Rheological properties of naturally structured and homogenized sod-podzolic soil and typical chernozem under various land use

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Abstract. The rheological parameters of naturally structured and homogenized sod-podzolic soils and typical chernozems under various land use were studied by the amplitude sweep test. The range of elastic behavior at low loads in which the microstructure does not break down (LVE-range) and the strength (stability) of natural structural bonds (storage modulus in this range) in naturally structured samples are greater than in homogenized samples but homogenized soils have a bigger range of plastic behavior (Crossover) at measurement conditions. Differences in rheological behavior between horizons are more pronounced for naturally structured soils. However, further studies and selection of optimal measurement conditions are necessary for better understanding of rheological behavior of naturally structured soils.

1. Introduction
Rheological properties are a functional expression of the interaction of the solid and liquid phases of the soil. The assessment of the properties of the soil structure by rheological methods is extremely promising because it allows evaluating the soil structure by quantitative parameters. The structure of the pore space, the number and size of pores, the number and strength of the contacts between soil particles determine the strength of the soil structure and its behavior under load [1]. In most cases, the determination of rheological properties was carried out on homogenized samples [2-5] and therefore the natural structure was destroyed. In the process of hydration, artificial structure formation occurs. This artificial structure was subjected to the determination of rheological properties. In this case, the ability of soil particles to establish bonds, the influence of various factors were evaluated such as the content of organic matter, the dispersity of the solid phase, the presence of salts, etc. The data obtained in these studies show us the potential ability of the soil to structure formation. Oscillatory methods carried out on MCR rheometers have been used and recommended for studying the rheological properties of soils by W. Markgraf [2] as suitable methods for determining the rheological behavior of soils, especially in the viscoelastic range before passing to the viscous flow range. One of the advantages of using oscillatory methods on a MCR rheometer is the ability to test samples of naturally structured soil (monoliths) [6-8]. The goal of this work was to compare the rheological behavior in homogenized soil (pastes) and naturally structured soil (monoliths) of sod-podzolic soils of the Moscow region and typical chernozems of Kursk region (Russia).
2. Objects
The studies were carried out on the soils of three land use options for typical chernozems: an oak forest, a forest belt (N 51.621347° E 36.256665°) and an arable field (N 51.621370° E 36.257558°) and two options for sod-podzolic soils: an old arable field (N 56.133261° E 37.802355°) and a forest (N 56.098272° E 37.821603°). A typical chernozem under the oak forest is located on the territory of the V.V. Alekhin Central Chernozem State Reserve, the arable field is located on the territory of the Kursk Research Institute of Agricultural Production and in the forest belt adjacent to it. The following horizons were identified in the typical chernozem of the oak forest - A1, A1B, Bca, in the typical chernozem of the arable field - Ap1, Ap2, A1B, Bca, in the typical chernozem of the forest belt - A1`, A1B`, A1B``. According to the WRB 2014 classification, these soils are Haplic Chernozems (Pachic) and Haplic Chernozems (Aric, Pachic). The sod-podzolic soil of the forest was in 700 m to the west of the Daryino village, Pushkin District, Moscow Region. The sod-podzolic arable soil was located on the experimental field of the Zelenograd station of the V.V. Dokuchaev Soil Science Institute. According to the WRB 2014 classification, the soils are called Albic Glossic Retisols (Lomic, Cutanic) and Albic Glossic Retisols (Lomic, Aric, Cutanic) respectively.

3. Methods
Determination of total, organic and inorganic carbon in soil samples was carried out by the express analyzer AN–7529 (Measuring instrument, Belarus) using the method of dry combustion in oxygen flow [9]. The inorganic carbon content was determined as the difference between the total carbon and organic carbon content after removal of carbonates. The texture of the soil samples was measured by laser diffraction’s method of particles using an Analysette–22 (Fritsch, Germany) with preliminary ultrasonic processing of the soil suspension for 5 min [9]. The rheological parameters of soil samples were studied by an amplitude sweep method using a modular compact rheometer MCR–302 (Anton Paar, Austria) with a PP–25 measuring system (parallel plates with a diameter of 25 mm) [2]. The theory of the amplitude sweep method, the rationale for its use in soil studies and the available results are described in detail in [2, 10]. The main technical parameters of the method, in accordance with the methodology introduced by Markgraf [2], were: 30 deformation values points; 15 s – measurement duration of one point; frequency - 0.5 Hz; γ deformation range from 0.001 to 100% log; constant temperature -20°C. The measurements were carried out at controlling the normal force of the upper plateau < 5N for homogenized soils and <15 N for naturally structured soils. As a result of a measurement, the device software produces two curves: a storage modulus G’ and a loss modulus G” (figure 1).

