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Rheological study of concentrated dispersions. Application to the drilling fluid.

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Abstract. In order to understand the rheological behavior of concentrated aqueous Algerian bentonite dispersions of drilling (sodium bentonite of Mostaganem "m'zila"), rheological tests were carried out. By varying the concentration of bentonite, flow tests have allowed to estimate the yield stress and apparent viscosity for each concentration and to see their influence on the rheological behavior of these dispersions. In addition dynamic tests (oscillatory) are used to define the linear region of our samples, the state of our fluid (elastic solid or viscous liquid) and understanding the mechanisms of structuring of the particles constituting the material. In parallel, other tests coupled with rheological measurements such as x-rays diffraction to know the mineralogical composition and granulometry to estimate the bentonite particle size.

1. Introduction
During oil drilling, many problems can be encountered due to the interactions between the various geological formations crossed and the drilling fluid used; this latter is formulated on the surface by using various components where bentonite is the main one for water based mud. The composition of bentonite used and the study of its rheological behavior in dispersion in water represent a very important step to avoid problems during implementation.

The bentonite used in this study comes from Mostaganem (m'zila, west of Algeria). It currently indicates the name of smectites which consist essentially of montmorillonite where are found three levels of organization which are: the sheet, the primary particle and the aggregate [1]. The sheets are composed essentially of atoms of silicium, oxygen, aluminum and magnesium [2].

In the literature, little research has been carried out on understanding the mechanisms of structuring of bentonite dispersions often used in drilling fluids in Algerian oil fields. It is with this objective that this study contributes by studying these mechanisms through rheological tests coupled with other methods such as X-ray diffraction (XRD) and particle size to better understand and reinforce the results obtained by identifying the origin of the observed properties.

2. Materials and methods

2.1. Materials and equipment
The bentonite used is primarily made up of sodium montmorillonite. This type of clay consists of an assembly of aggregates with a crystalline structure. It belongs to the phyllosilicate family 2: 1, i.e. it is

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formed of an octahedral layer located between two tetrahedral layers [2-9]. The Si^{4+} ions are positioned in the tetrahedral layers while the octahedral layer contains Al^{3+} ions. Compensating cations (Na^+, Ca^{2+}) are often found between the sheets. These minerals are more sensitive to hydration which causes partial swelling, and consequently changes in rheological properties [2, 10].

All the rheological tests were carried out in an imposed stress rheometer (Physica MCR301) having a geometry of coaxial cylinders (Re=14.464 mm, Ri=13.325 mm, Re Ri=1.088, h=39.997 mm) and were maintained at a constant temperature at 25±0.1°C.

The mineralogical composition of bentonite was determined by x-rays diffraction. The measurement of bentonite particle size was made using a granulometry test in laser instrument (Malvern Mastersizer 2000).

2.2. Experimental protocol

Since the protocol of preparation of such suspensions has a great effect on the final state of the suspension and consequently, on its rheological behavior [3], the samples are prepared as follows: initially a quantity of given demineralised water is stirred, then a mass of bentonite of variable concentrations (3, 4, 5, 6 or 7%) are versed in water in way closely to avoid the formation of aggregates of clay particles. In order to homogenize the obtained system, the suspensions are left under magnetic agitation during 24h.

Before each rheological test, the suspensions are subjected to a pre-shear then a rest time in the measurement geometry to have the same structural state of reference. In the case of a long-term test (dynamic tests), the surface of the sample is covered with a film layer of paraffin oil to avoid evaporation problem.

The pH of our suspensions is checked systematically, it is between 9.5 and 10.5, which shows that the faces and edges of the clay particles are negatively charged, giving a first indication on the microstructure of our suspensions [11].

3. Results and Discussion

3.1. Rheological tests

3.1.1. Flow tests

This test consists of applying a stress along a rising ramp of 20s for each stage. It will inform us about the type of the rheological behavior of the fluid by estimating parameters such as the yield stress of flow, the dynamic viscosity of our sample, as well as the evolution of these parameters as a function of the bentonite concentration.

