

## Changes in water stability of soil aggregates in a solar greenhouse in different planted years and their relationship to iron oxide

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**The distribution and stability of soil aggregates and its relation to iron oxide during different plantation years are studied in a solar greenhouse. Soil samples were obtained from a vegetable bed within a greenhouse in Xinmin (Liaoning, China). The composition, water stability and percentage of aggregate destruction, as well as variations in different forms of iron oxides were evaluated at 5 time points (0, 2, 5, 8 and 16 years). Soil from a dry land in an open field was also collected for comparison. The quantity and water stability of >0.25 mm soil aggregates from 2 and 5 years were lower than those in the soil from the open field. These parameters significantly increased after five years. The contents of non-free iron oxides decreased under greenhouse cultivation, resulting in reduced aggregate stability. Free iron oxides were positively associated with >2 mm aggregates. Complex iron oxides were positively correlated with 2–0.25 mm and 0.25–0.053 mm aggregates but negatively correlated with <0.053 mm aggregates.**

**Keywords:** Iron oxide, soil aggregate, soil cementation process, solar greenhouse, water stability.

LONG-TERM sustainability and improvement in the fertility and tillage of agricultural ecosystems are being increasingly studied. In this regard, soil aggregate stability should also be elucidated. The formation and decomposition of aggregates in soil are affected by many factors. As an important component of active soils, iron oxide controls physical, chemical and biological properties of soil<sup>1,2</sup>. It improves tightness, aggregate stability, physicochemical properties, such as permeability and soil structure<sup>3–6</sup>.

Cationic bridges in soil are formed between ferric ion and organisms; the bridge represents an organic mineral complex, which improves the aggregate structure<sup>5,7</sup>. In particular, iron ions play a dominant role in the formation and stability of aggregates in soils with low organic matter content and high iron content<sup>8,9</sup>. Studies show the varied effects of iron oxide on aggregate stability<sup>10,11</sup>.

A high degree of free iron results in stronger aggregate stability under natural condition; amorphous iron oxides or weak crystals of iron oxide can also enhance the stability of aggregates compared to crystal-state iron oxides<sup>10</sup>.

Solar greenhouses are used for sustaining soil resources and improving crop yield. However, the ongoing deterioration of soil structure threatens sustainable crop yields and national food security<sup>12–14</sup>. Frequent and intensive tillage and extensive use of fertilizers and pesticides are needed to maintain solar greenhouses<sup>15</sup>. Tillage and fertilization disturbances can increase macroaggregate (>0.25 mm aggregates) turnover, inhibit microaggregate (<0.25 mm aggregates) formation within macroaggregates<sup>16</sup>, and indirectly impair aggregate stability<sup>17</sup>. Researchers have proposed that fertilization and tillage may facilitate physicochemical reactions among polyvalent cations, organic molecules and clay<sup>18</sup>. The relationship between aggregate stability and binding agents with cultivation has been studied in open vegetable plots. But, different binding mechanisms of stable aggregation in solar greenhouse soils have received minimal attention. In this type of soil, the mechanisms that control aggregate formation and stabilization may be influenced by specific management practices and conditions. The present study aims at investigating the effect of greenhouse soil management on soil aggregation and iron oxide in northeast China. We determined different forms of iron oxide in aggregate fractions and size distribution of water stability aggregates after 2, 5, 8 and 16 years of tillage in a greenhouse. The results elucidate the aggregation process during long-term greenhouse soil management and highlight the relationship between aggregate-associated iron oxide and soil aggregation.