![Figure 1. Storage modulus (G’) and loss modulus (G”) versus deformation γ.](image)

The storage modulus G’ is a measure of the energy that goes to the restoration of the structure, this module can be considered the modulus of elasticity. The loss modulus is a measure of the energy which is spent on heating, friction and other losses, which is not restored. The rheological properties of soils were characterized by the following parameters: (1) (figure 1) phase I – LVE-range is the linear
soil contains less fine fraction and value increases. This fact can be explained by the differentiation of physical properties: the eluvial horizon is four times as compared to the humus horizon, in the lower horizons the differentiation of the parameter values by depth is more pronounced in forest soil: the decrease in the eluvial horizon is four times as compared to the humus horizon, in the lower horizons the parameter value decreases. This fact can be explained by the differentiation of physical properties: the eluvial horizon contains less fine fraction and the total carbon. For monolithic samples, a positive correlation

4. Results
According to the USDA classification, typical chernozems are silt loam. For the typical chernozems the differentiation of the fraction<0.01 mm content by depth is weak (table 1).

The field cut soil samples with natural structure (monoliths) of size 9.5 x 7.5 x 4 cm were tightly wrapped with tape and stored in a refrigerating chamber until the beginning of the experiment. Before testing the monoliths were placed on a plate covered with filter paper, the ends of which were lowered into distilled water for capillary saturation for three days before water appeared on the monolith’s surface. After water saturation, a testing monolith was cut out from the prepared surface of the monolith into a circular shape with a diameter of 25 mm for research. Bulk samples were ground with a rubber-tipped pestle and sieved through a 1 mm sieve. Then, 3 grams of an air–dry sample was placed in plastic cylinders with a diameter of 25 mm with a water-permeable bottom, the surface of the soil tablet was leveled with a piston and slightly compacted. Then the cylinders were also placed on wet filter paper with the ends lowered into distilled water for capillary saturation during the one day. The moisture of the test samples was determined by the gravimetric method immediately after the amplitude test by using the MX-50 moisture analyzer (table 1). The measurements were carried out in triplicate for pastes and five to seven times for monoliths; the average and confidence intervals for the average were calculated for all rheological parameters.

In pastes of typical chernozem range of the linear viscoelastic behavior varies from 0.007% deformation in the A1 horizon of the oak forest (figure 2) to 0.004% in the Bca. In the oak forest and the forest belt samples the linear viscoelastic range decreases down the profile, unlike the field where this value is slightly less in the upper arable horizon than in the underlying horizons.

In monoliths of typical chernozem, the LVE-range value is the largest in the A1’ horizon of the forest belt and it is 0.011% of deformation. On average, the excess of LVE-range of monoliths over pastes by 1.2 times. There is a tendency to a decrease in this value down in depth, which is associated with a decrease of the organic matter and fraction<0.01 mm content (Pearson correlation coefficients $r = +0.9$ and $+0.7$ respectively, $P = 0.95$) and the appearance of carbonates (Pearson correlation coefficient $r = −0.6$, $P = 0.95$).

Sod-podzolic soil’s pastes are characterized by a small range of linear viscoelastic behavior (or resistance to stress), not exceeding 0.004% deformation (figure 2). Down the profile of the arable soil the differentiation of this parameter is poorly pronounced. In forest soil, a decrease of LVE–range is observed in the horizons A2 and A2B and after it increases down the profile. The monoliths of the sod-podzolic soil are characterized by a considerably large range of linear viscoelastic behavior, the values of which vary from 0.002% in the eluvial horizon A2 to 0.009% in the A1 horizon. Generally, the excess of the linear viscoelastic behavior range in monolithic samples over pastes is 1.7 times. Simultaneously, differentiation of the parameter values by depth is more pronounced in forest soil: the decrease in the eluvial horizon is four times as compared to the humus horizon, in the lower horizons the parameter value increases. This fact can be explained by the differentiation of physical properties: the eluvial horizon contains less fine fraction and the total carbon. For monolithic samples, a positive correlation
was found between the linear viscoelasticity range and the total carbon content (Pearson correlation coefficient $r = +0.7$, $P = 0.95$).

