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MAGNETIC PROPERTIES OF DIFFERENT-AGED CHERNOZEMIC SOIL PROFILES

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ABSTRACT

In order to estimate the rate of magnetic susceptibility enhancement in automorphic temperate soils, magnetic properties and mineral weathering degree of different-aged chernozemic soils derived from a uniform parent material have been studied. In this work, layer samples of mature virgin leached chernozem and young chernozemic soils formed on the embankment of an earthy archaeological monument were used. Magnetic, physical and chemical and mineralogical analysis show that magnetic susceptibility enhancement in organogenic soils is associated with increase in loss on ignition, cation exchange capacity, degree of dispersion, as well as with decrease in amphiboles/zircon, amphibole/rutile and amphibole/ilmenite ratios. Magnetic susceptibility enhancement in different-aged chernozemic soils results from maghemite (or maghemite associations) formation. After 750-800 years, magnetic susceptibility in organogenic soils reached only about half of its value in a mature chernozem. These results indicate that the formation of mature magnetic profile in automorphic temperate soils is a very long process. The newly formed chernozemic soils are now at the stage of active formation of secondary magnetic minerals, but the resource of primary ferrous silicates (which are less resistant to weathering) is not exhausted in mature virgin chernozem yet.

Keywords: soils of earthy archaeological monument, virgin chernozem, newly formed chernozemic soils, magnetic susceptibility, soil profile distribution.

1. INTRODUCTION

Actual data on magnetic properties of the soil show that magnetic characteristics in the zonal series of the Russian plain soils naturally vary with depth, showing a close relationship to the type and intensity of the soil-forming process [1]. Magnetic properties depend primarily on the iron content, its phase composition and dispersion [2]. Ferrimagnetic minerals in most of the modern automorphic soils in the northern hemisphere, formed in the intensively altered unconsolidated sediments of the Quaternary age, can be detrital and authigenic. The majority of authigenic magnetic minerals in soil are formed in the organogenic horizons where chemical and biological weathering is most intense [1]. According to modern concepts, ferromagnetic minerals can accumulate in the soil horizons due to various causes, which are not confined to the *in situ* pedogenic processes [3], [4].

However, despite the widespread use of magnetic measurements in the study of soils, as well as fluvial, limnic and marine sediments and other natural objects potentially containing material of soil profiles at different stages of maturity, the mechanisms of magnetic mineral formation in soils remain a subject of debate. Anyway, we can confidently state that magnetic profile of the soil results from complex processes of energy and mass transfer, leading to vertical differentiation of the original parent rock's composition and properties.

The time it takes to reach quasi-equilibrium under the current bio-climatic conditions can vary considerably for different characteristics (composition, properties) of the soil profile formed on the exposed unconsolidated deposits.

The time scale for the Holocene soil humus profile formation is no longer than thousands of years, because longer processes in the post-glacial period cannot be realized [5]. As to the magnetic profile, the current information on the rates and characteristic times of its development is insufficient and often contradictory.

The major part of the experimental data was interpreted in terms of magnetic susceptibility of soils, depending on time; the data were obtained in the study of soil chronosequences on river and marine terraces [6], [4] and modern loess-soils and loess-palaeosoils sequences [7], [8]. Information about kinetic parameters of the magnetic susceptibility enhancement in automorphic and well-drained temperate soils most consistently and thoroughly is summarized in the chemical kinetic model proposed by Boyle et al [9].

Magnetic susceptibility enhancement in this model is explicitly associated with weathering duration, and the main controlling variables are ferrous silicate concentration in the soil-forming rocks and mean annual precipitation, with a lesser role for mean annual temperature. The process kinetics consists of two stages. The first stage (the characteristic time of ~10^4 years) involves active production of secondary magnetic minerals from Fe^{2+} released by weathering of the less resistant primary minerals (e.g., chlorite). As the primary minerals deplete, magnetic susceptibility enhancement rate is reduced. The second stage (the characteristic time of ~10^5

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years) involves slow increase in the secondary ferrimagnetic minerals concentration, which is due to weathering of the less reactive minerals (e.g., feldspars). The contribution of dissimilatory Fe-reducing microorganisms to magnetic susceptibility enhancement in the model is ignored.

According to the model developers themselves, the chemical kinetic model possesses elements of uncertainty; however, following its logic, we can assume that the Holocene soil magnetic profile is currently at the stage of active formation of secondary magnetic minerals.

Experimental study of the soil profile formation dynamics is complicated by the long duration of soil-forming processes. To date, however, a considerable expertise has been built up on estimation of the soil profile formation rate resulting from the study of soils developed on naturally overgrown dumps of different age and soils of earthy archaeological monuments [10], [11] [5] [12] [13],[14].

It was also found that the characteristic time of chernozem humus profile formation is $3\text{-}4\times10^3$ years, which is shown by the example of the East European Plain soils formed on the earth embankments of different ages [5]. Under specific conditions of the Southern Trans-Urals forest-steppe zone, total thickness of the newly formed chernozemic soil humus profile - even after 4×10^3 years – has reached less than 3/4 that of the background leached chernozem humus profile [14]. However the kinetic parameters of magnetic profile formation for chernozems have not been studied in details yet.

This paper is devoted to the study of magnetic properties and degree of mineral weathering in profiles of different-aged chernozemic soils derived from a uniform parent material. In this work, layer samples of virgin leached chernozem and chernozemic soils formed on the embankment of an earthy archaeological monument were used.

2. MATERIALS AND METHODS

Experiments were conducted on different-aged chernozemic soils taken from Bol'she-Klyarinskoe ancient settlement (a fort) which was founded in Volga Bulgaria between the 12th and 14th centuries [15].

It is located on a gentle slope near the Sukhaya Ulema River (Kamsko-Ust'inskii district, Central Volga region) and survived to the present day in a form of ruined defensive earthworks and fosses (Figure-1). Before this fort was built, the soil cover in the area was represented only by medium-thick clay loamy leached chernozem on yellowish brown heavy deluvial loam.