A long-term greenhouse experiment was set in the village of Fangjinniu (lat. 41°32'N, long. 120°23'E), in the Damintun Town (Shenyang City, Liaoning province, China). The study site conditions and sampling method were identical to those in the long-term study<sup>19</sup>. The soil is classified as Typic FiMi-Orthic Anthrosol<sup>20</sup> and sandy loam<sup>21</sup>. The main properties of soil samples in different greenhouse aged plots (0–20 cm) are summarized in Table 1. The treatments were assigned to different cropping durations. The treatments consisted of (i) A0 (control, open vegetable plot), (ii) A2 (2-year greenhouse age), (iii) A5 (5-year greenhouse age), (iv) A8 (8-year greenhouse age) and (5) A16 (16-year greenhouse age)

Sample pH and EC were determined for water and soil suspension samples (2.5 : 1)<sup>22</sup>. The amounts of total phosphorus (TP) in soil samples were determined through NaHCO<sub>3</sub>–NaOH method<sup>23</sup>. Inorganic phosphorus was fractionated according to the sequential extraction procedure of Jackson<sup>23</sup>. Soil samples were physically fractionated into different water-stable aggregates (WSA) and mechanical stable aggregates according to the procedure reported earlier<sup>19,24,25</sup>. The mass proportion of each

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**Table 1.** Soil properties of virgin soil samples in different greenhouse aged plots (0–20 cm)

Planted years	CK		2a		5a		8a		16a	
	Value	Std. dev								
pH	6.31	0.33	5.95	0.14	5.93	1.37	6.18	1.25	7.04	0.40
EC ( $\mu\text{m cm}^{-1}$ )	49.32	6.90	333.83	5.07	719.33	17.56	520.70	18.23	272.33	13.93
TDS ( $\text{mg l}^{-1}$ )	24.60	3.48	166.90	15.38	359.28	14.11	261.00	11.16	136.08	16.86
Organic matter ( $\text{g kg}^{-1}$ )	16.90	1.11	16.46	1.48	18.53	2.43	19.55	2.24	21.71	5.62
TP ( $\text{g kg}^{-1}$ )	0.76	18.60	0.86	16.62	1.30	15.87	2.03	18.05	1.87	17.75
Al-P ( $\text{mg kg}^{-1}$ )	131.48	16.53	155.46	15.51	313.30	17.99	356.00	23.93	304.23	8.73
Fe-P ( $\text{mg kg}^{-1}$ )	131.95	14.37	172.23	18.88	98.59	23.61	128.84	16.56	86.08	18.74
O-P ( $\text{mg kg}^{-1}$ )	156.26	19.75	163.18	12.06	212.28	12.26	274.96	14.75	190.00	14.58
Ca-P ( $\text{mg kg}^{-1}$ )	163.61	17.72	173.63	18.67	204.51	17.84	245.56	12.54	283.40	14.82

fraction size of stable aggregates was calculated. Results were expressed as WSA (%) with mean weight diameter (MWD, mm) and percentage of aggregate destruction (PAD, %).

$$\text{MWD} = \frac{\sum_{i=1}^n (\bar{R}_i w_i)}{\sum_{i=1}^n w_i},$$

where  $\bar{R}_i$  is the average diameter of a specific level of aggregate (2.0, 1.125, 0.1515 and 0.053 mm) and  $w_i$  is the dried weight of the specific level of aggregate.

$$\text{PAD} = (w_d - w_w)/w_d,$$

where  $w_d$  is the proportion of >0.25 mm dried aggregates and  $w_w$  is the proportion of >0.25 mm WSA.

Free iron oxide was determined according to Filimonova *et al.*<sup>5</sup> and Wu *et al.*<sup>8</sup>. Briefly, 0.50 g of air-dried soil sample was passed through a 0.25 mm sieve and placed in a 50 ml centrifuge tube. Ten millilitre of 0.3 mol l<sup>-1</sup> sodium citrate and 1.25 ml of 1 mol l<sup>-1</sup> NaHCO<sub>3</sub> were added to the tube. The sample was heated in a water bath at 80 ± 5°C. A solution of 0.5 g Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O was added and stirred using a glass rod for 15 min. After cooling, the solution was separated through centrifugation (3000–4000 rpm). The clear liquid was poured into a 50 ml volumetric flask. This treatment was performed twice. Finally, 1 mol l<sup>-1</sup> NaCl was used to wash the residue twice. The washing liquid was poured into the same volumetric flask and used to determine the free state of iron.

