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Des défis du Nord au Sud

A rheological approach to the mechanical behavior of some marls samples in Haiti

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ABSTRACT
Soils can be considered as viscoelastic materials having an intermediate behavior between Newtonian fluids and the perfectly elastic bodies. This type of behavior is the basis of the birth of rheology which studies the flow, deformation and viscosity of materials under external stress taking into account its speed rate. Haiti is a country where seismic risk is very high. It is therefore important to study dynamically the mechanical properties of soils. This paper presents how rheology has been used to study the deformation and structural collapse of marl samples of the "Morne Delmas" geological formation in Haiti.

RÉSUMÉ
Les sols peuvent être considérés comme des matériaux viscoélastiques ayant un comportement intermédiaire entre les fluides Newtoniens et les corps parfaitement élastiques. Ce type de comportement est à la base de la naissance de la rhéologie qui étudie l'écoulement, la déformation et la viscosité des matériaux sous l'action d'une contrainte en tenant compte de sa vitesse d'application. Haïti est un pays où le risque sismique est très élévé. Il est donc important d'étudier les propriétés mécaniques des sols de façon dynamique. Cet article présente la façon dont la rhéologie a été utilisée pour étudier la déformation et la rupture d'échantillons de marnes de la formation géologique « Morne Delmas » en Haïti.

1 INTRODUCTION

The shear modulus is the parameter used to characterize the deformation caused by shearing forces. In soil mechanics, this parameter can be determined using the triaxial or simple shear test at the shear box. However, they are static measures and for a country like Haiti where the seismic risk is important, we must find a way to know the value of these parameters when under dynamic loads. The fact that our study site is located in a flat plain may involve, with the level fluctuation of the water table, liquefaction problems. We have therefore chosen to work with a less common approach used in civil engineering: rheology at micro scale. This science is already widely used in the polymer industry and ceramics (Markgraf et al. 2006) but it can fit very well with other material like soils.

The word "rheology" was created in the United States of America in 1929 by Eugene Cook Bingham (Persoz 1960). It comes etymologically from the Greek "rheo" which means "flow" and "logos" witch mean "study". So Rheology is a science that studies the flow, deformation, viscosity of materials under the action of a stress taking into account its application speed or more generally its dependence with time (Kohl).

In continuum mechanics, in general elastic solids (solid Hooke) and Newtonian fluids are obedient to the theories of elasticity and fluid mechanics. However, there are other materials that have an intermediate behavior between Newtonian fluids and perfect elastic solids; this is the case with materials such as granular pastes, polymers for example. These are viscoelastic bodies. It is the study of this type of behavior that is the basis for the birth of this discipline: rheology. Table 1 shows the different fields covered by the study of rheology.

Table 1: Application fields of rheology

Continuum mechanics			
Solid mechanics		Fluid mechanics	
Elasticity	Plasticity	Non-Newtonian fluids	Newtonian fluids
-	Rheology		-

Works on micro scale soil rheology are very few in the rheological literature. Links with geotechnical are even rarer. Among the first research on the subject, Markgraf et al. sought to evaluate the effects of agricultural rolling machinery on soil stability, the role of water content or the presence of ions in the soil (probably from fertilizer use). This science that studies the flow of the body under stress has more frequent use in the chemical industry (food, polymers …). Before presenting the rheological tests on the ground Morne Delmas (procedures and results), it is important to make an introduction of the main physical, chemical and mechanical aspects involved in rheology of soils. This section of the paper will address the association between them, soil particles, the forces acting between them and the interpretation of the action of these forces in rheology.

The soil is a very heterogeneous material: sand, silt, clay with pores containing air or water depending on the saturation degree (Figure 1). This is the case for example of the marl of the Morne Delmas formation.