Stratigraphic analysis of the outer earthy wall of the fort was carried out in 2004 by archaeologists of the National Centre of Archeological Researches (Institute of History, Tatarstan Academy of Sciences). It was found that the wall was made by placing earth from the adjacent fosses (from both sides) onto the buried soil surface between them. The upper part of the wall was covered with parent rock material. Younger soils had been forming on the top of the earth wall since the fort was fully constructed.

Experiments were conducted on samples taken from the profiles of virgin leached chernozem in the area adjacent to the north side of the fort, and from the chernozemic soils formed on the external slope, on the top and on the internal slope of the outer earth wall in the lower part of the fort (Figure-1).

Diffraction spectra obtained through the use of powder mounts show high similarity between the parent rocks in the entire area studied [16]. Initial vertical uniformity of the parent rock material can be seen from the concentration ratio of Ti, Zr, and Y [17].

Preparation of soil samples was carried out according to the recommendations of the International Organization for Standardization [18]. To remove carbonates, samples were treated with 1 mol/l CH_3COOH solution (for 24 hours) and washed off with distilled water. Then, to remove organic matter (OM), the samples were treated with 30% H_2O_2 at room temperature for 15-20 days. The precipitate was separated by centrifugation, then dried and homogenized. Carbonates and OM were also removed before separation of particles with Stokes diameter of <2.5 μm and heavy mineral fractions with >2.9 $g\times cm^{-3}$ density was performed. The clay fraction was separated by the elutriation after sedimentation in a liquid column. The heavy mineral fraction was separated by repeated centrifugation in bromoform.

Magnetic susceptibility values were determined through the use of Bartington Instruments MS2B magnetic susceptibility meter. Each sample was studied in two modes - at high and low frequency (0.460 kHz for χ_{lf} and 1.65 kHz for χ_{hf}). To do this, the samples were crushed in an agate mortar and mass normalized. Thermomagnetic analysis (TMA) was performed with the aid of Curie express balance [19]. Intensity of inductive magnetization at different temperatures (heating rate – 100 °C/min, up to 800 °C) in the constant magnetic field of 0.2 T was measured. The TMA curves were recorded for the first and second runs to 800 °C.

Coercive spectra of normal magnetization in the magnetic fields of up to 0.5 T were obtained by means of coercive spectrometer («J_meter») [20], which allows separate recording of remanent and inductive magnetization at room temperature. The samples were magnetized from their natural state.

Bruker Corporation D2 Phaser Powder Diffractometer (Bragg-Brentano θ-θ geometry, 30 kV and 10 mA on X-Ray tube, Ni-filtered CuKα, goniometer radius - 141.4 mm, fixed slits, 1-dimensional LYNXEYE detector, sample rotation) was used to obtain diffraction spectra from powder samples. DIFFRAC.SUITE software pack was used for spectra processing. DIFFRAC.EVA (v-3.1) along with ICDD PDF-2 Release 2013 diffraction data base was used for qualitative analysis. Quantitative analysis of bulk samples was made using the RIR method [21]. Crystal phase ratio calculation for the >2.9 $g\times cm^{-3}$ fraction was made using the Rietveld method with DIFFRAC.TOPAS (v-4.2).

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Cation exchange capacity (CEC) was determined using the CINAO modification of the Bobko-Askinazi method [22]. "Amorphous" iron oxides (Fe_O) were extracted in the dark using ammonium acid oxalate (Tamm reagent) [23]. Iron content was determined photometrically with sulphosalicylic acid [24], [25]. Free iron oxides (Fe_D) were extracted with dithionite-citrate-bicarbonate (Mehra-Jackson reagent) [26]. Iron content was determined photometrically with potassium thiocyanate [23]. Humus content was determined using the Tyurin method [27], loss on ignition (LOI) was measured at 900 °C.

