Rheological Characterization of Oil Cement Suspensions

MELLAK Abderrahmane\textsuperscript{1} and AITOUCHE Moh-Amokrane\textsuperscript{2}

\textsuperscript{1}Prof. Dr in Faculty of Hydrocarbons and Chemistry, Boumerdes University, Algeria
Physics Laboratory of Petroleum Engineering. Department Mining and Oil Deposits.
\textsuperscript{2}Dr. Earth Physics Laboratory Geophysics Department
Faculty of Hydrocarbons and Chemistry. University of Boumerdes - 35000 – Algeria

Emails: mellakbder@yahoo.fr

Abstract: This study is a contribution to the study of the rheological behavior of cement suspensions. An oil well is drilled, cased, cemented and set completion. The well drilling is done in several phases then at various diameters to isolate the following problems like land fragile subsidence and poorly consolidated aquifer formations, loss of the movement in the porous and permeable formations. Therefore, it would go down a casing and cementing to work safely. The materials studied were chosen to satisfy the requirements and the problems encountered in real applications in the oil field (casing cementing wells). So it was used an oil hydraulic binder "G". This systematic study of rheological properties of cement Class "G" standardized API (American Petroleum Institute) deal with a formulation which is compatible with the surrounding environment taking account an optimal efficiency.

1. INTRODUCTION.
Cementing an oil well requires the establishment of slurry cement suitable, in the annulus between the borehole and the casing in place to provide a seal between various incompatible layers and between the casing and the geological formations. The cement slurry in water is as solid-liquid two-phases. It constitutes a system having a structure more or less complex depending on its composition parameters. The improved knowledge of these materials, in the physico-chemical and rheological areas is currently the subject of much research works in terms of fundamental subject research and in the field of applications [1, 2]. In order to provide a barrier between aquifer sand salt-bearing in oil wells and sometimes in deep water drills. The choice of grout must be compatible with the conditions inherent in the site. Therefore the knowledge and control of rheological properties are imperative. A physico-chemical study of these materials has been carried out previously [3] with a high use of hydraulic binder "G" which exhibits acquired properties needed for cementing oil. This work focuses on the rheological behavior of different grout in the liquid state.

2. MATERIALS USED

2.1 The hydraulic binder
The oil cement class "G", normalized by the API (American Petroleum Institute) was used. Its chemical composition is shown in Table 1. The hydraulic binder "G" is expected to reach great depths under compatibility after adding with various materials. It can be used with accelerators or retarders to cover a wide range of pressures and temperatures. This cement resists to sulfates [4].
Table 1: Chemical composition of cement "G" (in %).

|     | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO  | MgO  | SO₃  |
|-----|------|-------|-------|------|------|------|
|     | 21,30| 3,60  | 5,40  | 64,20| 0,90 | 2,20 |

Table 2 shows the average characteristics of the hydraulic binder "G".

Table 2: Characteristics of Cement "G".

| Specifications | Cement class « G » |
|----------------|--------------------|
| Particule size (µm) | 1 – 80             |
| Average diameter (µm) | 14                 |
| Specific surface (m².g⁻¹) | 0,3               |
| Real density (µm) | 3,18               |

2.2 Additions
The mechanical properties and the rheological behavior of the cement slurry can be modified by adding of various components and other additives. This includes:

1.2.1. Bentonite
Recall that the bentonite is a clay rock that has the ability to increase expand its volumes from 10 to 20 times by absorbing about 6 times its own mass of water causing swelling [5, 6]. Clay bentonite is used to reduce cement slurries to avoid fracturing of the low resistance formations.

1.2.2. Product Weighting
Baryte (BaSO₄) is the primary product used as weighting, with 4.20 as approximate average specific gravity. It is a low cost product. This inert product, also used in drilling mud, shall act through the mass effect to increase the density of cement slurries.

2.3 Mixing water
The mixing water used is a water having specific gravity 1.00. In the case of a "hard" water containing a lot of salt, magnesium and calcium, such as the oil fields of Hassi Messaoud (Algerian Sahara), it should be to precipitate the salts of calcium and magnesium by adding 1 to 2 kg NaOH per m³ of water to increase the performance of cement grout. Mixing the cement class "G" with freshwater in the proportions of 44% provides cement slurry with specific gravity of 1.85 [7].

2.4 Thinning
The main aim of the thinner is to reduce the shear threshold and the viscosity of the cement paste effect. It works by de floculating cement grains. This acid organic product is used in the proportion of 1% according to the weight of the dry cement.

2.5. Anti-foam
An anti-foam agent is added to prevent excessive foaming (micro bubbles) produced when mixing cement, adversely affecting the operation of the mud pumps. An antifoaming agent is incorporated in the proportion of 0.6% relatively to the weight of the cement and to the cement slurry.