From the flow curves obtained (Fig. 1), it can be seen that our bentonite suspensions exhibit a non-Newtonian behavior, most often a shear thinning (pseudoplastic) behavior, as that is illustrated in figure 1. Similar results were obtained by [4].

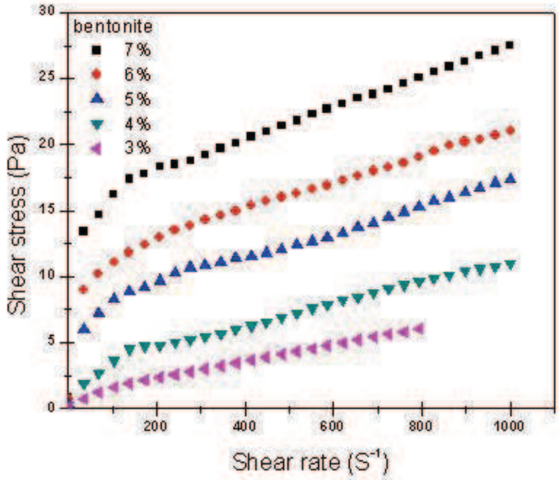

Figure 1. Rheograms of bentonite suspensions (3 to 7%) immediately after preparation (aged 24h).

The observed shear thinning behavior results in a decrease in the apparent viscosity of the suspensions with increasing the shear rate (Fig. 2) and this can be explained by the deformation or disaggregation of the suspended structural units under the effects of hydrodynamic forces.

All the suspensions have a yield stress which increases with the concentration (Fig. 3), below this yield stress the particles are linked together and the applied stress is insufficient to break these bonds and cause the flow of the sample which opposes resistance to the latter by its intact microstructure translating a solid behavior. As soon as this yield stress is exceeded, the sample is destroyed and there will be a flow, so it passes from a solid state to a liquid state.

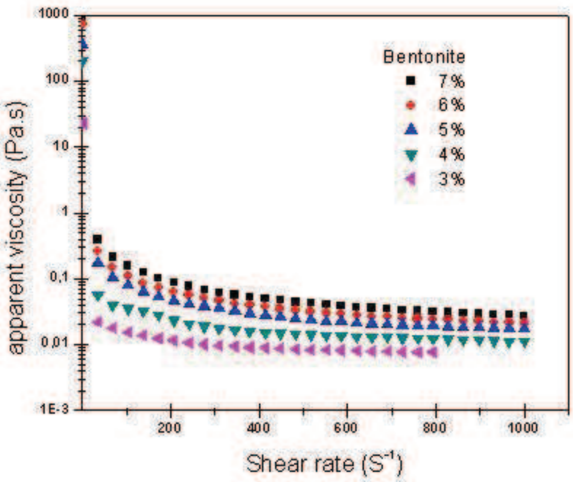

Figure 2. Variation of apparent viscosity as a function of shear rate of bentonite suspensions (3 to 7%).

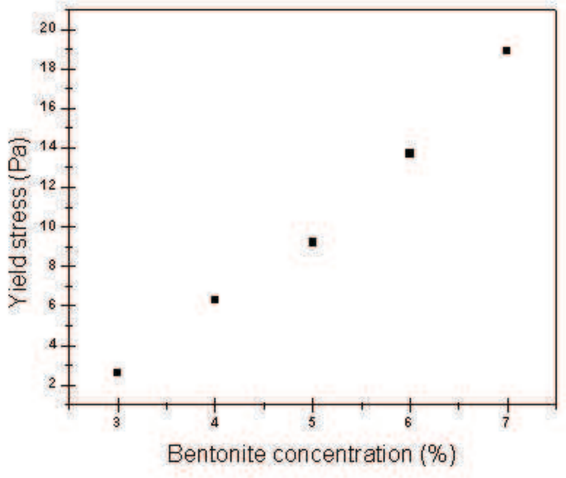

Figure 3. Variation of the yield stress of bentonite suspensions as a function of concentration.

Concerning the viscosity of the suspensions, it increases with increasing concentration (Fig. 4).