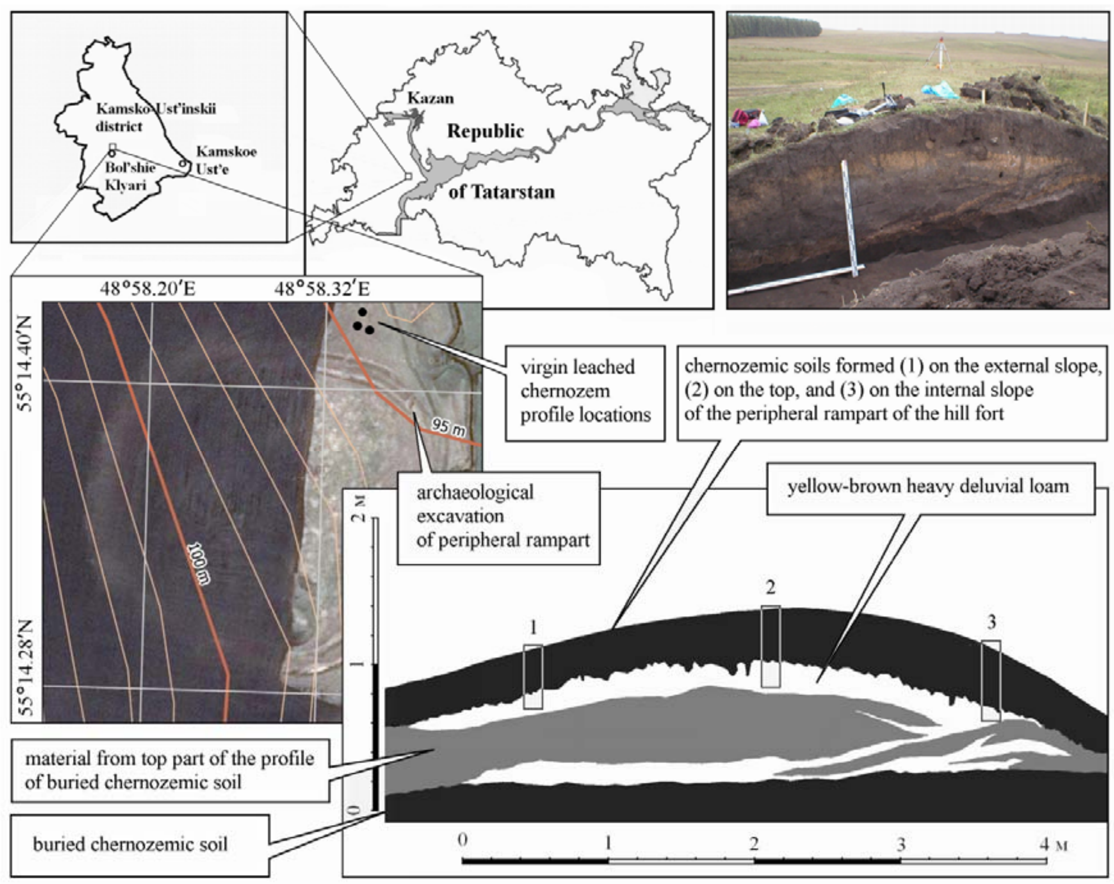

Figure 1. Studied site and location of the sampled soil profiles.

The data obtained were processed using MS Excel. According to the Shapiro-Wilk test criterion for the significance level of $\alpha = 0.05$, experimentally measured values of magnetic susceptibility, CEC, Fe_O, Fe_{D-O}, LOI and humus content were characterized by normal distribution in all profiles of different-age chernozemic soils. Therefore, parametric indexes were used to assess relationship between the measured data.

3. RESULTS AND DISCUSSIONS

Graphical analysis shows that the background virgin chernozem is characterized by cumulative type of magnetic susceptibility and humus content distribution profiles (Figure-2). Selective OM removal has no effect on the magnetic susceptibility profile differentiation. Somewhat elevated absolute values of χ_{lf} at the top and bottom of the profile may be attributed to removal of OM and carbonates, because magnetic susceptibility measurement was conducted on mass normalized samples. In the humus part of the soil profile magnetic materials are present predominantly in fine mineral particles, and their content in the <2.5 μm fraction is reduced toward the soil-forming rock.

Cumulative distribution of magnetic susceptibility in the virgin soil formed on the vertically uniform parent material presupposes pedogenic origin of magnetic materials. Pedogenic magnetic particles are usually fine grained, and the difference between magnetic susceptibility values (χ) at different frequencies reflecting the presence of superparamagnetic fine grains is considered an indicator of bio-inert interactions [29].

Calculation of the frequency-dependent magnetic susceptibility ($F = (\chi_{lf}-\chi_{hf})/\chi_{lf}100\%$) shows that the F-factor values range from ~2 to ~11% in original soil samples and from ~6 to ~15% in <2.5 μm fraction – in the soil-forming rock and the upper parts of profile, respectively. As in the case of magnetic susceptibility, the virgin chernozem is characterized by cumulative F-factor

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values distribution curve. It is not necessarily imply that magnetic properties and humus content are functionally dependent on each other. Note, however, that similarity in behavior of the profile curves may be partly due to OM and the contribution of biogenic accumulative organic surface horizons to the creation of optimal conditions for heterotrophic microorganisms capable of synthesizing magnetic nanoparticles [2].

Coercive spectra allow us to determine the contribution of dia-/paramagnetic (χ_p) and ferromagnetic (χ_f) component to the magnetic susceptibility [30]. χ_p and χ_f distribution curves clearly prove that magnetic susceptibility enhancement in organogenic horizons of virgin chernozem is due to the contribution of ferromagnetic components (Figure-2).

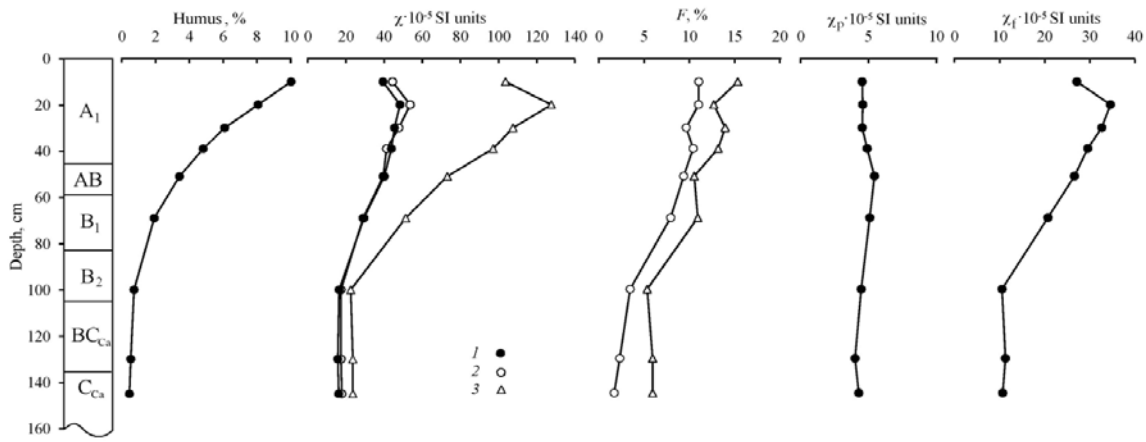

Figure-2. Humus content and magnetic properties as a function of depth in the profile of virgin leached chernozem: *1* – original soil samples; *2* - soil samples after the OM removal; *3* – <2.5 μm fraction isolated after the removal of carbonates and OM.

Magnetic susceptibility in soil profiles is often correlated with the iron content. Strong correlations (at a level of significance $\alpha = 0.001$) can be seen between magnetic susceptibility and oxalate-extractable Fe (Figure-3), and between magnetic susceptibility and Schwertmann's criterion values (Figure-4). There is no correlation between magnetic susceptibility and crystalline Fe oxides content (determined by the difference between contents of dithionite-citrate-bicarbonate-extractable Fe and oxalate-extractable Fe) (Figure-3).

