Influence of mineralogical compounds and organic matter on rheological properties: Classifying stiffness degradation in soils

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A rotational rheometer with a parallel-plate measuring device is used to achieve stress-strain parameters, which define soil as viscoelastic material according to rheological theory. Hence, data deriving from conducted amplitude sweep tests with controlled shear deformation on Rothamsted Soils (Broadbalk long-term experiment) will be presented. The application of farm yard manure over more than 120 years led to an accumulation of organic carbon, resulting into a more rigid microstructural stability – with respect to the three-phase system soil – primarily due to a network of micro roots, and an increased water holding capacity and cation exchange capacity. Water content, texture, organic matter compounds, fungi and hyphae, contents and kinds of clay minerals, carbonates, (hydr)oxides, and cations have an effect on the microstructural stability (rigidity, stiffness), and shear behaviour. Storage modulus G’ (elasticity) and loss modulus G” (viscosity), the linear viscoelastic range (LVE), pre-yielding and yield point (intersection of G’ and G”) characterise the three stages of microstructural degradation of soil on the particle-to-particle scale. A semi-quantitative classification of rigid-nonrigid or elastic-viscous material is applied considering the loss factor tan δ and integral z, delivering fundamental information about microstructural strength of investigated soil samples and their ‘internal network’.

Material and Methods

A silty loam from Rothamsted, UK (Table 1) (Powlson 1994; Watts et al. 2006), was taken and analysed, considering different properties such as texture, clay mineralogy, soil organic carbon (SOC) content, and manure application.

According to the method introduced by Markgraf et al. (2006), amplitude sweep tests were conducted with a modular compact rheometer MCR 300 to achieve data of stress-strain correlations on the micro scale (particle-particle contact). Representative recorded plots of an amplitude sweep test are shown in Figures 1a and b (Markgraf and Horn 2009). If tan δ<1, G’ prevails G”, a gel character is given. Viscous behaviour is defined in case of tan δ>1, G” predominates G’. Furthermore, a correlation between in Figure 1a presented stages (Phases I-III) and phases of stiffness degradation in Figure 1b become obvious. Due to a decrease of G’, the ratio of G’’/G’ (= tan δ) increases; if tan δ=1 is reached, elastic and viscous parts are equivalent, and an absolute yield point (=cross-over) is given at a defined deformation (%). For further comparison, the integral z of tan $\delta(\gamma)$ lim γ=0.001% to lim γ="cross over point", with tan δ=1 as defined limit on the y-axis can be calculated.

Table 1. Physicochemical characteristics of investigated substrates, Rothamsted (Broadbalk)

	Sand	Silt	Clay	SOC
		(%)		
FYM*+ N_2	21	58	21	2.7
FYM				2.8
N_1PK(Na)Mg	19	58	23	1.0
N_2PK(Na)Mg				1.0
N_4PK(Na)Mg[‡]				1.1
N_6PK(Na)Mg[†]				1.2
Wilderness (grass)	21	57	22	4.0
Bare fallow[§] (Highfield)	7	68	25	1.1

*FYM farm yard manure (since 1885; N_2 added since 1968)
[‡]since 1968
[†]since 1985
[§]Bare fallow – plough/autumn (since 1959)

