# RHEOLOGICAL INVESTIGATIONS OF FOUR BRAZILIAN OXISOLS AND A VERTISOL

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#### SUMMARY

Rheological investigations of bentonite suspensions (Ibeco Seal-80), silt- and clay-rich substrates corroborate the hypothesis, that the application of a parallel-plate-rheometer (MCR 300, Anton Paar) is appropriate for a description and quantification of microstructural changes on a particle-particle-scale. Amplitude sweep tests with controlled shear deformation (CSD) have been conducted on homogenised substrates deriving from four montmorillonitic, Fe-oxide-rich Brazilian Oxisols and a kaolinitic Vertisol confirm the applicability of such measuring device in soil mechanics. The influence of soil organic matter (SOM), Fe-oxides, and clay minerals on micromechanical shear behaviour under oscillation has been tested under saturated and drained (@-60hPa) conditions. From collected data, which include parameters as G' (storage modulus), G" (loss modulus), linear viscoelastic (LVE) range and a deformation limit (gL), the dissipation of elasticity in a viscoelastic substance i.e. soil, can be retraced. A comparison of this data has led to the assumption that Fe-oxides are supposed to have a more stabilising effect (s. occurance of *pseudosand*) than SOM.

Keywords: Rheology; amplitude sweep tests; microstructure

# INTRODUCTION

In soil mechanics general stress-strain relations are described in e.g. Mitchell and Soga (2005), using terms as the shear modulus G [MPa], shear strain g [%] or the strain rate  $\varepsilon$  [%] in dependency of time or frequency. Beside well established methods as i.e. direct shear or triaxial tests, rheometry seems to be a suitable application, whenever mechanical behaviour between single particles (tactiles, platelets, grains) needs to be measured, including possible effects due to both soil physical and chemical factors or pre-conditions.

Based on fundamental works of e.g. Vyalov (1986) and findings of Ghezzehei and Or (2001), Markgraf et al. (2006) and Markgraf and Horn (2006) investigated the influence of soil texture, natural given physicochemical properties, water content and valency effects (hydration mechanisms) on the micromechanical shear behaviour. Hence, in this work the aspect of Fe-oxide and SOM affected structural changes are included in order to proof the use of a parallel-plate-rheometer in soil (micro)mechanics once more. By executing oscillatory tests (amplitude sweep tests) with controlled shear deformation (CSD), which simulate deformation effects due to vibrations that are produced by i.e. farm implements, the microstructural stability of four kaolinitic Oxisols, with different Fe-oxide, soil organic matter (SOM) and clay contents as well a montmorillonitic Vertisol have been tested. The comparison of curve characteristics as well as single parameters as G' (storage modulus), G" (loss modulus), the calculated linear viscoelastic (LVE) range and deformation limit (gL) enable a quantification of dissipating elasticity in soils on a microscale (particle-particle scale).

# MATERIAL AND METHOD

Homogenised air-dried substrates <2mm were repacked in 45cm<sup>3</sup>-cylinders (ungefähre Lagerungsdichte?) and completely saturated with distilled water with two replicates and drained at -60 hPa. Physicochemical properties of the tested material are summarised in **Tab. 1**. The Oxisols are predominated by a kaolinitic clay friction, whereas an montmorillonitic clay mineralogy is given in case of the Vertisol. Here, a typical high content of Mg2+ and Ca2+ becomes obvious (ratio 1:2.5). Oxisols from Cruz Alta show textural differences, while microstructural changes in the Clayey Oxisols from Santo Angelo may be rather influenced by differences in soil organic matter (here Ct). Analyses were made according to standard methods in Schlichting et al. (1995).

	Sand	Silt	Clay	Na	Mg	Са	рН	Ct	Fe2O3	Feo	Fed	Feo/Fed
		[%]		[n	molc/	kg]	CaCl2	%		‰		[]
Sandy Oxisol (Cruz Alta)	75	7	18	0,4	2	5	4,1	0,6	2,0	0,6	14	0,04
Clayey Oxisol (Cruz Alta)	46	9	45	0,3	17	28	5,5	1,0	2,6	1,1	59	0,02
Clayey Oxisol 7 FFB (Santo Angelo)	6	25	69	1,5	17	41	4,4	6,6	3,9	2,5	99	0,03
Clayey Oxisol 7 NT (Santo Angelo)	5	19	75	1,1	21	43	4,9	1,1	4,1	2,6	102	0,03
Vertisol (Santana do Livramento)	3	32	65	2,7	157	396	5,5	3,4	n.v.	n.v.	n.v.	n.v.
FFB natural forest; NT no tillage										value	n. Ə	v. no

Tab. 1 Physicochemical properties of investigated substrates

A parallel-plate-rheometer MCR 300, (Anton Paar Comp., Germany), has been used. During all tests a constant temperature of 20°C is given, regulated by a PeltierUnit. **Figures 1a) and b)** show the application of the prepared samples (1a) and the measuring device (1b). Amplitude sweep tests under oscillatory conditions were conducted, with controlled shear deformation (CSD) g = 0.0001... 100%, an angular frequency w = p 1/s (f = 0.5 Hz) and 30 measuring points, which leads to an average test duration of 15 minutes. A plate distance of 4mm was preset according to a plate radius of the rotating bob of 25mm and the given texture (>2µm). The tests are controlled by the software USD 200. for calculations of the deformation limit gL and the yield stress ty "yield stress II" analyses were executed after each completed test run.