This, however, does not exclude the possibility of strong magnetic iron oxides to form in the surface horizons. Correlation analysis results can be technically regarded as evidence of dominating contribution of "amorphous" and poorly crystalline Fe oxides to humus profile magnetic properties. However, it is well known that magnetite is poorly soluble in Mehra-Jackson's reagent and, at the same time, is highly soluble in Tamm's reagent, especially in the form of dispersed particles [2].

The soils formed on the outer wall of the fort are morphologically similar to the background leached chernozem. Despite the fact that humus profile of the newly formed chernozemic soils is less thick than that of the background soil, it includes a set of morphologically recognizable organogenic horizons (A_d+A_1+AB). When comparing layer samples, no significant differences in the humus content of A_d, A_1 and AB horizons can be found (Figure-5).

At the same time, the absolute values of χ_{lf} in humus profiles of the newly formed chernozemic soils are considerably less than that in humus profile of the background chernozem. Independent two-sample t-test results show significant differences between magnetic susceptibility of organogenic horizons in the leached chernozem and the chernozemic soils formed on the outer wall of the fort ($t_{cal} = 11.26$, $t_{tab} = 2.04$). Thus, we can conclude that in the organogenic horizons of different-aged chernozemic soils humus content increases much rapidly than magnetic susceptibility.

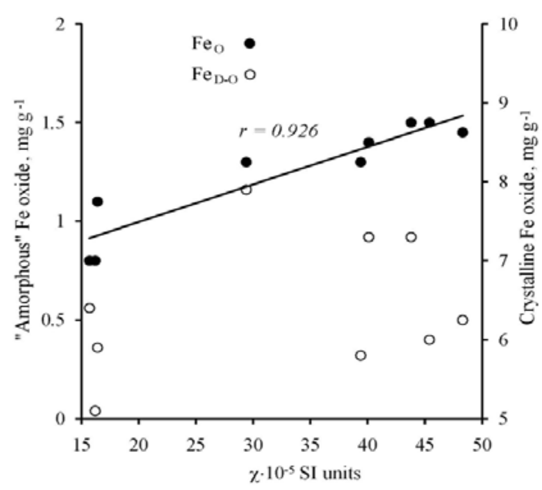

Figure-3. Relationship between magnetic susceptibility and contents of "amorphous" (Fe_O) and crystalline (Fe_{D-O}) Fe oxides in layer samples of virgin leached chernozem.

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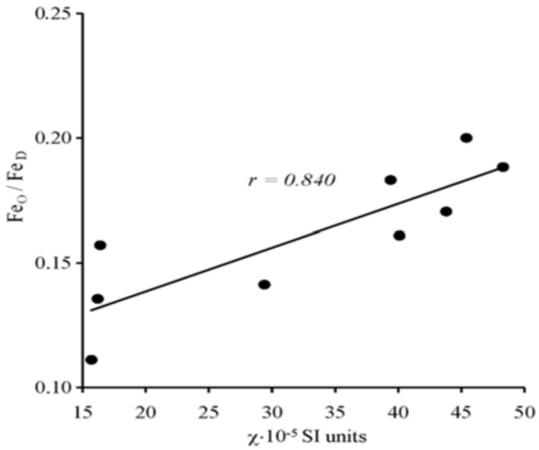

Figure-4. Relationship between magnetic susceptibility and crystallinity degree of free Fe in layer samples of virgin leached chernozem.

Other conditions being equal, this may be due to differences in kinetic parameters of humus formation and structural transformation of the parent rock.

Since there are no significant differences in humus content between organogenic horizons of different-aged chernozemic soils, LOI and CEC can be used as indirect indicators of mineral weathering degree. The change in the sample mass during the heating can be both due to OM loss and mineral dehydration and dehydroxilation. CEC is determined by the number of functional groups on the surface of both organic and mineral phases. Therefore, both characteristics depend on composition and structural properties of the soil mineral matrix. LOI is used to evaluate rock weathering degree either separately [31] or as a component of the equations for chemical weathering indices calculation [32].

Heating up to 900°C leads to considerably smaller mass losses in the middle part of the humus profile (A_1 horizon) of the newly formed soils comparing to the background chernozem (Figure-5). According to the independent two-sample t-test results, this difference is significant (t_{cal} = 2.18, t_{tab} = 2.10). Similarly, CEC is less in the middle part of the humus profile (A_1 horizon) of the newly formed soils than in the middle part of the virgin chernozem humus profile (t_{cal} = 4.69, t_{tab} = 2.10).

CEC in humus profiles of chernozemic soils usually correlates with LOI and humus content. For all the data, taken as a whole, there is a low (Figure-6a), or close to medium correlation (Figure-6b). However, if we analyze the data separately for the background and the newly formed soils, we can see that correlation coefficients are significantly increased. They show medium (for the newly formed soils) and strong (for the background soil) correlation between the compared parameters at a level of significance α = 0.01 and α = 0.001, respectively. Two point clouds – for the background chernozem and the newly formed soils – can be observed in the correlation field (Figure-6b). Moreover, two distinctive and almost non-overlapping point clouds can be seen in the correlation field of magnetic susceptibility, LOI and CEC (Figure-7).