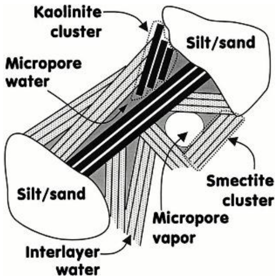

Figure 1: Representation of the heterogeneity of the soil (Ghezzehei and Or 2001)

The clay particles form a link between the sand or silt grains so that the mechanical properties of the material as a whole will depend on the nature of the clay bridges also depend on the type of mineral contained in the clay. In the case of swelling clays, water can be adsorbed in the space between the sheets, thereby increasing the thickness of these minerals. However, the interlayer space of the non-swelling minerals cannot be hydrated due to the strong attraction between these sheets, thereby promoting high rigidity of these minerals. In this case, there is therefore very little contact between the soil particles except in the case of swelling minerals. This is of vital importance in the understanding of soil behavior during application of shear stress.

The granular skeleton of non-swelling clay materials is very rigid in general. This is the case for example of kaolinites that have an internal friction angle relatively large. This granular structure is shown schematically in Figure 2. This structure allows the material to oppose a certain resistance to an external force which, when applied to this material tends to densify it, thus reducing the void volume. The material then has a behavior similar to that of an elastic solid. This status disappears when the intergranular forces are defeated by the external force. The material is in a state that is comparable to that of a more or less viscous fluid and no longer offers any shear strength (second part of the figure). The same mechanism is applied in the case of swelling minerals, but because of their lower stiffness, they reach the viscous state for smaller deformations in the case of non-swelling minerals.

The objectives of this paper is to study the mechanical properties of the Morne Delmas soil at the micro scale using rheology and the effect of water content and vibration frequencies on those properties.

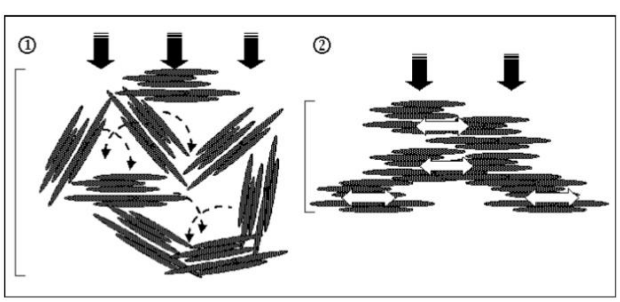

Figure 2: Mechanical behavior of swelling soils under oscillatory stress (Markgraf 2006)

2 METHODOLOGY

This section presents the soil samples used for this research, the material and how it is used to determine the rheological properties of two samples from the "Morne Delmas" geological formation.

2.1 Principles of rheology

The Maxwell model represented by a viscous damper and an elastic spring connected in series is one of the simple mechanical analogy used to represent the behavior of viscoelastic bodies (Figure 3).

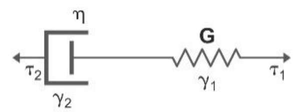

Figure 3: Maxwell model (Courraze and Grossiord)

Let's suppose that γ_1 and γ_2 as τ_1 and τ_2 are respectively the strains and stresses of the spring and damper. Since these two devices are placed in series, the rheological Maxwell equation [3] can be deduced from equations [1] and [2]:

$$\gamma = \gamma_1 + \gamma_2 \quad [1]$$

$$\tau = \tau_1 = \tau_2 = G\gamma_1 = \eta\dot{\gamma}_2 \quad [2]$$

$$\dot{\gamma} = \dot{\gamma}_1 + \dot{\gamma}_2 = \frac{1}{G}\dot{\tau} + \frac{1}{\eta}\tau \quad [3]$$

Where G is the shear modulus of the spring and η the dynamic viscosity of the damper.