The method proposed earlier<sup>5,8</sup> was used to measure the amount of amorphous iron oxide. Briefly, one gram of air-dried soil sample was passed through a 0.25 mm sieve and placed in a 100 ml Erlenmeyer flask. For flask 50 ml of H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub> was added in a soil to liquid ratio of 1 : 50 at 20–25°C. The flask was placed in a bag (away from sunlight) and oscillated in the machine for 2 h. The mixture was filtered and used as the amorphous Fe sample solution.

Complex iron oxide was determined according to procedure described earlier<sup>5,8</sup>. Briefly, 0.5 g of air-dried soil

sample was passed through a 0.25 mm sieve into a 50 ml centrifuge tube. Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> (25 ml) was added at 20–25°C to form a mixture which was centrifuged (4000 r/d) for 14 h to 16 h. The clear liquid was separated and used for complex iron testing. Iron was determined through phenanthroline colorimetry.

The statistical analyses were carried out using SPSS program (version 18.0), and the results expressed as mean values. Analysis of variances (ANOVA) was used to evaluate the difference among soil samples of different greenhouse ages. Duncan's multiple range tests were used to determine the differences among means. The relationships and interactions between soil examples and iron oxides obtained through different methods were evaluated with correlation analyses. Unless otherwise stated, the significant levels were set at  $P < 0.05$ .

WSA exhibits water erosion resistance, which reflects the water stability of soil aggregates, and also plays an important role in the stability of soil structure. As shown in Table 2, the proportions of WSA in >0.25 mm size classes for A2, A5, A8 and A16 treatments are lower than those in A0 treatment. This indicates that soil aggregates face more destruction under greenhouse cultivation than those under open-field cultivation. Fewer (>0.25 mm) macroaggregates were present in both the greenhouse and exposed soils, with a maximum value of approximately 9.21%. The particle size usually ranged from 0.25 to 0.053 mm. Overall the soil structure was poor. A majority of aggregates were nonwater stable, only a few were water stable.

The contents of >0.25 mm macroaggregates decrease in soil with greenhouse age lower than A5. The content begins to significantly increase ( $P < 0.05$ ) at planting time longer than A5 and continues to increase with time. The content increases by 9.38% between A8 and A5 and by 12.08% between A16 and A8. Under prolonged cultivation, aggregates increase but remain lower than that in the open-field soil. At greenhouse age lower than A5, the contents of <0.25 mm microaggregates show significant increase ( $P < 0.05$ ). The contents decrease after A5, but the decrease is not significant. At shed age lower than

**Table 2.** Composition of aggregates in different planted years (wet-sieving method)

Treatments	Aggregate size			
	> 2 mm	2–0.25 mm	0.25–0.053 mm	< 0.053 mm
A0	0.60 e <sup>a</sup>	8.61 a	76.12 a	14.67 e
A2	1.88 d	5.78 b	74.70 b	17.64 d
A5	2.02 c	5.02 c	73.19 c	19.77 c
A8	2.61 b	5.09 c	68.27 d	24.04 b
A16	2.92 a	5.71 b	63.92 e	27.45 a

<sup>a</sup>Same letters in the same column indicate no significant difference at  $P < 0.05$  level.

**Table 3.** MWD and PAD in different planted years

Treatments	MWD (mm)	PAD (%)
A0	0.232 a <sup>a</sup>	89.20 d
A2	0.225 b	90.63 b
A5	0.218 c	90.97 b
A8	0.226 b	91.35 a
A16	0.234 a	90.31 c

<sup>a</sup>Same letters in the same column indicate no significant difference at  $P < 0.05$  level.

A5, exogenous hydraulic action occurs and large-sized aggregates are broken into microaggregates in the greenhouse soil. At cultivation time higher than A5, microaggregates and cement combine and form large aggregates. Table 2 demonstrates that short-term greenhouse planting mode specifically affects the transformation of soil aggregates.

