MICROMORPHOLOGY OF SYNTHETIC SOIL AGGREGATES

by

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Introduction

Calcium carbonates as well as iron oxides are sup - posed to stabilize soil aggregates. The latter is sugges ted by the fact that soils rich in iron oxides often show high aggregate stability. Also, this effect was demons - trated in various studies (see references in Schahabi & Schwertmann 1970 a). By contrast in some Australian soils where iron oxides were present as single crystals no aggregating effect or iron was observed, and Al-oxi des were shown to be responsible for aggregation (Geen land et al. 1968, Deshpande et al. 1968).

The effect of liming on aggregate stability is still disputed. Positive as well as negative effects have been observed (Scheffer 1970, Becher and Schwertmann 1973).

The aim of this paper was to investigate the spatial distribution of "amorphous" and crystalline iron oxides and calcium carbonate in synthetic soil aggregates on which aggregate stability measurements have revealed widely different aggregation effect (Schahabi and Schwert- mann 1970 a, b).

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SYNTHETIC SOIL AGGREGATES

Materials and methods. –

Synthetic soil aggregates were prepared by mixing silty materials of extremely low aggregate stability with increasing proportions of various synthetic iron oxides and calcium carbonate. Some analytical data of the two soils an A_1 horizon of a Parabraunerde and an A_{1g} horizon of a Pseudogley both derived from loess are given in table 1.

The aggregates were prepared as follows: The iron oxides were washed free of anions and mixed with the air dried soil to yield 1 to 4 weight per cent of Fe. Then additional water was added until the m i x t u r e contained 40 wt. % water. After this procedure the mixture were dried at 35º C and sieved through a 2 mm sieve. For preparation of $CaCO_3$ treated aggregates 1 Kg of soil was mixed with water and CaO until a pH of 7. 0 was obtained. The samples were then mixed three times with $CaCO_3$ (5 wt. %) dried and sieved (2 mm). Further details are given in Schahabi and Schwertmann (1970 a, b).

Thin sections of aggregates were prepared with VESTOPAL 150 using Altemüller's method (1962). Quantitative estimations were carried out using the point counting method.

Results.

Untreated aggregates exhibit a well dispersed, pale plasma. Large, circular voids (vescicles) of 100–300 μ m in diameter occur in nearly all aggregates (Fig. 1). Ferruginous nodules and ferriargillans are present in some aggregates, probably relicts of soil formation.

Aggregates with 1. 4 % and 1. 0 % Fe added as ferrihydrite (formerly called amorphous ferric hydroxide) , still contain numerous large voids. In contrast to the untreated samples the voids often show an irregular shape.

The plasma of these aggregates has a higher hue. Flaky iron oxide aggregates which tend to form ferrans are embedded between the skeleton grains.

After addition of a 2 % Fe as ferrihydrite nearly all skeleton grains are covered with an iron oxide coating. Thin sections show that the improved water stability of these aggregates might be due to formation of ferrans throughout the sample (Fig. 2, black arrows). Larger voids are very rare in these aggregates indicating a higher degree of cementation.

Addition of larger amounts of Fe (> 2 % Fe) does not seem to improve cementation of skeleton grains, again in agreement with wet sieving analysis. Ferrans appear to be somewhat thicker in these aggregates (Fig. 3) but, these is no evidence in the thin sections for a better aggregation. Excess of iron oxides is obviously accumulated as discrete iron oxide particles rather than as coatings (Fig. 4.). Schahabi and Schwertmann (1970 a) observed that at 4 % Fe discrete iron oxide particles separate out if the mixture is suspended in an excess of water. - These was no difference in size and shape between aggregates formed from the A_1 and A_{1g} horizon materials.

Aggregates which were prepared using well crystalline iron oxides exhibit different fabrics. Large circular voids are present in hematitic and goethitic aggregates. In contrast to the ferrihydritic aggregates they show an irregular distribution of Fe-oxides (Fig. 5) indicating that a homogenous retention of the Fe oxide particles at the surface of the silt grains does not take place to the same extent.