2.2 Linear viscoelastic materials subjected to sinusoidal stress and strain

In linear viscoelasticity, strain and stress are sinusoidal and they evolve at the same frequency according to equations 4 and 5 were δ is the phase shift angle between the stress and the strain.

$$\tau(t) = \hat{\tau}\sin\omega t + \delta \quad [4]$$

$$\gamma(t) = \hat{\gamma}\sin\omega t \quad [5]$$

These values (real) of stress and strain can be replaced by complex numbers τ^* and γ^* considering that it is question of sinusoidal linear phenomena (Couarraze and Grossiord 1991). Thus, we have:

$$\tau^*(t) = \hat{\tau}e^{(i\omega t+\delta)} \quad [6]$$

$$\gamma^*(t) = \hat{\gamma}e^{(iwt)} \quad [7]$$

The dynamic complex shear modulus can be calculated as a ratio between τ^* and γ^*:

$$G^*(\omega) = \frac{\tau^*(t)}{\gamma^*(t)} = \frac{\hat{\tau}}{\hat{\gamma}} e^{i\delta} \quad [8]$$

The complex shear modulus can be divided into a real part (G': storage modulus) and an imaginary part G'': loss modulus) and we have:

$$G^*(\omega) = G'(\omega) + iG''(\omega) \quad [9]$$

For a perfect elastic body, G*(ω) = G and we have:

$$G'(\omega) = G \quad [10]$$

$$G''(\omega) = 0 \quad [11]$$

In case of Maxwell fluid, G*(ω) = iωη and we can write:

$$G'(\omega) = G \quad [12]$$

$$G''(\omega) = \eta\omega \quad [13]$$

In the case of a Newtonian viscous fluid, we have:

$$G^*(\omega) = \frac{i\omega}{\frac{1}{\eta} + i\frac{\omega}{G}} = \frac{i\omega}{1 + i\omega\frac{\eta}{G}} \quad [14]$$

If we say θ = η/G, the value of G* = G'+ iG'' is:

$$G^*(\omega) = \frac{\omega^2\eta\theta}{1 + \omega^2\theta^2} + i\frac{\omega\theta}{1 + \omega^2\theta^2} \quad [14]$$

2.3 Amplitude sweep test

Within the framework of this research, amplitude sweep tests were conducted with constant vibration frequency set at the beginning of the tests. This type of test is recommended in the case of material with high viscosity like soils and give more information on the role of the structure, the nature and the water content on their viscoelastic behavior (Markgraf, W. et al. 2006).

Figure 4 illustrates the principles of oscillatory shear test. We can distinguish two phases. The first one represents the phase shift between the strain γ and the shear rate $\dot{\gamma}$ and the second one, the phase shift between a perfectly elastic behavior and a Newtonian fluid behavior with, between the two, a viscoelastic behavior. In the first graphic, the solid line curve with the numbers 1, 2, 3 and 4 represents the different phases of the sinusoidal strain. When the strength F is applied, the strain is produced by an oscillation ($\omega = 2\pi f$) in a given direction where f (Hz) is the vibration frequency. A deflection $s(t)$ can be measured.

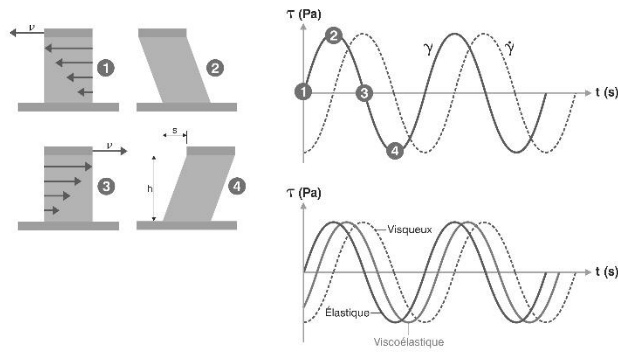

Figure 4: Principles of sinusoidal strain measurements (Markgraf and Horn)

The strain and the shear rate are calculated from the maximum deflection s_{max} according to the equations x to x (Markgraf et al.), (van der Vaart, 2010). The phase shift angle between perfectly elastic behavior and Newtonian behavior is named δ and is equal to 90°. The shear rate is calculated as show below:

$$\gamma = \frac{s(t)}{h} = \hat{\gamma} \sin \omega t \quad [1]$$

$$\hat{\gamma} = \frac{s_{max}}{h} \quad [2]$$

$$\dot{\gamma} = \frac{d\gamma}{dt} = \hat{\gamma} = \omega \cos \omega t \quad [3]$$

In order to study the effects of vibration frequencies on the "Morne Delmas" soil samples tests were conducted at 0,5Hz, 1Hz and 10Hz for strain between 0,1 and 100%. Each percentage of strain is equivalent to a rotation angle of the lower (mobile plate) of the rheometer. In this case, the relation between strain an rotation angle is shown in figure 1.

