mm, and annual average precipitation of 434.5 mm. The soil is classified as solonetz based on the FAO World Reference Base for Soil Resources, with a clay texture (26% sand, 22% silt, 52% clay)<sup>[19]</sup>. The physicochemical properties of the soil and cattle manure at the study site before planting are listed in Table 1.

**Table 1 Physico-chemical properties of the soil and cattle manure at the study site before planting**

Soil		Cattle manure	
pH	9.56	pH	8.11
EC/dS·m <sup>-1</sup>	6.23	EC/dS·m <sup>-1</sup>	9.07
SOM/g·kg <sup>-1</sup>	11.0	Na/g·kg <sup>-1</sup>	0.4
TN/g·kg <sup>-1</sup>	0.4	K/g·kg <sup>-1</sup>	14.2
TP/g·kg <sup>-1</sup>	0.3	Ca/g·kg <sup>-1</sup>	9.1
Available N/mg·kg <sup>-1</sup>	39	Mg/g·kg <sup>-1</sup>	3.8
Available P/mg·kg <sup>-1</sup>	12		
Available K/mg·kg <sup>-1</sup>	125		
Sand/%	26		
Silt/%	22		
Clay/%	52		

Note: EC is electrical conductivity; SOM is soil organic matter; TN is total N; TP is total P.

### 2.2 Experimental design

According to the manure application history, four different application years were specified in a randomized complete block design with three replicates. Manure was applied to the saline-sodic soil in 2000, 2004, and 2011, and the soil samples from all treatments were collected in 2016. Thus, saline-sodic soil samples to which manure had been applied for 16 years, 12 years, and 5 years were used as the experimental treatments, and saline-sodic soil without manure was used as the control treatment (CK) at the 2016 sampling. In every late April, cattle manure was applied in the same experimental plot at a rate of 10 000 kg/hm<sup>2</sup> on an oven-dry weight. After being sprinkled on the topsoil, cattle manure was mixed by plowing with the 0-20 cm soil layer. The study area adopted corn (*Zea mays* L.) succession cropping for all treatments, and urea (N=46%) was applied to the corn in the elongation stage at a rate of 400 kg/hm<sup>2</sup>.

### 2.3 Soil sampling

Undisturbed soil samples at depths of 0-20 cm and 20-40 cm

were collected from three random points in each plot (10 m×6.5 m) on October 8, 2016, using a spade. After transporting all samples to the laboratory in their intact form, roots, stones, and other debris were discarded from the soil samples before they were naturally air-dried. Undisturbed soil samples were broken into small pieces (~8 mm) along natural cracks to measure the WSAs during the process of air-drying. The air-dried soil was pushed through 0.25 mm and 1 mm diameter sieves to analyze the SOM and the pH and EC of the soil.

### 2.4 Laboratory methods

Soil aggregates in the samples were determined by wet sieving using an aggregate analyzer<sup>[20]</sup>. One hundred grams of air-dried soil (~8 mm) were spread on the top of a set of sieves with mesh sizes of 2.000 mm, 1.000 mm, 0.500 mm, 0.250 mm, and 0.106 mm from top to bottom. The set of sieves was immersed in water and shaken with a frequency of 30 min<sup>-1</sup> for 30 min. Each size fraction was washed into an evaporating dish with a known mass and then weighed after drying in a cabinet at 55°C. WSAs >0.25 mm were considered to be macroaggregates, and WSAs <0.25 mm were specified as microaggregates<sup>[21]</sup>. The mean weight diameter (MWD) and geometric mean diameter (GMD) were calculated as follows:

$$\text{MWD} = \sum_{i=1}^n X_i W_i \quad (1)$$

where, MWD is the mean weight diameter, mm;  $X_i$  is the average diameter of the  $i$ th size fraction of the aggregates, mm;  $W_i$  is the weight of the aggregates in that size range as a fraction of the weight.

$$\text{GMD} = \exp \left( \frac{\sum_{i=1}^n W_i \ln X_i}{\sum_{i=1}^n W_i} \right) \quad (2)$$

where, GMD is the geometric mean diameter, mm;  $X_i$  is the average diameter of the  $i$ th size fraction of the aggregates, mm;  $W_i$  is the weight of the aggregates in that size range as a fraction of the weight.