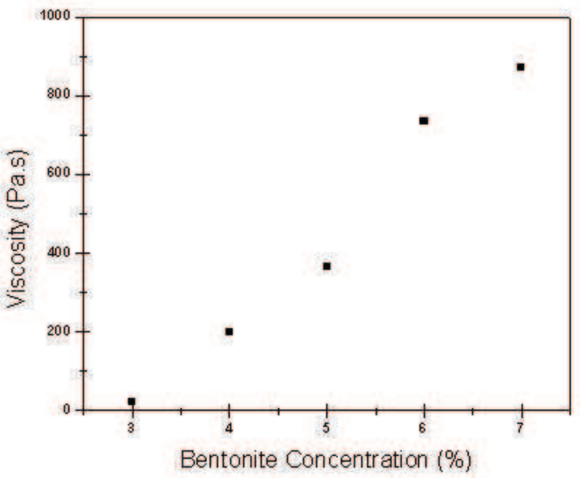

Figure 4. Variation of dynamic viscosity of bentonite suspensions as a function of concentration.

3.1.2. Dynamic tests
Rheological tests in dynamic regime consist in measuring the viscoelastic properties of given samples by studying the elastic G' and viscous G'' modulus as a function of stress, frequency and time.

a. Sweeping in stress
In order to know the linear viscoelasticity domain of the studied bentonite suspensions, i.e. measuring the evolution of the two modulus storage (G') and loss (G'') using a sweeping in stress for a given frequency (in our case 10 rad/s). The test consists in determining a critical stress beyond it there will be the rupture of the three-dimensional structure or of the network formed by the particles.

According to the Fig. 5 which represent the variation of G' and G'' as a function of stress at a frequency of 10 rad/s, it is noted that the values of the two modulus G' and G'' remain constant as long as the stress is less than a limiting value and beyond this value there is a collapse of the structure. That means the transition from the solid behavior to the liquid.

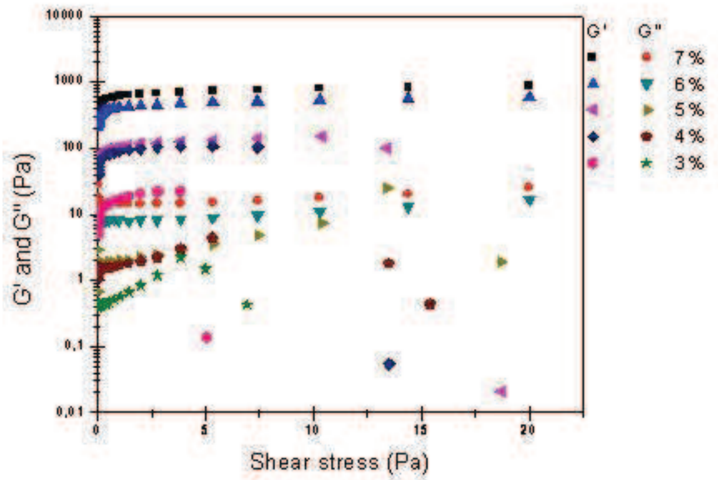

Figure 5. Evolution of viscoelastic modulus as a function of stress for different bentonite concentration.

b. Sweeping in frequency

Sweeping in frequency tests, which consist of measuring the evolution of the two modulus storage (G') and loss (G'') as a function of frequency, were carried out for various bentonite suspensions, choosing a frequency included between 0.01 and 100 rad/s and fixing the stress at a value belongs to the linear domain.

According to the results obtained (Fig. 6), it is noted that G' remains always higher than G'' for all the bentonite suspensions, this is a signature of an elastic solid behavior. In addition, the two viscoelastic moduli also increase according to the bentonite concentration. Similar results were obtained by other authors [5].