Aggregates with hematite and goethite show large areas without any iron (Fig. 6+7). In goethitic aggregates these parts are often separated by ferrans.

Although some ferrans were formed in hematitic aggregates it looks as if only a weak association exists bet -

ween skeleton grains and oxides. Aggregates high in Fe exhibit a diffuse network of iron oxides which loosely co ver the skeleton grains (bottom of Fig. 8).

Lepidocrocitic aggregates do not show any formation of ferrans. Small lepidocrocite aggregates are scattered through the pale soil plasma (Fig. 9). There is no indi- cation whatsoever for cementation of skeleton grains by the iron oxide. The lack of association of the well crys_ talline iron oxides with the silt particles explains why these oxides fail to improve aggregate stability.

Because it was assumed that aggregate stability   is mainly produced by the amount of plasma lying between the skeleton grains, plasma and voids were counted on the thin sections. However, by this method a distinction between plasma active in cementation (e. g. ferrans) and mert plasma (e. g. Fe—xide flocs)cannot be made. Results (tabl. 2) indicate that untreated (No. 26, 37) and ferrihy dritic aggregates with 1. 4 and 1. 0 % Fe added (No. 14, 18) contain less plasma and more skeleton grains. There are only slight differences between plasma portions of samples treated with 2. 0 to 4. 0 % Fe of ferrihydrite (No. 15–17, 19–20). Lower amounts of plasma in sample No. 21 result from formation of large iron oxide parti – cles which occure frequently in this sample.

In calcareous aggregates large circular voids also occur abundantly (Fig. 10). There seems to be no diffe- rence between these aggregates and the untreated ones. At high magnifications (300 x) a random distribution of small calcite particles (6– 20   m) are recognized under crossed polarizers (Fig. 11 small white spots). No indi cation was obrained for cementation of skeleton grains by these calcite grains again in aggreement with results from wet sieving analysis (Schahabi and Schwertmann , 1970 b).

Discussion and conclusions

Results of aggregate stability measurements by Scha habi and Schwertmann (1970 a, b) can be summarized as follows (see also Fig. 12 and 13):

1. - Addition of $CaCO_3$ to loessial soils which have lost most of their clay and iron oxides by clay migration and iron reduction did not improve efficiently their aggregation.

2. - Addition of poorly cristalline ferrihydrite (1-4 % Fe) improved aggregate stability significantly, up to about 2 % Fe.

3. - Addition of well crystalline iron oxides did not impro ve aggregate stability at all.

Micromorphological investigations confirm these re-sults. The presence of large circular voids in untreated soil aggregates and in those to which $CaCO_3$, and well crystalline iron oxides were added indicate the instabili ty of these aggregates. It is supposed that the circular voids were formed by air bubbles during aggregate for-mation.

The strong aggregation effect of ferrihydrite can be atributed to ferran formation indicating a strong tenden-cy for the high surface area of ferrihydrite ($\sim$250-300 m^2/g) to associate with the silt particle surface. The fact that aggregate stability did not increase beyond 2 % Fe was substantiated by the observation that discrete iso lated oxide particles (aggregates) occur at this Fe level. Moreover it was noticed that ferran formation was com-pleted at about 2. 0 % Fe.

The ineffectiveness of well crystalline Fe oxides (crystal size from EM 0. 05 - 1μm) on soil aggregation as observed by Schahabi and Schwertmann (1970 a), Greenland et al. (1968), and Deshpande et al. (1968)must then be attributed to the lack of ferran formation, i. e.

insufficient ability of the crystals to be retained at the
surface of silt particles. In the same manner the poor
aggregation effect of calcite crystals may be explained.
The general observation that Fe oxide cemented bodies
in soils (nodules, crusts, pipe stems) quite often contain
well crystalline Fe oxides does not necessarily conflict
with these results. Firstly, the amount of Fe in these for
mations is in general much higher than in the synthetic
aggregate so that the skeleton grains are embedded in
iron oxides and secondly, in contrast to the synthetic
aggregates the iron oxide crystals presumably grow in
the presence of the skeleton grains to be cemented.