N_1, N_2, N_4, N_6 = 48, 96, 192, 284 kg N as ammonium nitrate

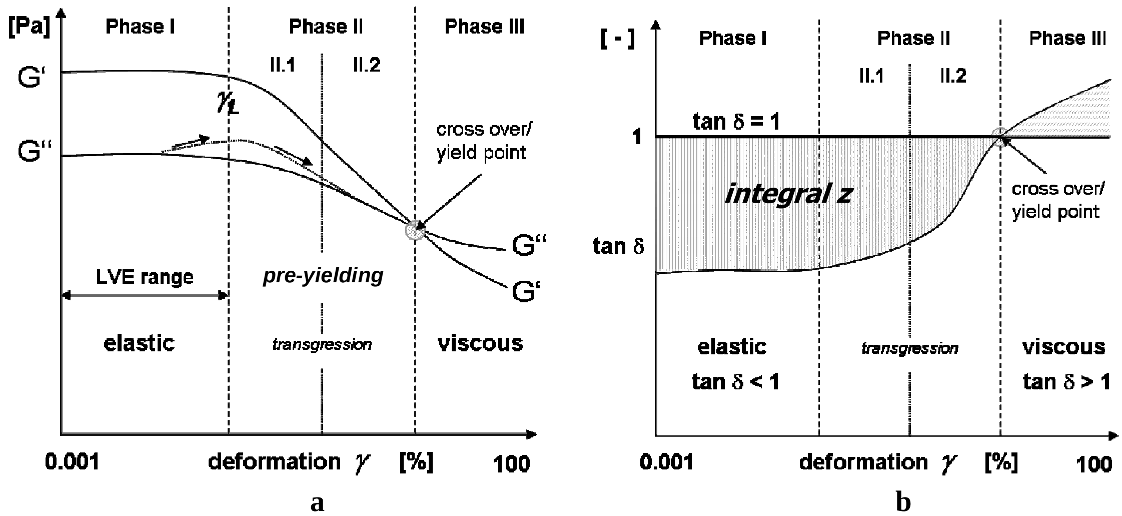


Fig. 1a. Idealised generated plots of storage modulus G' (Pa) and loss modulus G" (Pa) vs. deformation γ (%). In general, three stages of elasticity loss can be defined, showing a gradual transition of an elastic (G'>G") to a viscous (G'<G") character.
Fig. 1b. Deriving from one data set, results can be plotted in a loss factor vs. deformation coordinate system. Loss factor tan δ (-) equals the ratio of loss modulus to storage modulus (=G''/G'), and may function as analogue expression of elastic (tan δ <1), viscoelastic (tan $\delta \leq 1$) or viscous (tan δ >1) behaviour.