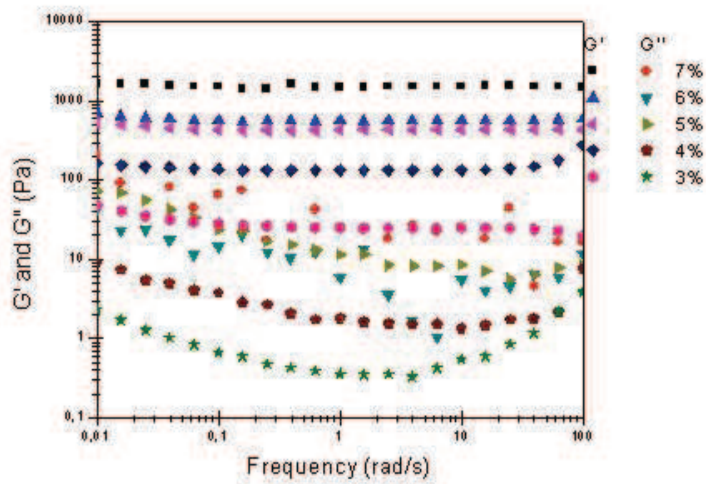

Figure 6. Evolution of viscoelastic modulus as a function of frequency for different bentonite concentration.

The results obtained in dynamic regime of Mostaganem bentonite confirm well those found previously in stationary regime. The behavior of these suspensions is an elastic solid even if for the weak mass concentrations (3%).

c. Sweeping in time (followed kinetic)
For better controlling the rheological properties of a material over time, firstly the structuring or the restructuring mechanisms of the particles and aggregates that make up this material must be understood [8]. These mechanisms were apprehended by a kinetic follow-up which consists in observing the evolution of the storage (G') and loss (G'') modulus by carrying out a sweeping in time. The results obtained for the concentrations (4, 5, 6 and 7%) are identical. Indeed, the suspensions present a behavior of a gel at the initial state (G' remains higher than G''). On the other hand for the suspensions 2, 2.5 and 3%, these present a time of gel approximately 76 min, 71min and 109 S respectively representing the moment where the crossing of the viscoelastic modulus is carried out. This time of gel physically represents the passage of the liquid state into a solid state and it decreases with increasing in bentonite concentration. For illustrating, just the figures 7, 8, 9 and 10 are represented which corresponding to the bentonite suspensions of 2, 2.5, 5 and 6%.

For the suspensions 4, 5, 6 and 7%, it can be noted that the loss modulus (G'') oscillates but tends to stabilize at an average value which is due to the acoustic resonance phenomenon [6, 8], while that the storage modulus evolves little and stabilizes. This shows that the structuring kinetics of these suspensions is very rapid. On the other hand, for dilute suspensions such as 2, 2.5 and 3%, the structuring kinetics is more significant and the liquid-solid transition can also be noticed. The authors Overlaz and Coussot [7] explained this effect through a percolation phenomenon: the initial increase of G'' indicates the progressive formation of solid aggregates in the fluid. When the volume fraction of the aggregates reaches a critical concentration, the aggregates then form a continuous solid network which is reinforced over time (inducing a sudden increase in G'). The material then becomes aging while G'' decreases.

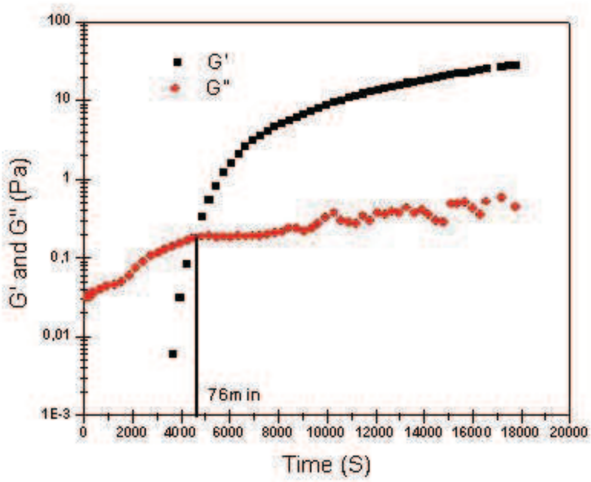

Figure 7. Variation of G' and G'' as function of time for the suspension of 2%.