In soils, aggregates with different particle sizes exhibit different effects on soil fertility conservation, aeration and hydraulic properties. Therefore, the distribution and structural quality of soil aggregates of different sizes are related to the quality of the soil structure<sup>26</sup>. MWD is usually used to reflect the distribution of soil aggregates. Table 3 shows that the values of MWD are low, indicating that most of soil aggregates are non-water stable and can be broken through wet sieving. Thus, the MWD of aggregates can effectively reflect the stability of the soil structure. MWD increases and then decreases with increasing age. This finding is similar to the varied characteristics of >0.25 mm macroaggregates, indicating that the water stability of soil aggregates decreases during early cultivation. After A5, the water stability increases. However, the differences in the changing amplitude and MWD values of aggregates are not evident. The MWD in A5 decreases by 3.11% compared with that in A2 and increases by 3.67% and 7.34% compared with that in A8 and A16 respectively. In addition to A16, the three other ages demonstrate MWD values lower than those in the open-field soil. Nevertheless, the difference between soil in A16 and exposed soil is not evident. Hence, the water stability of the greenhouse soil is lower than the soil from open-field. With increasing cultivation time, the soil

structure and water stability of the greenhouse soil improves minimally.

PAD is significantly correlated with the stability of the soil structure. Poor soil structure has high PAD levels. By contrast, the soil structure is stable at low PAD values. Table 3 shows that high PAD levels are found (on an average of 90.49%) in the exposed and greenhouse soils. This finding indicates that soil erosion resistance and soil structure are weak. Similar trends are observed in MWD.

The PAD values in different years in greenhouse soil are higher than those in the open-field soil and increase by 1.60%, 1.98%, 2.41% and 1.24% with time. This finding may be due to greenhouse farming that reduces the water stability of the soil structure. From A2 to A8, the PAD of greenhouse soil increases, but the variation is not evident because the increase is only 0.79%. After A8, the PAD value decreases by 1.14% compared with that between A8 and A16. The water stability of aggregates improves with increasing age.

Iron oxides are an important inorganic cementing material in soil aggregates and are influenced by external factors under soil cultivation. Iron oxide content changes with increasing planted time. As shown in Figure 1, the contents of iron oxides, including free, complex, or amorphous form, are low in each period. In general, free iron oxides > amorphous iron oxides > complex iron oxides. This finding could be attributed to the type of soil in the research area which is northern cinnamon soil. This type of soil is rich in organic matter and poor in iron. Aggregates with <0.053 mm size exhibit the highest content of the three forms of iron oxides. This shows that iron oxide mainly exists in microaggregates.

In this study, the content of amorphous iron oxides in >2 mm and 0.25–0.053 mm aggregates increases with age from A2 to A5. The content also decreases with increase in cultivation time. Amorphous iron oxide in 2–0.25 mm aggregates increase with time, but the change is small. The content of amorphous iron oxide in aggregates <0.053 mm decreases significantly by 46.3% from A2 to A5 ( $P < 0.05$ ). Complex iron oxides in various grain sizes decrease with increase in cultivation years ( $P < 0.05$ ). In open-field soils, iron oxide mainly exists in aggregates <0.053 mm from A2 to A5. The content significantly decreases by 52.5% after A5 ( $P < 0.05$ ). Nevertheless, the