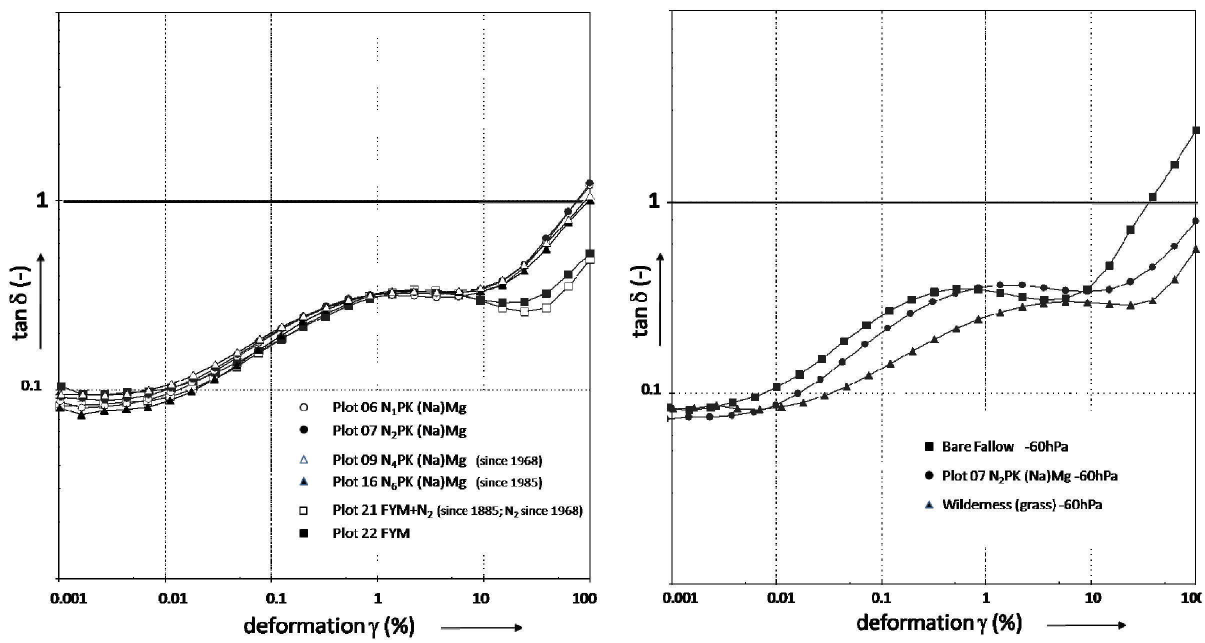
Results

Collected rheological data from conducted AST was used to achieve information about the influence of different manure applications on microstructural stability, in dependence on soil organic carbon (SOC) as well as inorganic nitrogen applications in combination with farmyard manure (FYM). In Figures 2a and b results from conducted AST with samples deriving from the Rothamsted Broadbalk long term experiment are shown. In general, curve characteristics are influenced by (i) water content: saturated (Fig. 2a); unsaturated (Fig. 2b), (ii) by treatments: N_xPK(Na)Mg, farm yard manure (FYM), bare fallow or wilderness (grass), (iii) SOC content, and (iv) texture. In comparison, different curve shapes are characteristic: in Figure 2a tan(δ) increases slightly within the section of γ=0.01…1%; an intersection with the tan(δ)=1-line does occur in few cases only: N_2PK(Na)Mg, N_4PK(Na)Mg, and bare fallow (unsaturated) variations. Furthermore, a slight increase in structural stability can be found as given in Figure 2a: FYM+N_2 > FYM >> N_6PK(Na)Mg ≥ N_4PK(Na)Mg > N_2PK(Na)Mg ≥ N_1PK(Na)Mg; samples which have been treated with farm yard manure since 1885 show a higher degree of stiffness compared to those which have been treated with N_xPK(Na)Mg within the last 40 years (e.g. N_4PK(Na)Mg). Secondly, differences in soil organic carbon content may have an effect on the microstructural stability as organic matter leads to a higher water retention, which results into a more stable system. This instant is well defined in Figure 2b. Pre-drained samples of wilderness (grass) show a higher microstructural stability than bare fallow, and N_2PK(Na)Mg. If, in addition, FYM (pre-drained) plots in Figure 2a are considered, the influence of SOC becomes even more obvious: wilderness plots have the highest SOC contents (4.0%), followed by FYM (2.7%), bare fallow, and N_2PK(Na)Mg (Table 1). Although bare fallow and N_2PK(Na)Mg have similar SOC contents (1.1%), textural differences affect shear behaviour, and, deriving from this, stiffness degradation: a more silty texture in case of bare fallow (68% silt) occurs to be less rigid than a loamy clay (N_2PK(Na)Mg, 56% silt). By calculating integral *z*, these structural differences can be expressed in absolute numbers; according to Figure 2a, this results into:

$z_{N_x\text{PK(Na)Mg saturated}} < z_{\text{FYM; FYM+N2 -150hPa}}$,

in case of Figure 2b into:

$z_{\text{bare fallow -60hPa}} < z_{N_2\text{PK(Na)Mg -60hPa}} < z_{\text{wilderness -60 hPa}}$



a **b**

Fig. 2a. Resulting graphs with tan δ (γ) of conducted amplitude sweep tests (AST) with Rothamsted Broadbalk samples: N_xPK(Na)Mg (saturated), and farm yard manure (FYM; pre-drained) treatments.
Fig. 2b. Resulting graphs with tan δ (γ) of conducted amplitude sweep tests (AST) with Rothamsted Broadbalk samples under unsaturated conditions (pre-drained at -60hPa): bare fallow, N_2PK(Na)Mg, and wilderness (grass).

Conclusions

Investigated Rothamsted samples, which have been treated with farmyard manure since 1885, showed a high degree in microstructural stability, as well as wilderness plots. As Haynes and Naidu (1998) pointed out, that 'there is a strong correlation between the amount of fertilizer N applied annually and the quantity of organic C accumulated in the soil'. This instant is also true, if N_xPK(Na)Mg-treated plots are considered, higher N-contents are correlated to a slight increase in C; due to this, microstructural stability is increased stepwise from N_1, N_2, to N_4, and N_6PK(Na)Mg treatments. In general, soil organic carbon improves soil structure and its functionality (Lemmermann and Behrens 1935, Tisdall and Oades, 1982; Haynes and Naidu 1998, Watts et al., 2006). Occurring menisci forces, which are formed due to drainage, maintain pore continuity, and as mentioned above, soil structure. It can be concluded, that rheological techniques and resulting parameters such as G', G", tan (δ) and z are a useful tool not only to describe and quantify microstructural stability, but also to link structuring processes, which are relevant for up-scaling considerations e.g. improving soil aggregation.

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