

How far are rheological parameters from amplitude sweep tests predictable using common physicochemical soil properties?

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Abstract. A basic understanding of soil behavior on the mesoscale resp. macroscale (i.e. soil aggregates resp. bulk soil) requires knowledge of the processes at the microscale (i.e. particle scale), therefore rheological investigations of natural soils receive growing attention. In the present research homogenized and sieved (< 2 mm) samples from Marshland soils of the riparian zone of the River Elbe (North Germany) were analyzed with a modular compact rheometer MCR 300 (Anton Paar, Ostfildern, Germany) with a profiled parallel-plate measuring system. Amplitude sweep tests (AST) with controlled shear deformation were conducted to investigate the viscoelastic properties of the studied soils under oscillatory stress. The gradual depletion of microstructural stiffness during AST cannot only be characterized by the well-known rheological parameters G' , G'' and $\tan \delta$ but also by the dimensionless area parameter integral z , which quantifies the elasticity of microstructure. To discover the physicochemical parameters, which influences the microstructural stiffness, statistical tests were used taking the combined effects of these parameters into account. Although the influence of the individual factors varies depending on soil texture, the physicochemical features significantly affecting soil microstructure were identified. Based on the determined statistical relationships between rheological and physicochemical parameters, pedotransfer functions (PTF) have been developed, which allow a mathematical estimation of the rheological target value integral z . Thus, stabilizing factors are: soil organic matter, concentration of Ca^{2+} , content of CaCO_3 and pedogenic iron oxides; whereas the concentration of Na^+ and water content represent structurally unfavorable factors.

1. Introduction

Soils are the key life resource for human beings, where food and forage crops are grown and water is cleaned by percolation. Furthermore soils provide renewable resources as well as construction grounds, and raw materials can be either extracted or captured from soils. At the same time, soils are important governing factors for local and global cycles of substances and water. Finally, soils are an essential component of ecosystems: they represent an important habitat for a giant community of plants and animals. Therefore soils are a substantial basis for biodiversity.

Nowadays degradation of soils by deformation and soil erosion are severe problems for world food supplies, hence avoidance of soil loss and deterioration as well as contamination and desertification is necessary in order to preserve beneficial soil functions.

Therefore an important consideration is the conservation of soil structure and convenient pore geometry, which guarantee exchange and supply with water and air. If the soil structure and thus pores and pathways are destroyed by external stresses (e.g. passage with heavy agricultural machines), the



infiltration of water into the soil declines. As a consequence surface runoff and soil erosion increase enormously, finally resulting in topsoil depletion. Not only the loss of fertile topsoil leads to problems, but also the formation of erosion gullies and the coverage of soils at the slope toe with colluvium. Moreover, parts of the erosion material are released into surface waters, where they deposit and accumulate. If intense sedimentation and siltation takes place in busy waterways, dredging is required. Also the large amount of pollutants and nutrients that are discharged with the sediment into surface waters is not negligible [1].

As soil structure possesses many functions, the quantification of structural stability is an important field of research and there have been ambitious projects and discussions on it for many decades [2, 3]. In classical soil mechanics the stability of soil structure is often characterized by investigating the compression and/ or the shear behavior. These analyses on the mesoscale, i.e. aggregate scale yield stability parameters e.g. precompression stress (P_c), angle of internal friction (ϕ) and cohesion (c) which provide information about the reaction of the regarded total soil volume towards stresses at defined boundary conditions. However, no conclusions regarding the causes of mechanical stability are possible [4]. The reasons for different strength properties especially in visually comparable soils are to be found on the microscale, i.e. particle scale and are manifested in microstructural processes and interactions. To achieve the required knowledge of the processes at the microscale, in recent years rheological studies of natural soils have received growing attention [i.a. 5, 6-8].

Since rheological analyses detect the dynamic interaction between single soil particles, entirely new insights on the combined effects of soil stabilizing factors can be gained. Due to the numerous variety of factors influencing the microstructure of soil, the following questions arise: (1) what are the relevant factors influencing microstructural stability of riparian soils? And moreover (2) can these factors be parameterized in pedotransfer functions (PTFs) and is thereby the rheological parameter *integral z* – which quantifies the microstructural stiffness – predictable?