**Table 1.** Physical and chemical properties of typical chernozems and sod–podzolic soils.

| Soil, horizon, depth, cm | Texture: fraction’s content <0.01 mm (%) | TC $^a$ (%) | IC $^b$ (%) | OC $^c$ (%) | MC $^d$ nat.str. homog. soil (%) |
|--------------------------|-----------------------------------------|--------------|--------------|--------------|---------------------------------|
| **Typical Chernozem (forest belt)** | | | | | |
| A1’ (0-20) | 55.11 | 3.86 | 0.00 | 3.86 | 44.60 | 80.37 |
| A1B’ (60-80) | 50.94 | 2.11 | 0.00 | 2.11 | 37.89 | 80.93 |
| A1B” (80-110) | 48.90 | 1.54 | 0.01 | 1.53 | 43.52 | 78.06 |
| Bca (110-140) | 49.79 | 2.41 | 1.51 | 0.90 | 44.08 | 71.74 |
| BCca (140-180) | 47.82 | 2.57 | 2.10 | 0.47 | 42.73 | 72.35 |
| **Typical Chernozem (oak forest)** | | | | | |
| A1 (0-50) | 53.93 | 3.30 | 0.01 | 3.29 | 40.45 | 63.48 |
| A1B (50-95) | 46.87 | 1.40 | 0.02 | 1.38 | 43.59 | 72.48 |
| Bca (95-170) | 49.90 | 2.23 | 1.85 | 0.38 | 34.02 | 59.78 |
| **Typical Chernozem arable** | | | | | |
| Ap1 (0-20) | 58.06 | 2.65 | 0.10 | 2.55 | 35.76 | 74.67 |
| Ap2 (20-30) | 52.94 | 2.99 | 0.01 | 2.98 | 30.43 | 68.45 |
| A1B (60-97) | 48.72 | 1.90 | 0.01 | 1.89 | 41.79 | 71.56 |
| Bca (97-120) | 46.97 | 1.62 | 0.92 | 0.70 | 43.14 | 76.42 |
| **Sod - podzolic soil** | | | | | |
| A1 (4-15) | 41.69 | 2.43 | - | - | 51.63 | 64.17 |
| A2 (15-28) | 34.38 | 0.31 | - | - | 26.39 | 37.95 |
| A2B (28-35) | 41.72 | 0.18 | - | - | 24.02 | 49.20 |
| B1 (35-70) | 46.50 | 0.13 | - | - | 22.66 | 48.90 |
| B2 (70-100) | 52.62 | 0.12 | - | - | 22.73 | 49.18 |
| **Sod - podzolic arable soil** | | | | | |
| Ap1 (0-26) | 40.18 | 1.46 | - | - | 31.3 | 51.94 |
| Ap2 (26-30) | 39.71 | 1.19 | - | - | 27.4 | 51.07 |
| A2B (30-65) | 40.65 | 0.27 | - | - | 23.5 | 50.52 |
| B1 (65-85) | 45.08 | 0.23 | - | - | 33.4 | 56.92 |
| B2 (85-115) | 48.81 | 0.22 | - | - | 24.0 | 51.62 |
| BC (115-160) | 48.27 | 0.19 | - | - | - | 60.53 |

$^a$ TC - Total carbon
$^b$ IC - Inorganic carbon
$^c$ OC - Organic carbon
$^d$ MC - Moisture content
The range of linear viscoelastic behavior of naturally structured (monoliths) and homogenized (pastes) samples of the typical chernozems and the sod-podzolic soils.

The range of linear viscoelastic behavior or resistance to loads in typical chernozem arable soil is 1.4 times larger than in sod-podzolic arable soil both in paste specimens and in monolithic ones. Resistance to stresses in forests considerably exceeds the stability of arable soils of both sod-podzolic and chernozem soils. Generally, the resistance to loads of monolithic samples of sod-podzolic soil is more than pastes by 1.7 times, for chernozem - by 1.2 times.

Figure 3 presents the values of the storage modulus in the range of linear viscoelastic behavior, which characterizes the strength of the structure in the range of elastic behavior. As can be seen, the storage modulus values of the monoliths are significantly higher than of paste samples, on average by 5 times. In a series of pastes of different horizons of typical chernozem, no statistically significant differences were found (figure 3).

For monoliths of the forest belt there is a tendency to increase in the parameter in the lower horizons. Most likely, this is due to the appearance of carbonates in the lower horizons of the profile, which have a cementing effect during the formation of strong bonds. Simultaneously, the organic matter of the
humus horizons prevents the formation of strong contacts between particles during swelling (Pearson correlation coefficients $r = +0.6$ and $-0.7$ respectively, $P = 0.95$). Monolithic samples have a greater storage modulus in the range of viscoelastic behavior than pastes; the values of storage moduli in monolithic samples exceed these values in pastes by 3–7 times.