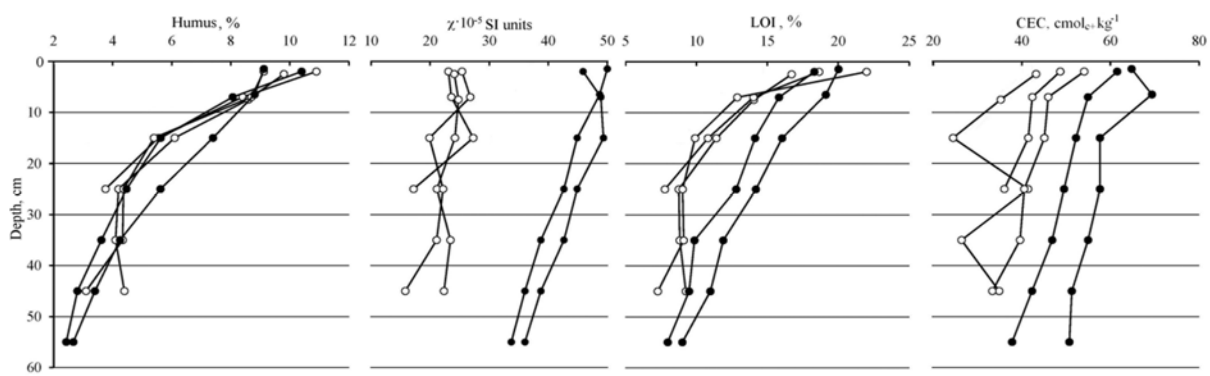

Figure-5. Humus content, magnetic susceptibility, loss on ignition and cation exchange capacity as a function of depth in the humus profiles of the virgin leached chernozem (●) and the newly formed chernozemic soils (○)

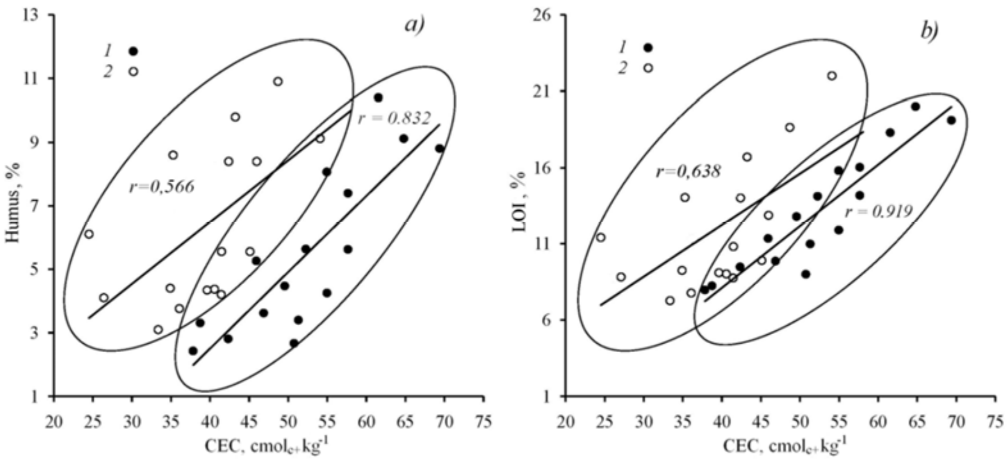

Figure-6. Relationship between cation exchange capacity and (*a*) humus content, (*b*) loss on ignition for layer samples taken from the humus profiles of the different-aged chernozemic soils: *1* – virgin leached chernozem *2* – newly formed chernozemic soils.

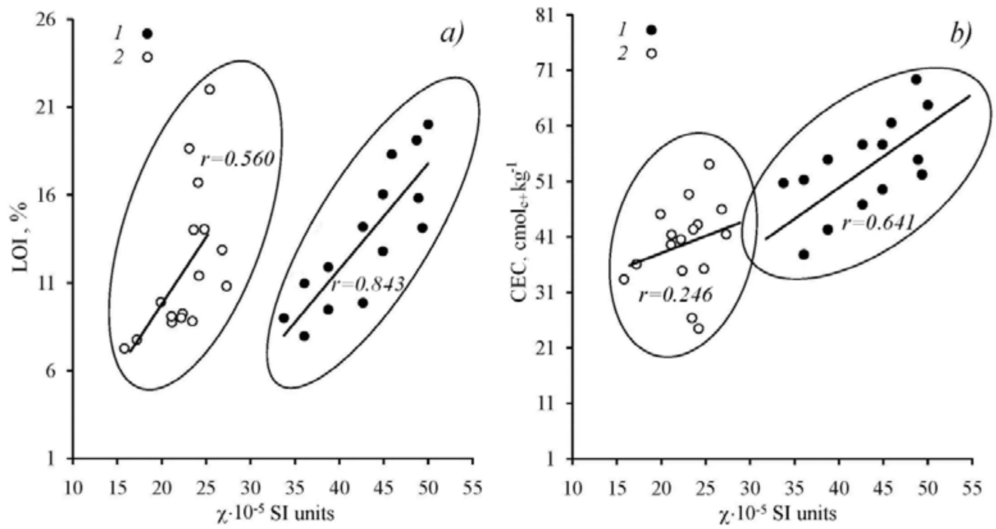

Figure-7. Relationship between magnetic susceptibility and (*a*) loss on ignition, (*b*) cation exchange capacity for layer samples taken from the humus profiles of the different-aged chernozemic soils: *1* - virgin leached chernozem *2* - newly formed chernozemic soils.

The phenomenon observed obviously comes from the age differences between the studied humus profiles, and it cannot be explained by differences in the OM content. The most likely explanation for the differences between the background chernozem and the newly formed soils is the difference in fine dispersed mineral components characteristics.

The diffraction spectra of the powder mounts (Figure-8) show that the parent rock contains not only clay minerals, but also quartz, calcite, potash and sodium feldspars, as well as small amounts of dolomite. It also contains a small admixture of amphiboles and ferrous sulfate, which are characterized by low resistance to chemical weathering [33]. There is a tendency for the amphibole reflection to reduce (at 0.842 nm) from the parent rock toward the upper part of the humus horizon of different-aged chernozemic soils.

The weathering degree should affect the crystallite size of minerals resistant to weathering, such as quartz. The average size of coherent scattering regions shows that quartz crystallite size decreases toward the bottom of the different-aged chernozemic soil horizons (Table-1).

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Table-1. The crystallite size of quartz in different-aged chernozemic soils.