2. Material and methods

2.1. Soil material

Marshland soils of the riparian zone of the Elbe estuary (North Germany) were used. 22 sites along the estuary were chosen to depict the gradient in salinity as well as in sodicity. At each site three soil pits were dug in the embankment foreland in dependence on the mean water levels: profile 1 above mean high tide (MHWL), where generally grassland had developed; profile 2 in the range of MHWL with partially extensive reed vegetation and profile 3 below MHWL, where vegetation is mostly lacking (Figure 1).

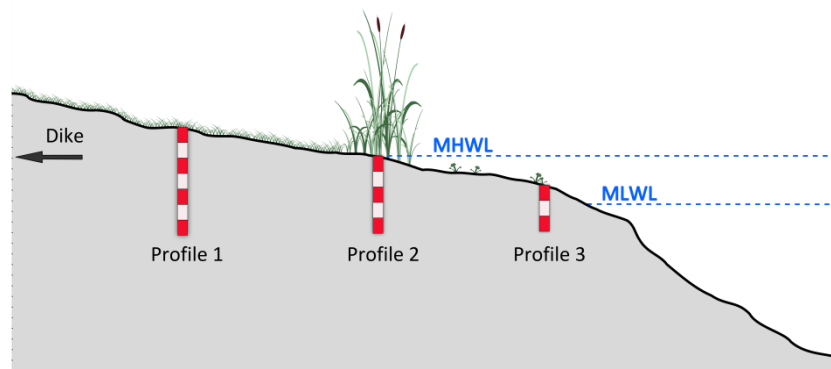


Figure 1. Schematic overview of the sampling design

Disturbed soil samples were taken from defined depths: in profile 1 in 10, 30, 50 and 70 cm; in profile 2 in 10, 30 and 50 cm; in profile 3 in 10 and 30 cm beneath ground level. The soil material was collected and transported in properly sealed plastic bags.

2.2. Standard laboratory analyses

Prior to physicochemical analyses, which were performed according to Blume, Stahr and Leinweber [9] and Schlichting, Blume and Stahr [10], samples were air dried, homogenized and sieved through a 2 mm sieve. The screened soil material was taken to measure pH and electric conductivity (EC) in 1:2.5 (w/w) suspensions using 0.01 M CaCl_2 respectively deionized water. Particle size distribution was determined by sieving and sedimentation applying the pipette-method, whereby at first soil organic matter is destroyed with H_2O_2 (30%), and calcite (CaCO_3) is dissolved by HCl. Total soil carbon was measured by grinding a little of the sieved soil in a ball mill and afterwards about 200 mg of the material was ashed in a Ströhlein apparatus based on the principle of coulometry. CaCO_3 was analyzed according to the Scheibler method by gas volumetric determination of the released CO_2 . The difference between inorganic and total carbon corresponds to the organic C. Exchangeable cations were extracted by 0.1 M BaCl_2 . Concentrations of Ca^{2+} , Mg^{2+} , K^+ and Na^+ were measured using an atomic absorption spectrometer.

The total amounts of pedogenic iron oxides (Fe_d), which develop during aging of soils, were determined by extraction with 0.3 M Na-citrate solution, 1 M NaHCO_3 solution and Na-dithionite according to the method described by Mehra and Jackson [11]. The iron concentration was determined by atomic absorption spectrometry as well.

2.3. Rheometry

Amplitude sweep tests (AST, oscillatory mode) with controlled shear deformation were conducted with a modular compact rheometer MCR 300 (Anton Paar, Ostfildern, Germany) with a profiled parallel-plate measuring system at a plate distance of 4 mm. The configurations of the tests were carried out in accordance with Markgraf [12] (Table 1).

Table 1. Configuration parameters amplitude sweep test with controlled shear deformation

<i>parameter</i>	
plate distance (h)	$h = 4 \text{ mm}$
shear deformation (γ)	$\gamma = 0.0001 \text{ to } 100\%$
frequency (f)	$f = 0.5 \text{ Hz } (\omega = \pi \text{ rad/s})$
temperature (T)	$T = 20 \text{ }^\circ\text{C}$ (regulated by Peltier unit)
measuring points	30 pts.
test duration	appr. 15 min.

Air dried, homogenized and sieved ($< 2 \text{ mm}$) samples from all sites and depths were repacked into small cylinders (45 cm^3) to a standard bulk density ($\rho_t = 1.4 \text{ Mg m}^{-3}$) and capillary saturated with deionized water to achieve quasi-saturated conditions (0 kPa). Subsequently a portion of the prepared cylinders were drained and equilibrated to defined matric potentials (-6 or -15 kPa). Thus rheological analyses for every sampled depth were performed at three different matric potentials.