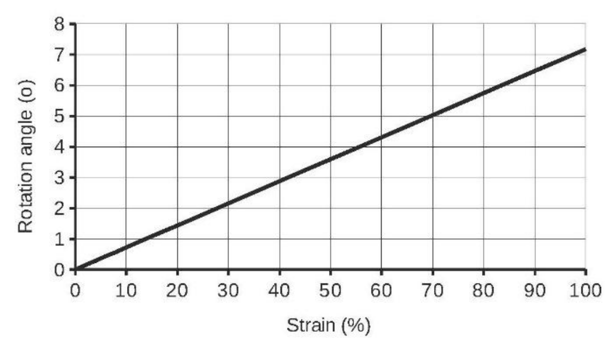

Figure 5: Relation between strain and rotation angle

Thus, as we have seen before, a storage modulus G' (elastic behavior) and a loss modulus G'' (viscous behavior) are measured during the amplitude sweep test. Figure 6 (Markgraf et al. 2012) illustrates the results that can be obtained.

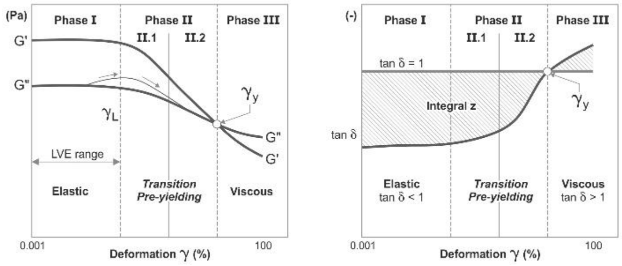

Figure 6: Storage modulus (G') and loss modulus (G'') in the left ; tan(δ) in the right (Markgraf et al. 2012)

On the left, we have G' and G'' in relation with the strain γ. The diagram is divided into three phases. The first one corresponds to a linear viscoelastic area. The two curves are practically parallel. The second phase is transition area where we can observe a convergence of the two curves at the point γ_y. At the third phase, the structure of the material does not allow it to resist to a shear stress. At this stage, it has a viscous behavior and the deformation is irreversible. The picture on the right expresses the left graphic in term of tan(δ). "Integral z" is the surface between the line tan(δ) = 1 and the curve tan(δ) = f(γ). The greater this surface is, the more the elastic behavior prevails on the viscous one.

2.4 The SIS-V50 biconical rheometer

A SIS-V50 biconical rheometer was used to perform the oscillatory tests. The material owes a range of amplitude from $0{,}01^o$ to 180^o and a range of frequencies from 0,001 to 50 Hz. The tests can be performed at constant temperature between 30^oC and 220^oC. The volume of the chamber is equal to $4{,}5cm^3$ and the space between upper (fixed) and lower (mobile) plates after the closure of the machine just before the beginning of the test is 4mm. Thus, the sheared soil will be confined in a pressured reservoir throughout the test period. Figure 7 shows the SIS-V50 rheometer and Figure 8 shows a section of the rheometer after the closure of the plates (SCARABAEUS website, 2012).