The SOM content was determined by dichromate oxidation with heating ( $\text{K}_2\text{Cr}_2\text{O}_7\text{-H}_2\text{SO}_4$ )<sup>[22]</sup>. The soil pH and EC were measured using a pH-meter electrode and a conductivity meter, respectively, at a 1:5 soil-to-water ratio. The cation exchange capacity (CEC) was measured using the method described by Bao<sup>[22]</sup>. Exchangeable  $\text{Na}^+$  and calcium ( $\text{Ca}^{2+}$ ) were determined using atomic absorbance after extraction with 1 mol/L  $\text{NH}_4\text{OAC}$  at pH 7.00. Soil  $\text{CaCO}_3$  was measured using the gas-volumetric method<sup>[23]</sup>.

### 2.5 Statistical analysis

All statistical analyses were performed using SPSS 17.0 (Statistical Package for Social Science), and the data were analyzed using Duncan's multiple comparison test. The correlation between the stability and distribution of WSAs was tested with multiple linear stepwise regression. The correlation matrix was used to analyze the relationship among the soil properties.

## 3 Results

### 3.1 Distribution of WSAs size

Compared with the CK treatment, water-stable micro aggregates (<0.106 mm and 0.106-0.25 mm WSAs) decreased and water-stable macroaggregates (0.25-0.5 mm, 0.5-1.0 mm, 1.0-2.0 mm, and >2.0 mm WSAs) increased in the treatments with applied manure (Table 2). At depth of 0-20 cm, the 0.25-0.5 mm, 0.5-1.0 mm, 1.0-2.0 mm, and >2.0 mm WSAs in the 5-year,

12-year, and 16-year treatments increased, and the 0.5-1.0 mm, 1.0-2.0 mm, and >2.0 mm WSAs were significantly higher in the 12-year treatment than in the CK treatment ( $p<0.05$ ). There was an insignificant decrease in the <0.106 mm and 0.106-0.25 mm WSAs in the 5-year, 12-year, and 16-year treatments compared to those in the CK treatment. For the treatments with applied manure, the 0.5-1.0 mm, 1.0-2.0 mm, and >2.0 mm WSAs decreased in the 16-year treatment compared to those in the 12-year treatment. The proportion of >2.0 mm WSAs was greater than the proportions of the other sizes within the water-stable macroaggregates in the 5-year and 12-year treatments; however, the proportion of 0.25-0.5 mm WSAs was the highest within the water-stable macroaggregates in the 16-year treatment.

At depths of 20-40 cm, the proportions of the 0.25-0.5 mm, 0.5-1.0 mm, 1.0-2.0 mm, and >2.0 mm WSAs increased under the different manure applications (Table 2): the proportions of the 0.5-1.0 mm, 1.0-2.0 mm and >2.0 mm WSAs were significantly greater in the 12-year treatment than in the CK treatment ( $p<0.05$ ). Compared with the 5-year treatment, the 0.106-0.25 mm and 0.25-0.5 mm WSAs decreased in the 12-year treatment and increased in the 16-year treatment. Contrarily, the 0.5-1.0 mm, 1.0-2.0 mm, and >2.0 mm WSAs increased in the 12-year treatment compared with those in the 5-year treatment and decreased in the 16-year treatment. The proportion of >2.0 mm WSAs was greater than the proportions of the other sizes within the water-stable macroaggregates in the 5-year, 12-year, and 16-year treatments.

**Table 2** Distribution of water-stable aggregates size under different manure applications at depths of 0-20 and 20-40 cm (%)