A small amount of soil (4 cm^3) was taken from each cylinder with a spatula and put on the fixed measuring plate of the rheometer for the actual measurement. After lowering the upper plate to the defined measuring gap (4 mm), the normal force was checked to maintain an undisturbed quasi-elastic soil structure (i.e. $F_N \leq 12 \text{ N}$). Additionally a waiting time of 40 sec prior each test was implemented. Operation of the rheometer, monitoring of the ASTs and generation of the rheological parameters (G' , G'' , $\tan \delta$, integral z) were carried out by the software Rheoplus/32 V3.21. Per depth and dewatering state five replicates were performed. Before and after each test the water content (w/w) was determined.

2.3.1. Loss factor $\tan \delta$ and integral z .

According to Markgraf [12] soil can be defined as viscoelastic substance. Hence amplitude sweep tests are an appropriate instrument to investigate the structural character of soils. During ASTs soil samples experience a gradual depletion of microstructural stiffness (Figure 2). This degradation of internal structure can be characterized by the well-known rheological parameters storage modulus (G'), loss modulus (G'') and the linear viscoelastic range (LVE). Moreover the microstructure of soils can be specified by the loss factor $\tan \delta$, which equals the ratio of G'' to G' , i.e. the imaginary part to the real part of the deformation energy. If $\tan \delta < 1$, G' prevails over G'' , and the soil exhibit a quasi-elastic behaviour: applied deformation leads to full or at least partial microstructural regeneration. When $\tan \delta = 1$ the flow point ("cross-over") is reached ($G' = G''$). If the flow point is exceeded a viscous character predominates, $\tan \delta > 1$ (i.e. $G'' > G'$) and irreversible microstructural collapse occurs [13].

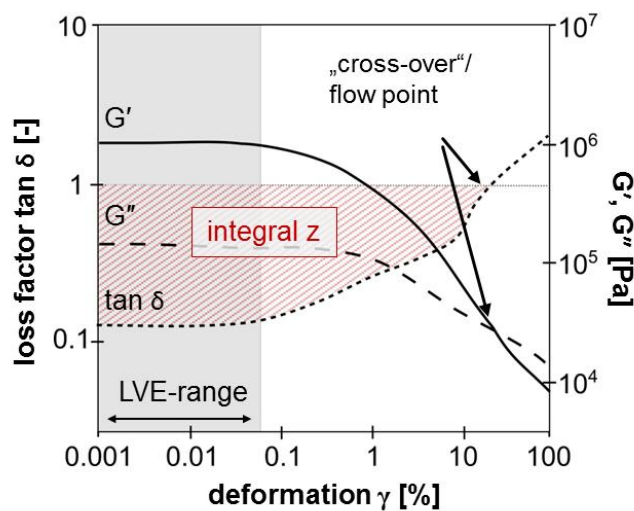


Figure 2. Diagram of an amplitude sweep test as a function of deformation γ . Loss factor $\tan \delta$ serves as analogue expression of quasi-elastic ($\tan \delta < 1$) or viscous ($\tan \delta > 1$) behaviour. For further comparison the integral z can be calculated for the interval from $\gamma = 0.001\%$ to "cross-over" as difference of the integrals of the 1-function and the $\tan \delta$ function.

For further comparison of the quasi-elastic range ($\tan \delta < 1$) in absolute terms, the dimensionless area parameter integral z can be used. The greater the value of integral z , the more elastic resp. rigid the soil is [7]. Thus integral z quantifies the structural strength in consideration of loss of elasticity due to increasing shear deformation over time. The integral of $\tan \delta(\gamma)$ with $\tan \delta = 1$ as defined limit on the ordinate was calculated from equation (1).

$$\int_{0.001}^{\text{"cross-over"}} (1 - \tan \delta) d\gamma \quad (1)$$

2.4. Statistics

The statistical evaluation was accomplished with the free software environment R (Version 3.1.1) [14] based on mixed models [15, 16] by means of analyses of covariance and variance (ANCOVA resp. ANOVA).

After testing all eligible chemical parameters on multicollinearity the explanatory variables shown in Table 2 have been selected as initial input parameters to run ANCOVA. Subsequently performed ANOVA permits the identification of the significant predictor variables.