Sample	**Crystallite size L** (nm)	
	Reflex (101) 3,343Å	**All reflexes** (average size)
C_{Ca} (140-150 cm) (parent rock)	188.6	160,3
A_1 (5-10 cm) (virgin leached chernozem)	152.2	126,7
A_1 (4-10 cm) (soil formed on the outer slope of the fort wall)	149.1	132,7

The mineral weathering degree in humus profiles derived from a uniform parent material can be estimated using the concentrations of minerals that sharply differ in their resistance to chemical weathering. The ratio of amphibole concentration to concentration of any mineral that is more resistant to weathering (e.g., zirconium and titanium minerals) can be calculated using the X-ray phase analysis of the heavy fraction. Less resistant minerals will be destroyed in the process of soil formation, while more resistant minerals will accumulate at the top of the profile. When isolating heavy minerals, additional purification operations (from light mineral admixture) were not performed. The basis for this decision was the assumption that a small admixture of light minerals ensures minimal losses during separation in bromoform.

Accessory zirconium and titanium minerals represented on the X-ray spectra (Figure-9) and characterized by a complete set of reflection intensities include zircon and rutile (very resistant) and ilmenite (resistant). The ratios of amphibole concentration to concentration of weathering-resistant minerals are shown in Figure-10. Histograms show that the most accessible sources of Fe^{2+} are actively involved in the initial stage of the magnetic profile formation when they form secondary magnetic minerals.

A negative correlation between total magnetic susceptibility and its dia-/paramagnetic component also indicates active weathering of primary ferrous silicate in humus profiles of different-aged chernozemic soils (Figure-11a). At the same time, a strong positive correlation between total magnetic susceptibility and its ferromagnetic component implies that weathering of parent dia-/paramagnetic minerals is associated with secondary magnetic minerals formation (Figure-11b). In both cases, two sets of data - for the background chernozem and for the newly formed soils - form two non-overlapping point clouds in the correlation field.

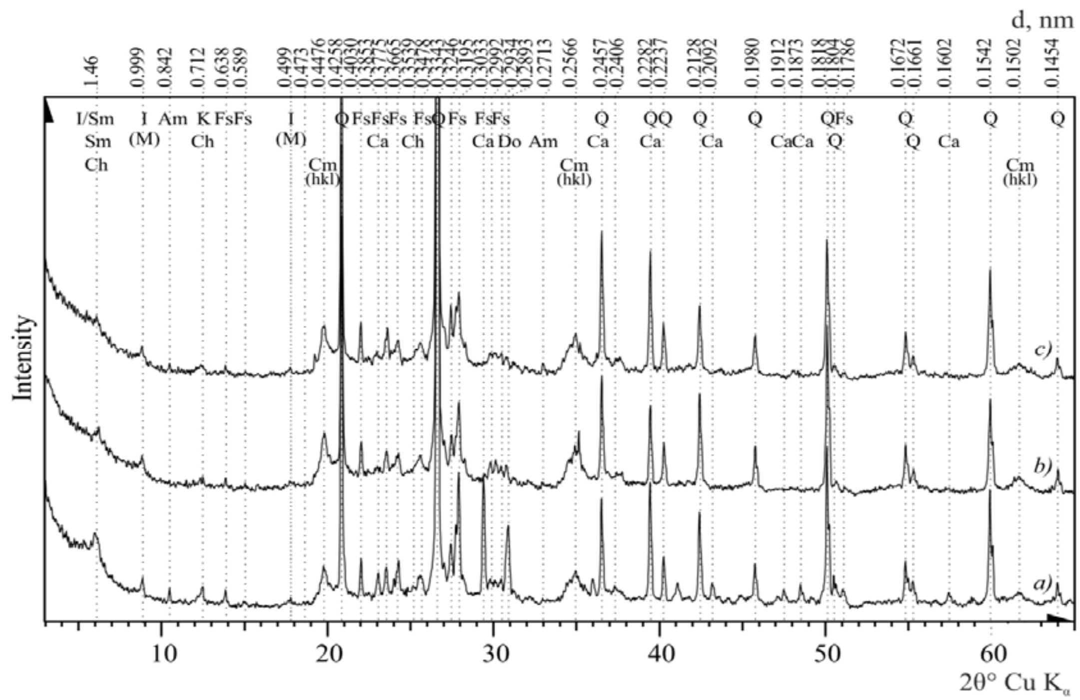

Figure-8. X-ray diffraction patterns of powder mounts from parent rocks (*a*) and upper layers of the chernozemic soil formed on the outer slope of the fort wall (*b*), and the virgin leached chernozem (*c*): (I/Sm) illite–smectite; (I) illite; (M) muscovite; (Am) amphiboles; (Ch) chlorite; (K) kaolinite; (Fs) feldspars; (Q) quartz; (Ca) calcite; (Do) dolomite; (Cm) clay minerals.

