Linear Viscoelastic Behavior of Bentonite-Water Suspensions

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Abstract:
Bentonite are extensively used materials in a wide range of applications. Creep and oscillatory shear experiments in the linear viscoelastic domain were carried out on bentonite-water suspensions at different solid fractions. It was found that bentonite dispersions exhibit important viscoelastic behavior which could be represented by the generalized Kelvin-Voigt mechanical model. It is well known that an exhaustive study of colloidal dispersions may require the determination of its viscoelastic properties over a wide frequency scale. Unfortunately, due to microstructure changes, the experiments are limited in time. In order to avoid such limitation, oscillatory data were deduced from creep curves - without actually vibrating the clay dispersions - because a periodic experiment at frequency ω is qualitatively equivalent to a creep test at time $1/\omega$. That is, it was possible to complete the dynamic response in the low-frequency range using data obtained from the transient response in creep.

Zusammenfassung:
Bentonit ist ein häufig verwendetes Material mit breitem Anwendungsspektrum. Dehnungs- und oszillatorische Scherexperimente wurden im linearen viskoelastischen Bereich für Bentonit-Wasser-Suspensionen mit unterschiedlichen Feststoffgehalten durchgeführt. Wir finden, dass diese Materialien ein viskoelastisches Verhalten aufweisen, das sich durch das generalisierte Kelvin-Voigt-Modell beschreiben lässt. Es ist wohlbekannt, dass die erschöpfende Untersuchung kolloidaler Suspensionen die Kenntnis ihrer viskoelastischen Eigenschaften über einen grossen Frequenzbereich erfordert. Unglücklicherweise, bedingt durch mikrostrukturelle Umordnungen, sind die Experimente nur während eines beschränkten Zeitintervalls durchführbar. Um diese Beschränkung zu umgehen, wurden Schwingungsdaten von den Ausdehnungkurven abgeleitet – ohne die Dispersionen in Schwingungen zu versetzen – da ein periodisches Experiment bei Frequenz ω einem Zeitstandversuch zur Zeit $1/\omega$ qualitativ gleichwertig ist. Das heisst, es war möglich, die dynamische Resonanz im Niederfrequenzbereich zu ermitteln, indem Daten verwendet werden, die aus der zeitabhängigen Antwort während der Ausdehnung erhalten werden.

Résumé:
Les bentonites sont des argiles couramment utilisées dans diverses applications industrielles. Les expériences en fluage et en régime dynamique ont permis de mettre en évidence le caractère viscoélastique des suspensions de bentonite. On a notamment montré que ces propriétés viscoélastiques peuvent être correctement décrites par un modèle analogique de type Kelvin-Voigt. Il est admis qu'une étude complète des suspensions colloïdales nécessite la connaissance des propriétés viscoélastiques sur une gamme de fréquences très large (souvent de 10 à 12 décades). En raison des modifications structurales intervenant pendant la mesure, les expériences sont limitées dans le temps. Pour contourner ce problème, et en se basant sur le postulat qu'une expérience en dynamique à une fréquence ω est qualitativement équivalente à une expérience en fluage à un temps $1/\omega$, il a été possible de calculer les grandeurs dynamiques, aux faibles fréquences, à partir des mesures faites en fluage, sans passer par l'expérience.

Key words: bentonite suspensions, viscoelastic properties, creep curve, storage and loss moduli

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1 INTRODUCTION

Montmorillonite clays, known as widespread thickening agents for industrial fluid formulations, have gained great attention in recent years. This is due to the wide range of applications to which they have been put, and their potential for novel uses in the future. Among these applications are Civil Engineering like soil boring, slurry walls or cast-in-places piles and industrial applications including cosmetics (creams), chemical (paints), food products (wine) or petroleum (water-based drilling fluids) [1 - 3] where they are subjected to sizable deformations relative to the stress required for flow.