Table 2. Selected input parameters

<i>parameter</i>	<i>acronym</i>	<i>unit</i>
electric conductivity	EC	mS cm ⁻¹
organic matter	OM	g 100 g ⁻¹
calcite content	CaCO ₃	g 100 g ⁻¹
pedogenic iron oxides	Fe _d	g kg ⁻¹
calcium concentration	Ca ²⁺	cmol _c kg ⁻¹
sodium concentration	Na ⁺	cmol _c kg ⁻¹
water content	Θ _g	w/w
depth beneath ground level	depth	cm

3. Results

The rheological response of soils under oscillatory conditions is influenced strongly by particle size distribution and water content (Figure 3). The texture effect is depicted by the median of grain size for all top soils (10 and 30 cm depth) in Figure 3: under quasi-saturated conditions (0 kPa) the integral z values become greater the greater the median of grain size gets (figure 3a). Because all discovered textures, which could differ concerning further chemical parameters, were taken into account, the coefficient of determination (R^2) indicates just a weak association between integral z and matric potential depending on texture. However, it can be stated that in fine-grained soils the microstructural stiffness is smaller than in sandy soils when saturated with water.

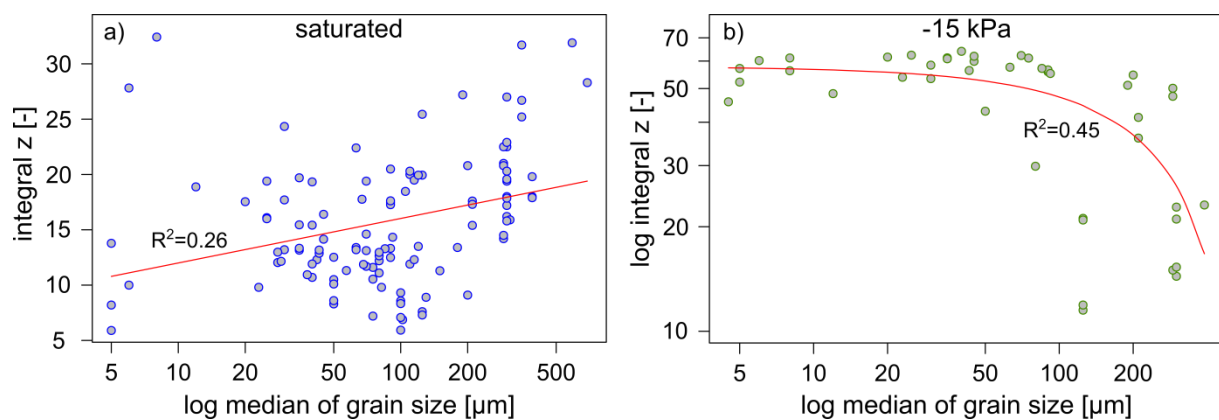


Figure 3. Texture effect depicted by the median of grain size for all top soils (10 and 30 cm);
a) quasi-saturated conditions and b) matric potential of -15 kPa

At a matric potential of -15 kPa first of all greater integral z values were determined and in addition the integral z values become greater the smaller the median of grain size is (figure 3b). Consequently the desiccation has a stabilizing effect on the soil microstructure. This effect becomes more pronounced, the more fine-grained the soil is.

Due to the considerable impact of particle size distribution, the textures were grouped according to the German soil texture classification (Figure 4).

Based on the defined texture groups the statistical analyses (ANCOVA, ANOVA) were conducted and the physicochemical factors shown in Table 3 have been identified to significantly affect microstructural stability.

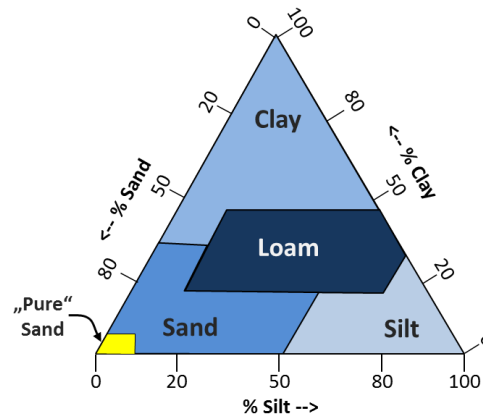


Figure 4. German soil texture classification (after [17])

Table 3. Physicochemical factors significantly influencing soil microstructure.