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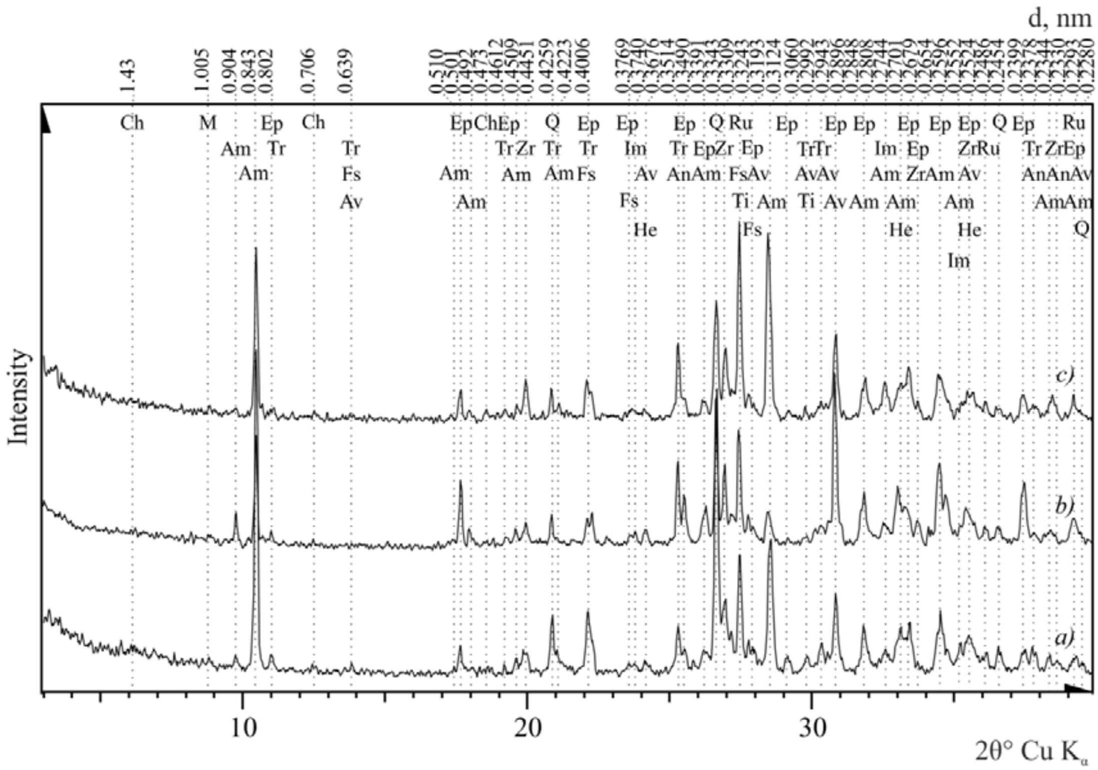

Figure-9. X-ray diffraction patterns of heavy mineral powder mounts from parent rocks (*a*) and upper layers of the chernozemic soil formed on the outer slope of the fort wall (*b*), and the virgin leached chernozem (*c*): (Am) amphiboles; (M) muscovite; (Ch) chlorite; (Ep) epidote; (Tr) tourmaline; (Zr) zircon; (Q) quartz; (An) anatase; (Ru) rutile; (Im) ilmenite; (Av) augite; (Ti) titanite; (He) hematite; (Fs) feldspars.

The study of magnetic and mineralogical characteristics of bulk samples and <2.5 μm fraction was performed through differential TMA. Figures 12 and 13 show cumulative (in the upper part of the figures) and differential (in the lower part of the figures) curves. The first heating curves are shown in the left part of the figures. The right part contains the second heating curves obtained after cooling and re-heating the same sample under the same conditions. The first peak of the differential TMA curve for bulk samples (100-150° C) is associated with the removal of free and bound water (Figure-12).

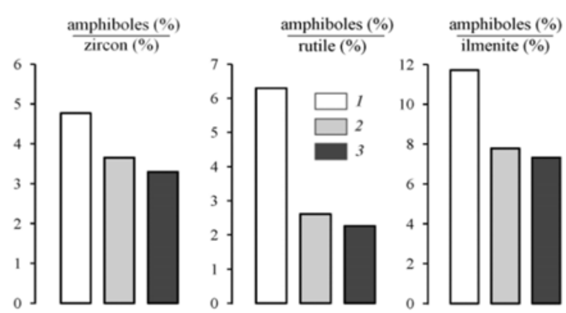

Figure-10. Histograms of heavy mineral ratios for the parent rock (*1*) and the upper layers of the chernozemic soil formed on the outer slope of the fort wall (*2*), and the virgin leached chernozem (3).

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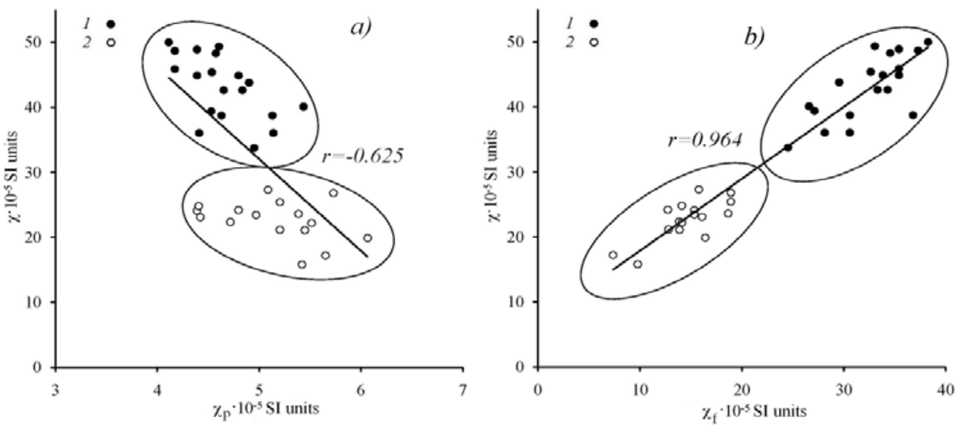

Figure-11. Relationship between magnetic susceptibility and (*a*) loss on ignition, (*b*) cation exchange capacity in layer samples from the humus profiles of the different-aged chernozemic soils: *1* – virgin leached chernozem *2* – newly formed chernozemic soils.