Figure 7 : SIS-V50 - SCARABAEUS, Wetzlar, Germany

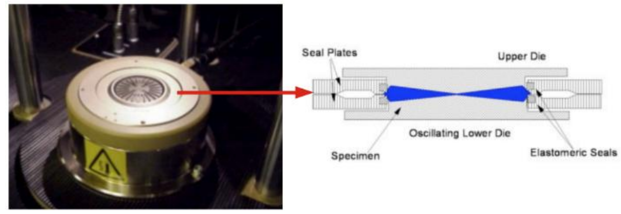

Figure 8 : Lower plate of the SIS-V50 rheometer - Section of the closed rheometer

With the biconical rheometer, the shear rate only depends on the angular velocity (ω) and the cone angle while in the cone-cone rheometer for example, the shear rate is not uniform. It varies from zero at the center to his maximum value at the outer edge (Markgraf, 2013; White, 2012).

2.5 Soil samples used

Two soil samples SONAPI-09 taken from the SONAPI industrial park and BME-11 taken from the Bureau of Mines and Energy were used for this research. The samples were taken at 2 m depth in a hand-dug well. The physical characteristics of these materials are summarized in table 1

Table 1: Physical properties of the soil samples used

Sample	S-09	B-11
$CaCO_3$ (%)	57,2	76,2
γ_s (kN/m^3)	26,7	26,6
Clay (%)	45,0	30,0
Silt (%)	36,0	57,0
Fine sand (%)	10,0	10,0
Coarse sand (%)	09,0	03,0
Gravel (%)	00,0	00,0
LL	47,6	38,0
PL	28,6	23,5
PI	19,0	14,5

$CaCO_3$: carbonate content ; γ_s : specific density; LL : liquid limit ; PL : plastic limit ; PL : plasticity index

According to the percentages of clays and carbonates, these three soils can be classified as marl (Calembert and Pel, 1972). The Casagrande diagram classifies S-09 and B-11 as medium plastic inorganic clays (Costet and Sanglerat 1969).

In addition, mineralogical tests (X-ray diffractometry, Scanning Electron Microscopy and Environmental Scanning Electron Microscopy) performed on this soil samples have shown the presence of swelling minerals in the "Morne Delmas" formation soil (Guerrier 2013).

Particles whose size is less than 53µm samples were used for rheological tests. Four main reasons are behind this choice. The first reason is the need to avoid putting between the rheometer plates too coarse elements to maximize the chances of having a sample as homogeneous as possible. The second reason is that as part of this thesis concerning the swelling soil, only that fraction of the soil consists mainly of clays and silts is relevant for testing. The third reason is that higher particle sizes have no effect on some rheological properties such as thixotropy (Ferroir et al., 2001). Furthermore, the authors who have worked on the issue, did it with the clayey and silty part of the materials used. This will make a good basis for comparison with our results. The fourth and final reason is that these tests are part of the result of previous work on the rheology of the soil Delmas (Simon, 2009 and Breye, 2011).

3 RESULTS AND DISCUSSION

The tan(δ) versus strain (γ) curves are presented below for the soil samples used. The general shape of tan(δ) curves are illustrated in figure 9 (Markgraf et al.). The curves obtained for the Morne Delmas soils have a shape similar to the intermediate curve (silty texture) between sandy and clayey texture.

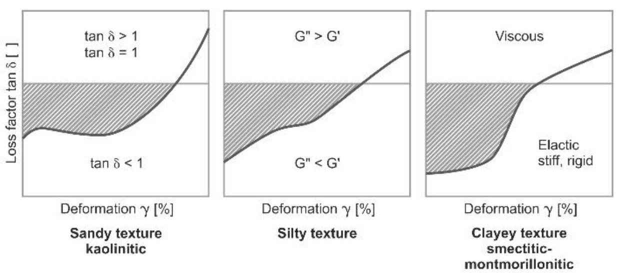

Figure 9: General shape of the tan(δ) versus strain according to the soil texture (Markgraf et al.)

The two following graphics represent the tan(δ) as a function of the strain for S-09 and B-11 at 20% and 40% of water content and for three different frequencies.