<i>parameter</i>	<i>acronym</i>	<i>unit</i>
organic matter	OM	$\text{g } 100 \text{ g}^{-1}$
calcite content	CaCO_3	$\text{g } 100 \text{ g}^{-1}$
pedogenic iron oxides	Fe_d	g kg^{-1}
calcium concentration	Ca^{2+}	$\text{cmol}_c \text{ kg}^{-1}$
sodium concentration	Na^+	$\text{cmol}_c \text{ kg}^{-1}$
water content	Θ_g	w/w
depth beneath ground level	depth	cm

Furthermore, the determined statistical relationships between the rheological parameter integral z and parameters influencing microstructural stiffness facilitated the development of pedotransfer functions, which allow a mathematical estimation of the microscale stability parameter integral z for the defined texture groups. Since the effect of the individual parameters vary depending on texture, those parameters, which have a minor impact on the soil microstructure, remain unconsidered, without deterioration of model quality resp. accuracy of PTFs. Hence, not every parameter is required for each defined texture group to predict the integral z value. Table 4 summarizes the developed PTFs for each texture group.

Table 4. Pedotransfer functions to predict microstructural stiffness through mathematical estimation

<i>texture group</i>	<i>pedotransfer function</i>	<i>R²</i>
sand	$\text{integral } z = 40.2 + 3.2 \text{ OM} + 2.1 \text{ Ca}^{2+} + 2.7 \text{ Fe}_d - 1.2 \Theta_g - 0.1 \text{ depth}$	0,64
loam	$\text{integral } z = 67.9 + 4.6 \text{ OM} + 0.2 \text{ CaCO}_3 - 1.4 \Theta_g$	0,56
silt	$\text{integral } z = 41.3 + 6.8 \text{ OM} + 0.5 \text{ CaCO}_3 + 2.3 \text{ Fe}_d - 1.2 \Theta_g$	0,50
clay	$\text{integral } z = 46.7 + 5.0 \text{ OM} + 2.3 \text{ CaCO}_3 - 3.6 \text{ Na}^+ - 0.8 \Theta_g - 0.2 \text{ depth}$	0,62

It turns out that when the combined effects of the physicochemical parameters are considered, the stabilizing factors are: soil organic matter, concentration of Ca^{2+} , content of CaCO_3 and pedogenic iron oxides (Fe_d); whereas the concentration of Na^+ , water content and the depth beneath ground level represent structurally unfavorable factors.

4. Discussion

4.1. Texture effect

Under oscillatory stress a characteristic degradation of internal structure depending on particle size distribution takes place. The textural effect, especially the importance of particle shape and mineral surface properties, was already emphasized by Markgraf and Horn [7] and Baumgarten [18] and is reflected in the results presented here. Coarse-grained textures exhibit a fast reduction of elasticity with increasing deformation at first. But as a consequence of relative motions (rotation, interlocking, reorganization) coarser particles are able to attain a more stable resp. an energetically more favorable arrangement, whereby the complete collapse of microstructure is delayed.

The more fine-grained the soil is, i.e. the higher the silt and/ or clay content, the less often interlocking between particles occurs and elasticity decreases gradually with increasing shear deformation [18]. While rounded and angular particles show rolling shear behavior [19], an increase in platy particles leads to sliding shear behavior [20], assuming a completely homogenized system. Sliding shear behavior and formation of smooth slickensides results in a reduced shear strength [21] caused by depletion of internal structure.

However, because of its smaller particle size, chemical surface properties prevail in clayey soils additionally to the induced shear behavior. Therefore clayey soils show an elastic character over a wide deformation range, until elasticity suddenly continuously decreases. Particularly the rheological properties may vary substantially because of diverse clay mineral associations, which react differently to shearing [22].

Furthermore, excess pore water pressure may arise under mechanical stress, which reduces effective stress at almost constant total stress. Convex menisci promote the displacement of particles relative to one another and therefore deformation of microstructures during shear strain. Depletion and/ or redistribution of positive pore water pressures are dependent on hydraulic conductivity, tortuosity of pore space and hydraulic gradient, thus reallocation of excess pore water pressures is closely connected with particle size distribution and (micro) aggregation of soils: short flow paths and/ or high conductivity favor the reduction of positive pore water pressure. Concave and thus contractive menisci are established when a new equilibrium is regained [23].