Rheology of bentonite-water suspensions is of paramount importance and its correct evaluation can provide useful information for the design of engineering applications, formulation and quality control of commercial production, storage stability, knowledge of the effects of mechanical processing on the structure of the suspensions [4, 5]. Therefore, the rheological behavior of bentonite suspensions has been an object of interest for a rather long time. The findings of these studies were reviewed in significant contributions: Luckam and Rossi [6], Duran et al. [7], Ramos-Tejada et al. [8, 9], Gao et al. [10], to name just a few.

Steady shear viscosity is a property of all fluids regardless of whether or not they exhibit elastic behavior; however, many phenomena cannot be described by the viscosity functions alone and elastic behavior must be taken into consideration to obtain information in conditions close to the unperturbed state and, consequently, to characterize their microstructure. In addition to this, the knowledge of the linear viscoelasticity functions is necessary for the prediction of the viscous flow through the development of suitable non-linear viscoelasticity models. Bentonite suspensions fall in this category of materials. Indeed, their consistency varies from fluid to almost solid. Dilute dispersions may behave as simple liquids, whereas those which contain a considerable amount of solid in the continuous phase may function essentially as solids. Between these two extremes lies an important category comprising the semisolids dispersions which exhibit simultaneously both liquid and solid properties in significant proportions and are therefore viscoelastic. Unsteady state shear measurements (transient and oscillatory) provide a dynamic means of evaluating viscoelasticity. Furthermore, it is well known that an exhaustive study of colloidal dispersions may require the determination of its viscoelastic properties over a wide frequency scale. Unfortunately, due to microstructure changes (evaporation, sedimentation), the experiments are limited in time and are inaccurate in the short time region because of instrumental inertia. It is therefore very difficult or rather sometimes impossible to get experimental viscoelasticity data over a wide range of frequencies [11 - 13].

The main objective of this work was to investigate the viscoelastic properties of bentonite suspensions (creep, oscillatory and spectral analysis) and to consider some fruitful methods which may be used in order to extend the dynamic (frequency sweep) results to lower frequencies.

2 MATERIALS AND METHODS

The bentonite investigated in this work is montmorillonite in its sodium form (from Maghnia, West Algeria). It is a powerful viscosifying natural clay which is employed for industrial fluid formulations [4, 6, 14, 15]. The crystal structure of these minerals, which present a plate-like geometry, belongs to the phyllosilicate 2:1 family and is formed by an octahedral layer sandwished between two tetrahedral layers [6, 16] (Fig. 1). The layers include a high negative charge compensated by interfoliaceous cations (Ca^{++}, Na^{+}). These minerals are highly sensitive to hydratation that causes particle swelling, and consequently induces modifications of their rheological characteristics [17]. Due to the very large side extension of the layers of the crystal, from 0.1 to

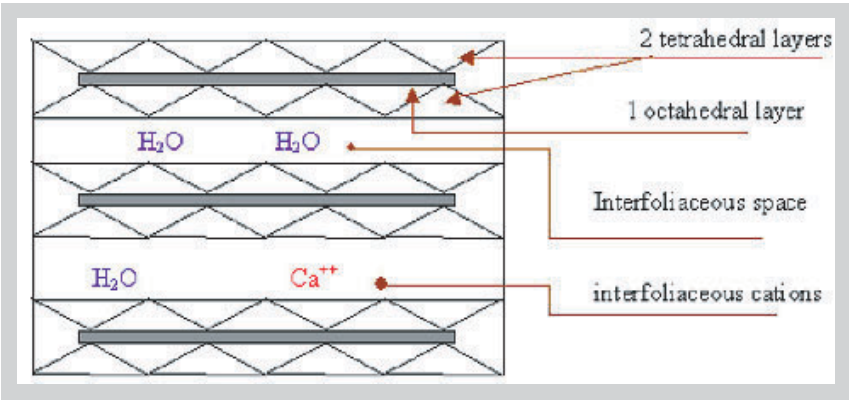


Figure 1: Layers disposition of 2:1 type for montmorillonite clays.

1 mm in lenght, a relative flexibility of the particles, and thus elastic behavior, can be observed [18, 19].