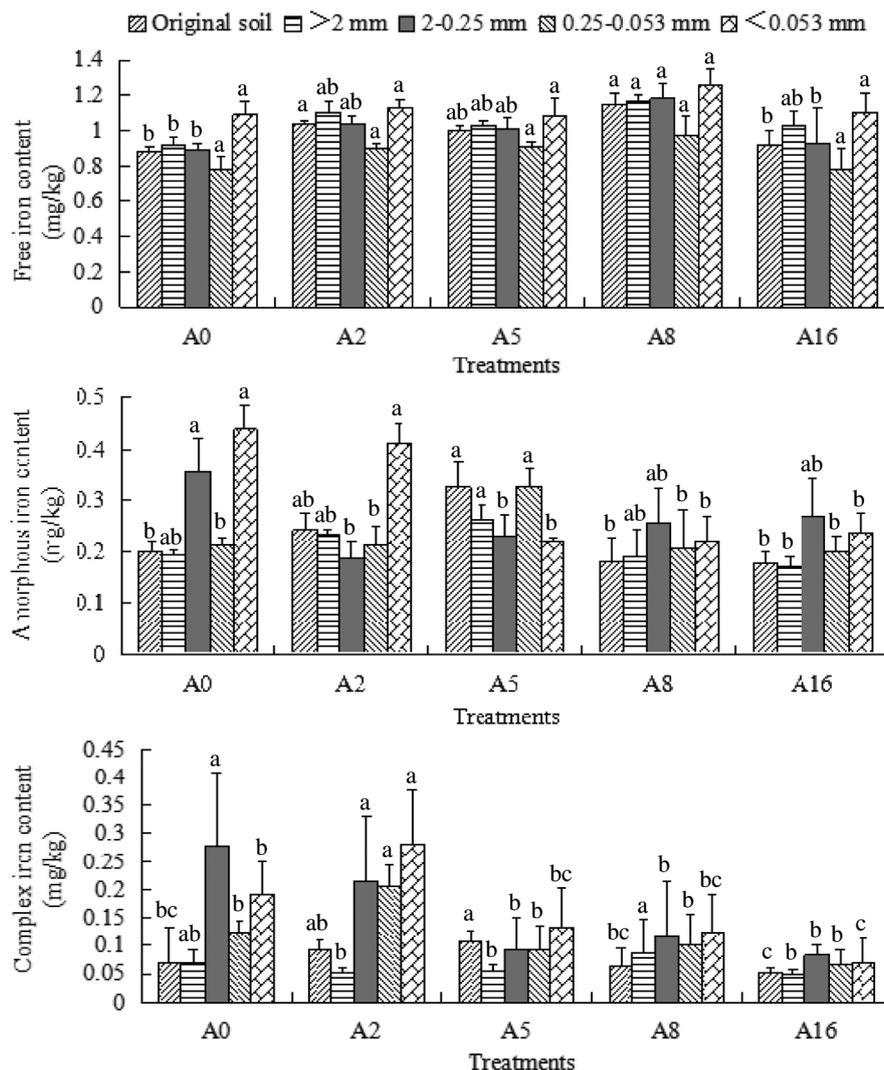


Figure 1. Contents of iron oxides in different treatments. Same letters refer to non-significant differences among different treatments ( $n = 45$ ) with LSD test ( $P < 0.05$ ).

decrease is weak. The contents of amorphous and complex iron oxides decrease considerably in the early stage of greenhouse cultivation. Free iron oxide shows no evident regularity.

The water stability of aggregates reflects the ability of the soil structure to resist erosion; therefore, correlation analysis was performed between the different forms of iron oxide composition and stability of WSA. Table 4 shows the correlation coefficients obtained for the three forms of iron oxide and aggregates with different particle sizes. Amorphous iron oxides are positively correlated with 2–0.25 mm aggregates. A high positive correlation exists between complex iron oxides and 2–0.25 mm and 0.25–0.053 mm aggregates. Free iron oxides show a good positive correlation with aggregates >2 mm; hence, free iron oxides exhibit a strong cementing ability in aggregates with large particle sizes. Amorphous and complex iron oxides are negatively correlated with aggregates

<0.053 mm, whereas free iron oxides demonstrate no evident correlation.

Greenhouses are used to achieve sustainable utilization of soil resources and improve crop yields. Studies show that excessive tillage and fertilization can seriously degrade the soil structure<sup>19,24</sup>. This study shows that for 5 years macroaggregates >0.25 mm soil decrease with increasing age in a greenhouse. But as the aggregates <0.25 mm increase, the water stability of the aggregates decrease. This indicates that macroaggregates may partially break down into microaggregates within 5 years of converting open vegetable fields to greenhouse<sup>19</sup>. After 5 years, the content of >0.25 mm aggregates increases. This indicates that microaggregates combine to form large aggregates and this improves the stability of aggregates. This improves the stability of aggregates. This result was consistent with previous reports, in which the proportion of microaggregates decreases with rise in

**Table 4.** Correlation coefficients between iron oxide and the content of water-stable aggregates

	>2 mm	2–0.25 mm	0.25–0.053 mm	<0.053 mm
Free iron oxides	0.694*	–0.686*	0.038	–0.105
Amorphous iron oxides	–0.186	0.799*	0.307	–0.436
Complex iron oxides	–0.088	0.844**	0.671*	–0.743*

\* $P < 0.05$ ; \*\* $P = 0.01$ .

macroaggregates through the action of temporary and transient binding agents<sup>24,27,28</sup>. The PAD level of greenhouse soil is high, water erosion is high, and the overall water stability is low; furthermore, changes in aggregate stability differ minimally between different years.