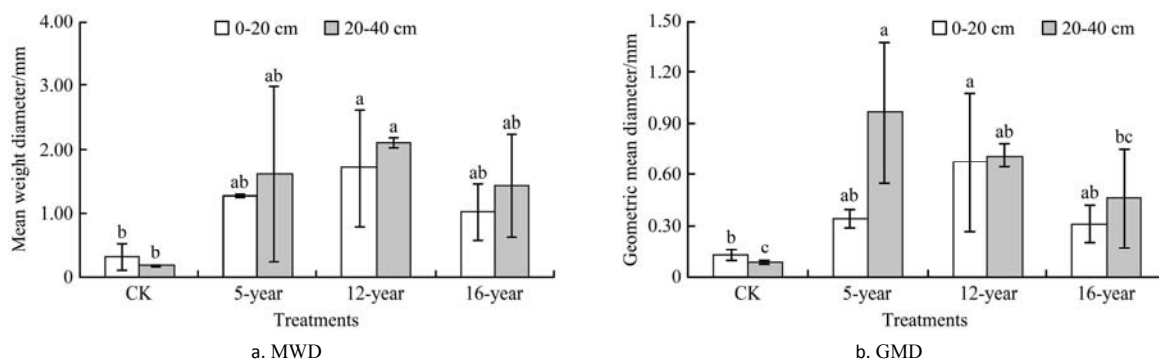
Treatments		Soil aggregate size/mm					
		<0.106	0.106-0.25	0.25-0.50	0.50-1.00	1.00-2.00	>2.00
0-20 cm	CK	49.23±0.23 <sup>a</sup>	33.16±0.24 <sup>a</sup>	8.95±0.05 <sup>a</sup>	4.88±0.06 <sup>b</sup>	0.77±0.01 <sup>b</sup>	3.02±0.04 <sup>b</sup>
	5-year	32.97±0.06 <sup>a</sup>	15.59±0.03 <sup>a</sup>	15.69±0.05 <sup>a</sup>	10.97±0.04 <sup>ab</sup>	4.80±0.02 <sup>ab</sup>	19.98±0.02 <sup>ab</sup>
	12-year	23.93±0.08 <sup>a</sup>	8.08±0.07 <sup>a</sup>	13.38±0.09 <sup>a</sup>	18.78±0.04 <sup>a</sup>	8.70±0.05 <sup>a</sup>	27.13±0.18 <sup>a</sup>
	16-year	30.60±0.07 <sup>a</sup>	19.07±0.03 <sup>a</sup>	20.64±0.02 <sup>a</sup>	10.97±0.04 <sup>ab</sup>	3.65±0.01 <sup>b</sup>	15.08±0.09 <sup>ab</sup>
20-40 cm	CK	74.42±0.04 <sup>a</sup>	15.75±0.08 <sup>ab</sup>	3.53±0.03 <sup>b</sup>	4.78±0.05 <sup>b</sup>	0.60±0.01 <sup>b</sup>	0.93±0.01 <sup>b</sup>
	5-year	36.48±0.36 <sup>b</sup>	11.78±0.06 <sup>ab</sup>	8.69±0.08 <sup>ab</sup>	10.05±0.09 <sup>ab</sup>	5.28±0.04 <sup>ab</sup>	27.72±0.26 <sup>ab</sup>
	12-year	25.56±0.04 <sup>b</sup>	5.26±0.02 <sup>b</sup>	6.02±0.02 <sup>b</sup>	19.06±0.02 <sup>a</sup>	7.80±0.04 <sup>a</sup>	36.29±0.01 <sup>a</sup>
	16-year	25.46±0.05 <sup>b</sup>	19.10±0.04 <sup>a</sup>	19.04±0.09 <sup>a</sup>	7.94±0.01 <sup>b</sup>	4.82±0.01 <sup>ab</sup>	23.65±0.17 <sup>ab</sup>

Note: Different letters in same column indicate significant differences between different treatments in the same size of water-stable aggregates at the 0.05 level. Treatments 5-year, 12-year and 16-year represent soil with manure application for 5, 12, and 16 years, respectively. CK represents soil without manure application.

**3.2 Relationship between MWD, GMD, and WSAs**

Different treatments had a prominent impact on the MWD and GMD values of the WSAs. The MWD and GMD values increased in the sequence of the CK, 16-year, 5-year, and 12-year treatments as a result of the addition of manure to the soil at depth of 0-20 cm (Figures 1a and 1b). The MWD and GMD values in the 12-year treatment differed significantly from those in the CK treatment ( $p<0.05$ ). At depth of 20-40 cm, the MWD trend was

similar to that at depth of 0-20 cm, and the GMD values increased in the sequence of the CK, 16-year, 12-year, and 5-year treatments (Figures 1a and 1b). There was a significant difference in the MWD values between the 12-year and CK treatments ( $p<0.05$ ). The GMD values in the 5-year treatment were significantly greater than those in the CK treatment. However, there was a decrease in the MWD and GMD values in the 16-year treatment compared with those in the 12-year treatment at depths of 0-20 cm and 20-40 cm.



Note: Different letters indicate significant differences between different treatments at the 0.05 level. Treatments 5-year, 12-year and 16-year represent soil with manure application for 5, 12, and 16 years, respectively. CK represents soil without manure application. Error bars represent standard deviation ( $n=3$ ).