Bentonite suspensions with mass concentrations ranging from 4 to 10 % were studied. In order to obtain reproducible results, an identical experimental procedure was used for the preparation of all suspensions. Bentonite powder was first gradually added to the required amount of distilled water. Homogenization was obtained by continuous mechanical agitation during 24 hours. It was observable that the low concentrations mixtures (4 and 5 %) were stable up to 10 hours after preparation and considerably longer times for the high concentration mixtures (6 to 10 %). An average pH equal to 9 was measured for the suspensions resulting in the formation of a 'house of cards' structure [6, 8, 16, 20].

For purposes of this experimentation a controlled stress instrument (Carri-Med CSL 100, with a cone-and-plate measuring device : diameter = 4 cm, angle = 0.034 rad, gap = 0.06 mm) was used to determine the creep and oscillatory response of the bentonite suspensions [21]. Since the domain structure of bentonite suspensions is quite sensitive to shear deformation history, samples were carefully loaded on the rheometer plate with a spatula and then the plate was slowly raised. All measurements were conducted at controlled temperature of 20° ± 0.1°C. To prevent changes in composition during measurements due to evaporation of the suspending fluid, the experiments were carried out in a water saturated atmosphere. In addition, the response of the instrument was controlled using maltose solutions at different concentrations and a Newtonian standard oil of 0.2 Pas viscosity.

Previous measurements with bentonite suspensions [22] have shown wall slip effects due to inhomogeneous fluid properties at solid boundaries [23, 24]. To prevent this effect the surfaces of the cones and the plates were coated with bentonite powder glued on a thin layer of a silicon-based adhesive. The advantage of this technique is to allow the creation of isomorphism conditions at the walls without notable modification of the inertia and geometry of the measuring device.

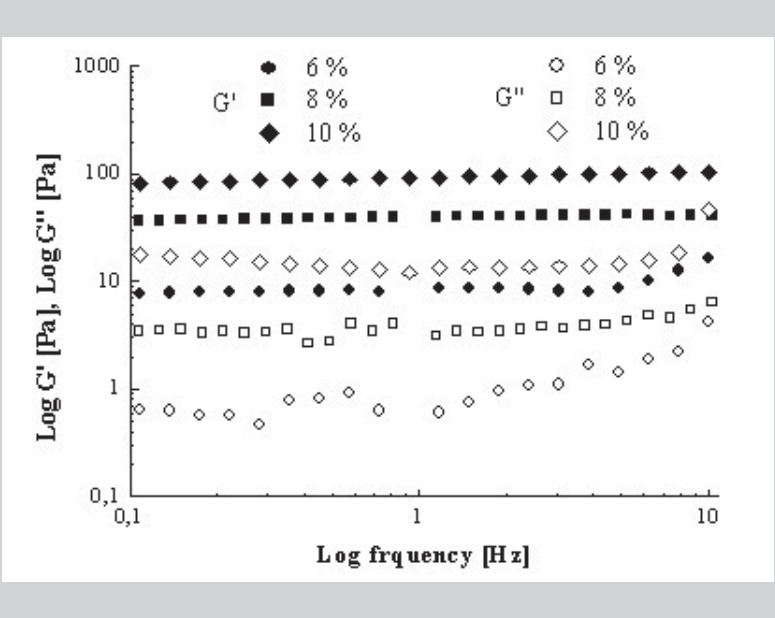


Figure 2: Dynamic experiments : variation of G' and G'' with frequency for three bentonite dispersions (6, 8 and 10 wt%).

3 EXPERIMENTAL RESULTS AND DISCUSSION

The viscoelastic properties of semi-solid systems are generally observed at low deformations to minimize the possibility of breakdown in structure. The test simply flexes organized structures as flocs or gel networks but does not disrupt them. At the end of the experiment, the fundamental structure of the material is unchanged and the test is therefore rheologically non-destructive. The data obtained from such an investigation may then be interpreted with linear viscoelatic theory [25, 26, 27].