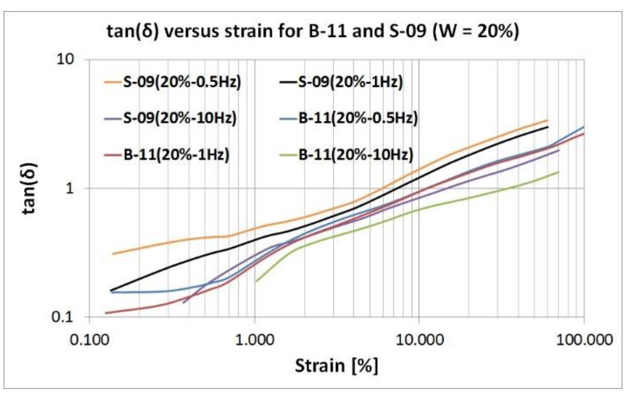

Figure 10: tan(δ) versus strain for B-11 and S-09 (W = 20%)

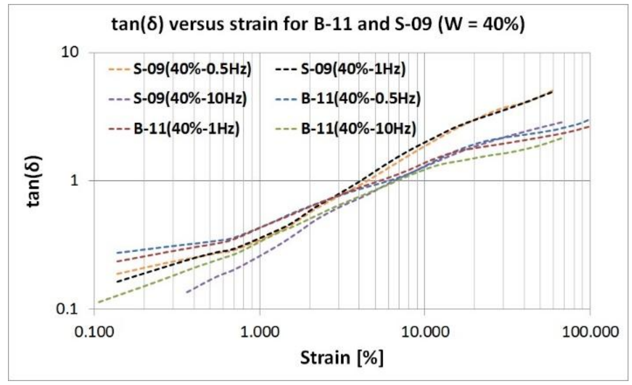

Figure 11: tan(δ) versus strain for B-11 and S-09 (W = 40%)

Table 2: Strain at the yield point for SONAPI-09 and BME-11 samples at 20% and 40% of water content and at different frequencies

W[%]	20			40		
f[Hz]	0,5	1	10	0,5	1	10
$\gamma_{yS\text{-}09}$	5,92	7,30	14,70	4,45	4,05	6,46
$\gamma_{yB\text{-}11}$	11,01	11,07	34,27	5,96	5,29	6,80

3.1 Effects of soil nature

If we consider the amount of CaCO3 in these 2 soil samples and Atterberg limits, we can assert that SONAPI-09 has a more significant swelling character than BME-11 (Guerrier 2014). At 20% of water content, for the same frequency, SONAPI-09 reached the yield point before BME-11. This behavior can be explained by the fact that the effect of clay minerals on the rheological properties of soils is directly related to the nature of the mineral found in those soils. In the case of non-swelling clays, there is no way to hydrate the inner surface of the particles because of the strength of links between the clay sheets. Much of the water contained in the soil remains between the particles which results in a distribution of the effective stress in the soil on very few bonds between soil particles. The elastic nature of the soil is more pronounced and the material remains within the elastic range for larger deformations (Markgraf 2006). This, therefore, gives a better stability at the microstructure of non-swelling soils with compared to swelling soils. On the contrary, the electrostatic force the space between swelling clays sheets is weak and they can be hydrated. This particularity does not allow the structure to resist to larger deformation (Ghezzehei 2001).

3.2 Effect of $CaCO_3$ content

At equal frequencies, strain at yield point for BME-11 is greater than that of the sample SONAPI-09 for the same water content. This is probably due to the nature of clay minerals, but also the percentage of carbonate contained in the two samples. The $CaCO_3$ has binding properties. Indeed, its presence in a clay soil changes the surface charge of the clay particles. This changes the structure of the double layer whose development was facilitated by the surface modified fillers by the presence of carbonates. The extension of the double layer is thus reduced, and therefore the apparent volume of soil particles, resulting in a contraction of the soil. This contraction results in clay flocculation and rearrangement of particles thereby removing pores. Calcium also create bridges between these clay flocculates. The plasticity index decreases, increasing the plastic range of the soil (Cabane 2005).