Figure 1 MWD and GMD of WSAs under different manure applications

The relationship between the WSAs and the MWD was explored by using linear regression models, which showed that the >2.0 mm, 1.0-2.0 mm, and 0.5-1.0 mm WSAs were significantly and positively correlated with the MWD, and the >2.0 mm WSAs were the predominant factor driving the changes in the MWD, as shown in Equation (3) ( $R^2=0.99$ ;  $p<0.01$ ).

$$Y_1 = 0.373 + 0.046X_1 + 0.011X_2 + 0.004X_3 \quad (3)$$

where,  $Y_1$  is mean weight diameter, mm;  $X_1$  is average WSA<sub>s</sub> of >2.0 mm;  $X_2$  is average WSA<sub>s</sub> of 1.0-2.0 mm;  $X_3$  is average WSA<sub>s</sub> of 0.5-1.0 mm.

The relationship between the WSAs and the GMD was determined by using linear regression models, which indicated that the 1.0-2.0 mm, and >2.0 mm WSAs had a significant and positive correlation with the GMD, and 1.0-2.0 mm WSAs were the crucial factor, as shown in Equation (4) ( $R^2=0.94$ ;  $p<0.01$ ).

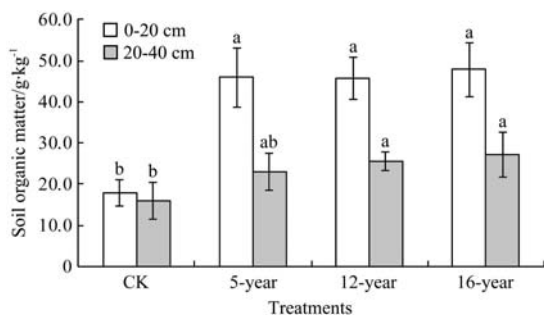
$$Y_2 = 0.014 + 0.016X_1 + 0.021X_2 \quad (4)$$

where,  $Y_2$  is geometric mean diameter, mm.

**3.3 The chemical properties of saline-sodic soil under different manure applications**

The SOM content increased in the different treatments

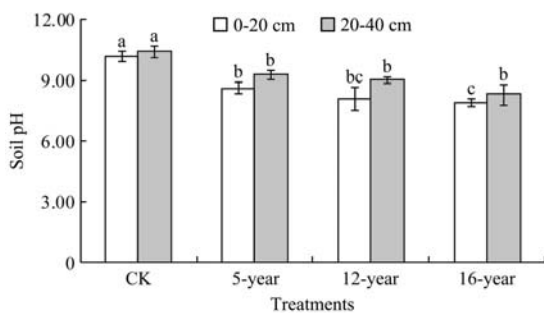
according to the number of years of manure addition (Figure 2). At depth of 0-20 cm, the highest SOM content was obtained in the 16-year treatment and was significantly higher in the 5-year, 12-year, and 16-year treatments than in the CK treatment ( $p < 0.05$ ). At depth of 20-40 cm, there was a significant increase in the SOM content in the 12-year and 16-year treatments compared with the CK treatment ( $p < 0.05$ ). However, there was no significant difference in the SOM content between the 5-year and CK treatments ( $p < 0.05$ ).



Note: Different letters indicate significant differences between different treatments at the 0.05 level. Treatments 5-year, 12-year and 16-year represent soil with manure application for 5 years, 12 years and 16 years, respectively. CK represents soil without manure application. Error bars represent standard deviation ( $n = 3$ ).

Figure 2 Content of SOM under different manure applications

At depths of 0-20 cm and 20-40 cm, a significant decrease in the soil pH in the 5-year, 12-year, and 16-year treatments compared with the CK treatment was obtained and was attributed to the application of manure (Figure 3). The soil pH in the 5-year, 12-year, and 16-year treatments was significantly lower than that in the CK treatment at both depths of 0-20 cm and 20-40 cm. There was a significant difference between the 5-year and 16-year treatments at depth of 0-20 cm ( $p < 0.05$ ).