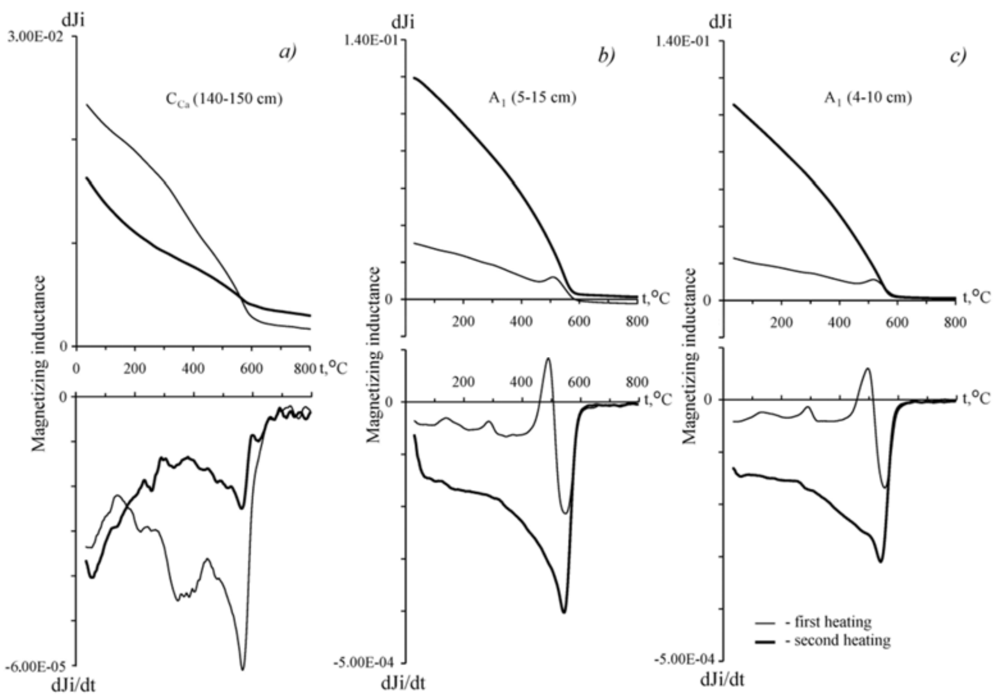

Figure-12. Thermomagnetic analysis results for the parent rock sample (*a*) and the samples taken from the upper layers of the virgin leached chernozem (*b*) and chernozemic soil formed on the outer slope of the fort wall (*c*).

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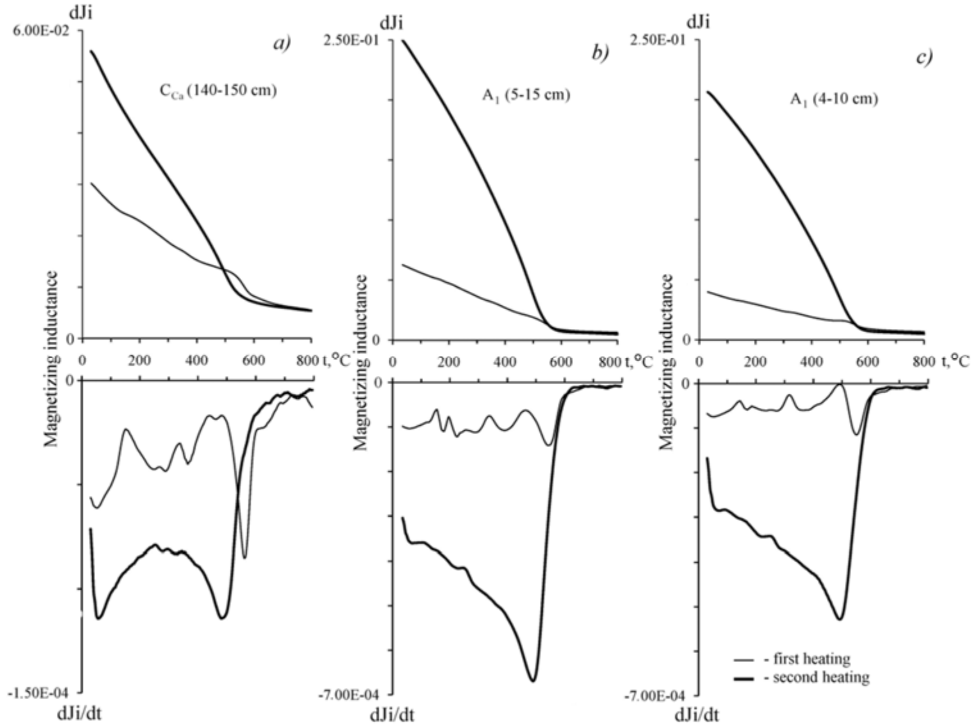

Figure-13. Thermomagnetic analysis results for <2.5 μm fraction after the removal of carbonates and organic matter from the parent rock (*a*) and upper layers of the virgin leached chernozem (*b*) and chernozemic soil formed on the outer slope of the fort wall (*c*).

The organic matter burn-off is reflected on the curves at 420-590 °C by the increase in magnetization due to magnetite formation. The peak of the differential curve at 300 °C can be explained by iron sulfide contained in the samples. This is also proved by the bend of the second heating differential curves at the same temperature. Moreover, the second heating curves (Figure-12, on the right) show the presence of fine magnetic grains - the newly formed magnetite, primarily, with slight traces of iron sulfide.

The differential TMA curves for <2.5 μm fraction after the OM and carbonates removal are more information rich (Figure-13). There is a peak at 180-210 °C (apart from the one associated with free and bound water) which is typical for maghemite and magnetite and usually appears due to the maghemitization stress relieving [19]. The peak is reduced from mature chernozem toward young soils, which is consistent with positive correlation between total magnetic susceptibility and its ferromagnetic component (Figure-11). The samples also contain iron sulfide, and the slight bend at 250-280°C is considered an indicator of lepidocrocite [19].

4. CONCLUSIONS

A comparative study of magnetic properties of the different-aged chernozemic soils showed that the formation of mature magnetic profile in automorphic temperate soils is a very long process.

The results show good agreement with the chemical model proposed by Boyle et al. [9], in which magnetic susceptibility enhancement is explicitly associated with the weathering duration.

After 750-800 years, magnetic susceptibility in the organogenic soils reached only about half of its value in the mature chernozem.

In terms of the chemical model, the newly formed chernozemic soils are now at the stage of active formation of secondary magnetic minerals, but the resource of primary ferrous silicates (which are less resistant to weathering) is not exhausted in mature virgin chernozem yet.

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