Table 1: Analytical data of soil material from two loess soils

material	particle size distribution (%)					pH (0.01 m CaCl$_2$)	Organic Material	CEC (me/100 g)	Fe$_d$ (% Fe)
	< 2	2-6	2-20	20-60	>60				
	µm								
A$_l$	5.5	2.8	25.3	60.9	4.5	3.7	1.30	9.2	0.34
A$_{lg}$	4.7	3.1	25.4	65.0	1.8	3.8	0.96	7.0	0.25

Table 2: Distribution of skeleton grains, plasma and voids of ferrihydritic synthetic soil aggregates

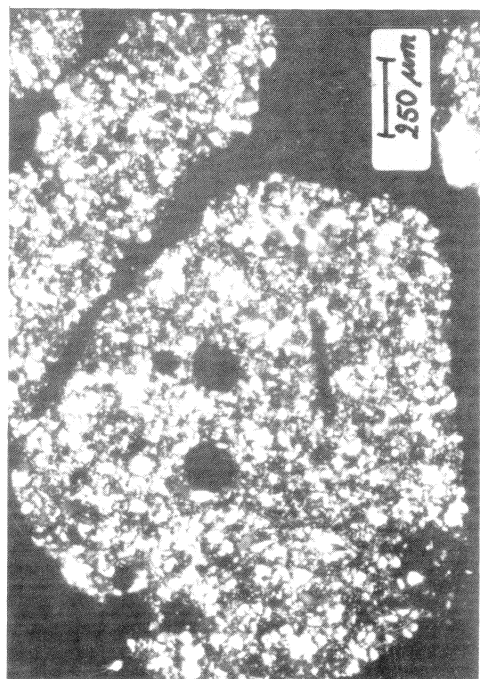| sample No.	iron added ($\%$ Fe_d)	skeleton grains	plasma (Vol $\%$)	voids	points counted
26	0.0	58	30	12	800
14	1.0	52	36	12	800
15	2.2	50	42	8	800
16	2.8	42	45	13	800
17	3.6	45	43	13	800
37	0.0	62	26	12	800
18	1.4	57	32	11	800
19	2.0	50	40	10	1000
20	2.9	47	42	11	800
21	4.0	46	38	16	800