3.3 Effect of the vibration frequency

As we can see in figure 12 and figure 13, the strain at the yield point is even higher than the vibration frequency is high. That means that the material keeps an elastic behavior for a large range of strain when increasing the vibration frequency. If we say that the shear modulus of the soil G is equal to ρV_s^2 (Filiatraux 1996), for two identic samples of soils (with the same density ρ), we have a higher value of G for a higher velocity of the shear wave (V_s). However, in dense media, high frequency shear waves travel more slowly than low frequency shear waves. It follows, therefore a reduction in the shear modulus and the material thus has less rigidity and can support larger deformations. Note that the samples placed in the rheometer are very dense because of containment that the geometry of this machine allows to impose.

Another explanation can justify those results. The elastic and plastic behaviors characterize homogeneous substances such as liquids or heterogeneous materials such as alloys. Several natural bodies are dispersions of a solid or liquid phase in a liquid phase (suspension, emulsion). This is the case of soil with a certain water content. Agitation of these bodies gives them a liquid behavior while at rest, they are in gel form. There is also a decrease in viscosity with increasing shear. However, some thixotropic soils tend to gel more quickly when they are agitated at a certain pace than when they are at rest. That is the case of rheopectic materials (Persoz et al. 1960). This could explain the fact that at high frequency vibration, the SONAPI-09 and BME-11 soils keep their gel state to larger deformations.

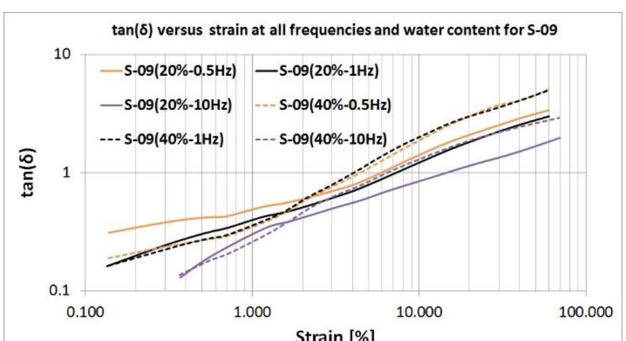

Figure 12: tan(δ) versus strain at all frequencies and water content for S-09

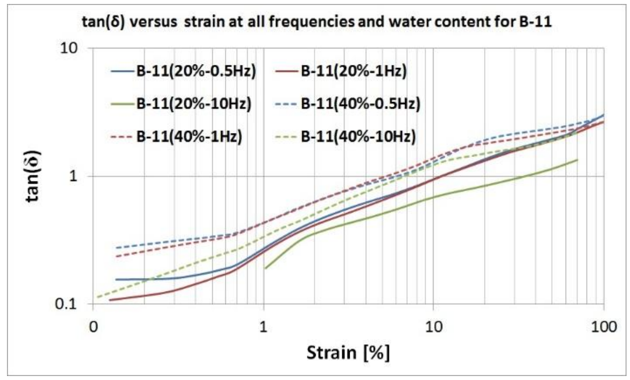

Figure 13: tan(δ) versus strain at all frequencies and water content for B-11

4 CONCLUSION

This paper presents a rheological approach to the study of mechanical properties of soils considered as a viscoelastic material. Two marl samples (S-09 and B-11) from the “Morne Delmas” geological formation were used for this research. Amplitude sweep tests were conducted with a SIS-V50 biconical rheometer (shear rate independent of the position along the rotor radius). Several conclusions can be drawn at the end of this research. The strain value at the yield point where G”>G’ depends on the nature of the clay minerals contained in the soil. It is lower for the swelling minerals than non-swelling minerals at the three working frequencies. An increase in the water content leads to a decrease in the strain value at the yield point. Soils with high calcium carbonate content show higher strain value at yield point than soils with lower calcium carbonate content. The stain value at yield point increases with an increase of the vibration frequencies.