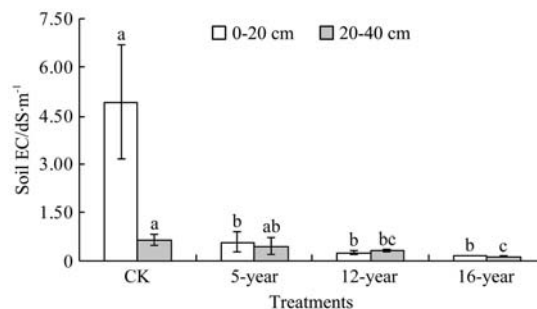
Note: Different letters indicate significant differences between different treatments at the 0.05 level. Treatments 5-year, 12-year, and 16-year represent soil with manure application for 5 years, 12 years, and 16 years, respectively. CK represents soil without manure application. Error bars represent standard deviation ( $n = 3$ ).

Figure 3 Soil pH under different manure applications

Different manure treatments had a significant impact on the soil EC, showing that there was a reduction in the EC in response to the application of manure (Figure 4). At depth of 0-20 cm, the soil EC was significantly lower in the 5-year, 12-year, and 16-year treatments than in the CK treatment ( $p < 0.05$ ). At depth of 20-40 cm, a significant decline in the EC was observed in the 12-year and 16-year treatments, whereas there was no significant difference between the 5-year and CK treatments or between the 12-year and 16-year treatments ( $p < 0.05$ ).

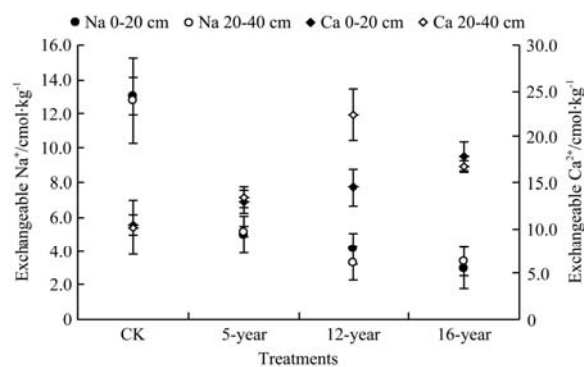
A significant decrease in the soil's exchangeable  $\text{Na}^+$  was observed in the treatments with manure compared with the CK treatment at depths of 0-20 cm and 20-40 cm ( $p < 0.05$ ) (Figure 5).

In contrast, the exchangeable  $\text{Ca}^{2+}$  in the treatments with manure was higher than that in the CK treatment (Figure 5). At depths of 0-20 cm and 20-40 cm, the exchangeable  $\text{Ca}^{2+}$  was significantly increased in the 12-year and 16-year treatments ( $p < 0.05$ ).



Note: Different letters indicate significant differences between different treatments at the 0.05 level. Treatments 5-year, 12-year and 16-year represent soil with manure application for 5 years, 12 years and 16 years, respectively. CK represents soil without manure application. Error bars represent standard deviation ( $n = 3$ ).

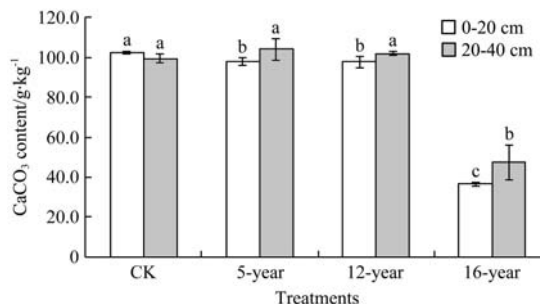
Figure 4 Soil EC under different manure applications



Note: Treatments 5-year, 12-year, and 16-year represent soil with manure application for 5 years, 12 years, and 16 years, respectively. CK represents soil without manure application. Error bars represent standard deviation ( $n = 3$ ).

Figure 5 Soil exchangeable  $\text{Na}^+$  and exchangeable  $\text{Ca}^{2+}$  under different manure applications

A decrease in the  $\text{CaCO}_3$  content was observed after the application of manure (Figure 6). At depth of 0-20 cm, the  $\text{CaCO}_3$  content showed a significant decrease in the 16-year treatment compared with the 5-year, 12-year, and CK treatments ( $p < 0.05$ ). At depth of 20-40 cm, the  $\text{CaCO}_3$  content in the 5-year, 12-year, and 16-year treatments was significantly lower than that in the CK treatment ( $p < 0.05$ ).



Note: Different letters indicate significant differences between different treatments at the 0.05 level. Treatments 5-year, 12-year, and 16-year represent soil with manure application for 5-year, 12-year, and 16-year, respectively. CK represents soil without manure application. Error bars represent standard deviation ( $n = 3$ ).