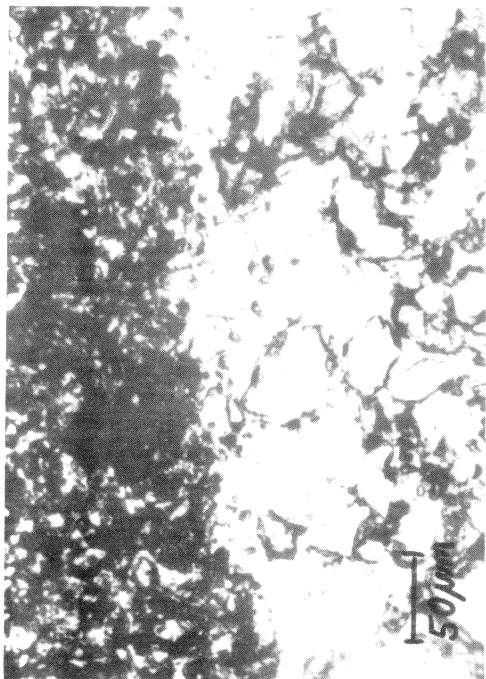Fig. II

Fig. 12

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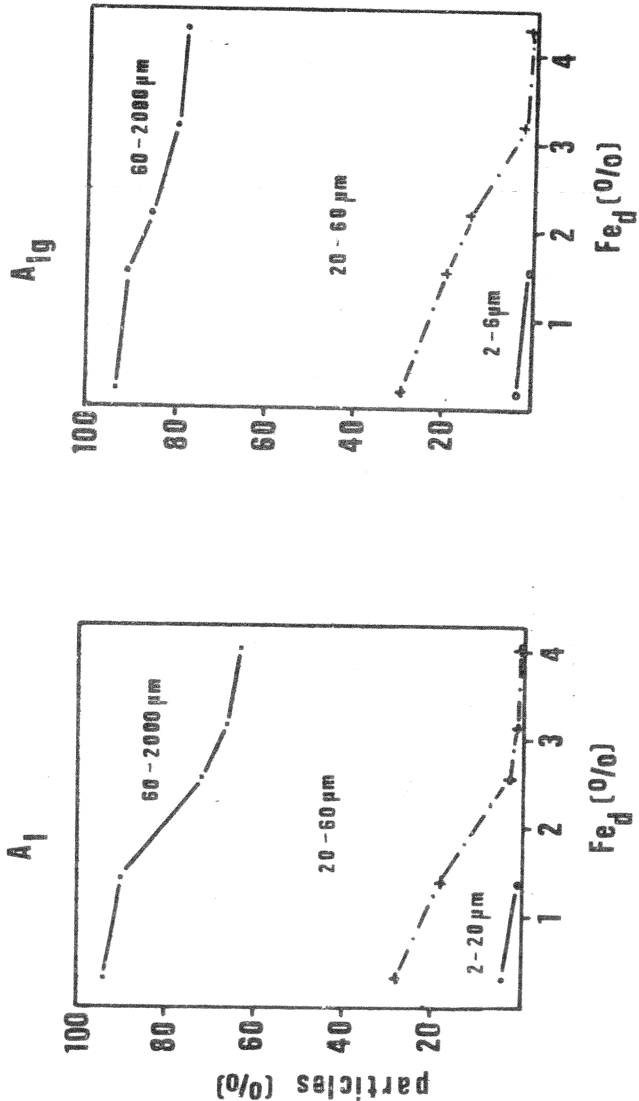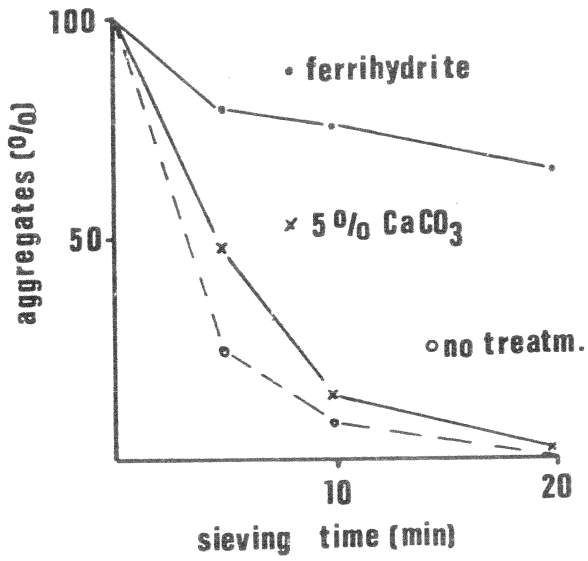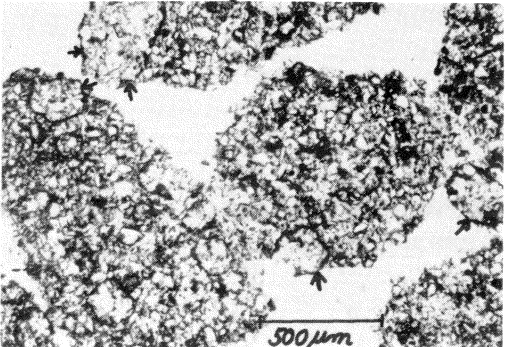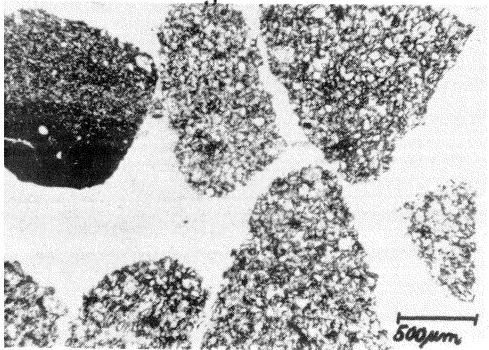