5 PERSPECTIVES

We have seen in this paper how we can use rheology to study mechanical properties of soils at a microscopic scale. Some works have to be done to make links between the mechanical parameters determined by rheology and those determined in classical soil mechanics.
The rheometer used is very accurate material but we still have to work on the reproducibility of the tests conducted. We showed an example of two amplitude sweep tests conducted on the same exact sample and the curves are similar but it is not sufficient to say categorically that the tests conducted within the framework of this research are perfectly reproducible.

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REFERENCES

Breye, N. 2011. *Étude chimique et rhéologique en fréquence des argiles de Morne Delmas (Haïti)*. Mémoire de Master, Université catholique de Louvain, Belgique.

Cabane, N. 2005. *Sols traités à la chaux et aux liants hydrauliques*. Thèse de doctorat, École Nationale Supérieure des Mines de Saint-Étienne, France.

Calembert, L. and Pel, J. 1972. Introduction à la géologie de l'ingénieur. *Notes de cours*, Faculté des Sciences Appliquées de l'Université de Liège, Liège, Belgique.

Costet, J. and Sanglerat, G. 1981. *Cours pratique de mécanique des sols*, Volume 1, 3ème ed., Dunod, Paris, France.

Couarraze, G. and Grossiord, J. L. 1991. *Initiation à la rhéologie*. Lavoisier – Tec & Doc, Paris, France.

Filiatraux, A 1996. Éléments de génie parasismique et de calcul dynamique des structures. Presses Internationales Polytechnique, Canada.

Ferroir, T., Khelifi, E. and Letellier, M. 2001. *Étude de la thixotropie des sols à partir d'exemple en région parisienne*. http://tristan.ferrooir.free.fr/index.php

Ghezzehei, T. A. and Or, D. 2001. Rheological properties of wet soils and clays under steady and oscillatory stresses. *Soil Science Society of America Journal*, 65:624 – 637.

Guerrier, K. 2013. *Étude des propriétés mécaniques et rhéologiques d'échantillons de marne de la formation géologique « Morne Delmas » en Haïti*. Thèse de doctorat, Université catholique de Louvain, Belgique.

Kohl, P. website visited in 2012. Rhéologie et formulation. http://patrick.kohl.pagesperso-orange.fr/rheologie/rheo_1.htm

Markgraf, W., Horn, R. and Peth, S. 2006. An approach to rheometry in soil mechanics – Structural changes in bentonite, clayey and silty soils. *Sciences Direct* 91: 1 – 14.

Markgraf, W., Watts, C. W., Whalley, W. R., Hrkac, T. and Horn, R. 2012. Influence of organic matter on rheological properties of soil. *Applied Clay Science*, 64: 25 – 33.

Markgraf, W. and Horn, R. consulted in 2012. *An introduction to rheology in soil mechanics - Structural behavior of bentonite against salt concentration and water content*. http://www.bodenkunde2.uni-freiburg.de/eurosoil/abstracts/id1133_Markgraf_full.pdf . Institute for Plant Nutrition and Soil Science - Christian-Albrechts-University of Kiel - Germany

Markgraf W. 2013. Personal communication.

Markgraf, W., 2006. Microstructural changes in soils rheological investigations in soils mechanics. Ph.D Thesis, Christian-Albrechts-Universität, Germany.

Markgraf, W., Horn, R. and Peth, S. 2006. An approach to rheometry in soil mechanics – Structural changes in bentonite, clayey and silty soils. *Applied Clay Science*, 91:1 – 14.

Persoz, B., Crussard, C., Durand, R., Goguel, J., Joly, M., Kauer, F., Képès, A., Lafuma, H., Meyer, A., Saucier, H., Thirion, O., DeVries, A. J., and Weiss, H. 1960. Introduction à l'''etude de la rhéologie, Dunod, France.

SCARABAEUS website 2012. http://www.scarabaeus-gmbh.de/en/home/

Simon, F. 2009. *Analyse physique et chimique d'argiles*. Mémoire de Master, Université catholique de Louvain, Belgique.

White, J.L. 1995. Rubber processing: technology, materials, principles, Hanser Publishers.