Figure 6 Soil  $\text{CaCO}_3$  content under different manure applications

#### 4 Discussion

The SOM content increased with the application of manure,

which was likely a result of the addition of organic matter containing C over a number of years. In this study, there were more macroaggregates in the treatments with manure compared with the CK treatment. The formation of soil macroaggregates is significantly correlated with the application of manure<sup>[24]</sup>, most likely because the concentration of SOM increases with the long-term addition of manure<sup>[25]</sup>, and cementation during the formation of soil aggregates is strengthened<sup>[26,27]</sup>. In addition, the decomposition of organic manure promotes microbial activation, which facilitates the formation of WSAs through the acceleration of soil particles entwined by mycorrhizal fungi, and increases the secretion of cementation substances by microorganisms<sup>[28]</sup>. Aoyama and Angers et al.<sup>[29]</sup> found that the application of manure improved the formation of water-stable macro aggregates.

Sodium ( $\text{Na}^+$ ) is the base cation in the saline-sodic soil of the Songnen Plain, and a higher  $\text{Na}^+$  content has been reported to increase the soil pH and EC and the dispersion of soil with water, which negatively impacts the formation of WSAs<sup>[30]</sup>. We also found that soil exchangeable  $\text{Na}^+$  was positively and significantly correlated with the soil pH ( $R=0.85$ ,  $p<0.01$ ) and EC ( $R=0.64$ ,  $p<0.01$ ); on the contrary, soil exchangeable  $\text{Na}^+$  was negatively and significantly correlated with the soil MWD ( $R=0.63$ ,  $p<0.01$ ) and GMD ( $R=0.54$ ,  $p<0.01$ ) (Table 3). Thus, negative factors such as the  $\text{Na}^+$  and pH in the saline-sodic soil adversely affected the formation and stability of soil aggregates. Gupta and Khan<sup>[31]</sup> also found that the pH and EC had a highly significant effect on the water-stable macro aggregates and the MWD of WSAs in distillery effluent-amended soils as a result of the exchangeable  $\text{Na}^+$ , the hydrolysis of which increased the soil pH. As a highly dispersive agent,  $\text{Na}^+$  leads to the breakup of soil aggregates<sup>[32]</sup>. The application of manure to saline-sodic soil decreases the content of sodium and the soil pH and EC, which provides positive conditions for the formation of soil macroaggregates<sup>[33]</sup>. Karami et al.<sup>[34]</sup> reported that the MWD and GMD increased following the application of sheep and cow manure. This result is similar to our results, which show that the MWD and the GMD increased after the application of manure. Thus, organic matter has a positive effect on the structural stability of WSAs<sup>[35]</sup>.

The increased exchangeable  $\text{Ca}^{2+}$  was caused by the application of manure, which supplied part of the  $\text{Ca}^{2+}$ .  $\text{Ca}^{2+}$  was also supplied by the dissolution of  $\text{CaCO}_3$ , which was caused by the acidic material released from the process of manure decomposition. Increased  $\text{Ca}^{2+}$  is beneficial for the decrease in exchangeable  $\text{Na}^+$

through ionic exchange. Ultimately, the soil exchangeable  $\text{Na}^+$  pH decreased after the application of manure. This finding could be observed from the correlation analysis, which revealed that  $\text{CaCO}_3$  was significantly and positively correlated with the pH ( $R=0.56$ ,  $p<0.01$ ), and exchangeable  $\text{Ca}^{2+}$  was significantly and negatively correlated with the pH ( $R=0.56$ ,  $p<0.01$ ) and exchangeable  $\text{Na}^+$  ( $R=0.73$ ,  $p<0.01$ ) (Table 4). Meanwhile,  $\text{Ca}^{2+}$  can improve the stability of WSAs through cationic bridging with clay particles and SOM<sup>[12]</sup>. In our study, exchangeable  $\text{Ca}^{2+}$  had a significant and positive correlation with the MWD ( $R=0.54$ ,  $p<0.01$ ) and the GMD ( $R=0.41$ ,  $p<0.05$ ), respectively (Table 4). Consequently, the increase in the WSAs, especially the macroaggregates, resulted from the decrease in the dispersive agent ( $\text{Na}^+$ ) and the increase in the cementing materials. SOM supplied by the application of manure was beneficial to the formation and stability of the WSAs. The application of organic matter affected the structural stability of the WSAs<sup>[36]</sup>. Additionally, the increased exchangeable  $\text{Ca}^{2+}$  also contributed to the stability of the WSAs. Kim et al.<sup>[37]</sup> found that the increase in  $\text{Ca}^{2+}$  in a soil solution with added gypsum increased the aggregate size, aggregate stability, and MWD.