However, rheological characteristics and depletion of soil microstructure are not exclusively regulated by soil texture. They are further influenced by varying soil chemical features and especially varying water contents by which the rheological properties are modified.

4.2. Physicochemical parameters

The obtained findings corroborate that the microstructure of soils is the product of a variety of chemical, physical and biological interactions. Taking the combined effect of physicochemical features into account it appears that soil organic matter, concentration of Ca^{2+} , calcite content (CaCO_3) and pedogenic iron oxides (Fe_o) represent stabilizing factors, whereas the concentration of Na^+ , water content and the depth beneath ground level stand for structurally disadvantageous factors.

The positive effect of the beneficial factors is mainly attributable to their gluing and cementing effect. Soil organic matter influences soil structure on different scales and by several mechanisms due to its complexity [24]. Extracellular polymeric substances (EPS), metabolites and other breakdown product produced by microbial decomposition act as binding agent, stick soil particles together and enhance soil (micro) aggregation [24, 25]. Ca^{2+} provokes the formation of a stable card house structure [26] and various authors reported on the stabilizing effect of Ca^{2+} [e.g. 27, 28]. Thereby the formation of Ca^{2+} bridges represents a long-term effect [29], which is further intensified through the addition of organic substances [30]. In this regard, it appears plausible that CaCO_3 is also included with a positive sign in the transfer function, as it serves as a Ca^{2+} source. Cementation of mineral particles also occurs with an increasing content of iron oxides [31]. Strong interactions between iron oxides and organic substances benefit the formation of organo-mineral complexes, which on the one hand stabilize (micro) aggregates and on the other hand soil organic matter [32]. Markgraf and Horn [33] found the

formation of pseudosand in association with the cementation by iron oxides. Moreover they reported a significant reduction in microstructural stiffness after extracting pedogenic iron oxides from the soils under study.

The pedotransfer function for clayey soils includes sodium with a negative sign, constitute the well documented dispersive effect of Na^+ [e.g. 34], which is attributed to its monovalency and its big hydrated radius. Although it is generally known that in natural soils the soil moisture is insufficient to completely hydrate exchangeable cations [31], Na^+ -clay-connections are inherently unstable, as Na^+ mainly forms ionic bonds [35]. Finally, the water content negatively affects the elasticity of soils: increasing water content weakens the microstructure of soils due to lower menisci forces.

4.3. Menisci forces

The soil microstructure is weakened by increasing water saturation. Furthermore, the more fine-grained the soil material is, the more sensitive it reacts towards shearing under saturated conditions. Under saturated conditions additional water layers are absorbed, which enlarge the distance between individual particles and act as lubricant. The more water layers are attached to the mineral surface, the greater the mobility of single particles [36]. If chemically active particles are available, existing electrostatic interactions are reduced to that effect and finally capillary suction disappears when complete saturation is reached [37].

On the contrary, increasing desiccation stabilizes the microstructure of soil by water menisci forces, whereby fine-grained soils show a stronger reaction towards dewatering. In unsaturated soils, i.e. at negative pore water pressure, water menisci between soil particles provoke a contractive force such that the matric potential acts as a binding force between particles and stable connections are formed at rest [38]. Therefore any change in matric potential also causes a change in pore water pressure. By successive drainage soil particles are pulled together [39] and apparent cohesion increases [40], accordingly the resistance of soils against external stresses is enhanced [41]. The observed stabilizing effect of menisci forces on the microscale was also found by other authors [18, 42, 43].

5. Conclusion

The microstructural stiffness of the investigated riparian Marshland soils was characterized by amplitude sweep tests with controlled shear deformation. The results show a clear dependence on soil texture and water content. Depletion of internal structure takes place in a characteristic manner influenced by particle size distribution. Coarse-grained textures show a fast reduction of elasticity with increasing deformation at first, but a delay of microstructural collapse is possible due to interlocking and/ or rotation of single particles. The higher the silt and/ or clay content the less often interlocking between particles occurs. Hence, elasticity decreases gradually with increasing deformation. Desiccation stabilizes the microstructure of soils through amplified menisci forces, whereas increasing water saturation weakens the microstructure. Moreover the presented results confirm that the microstructure of soil is the product of a variety of processes and that the consideration of the combined effects of physicochemical features is appropriate and allowed the development of the plausible pedotransfer functions.

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