3.1 DYNAMIC ANALYSIS

The linear viscoelastic properties of bentonite dispersions were first studied in terms of complex dynamic properties using oscillatory shear experiments. The amplitude of the excitation must be small enough not to exceed the linear limit. A sinusoidal stress $\tau = \tau_o \sin \omega t$ is applied to the sample whereas its corresponding sinusoidal strain $\gamma = \gamma_o \sin(\omega t + \delta)$is measured, ω is the frequency of oscillation in rad/s, γ_o is the strain amplitude, τ_o is the stress amplitude, δ is the phase angle between the stress and the strain (i.e.,the loss angle) and t is the time. The shear storage and shear loss moduli, respectively $G' = (\tau_o/\gamma_o)\cos\delta$ and $G'' = (\tau_o/\gamma_o)\sin\delta$, were calculated from the measured outputs, the strain amplitude γ_o and the loss angle δ. The storage modulus is a measure of the reversible, elastic energy, while the loss modulus is a measure of the viscous dissipation. The effect of frequency was investigated with a constant deformation of 10%.

The moduli *G'* and *G''* are plotted in Fig. 2 as a functions of frequency for three different bentonite suspensions (6, 8 and 10 %). It can be observed that both *G'* and *G''* are frequency independent, showing a pronounced elastic behavior of the suspensions and increase with bentonite concentration which indicates an increase of both elastic and viscous properties. This plateau region has been related to the formation of a three-dimensional network. Furthermore, for each concentration the storage modulus *G'* is larger than the loss modulus *G''* in the whole experimental frequency range studied showing that the elastic behavior of bentonite dispersions is dominant compared to the viscous behavior. This result is in good accordance with those obtained by other authors [8, 10, 28].

3.2 CREEP ANALYSIS

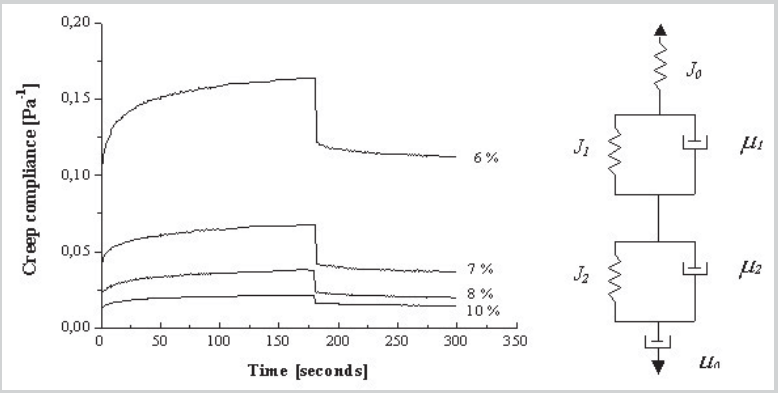
3.2.1 Discrete creep analysis

In addition to the transient properties discussed previously, creep tests were carried out. A creep curve is obtained by applying at time t_o a stress held constant and measuring the time evolution of the resultant deformation followed by a strain recovery curve obtained after removal of the constant stress. The elastic properties are defined by correlating the results with the classical viscoelastic models of Maxwell or Kelvin-Voigt which can be physically described by assemblies of dashpots and springs. From our experiments it was found that the viscoelastic behavior of the bentonite dispersions could be represented by the generalized Kelvin-Voigt model which combines a Maxwell unit (Newtonian dashpot in series with purely elastic spring) in series with one or more Kelvin-Voigt units (dashpots in parallel with springs). The creep compliance for the bentonite dispersions is then defined as :

$$f(t) = J_o + \sum_{i=1}^{n} J_i(1 - e^{-t/\theta_i}) + \frac{t}{\eta_N} \qquad (1)$$