Soil macroaggregates are positively correlated with soil structure stability and fertility<sup>29</sup>. Experimental data shows that when large aggregates exist in small proportions, soil stability is low. Aggregates with 2–0.25 mm and 0.25–0.053 mm sizes significantly decrease after 2a, and aggregates with 0.25–0.053 mm sizes demonstrate high proportion in the soil. With increasing planted years, the content of <0.053 mm aggregates increase but the content of >2 mm aggregates remains constant. These results explain that larger aggregates are decomposed into smaller microaggregates under greenhouse cultivation.

Amorphous and complex iron oxides are positively related to 2–0.25 mm and 0.25–0.053 mm aggregates, but the correlation is low. As compared to the amorphous form, complex iron oxide, despite its low content, exhibits high correlation with the aggregates. These results are in line with those of Filimonova *et al.*<sup>5</sup>, who found that Fe precipitation is closely linked to the soil formation processes. In addition, Powers and Schlesinger<sup>6</sup> also insisted that amorphous Fe was an index of noncrystalline clays and was negatively related to clay proportion. In our study, complex and amorphous iron oxides are negatively correlated with <0.053 mm aggregates, and the correlation of the complex is high. This shows that the increasing proportion of microaggregates is mainly due to the decomposition of large-sized aggregates. Moreover, the contents of complex iron oxides in 2–0.25 mm and 0.25–0.053 mm aggregates significantly decrease after more than 2 years, resulting in the collapse of large-sized aggregates into two particle fractions. The possible mechanism is that humic molecules are bound to clay particles via strong clay–humus complexes, and this process is mediated by pyrophosphate-extracted Fe<sup>30,31</sup>. Humic acids can also be coordinated with Fe<sup>3+</sup>, which forms bridges between the clay surface and humic acid<sup>31</sup>. These results reinforce the previous findings<sup>31,32</sup> that at the colloidal level, aggregates are formed and stabilized through polyvalent Fe found on the edges or faces of clay minerals.

Researchers confirmed the important functions of amorphous iron oxide in soil aggregation<sup>10,33,34</sup>. The cementing effect of free iron oxide is only slightly

important in solar greenhouse soil, as supported by Jozefaciuk and Czachor<sup>10</sup>. Organic matter can form a complex with iron oxides at low pH to form mobile, organic–metallic compounds, which decrease microbial access to soil organic carbon and mineralization<sup>33</sup>. Amorphous iron oxide and organic matter can form fine stable particles in soil<sup>9</sup>. Amorphous iron oxide that underwent simple sorptive processes on soil organic matter surface is more important than free iron oxide in terms of aggregate stability<sup>35</sup>. In the present study, complex iron oxide appears more prominent as aggregating agent than amorphous and free iron oxide. As a result, the aggregate structure of the main body deteriorates and WSAs are transformed into smaller particles. In the experiment, the water stability of aggregates increases after five years; this could be related to the cementation of organic matter in the soil<sup>36,37</sup>. However, the specific mechanism must be further studied.

The water stability of greenhouse soil aggregates was poor, and the proportion of >0.25 mm WSA was very low. The water stability first decreased and then increased after A5, but the change was not evident. In addition to A16, the water stability of aggregates in other years was lower than the open-field soil. Changes in the different forms of iron oxide varied. Free iron oxides did not differ significantly, but cementation with >2 mm aggregates was evident. Amorphous and complex iron oxides mainly existed in <0.25 mm microaggregates, and the contents decreased compared with those in the soil in A2. This size of microaggregates exhibited a positive correlation with complex iron oxides. The contents of amorphous and complex iron oxides decreased because of the high-strength cultivation of greenhouse soil, resulting in the conversion of microaggregates into small particle sizes. Hence, WSAs were destroyed, and their stability decreased.

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