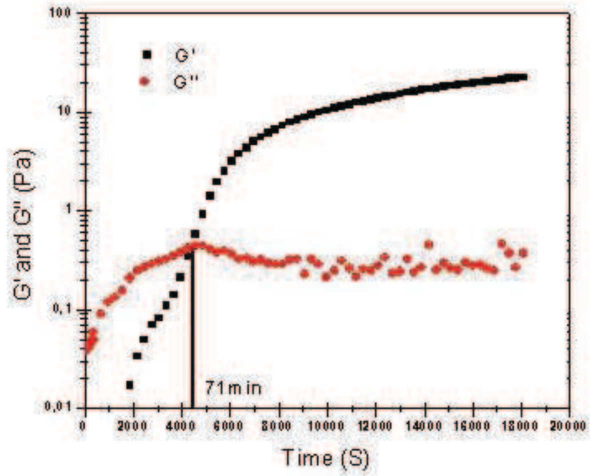

Figure 8. Variation of G' and G'' as function of time for the suspension of 2.5%.

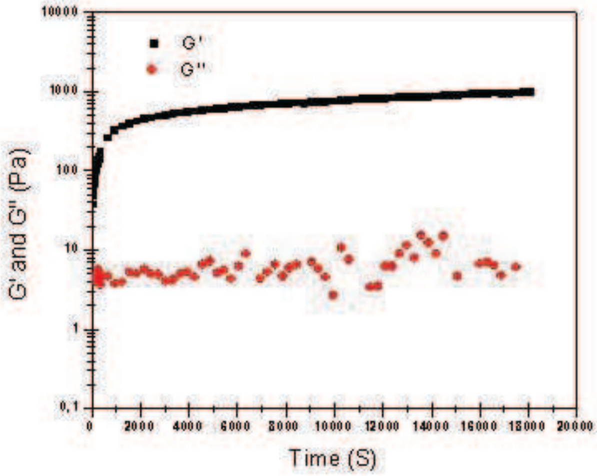

Figure 9. Variation of G' and G'' as function of time for the suspension of 5%.

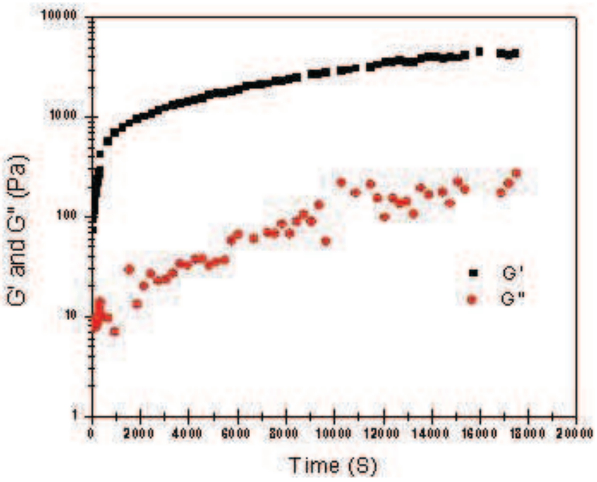

Figure 10. Variation of G' and G'' as function of time for the suspension of 6%.

3.2. Mineralogical analysis

In order to determine the various minerals constituting Mostaganem bentonite, a test of X-ray diffraction (DRX) is carried out.

The diffractogram (Fig. 11) was obtained by placing the sample of bentonite in the form of a powder in a sample holder. This figure shows several peaks in which the two principal ones correspond to montmorillonite which is the most encountered mineral in bentonite samples. The existence of other peaks shows that the sample is not pure and contains impurities which are: quartz at d = 4.16 Å, feldspath at d = 9.5 Å and d = 4.38 Å and calcite at d = 2.98 Å.

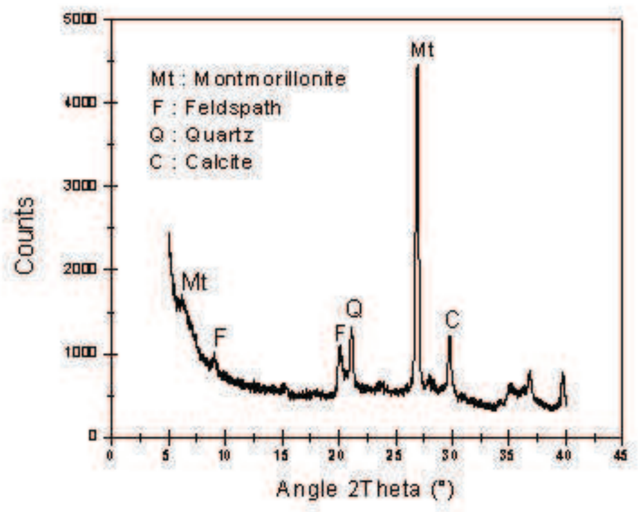

Figure 11. X-ray diffraction pattern of Mostaganem bentonite.