where *f(t)* is the over-all creep compliance at the time *t* during the test, J_o is the purely elastic contribution (or the instantaneous elastic compliance) and η_N is the purely viscous contribution, represented by the dashpot of the Maxwell model, i.e. the uncoupled or residual steady-state viscosity obtained from the creep curve at long times when the compliance curve is linear. The set $\{J_i, \theta_i;\ i = 1, ..., n\}$ forms the discrete distribution (or discrete spectrum) of retardation times θ_i where J_i is the contribution to retarded elastic compliance of the i^{th} component with a retardation time $\theta_i = J_i \eta_i$ and a viscosity η_i. Fig. 3 shows the creep and recoil curves obtained for different bentonite concentrations (6, 7, 8 and 10 wt%) and Tab. 1 summarizes the viscoelastic parameters derived from Eq. 1. One may observe a decrease of the elastic compliance with concentration, i.e. increase of the elastic modulus $E = 1/J$, indicating an increase of the viscoelastic properties. Also, it was found that two Kelvin-Voigt units ($i = 2$ in Eq.1) were necessary to describe the bentonite suspensions viscoelastic behavior by the mechanical model of Kelvin-Voigt, except for 4 wt% where $i = 1$.

The viscoelastic parameters were derived from the creep compliance-time curves by use of the well-known method initially developed by Inokuchi [29, 30]. In the analysis procedure, the instantaneous compliance J_i is set from the curve intercept on the compliance axis of the graph, and the Newtonian viscosity η_N is derived from the slope of the linear portion of the curve which represents the Newtonian shear of the sample. When these terms are removed from Eq. 1, only the exponential terms remain. Thereafter, a stepwise analysis of the retarded elasticity region of each curve yields discrete pairs of values of J_i, θ_i for *n* viscoelastic Kelvin-Voigt units.



Conc [wt%]	J_0 [1/Pa]	J_1 [1/Pa]	J_2 [1/Pa]	η_W [Pas]	η_1 [Pas]	η_2 [Pas]	θ_1 [s]	θ_2 [s]
4	1.9864	20.934	-	508	0.9	-	18.4	-
5	0.3766	0.1392	0.1055	5691	141.9	51	19.7	5.4
6	0.0964	0.0291	0.0251	12960	806	123.8	23.4	3.1
7	0.0373	0.0244	-	23710	1115	-	27.2	-
8	0.0229	0.0077	0.023	37760	3507	3524	27.1	8.2
10	0.0119	0.0055	0.0018	86870	4593	4482	25.1	8.1

Figure 3 (above): Creep compliance and recovery curves for different concentrations (6, 7, 8 and 10 wt%). A mechanical model which may be used to describe the creep results is also shown (a Maxwell unit in series with two Kelvin-Voigt units). Note that creep curves for 4 and 5 wt% concentrations are not represented here (J_{max}= 0.65 Pa^{-1} at t = 180 s for 5 wt%).

Table 1 (below): Viscoelastic parameters of the different dispersions.

3.2.2 Continuous spectral analysis

Instead of the mechanical model approach used above, we can convert a creep curve into a continuous retardation spectrum. This spectrum represents the viscoelastic behavior of bentonite suspensions in a more general way than a function such *f(t)* [11, 27, 31, 32]. The number of Kelvin-Voigt units is allowed to increase to infinity (i $\longrightarrow \infty$ in Eq.1) and the retardation spectrum $L(\theta)$ is defined by the continuous analog of Eq. 1:

$$f(t) = J_o + \frac{t}{\eta_N} + \int_{-\infty}^{+\infty} L(\theta)(1 - e^{-t/\theta}) d\ln\theta \qquad (2)$$

where $L(\theta)$ is of the nature of a distribution function with the dimensions of a compliance. Since the spectra cannot be determined directly, they must be obtained through mathematical means from the experimental data. Tschoegl [33] has thoroughly discussed approximation methods based on differential or finite difference calculus, for obtaining $L(\theta)$. In this work, the distribution function was derived using a first-order approximation method using Eq. 3:

$$L(\theta) = \frac{d}{d\ln t}\left[f(t) - \frac{t}{\eta_N} \right] \qquad (3)$$

The elastic part of the creep compliance derived from the creep curve ($f(t) - t/\eta_N$) is plotted as a function of ln *t*. The gradients of this curve at selected points provide data for Eq. 3 from which the required continuous spectrum is obtained. Typical continuous spectra, obtained for many bentonite-water suspensions, assume the appearance of bell-shaped curves, as illustrated in Fig. 4. Maxima in the spectrum reveal concentrations of time dependent elastic strain mechanisms operating at a molecular level, i.e. retardation processes, as determined by their contributions to the compliance. The height of each spectrum decreases with bentonite concentration, reflecting a consistency increase and thus, an increase of viscoelastic properties. Finally, it can be observed that, for high retardation times, the function $L(\theta)$ approaches zero, showing that the steady state flow is reached.