As shown in Table 3, significant positive correlations were observed between the MWD, the GMD, and macroaggregates ( $p<0.01$ ). Consequently, the increase in soil macroaggregates, especially for the  $>2.0$  mm and  $1.0\text{-}2.0$  mm WSAs, was the primary factor driving the improvements in the stability of the soil with the application of manure. As mentioned, the increased water-stable macroaggregates resulted from the cementation of SOM and micro aggregates. However, water-stable macroaggregates, the MWD, and the GMD decreased in the 16-year treatment compared with the 12-year treatment, but they were greater than the corresponding values in the CK treatment (Table 2, Figures 1a and 1b). There may be a transition from large macroaggregates ( $>2.0$  mm) to small macroaggregates ( $0.25\text{-}0.5$  mm) (Table 2) due to the increase of  $\text{K}^+$  from the annual application of manure to the soil (total potassium was  $14.2$  g/kg, Table 1). Our finding is consistent with the results of Guo et al.<sup>[38]</sup>, who illustrated that the application of pig and cattle manure in a vertisol significantly decreased the proportion of large macroaggregates ( $>2.0$  mm), which increased the exchangeable  $\text{K}^+$  content. Although the long-term application of manure could increase the ability of SOM to act as a binding agent, the increase of monovalent  $\text{K}^+$  as a dispersing agent could reduce soil aggregation<sup>[38,39]</sup>.

**Table 3 Pearson correlations between soil properties**

	Depth	MWD	GMD	pH	EC	SOM	$E_{\text{Na}^+}$	$E_{\text{Ca}^{2+}}$	$\text{CaCO}_3$	CEC	ESP
Depth	--										
MWD	0.16	--									
GMD	0.18	0.96**	--								
pH	0.30	-0.46*	-0.37	--							
EC	-0.34	-0.42*	-0.35	0.56**	--						
SOM	-0.64**	0.28	0.19	-0.70**	-0.33	--					
$E_{\text{Na}^+}$	-0.02	-0.63**	-0.54**	0.85**	0.64**	-0.56**	--				
$E_{\text{Ca}^{2+}}$	0.21	0.54**	0.41*	-0.56**	-0.46*	0.25	-0.73**	--			
$\text{CaCO}_3$	0.09	-0.01	0.07	0.56**	0.30	-0.40	0.44*	-0.33	--		
CEC	-0.02	0.37	0.30	-0.42*	-0.33	0.24	-0.46*	0.30	-0.21	--	
ESP	-0.02	-0.64**	-0.55**	0.85**	0.64**	-0.56**	0.99**	-0.71**	0.43*	-0.59**	--

Note: MWD is mean weight diameter; GMD is geometric mean diameter; EC is electrical conductivity; SOM is soil organic matter;  $E_{\text{Na}^+}$  is exchangeable  $\text{Na}^+$ ;  $E_{\text{Ca}^{2+}}$  is exchangeable  $\text{Ca}^{2+}$ ; CEC is cation exchange capacity; ESP is exchangeable sodium saturation percentage. \* means the correlation is significant at the 0.05 level; \*\* means the correlation is significant at the 0.01 level.

## 5 Conclusions

The long-term application of cattle manure to saline-sodic soil increased the amount of soil macroaggregates and the stability of WSAs. Additionally, a significant and positive correlation was observed between the structural stability of the WSAs and soil macroaggregates. Furthermore, the WSAs >2.0 mm were the predominant factor affecting the MWD, and the 1.0-2.0 mm WSAs were the predominant factor affecting the GMD. Exchangeable  $\text{Ca}^{2+}$  was positively correlated with the MWD and the GMD; however, the pH, EC, and exchangeable  $\text{Na}^+$  had a negative correlation with the MWD and the GMD. Soil macroaggregates increased with the long-term application of cattle manure, strengthened cementation during the formation of WSAs, and decreased the soil pH, EC and  $\text{Na}^+$  as a result of the increase in the SOM content due to the application of manure. The increased soil macroaggregates and their stability were conducive to improvements in the soil's physical properties and ensured a suitable environment for crop growth.

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