For pastes of sod-podzolic soils, no statistically significant differences were found (figure 3).

Monoliths of humus horizons have a minimum storage modulus, its increase is observed in the eluvial horizon of the forest sod-podzolic soil. For the arable soil, weaker differentiation is observed, but there is also a tendency to an increase in the value of the parameter. This is probably due to the fact that the organic matter of the upper humus horizons alienates the soil particles from each other, increasing the distance between them during the swelling of the samples. The decrease in the storage modulus in the lower horizons of forest sod-podzolic soil is probably associated with an increase in the content of $<0.01$ mm fraction and decrease of the total carbon content (Pearson correlation coefficient $r = -0.8$, $P = 0.95$).

The range of low deformation, characterized by a range of linear viscoelasticity and storage modulus in this range, shows differences in the behavior of monolithic samples with a natural structure and pastes with artificial structure, which is especially clearly manifested in samples of sod-podzolic soil. The range of stable behavior in which the microstructure does not break down and the strength (stability) of natural structural bonds in the range of linear viscoelasticity in monoliths at low loads is greater than in artificial pastes.

![Figure 4](image-url)

**Figure 4.** The deformation value at the intersection point of the storage modulus and loss modulus (crossover) of naturally structured (monoliths) and homogenized (pastes) samples of the typical chernozems and the sod-podzolic soils.

The point of equal values of the storage modulus and loss modulus indicates the deformation at which the structure is destroyed and the elastic-plastic behavior changes into a viscous flow. In all variants of the studied soils, this value for paste samples is higher than for monolithic ones, except for the upper humified horizons of sod-podzolic soil under the forest and forest belts for typical chernozem. For paste samples of horizons of sod-podzolic soil, no statistically significant differences were found. In the series of monoliths, the humus horizons have the highest value of the Crossover. The parameter value decreases in the lower horizons (figure 4), which is associated with a decrease in the content of organic matter. The positive correlation of this parameter for monolithic samples with the total carbon content was found (Pearson correlation coefficient = $+0.8$, $P = 0.95$).

For pastes of chernozems of the typical oak, the forest belt, and the arable field, no statistically significant differences were found. In the series of monoliths, the horizon A of the forest belt has the largest value of deformation, at significantly lower parameter values in the underlying horizons. For typical chernozem of the oak forest, no significant differences were revealed, there is a slight increase...
in the parameter value by the depth for arable field. Data analysis revealed a positive correlation between the Crossover deformation values for monoliths and the organic carbon content (Pearson correlation coefficient \( r = +0.6, P = 0.95 \)). In samples with a high organic carbon content, the contacts between particles during swelling are more plastic, which is manifested in a larger parameter value of the plastic behavior range.

Monolithic samples (with stronger structural bonds in the range of linear viscoelastic behavior at low deformation) show a more brittle behavior during load increasing compared to pastes which are characterized by a large value of the plastic behavior range.

5. Conclusion
Resistance to stresses of monolithic samples is greater than in paste samples, as evidenced by larger LVE-range values. The maximum excess is observed in the oak forest and forest belt soil. Resistance to loads of soil structure under the forests is much greater, perhaps this is due to a slightly different component composition of OM in the soils under the forest [11]. In the carbonate soil resistance of monoliths and pastes is almost equal.

Storage modulus as a measure of the strength of interparticle bonds in the LVE-range in naturally structured samples is much greater than in homogenized samples. Perhaps the reasons for this are: 1) unequal moistening of monoliths and pastes, the moisture content of the monoliths was significantly lower than the moisture of the pastes, despite the longer duration of wetting of the monoliths (3 days) compared to pastes (1 day). The tomographic studies of monoliths we carried out [12] earlier showed the presence of numerous closed pores, perhaps they do not allow the moisture to completely wet the sample; 2) a different normal force of the upper plateau was applied (for pastes \(<5\) N and for monoliths \(<15\) N). In the case of monoliths, it was necessary to apply in order to ensure that the plateau surface was completely adherent to the sample. However, an increase in the normal force increases the compression of the sample and, accordingly, the storage modulus is noted by Holthusen [7]. Thus, further studies of the rheological properties of monolithic samples and the selection of optimal measurement conditions are necessary to avoid errors.

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