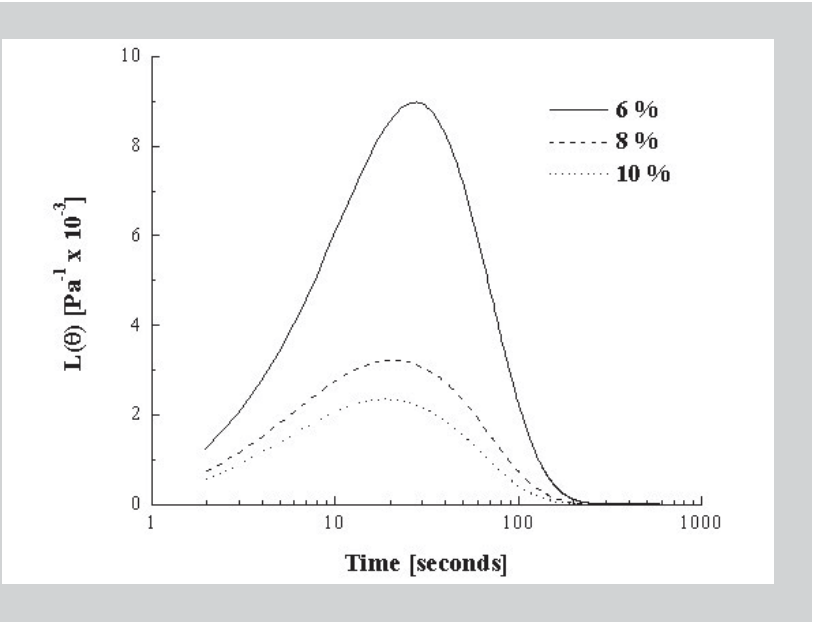


Figure 4: continuous retardation spectra for different bentonite concentrations (7, 8 and 10 wt%). Note that 5 and 6 wt% concentrations are not plotted here for ease of legibility of the graph. Values of maxima spectra are as follows: 75x10^{-3} Pa^{-1} (5 wt%) and 15x10^{-3} Pa^{-1} (6 wt%).

3.3 TRANSFORMATION OF CREEP DATA TO OSCILLATORY FUNCTIONS

An exhaustive study of clay suspensions may require the determination of their viscoelastic properties over a wide frequency (or time) scale. Unfortunately, the experiments are limited in time due to sedimentation and evaporation processes, and are inaccurate in the short time region because of instrumental inertia. However, the high-frequency (short time) range may be investigated by a dynamic method such as oscillatory while the long time range (low-frequency) is accessible by a transient method such as creep because a periodic experiment at frequency ω is qualitatively equivalent to a creep test at time $1/\omega$ ($t \Leftrightarrow 1/\omega$). Mathematical methods may then be used to unify data so as to describe the viscoelastic behavior over a sufficiently wide range of frequency (or time) to yield satisfactory determination of the spectrum [11]. That is, using only one type of response function, it was possible to complete the dynamic response in the low-frequency range (long time) using data from the transient response in creep. Creep compliance curves, analyzed by the discrete spectral method, may be employed to calculate storage and loss compliances (*J'* and *J''*) using the Fourier sine and cosine transformation. The exact interrelationships obtained are given by [27, 31, 32]:

$$J'(\omega) = J_o + \sum_{i=1}^{n} J_i \frac{1}{1 + \omega^2 \theta_i^2} \qquad (4)$$

$$J''(\omega) = \sum_{i=1}^{n} J_i \frac{\omega\theta_i}{1+\omega^2\theta_i^2} + \frac{t}{\eta_N} \quad (5)$$