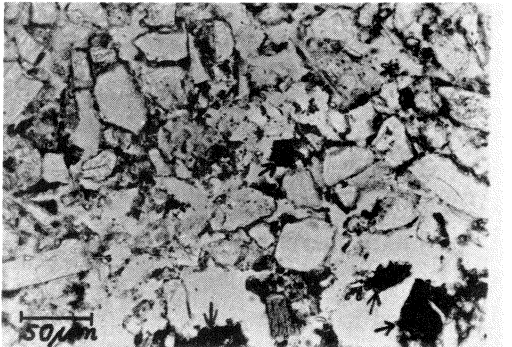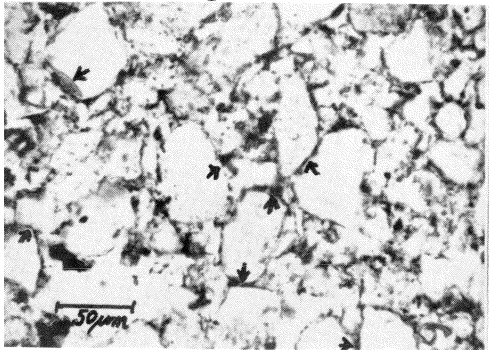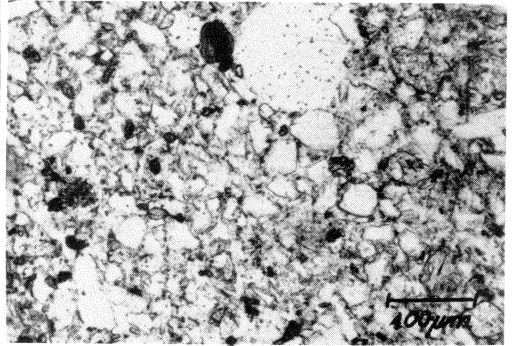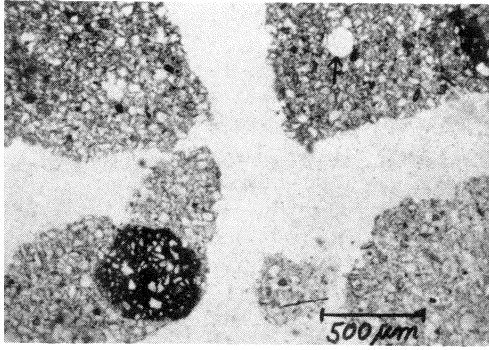
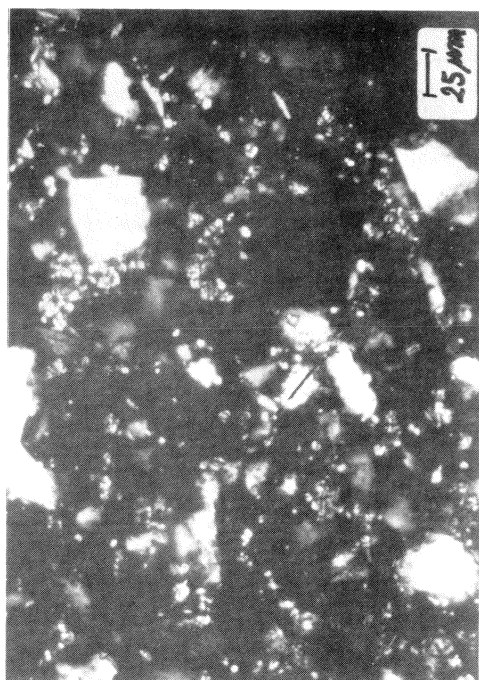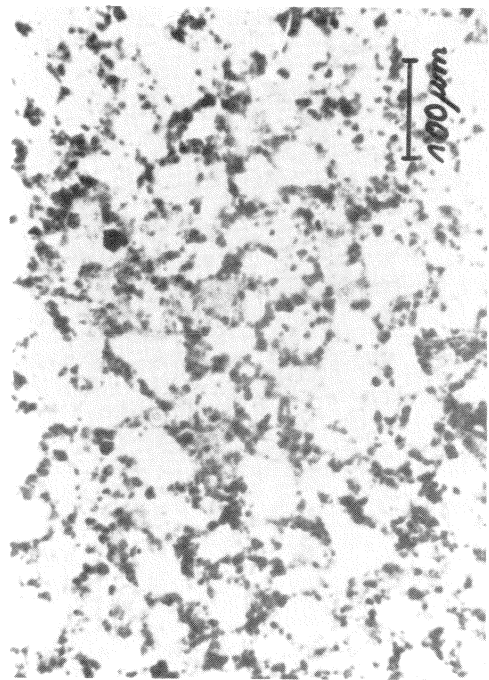## Captions of Figures