Fig. 1a) and b): Application of the prepared samples (1a), and the measuring device (1b).

# **RESULTS AND Discussion**

A representative result of an amplitude sweep test is shown in **Fig. 2**. The plots of the storage and loss modulus' (G' and G'') are generated automatically during a test. Three phases of elasticity loss are distinctive:

**Phase 1:** initial or plateau phase, G'>G", an elastic behaviour is given, represented by a spring for ideal elastic substances according to Hooke's law. A linear viscoelastic (LVE) range including a deformation limit gL

are characteristic parameters for the quantification of 'stored elasticity' of any viscoelastic substrate e.g. soils.

Phase 2: stage of transgression, intersection of G' and G".



**Phase 3:** final stage of structural collapse, G'<G", a viscous character is predominant, substances are creeping or running; this behaviour can be represented by a dashpot, an analogue for ideal fluids according to Newton's law.

**Fig. 2** Generated plots of G' (storage modulus)  $\square$  and G"(loss modulus)  $\square$ . Three stages of elasticity loss can be defined, showing a gradual transition of an elastic (G'>G") to a viscous (G'<G") character.

Due to comparing collected data microstructural effects of SOM and Fed depending on the actual water content w/w [%] become obvious. Values of gL and ty show significant differences (**Tab.2**).

untreated	gL	ty	w/w [%]	gL	ty	w/w [%]
	[%]	[Pa]	saturated	[%]	[Pa]	-60hPa
Sandy Oxisol (Cruz Alta)	0,00682	437	32	0,00661	941	18
Clayey Oxisol (Cruz Alta)	0,01037	499	37	0,01022	897	19
Clayey Oxisol 7 FFB (Santo Angelo)	0,01677	1105	41	0,00917	804	22
Clayey Oxisol 7 NT (Santo Angelo)	0,01277	1067	36	0,01277	919	18
Vertisol (Santana do Livramento)	0,02630	255	44	0,02835	532	42
SOM leached	gL	ty	w/w [%]	gL	ty	w/w [%]
	[%]	[Pa]	saturated	[%]	[Pa]	-60hPa
Sandy Oxisol (Cruz Alta)	0,00287	52	31	0,00560	288	25
Clayey Oxisol (Cruz Alta)	0,00584	101	46	0,00610	293	39
Clayey Oxisol 7 FFB (Santo Angelo)	0,00679	149	43	0,00771	350	34
Clayey Oxisol 7 NT (Santo Angelo)	0,00768	215	49	0,00856	502	43
Fed leached	gL	ty	w/w [%]	gL	ty	w/w [%]
	[%]	[Pa]	saturated	[%]	[Pa]	-60hPa
Sandy Oxisol (Cruz Alta)	0,00295	9,6	25	0,01487	140	16
Clayey Oxisol (Cruz Alta)	0,01413	49,7	37	0,02443	194	19
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Tab. 2 Summarised results from conducted amplitude sweep tests (CSD).

Clayey Oxisol 7 FFB (Santo Angelo)	0,01903	40,4	26	0,00585	114	23
Clayey Oxisol 7 NT (Santo Angelo)	0,02570	42,7	44	0,03423	208	17

In untreated samples, the combination of organic matter and Fe-oxides as strengthening factors lead to more stable conditions if compared to SOM or Fed leached samples. In the latter case, either the deformation limit and the yield stress become noticeable smaller, or, in other words: gL and ty untreated > SOM leached > Fed leached. This instance is illustrated representatively for Clayey Oxisol 7 FFB measured under saturated conditions in **Fig. 3**. **In addition, in almost every case,** gL and ty increase under pre-drained conditions (-60hPa, not illustrated). Herein, a secondary stabilising effect can be demonstrated. Levels of G'-60hPa and G" -60hPa plots are higher than under saturated conditions.



**Fig. 3** Results of conducted AST with samples of Clayey Oxisol 7 FFB, S. Angelo. Rectangles: untreated; circles: SOM leached; triangles: Fed leached. Filled symbols: storage modulus G' (■●▲), blank symbols: loss modulus G' (□or).

# CONCLUSIONS

By conducting amplitude sweep tests with a parallel-plate-rheometer, microstructural changes can be defined, which are influenced by i.e. Fe-oxides, textural differences (incl. clay mineralogy), or water content. Presented results show that Fe-oxides have in first instance an influence on the microstructural stability, followed by SOM. Regarding untreated Vertisol samples, relatively low values derive from these tests. In this case, differences in clay mineralogical properties have a major influence; the microstructural stability of montmorillonitic substrates is lower than of kaolinitic ones, but remain on a more or less stable level of viscoelasticity: turbulent (rotund shape) versus sliding shear behaviour (platy or aligned particles) become obvious (Markgraf et al., 2006).

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