Values for G' and G'' may be deduced using Eqs. 6 and 7 :

$$G' = \frac{J'}{J'^2 + J''^2} \quad (6)$$

$$G'' = \frac{J''}{J'^2 + J''^2} \quad (7)$$

The results in terms of variation of the storage modulus G' with frequency are illustrated in Fig. 5. The high frequency values are derived from oscillatory measurements with the Carri-Med rheometer (solid symbols) and the low frequency values are obtained by transforming creep data (open symbols). One may observe that the transformed dynamic functions compare well with the functions obtained directly from oscillatory measurements and that the observable frequency scale increases from two to four decades. This method was applied elsewhere in the case of other complex fluids like foams [34].

4 CONCLUSION

The viscoelastic properties were investigated in terms of creep flow and dynamic tests. It was found that the dispersions exhibit pronounced viscoelastic properties for concentrations higher than 6% by weight which can be represented by the mechanical Kelvin-Voigt model. It was necessary to combine transient and oscillatory measurements to determine the viscoelastic properties over a sufficiently wide frequency range: The dynamic response in the low frequency range (long time) was completed using data from the transient response in creep. The remarkable feature revealed by this method is that the transformed dynamic functions compare well with functions obtained directly from oscillatory measurements.

NOTATIONS:

G': Shear storage modulus [Pa]
G'': Shear loss modulus [Pa]
$f(t)$: Over-all creep compliance
$L(\theta)$: Retardation spectrum

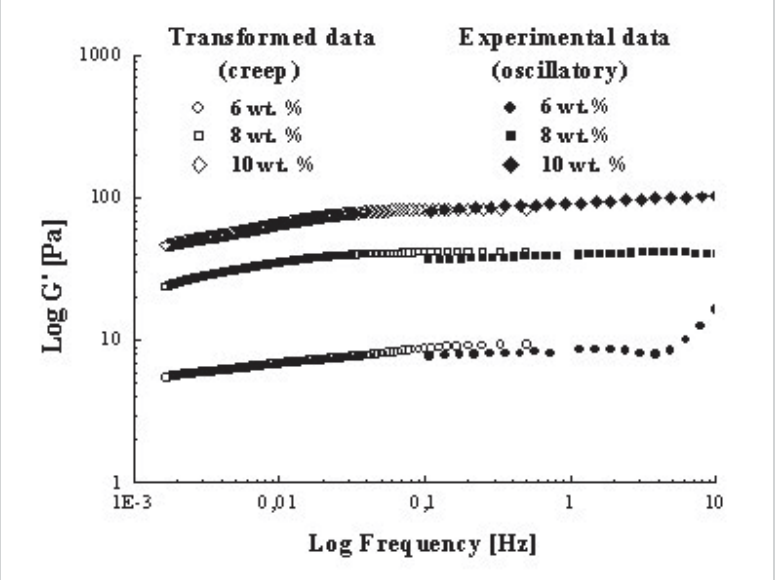


Figure 5: Unified plots of the variation of the storage modules G' with frequency for three bentonite suspensions (6, 8 and 10wt%). Low frequency values are derived by transforming creep data (open symbols), and high frequency values are measured by oscillatory measurements with a Carri-Med rheometer (solid symbols).

J_O: Instantaneous elastic compliance [Pa^{-1}]
J_i: Retarded elastic compliance of the i^{th} component [Pa^{-1}]
J': Storage compliance [Pa^{-1}]
J'': Loss compliance [Pa^{-1}]
t: Time [s]
δ: Phase angle between the stress and the strain (loss angle) [rad]
$\dot{\gamma}$: Shear rate [s^{-1}]
γ_O: Strain amplitude [-]
η_i: Viscosity of the i^{th} component [Pas]
η_N: Newtonian viscosity [Pas]
θ_i: Retardation time [s]
τ_O: Stress amplitude [Pa]
ω: Frequency of oscillation [Hz]

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