Fig. 1: Aggregates of untreated silty material (A_1 horizon No. 26.) with sesquioxidic nodule and circular void (arrow).

Fig. 2: Same as Fig. 1 at higher magnification (note the circular void).

Fig. 3: Thin section of silty material with ~ 2 % Fe added as ferrihydrite (No. 15). Skeleton grains covered with ferrans (arrows).

Fig. 4: Thin section of aggregate prepared by mixing 4. 0% Fe as ferrihydrite. (No. 21) showing discrete iron oxide particles (arrows).

Fig. 5: Aggregates after addition of hematite showing an irregular distribution of Fe-oxides.

Fig. 6: Aggregates with goethite showing large areas without any iron seperated by ferrans (arrows).

Fig. 7: Thin section of hematitic aggregate.

Fig. 8: Thin section of aggregate after addition of lepidocrocite.

Fig. 9: Aggregates prepared by mixing 5% $CaCO_3$ (crossed nicols).

Fig. 10: Same as Fig. 9 at higher magnification (crossed nicols).

Fig. 11: Particle size distribution (dispersion in water of A_1 and A_{1g} material as influenced by the addition of increasing amounts of ~ferrihydrite expressed as dithionite soluble Fe of the mixture (according to Schahabi and Schwertmann, 1970 a).

Fig. 12: Percentage of water stable aggregates 1–2 mm of the A_{1g} horizon as related wet sieving time before and after addition of $CACO_3$ and ferrihydrite (according to Schahabi and Schwertmann, 1970 b).

SINTHETIC SOIL AGGREGATES

SUMMARY

Soil aggregates were prepared by mixing various synthetic iron oxides or $CaCO_3$ with silty material from loess soils poor in clay and free iron. Micromorphological examinations on these aggregates showed that the sta bilizing effect of ferrihydrite (formerly called amorphous ferric hydroxide) can be attributed mainly to the formation of ferrans. After addition of about 2 % Fe nearly all ske leton grains were covered with Fe oxides and the maximum aggregation was produced. Addition of further ferrihydri te leads to formation of discrete iron oxide flocs. The low effectiveness of well crystallire Fe oxides (goethite, hematite, lepidocrocite) is due to incomplete association of these oxides with the surface of skeleton grains, so that the crystalline Fe oxides were distributed randomly between the skeleton grains. Addition of $CaCO_3$, which also was unable to improve aggregate stability, resulted in si milar random distribution of calcite crystals between the skeleton grains.

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