3.3. Granulometric analysis

The granulometric analysis of our bentonite illustrated in Fig. 12 revealed a particle distribution composed of 0.13% of sand (> 50 μm), 91.96% of silt (between 2 and 50 μm) and 7.86% of fine particles (< 2 μm).

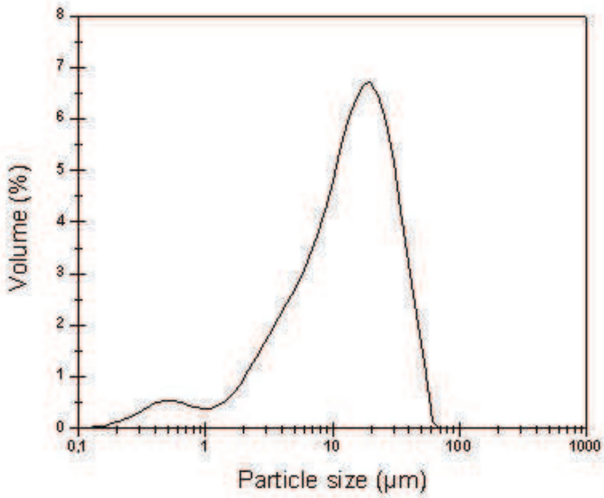

Figure 12. Granulometric distribution of Mostaganem bentonite particles (Algeria).

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4. Conclusion

The study of the rheological behavior of Mostaganem bentonite showed, on the one hand, that the behavior is shear thinning with a yield stress of flow, having a dynamic viscosity which increases as a function of the concentration of the clay particles and, on the other hand, The presence of the viscoelastic properties, i.e. the liquid / solid transition for the low concentrations and a gel state for the high concentrations. These results are in good agreement with the results of the mineralogy and the granulometry which show that montmorillonite is the main mineral constituting Mostaganem bentonite and that it contains less sand (0.13%) which gives it more important rheological properties.

5. References

[1] Le pluart L, Duchet J, Sautereau H, Halley P, Gerard J F 2004 *Applied clay science,* **219** 25
[2] Luckham P F, Rossi S 1999 *Colloid Interface Sci.* **82** 43
[3] Benchabane A 2006 Etude du comportement rhéologique de mélanges argiles - polymères. Effets de l'ajout de polymères, *Thèse de doctorat, Université Louis Pasteur de Strasbourg*
[4] Paumier S, Monnet P, Pantet A 2007 Etude des propriétés viscoélastiques de suspensions de smectite homoioniques à bi-ioniques (Ca^{2+}, Na^{+}) : influence de la concentration (2-100 g/l), *18ème Congrès Français de Mécanique, Grenoble*
[5] Bekkour K, Kherfellah N 2002 *Applied Rheology,* **12** 234
[6] Kaldasch J, Laven J, Stein H N 1998 *J. Rheol,* **42** (6) 1285
[7] Ovarlez G, Coussot P 2007 *Physical Review E.* **76** (1) 11406
[8] Gareche M 2013 Formulation et caractérisation rhéologique des suspensions de bentonite en présence d'additifs polymériques, *Thèse de doctorat, Université M'Hamed Bougara de Boumerdes*
[9] Grandjean J 1997 *J Colloid Interface Sci.* **185** 554
[10] Van Olphen H 1977 An introduction to clay colloid chemistry, *Willey,* New York
[11] Durán J D G, Ramos-Tejada M M, Gonzalez-Caballero F 2000 *J. Colloid Interface Sci.* **229** 107