2.6. Usual used cement
The cement injected into the formation depends essentially on the density to be compatible with the resistance of the injection formation. According to the API [7], there are currently used cement, slag light cement, (for to not fracture fragile formations) or heavy cement. This gives the following specific gravities summarized in the table 3
Table 3: Type of cement slurry

| Type                  | Specific Gravity | Addition                        |
|-----------------------|------------------|---------------------------------|
| Conventional cement   | 1.80 – 1.92      | Cement class « G »               |
| Light cement          | 1.40 – 1.80      | Cement class « G » + Bentonite   |
| Heavy cement          | 1.92 – 2.10      | Cement class « G » + Baryte      |

3. MATERIALS AND EXPERIMENTAL PROCEDURE

3.1 Equipment used
For the previous rheological studies, rotative rheometer is currently used with parallel plate geometry (Fig. 1) for the characterization of plastic fluids [8, 9]. The measurement geometry is easy to build and to align. It also has the advantage of having an adjustable sensitivity and it requires a very small volume of samples to be studied.

![Figure 1. Schematic representation of the measurement geometry.](image)

Table 4 shows the measurement geometry used and the main conditions under which these tests were conducted. All samples were studied in a geometry measurement plan/plan protected by an anti-evaporation chamber covering the experimental device to prevent loss of heat in the system.

Table 4: Measurement conditions of rheological parameters.

| Type of device                  | Carri Med CSL100 |
|---------------------------------|------------------|
| Value of the constraint         | 100 N/m²         |
| Geometry employed               | Plan/Plan        |
| Gap                             | 1200 µm          |
| Diameter                        | 4 cm             |
| Gradient deformation speed      | 0 à 750 s⁻¹      |
| Temperature                     | 20° C (±1°C)     |

3.2 Procedure:
The following procedure was applied for the preparation of all the case of cement slurry. Slow introduction of cement in the water, after mixing during two (2) minutes (by the use of a mechanical paddle and simultaneous introduction of other components (plasticizer, anti-foam agent, bentonite clay, barite according the slag to study). All the rheometry tests were performed under the same experimental conditions. For an admissible reliability of the measures, it is important that the fluid keeps its consistency throughout the duration of the test. In addition, the system must remain isothermal (changing the temperature may modify the behavior law), so a closed chamber saturated by water vapor covering the experimental device was used to prevent any evaporation. Temperature (20
°C±1 ° C) is controlled by Peltier effect involved in the system. These materials possess a strongly time-dependent behavior. It is important to obtain a state stationary. This protocol is used to measure the threshold for each grout, defined as the first value of the stress corresponding to a non-zero shear rate.

3.3. Rheological measures

The rheological behavior of the conventional grout was been widely studied then it is relatively well known [10-13]. The problem is fairly not the same with special cement grout. These materials are poorly studied because of their very complex structure and difficulties in their implementation (rapid change of state, rapid transition from slurry to a solid material).

3.3.1 Measures flow

The rheological characterization according to the density of the slag cement "G" was performed for class three grouts. They are the light cement (L1) with a density equals to 1.50, L2 as conventional slag cement with a density equals to 1.90 and heavy slag cement (L3) for a density equivalent to 2.00. The flow curves obtained are shown in Figures 3, 4 and 5. The rheological characterization of slag cement reduced L1 is characterized by the following flow chart (Fig. 2):

![Figure 2. Rheogram slag cement case L1 (density 1.50).](image)

The rheological behavior of slag cement L1, whose rheological equation is: $\tau = 0.512 + 0.284 \gamma^{0.72}$ can be described fully by the Herschel-Bulkley model [14] ($\tau = \tau_0 + K \gamma^n$), for a shearing speed from 0 to 700 s$^{-1}$.

Where:
- $\tau$: is the shear stress (Pa);
- $\tau_0$: the flow threshold (Pa);
- $\gamma$: the shear rate (s$^{-1}$);
- $n$: behavior index ;
- $k$: the consistency index (Pa.sn).

The rheological characterization of cement slurry currently used L2, for a density equals to 1.90 is illustrated by the following flow chart (Fig. 3).
Its rheological equation is $\tau = 0.265 + 2.227 \gamma^{0.47}$ and responds to the Herschel-Bulkley’s model in the shear range 0-240 s$^{-1}$ and greater than 240 s$^{-1}$ with the rheological equation $\tau = 12.27 + 0.082 \gamma$. It follows the Bingham’s model ($\tau = \tau_o + \mu_{pl} \gamma$) [15], here $\mu_{pl}$ defines the plastic viscosity (Pas).

The rheological characterization of the heavy slag cement L3 is characterized by the following flow chart (Fig. 4).

In the range of shearing speed greater than 35 s$^{-1}$, the rheological equation $\tau = 24.32 + 0.14 \gamma$ verifies the Bingham’s model ($\tau = \tau_o + \mu_{pl} \gamma$).

3.3.2. Discussion

The analysis of these curves shows that the rheological behavior of the studied materials can be represented adequately by either the Bingham’s model ($\tau = \tau_o + \mu_{pl} \gamma$), or by the Herschel-Bulkley’s model ($\tau = \tau_o + K\gamma$). There is an increase in viscosity with the increasing density. Cements L2 and L3 have a rheological behavior of Bingham type from a certain shear rate. In the range of low shear speed, the grout L1 and L2 satisfy the Herschel-Bulkley’s model while L3 have a grout rheological behavior undefined by the conventional models. In all cases, we can see that the behavior of the grout as a yield stress (first stress value associated with the existence of a non-zero shear rate) and is shear-thinning ($n < 1$). Then we can argue that the viscosity decreases with the shear rate. This behavior could be explained by the structure of such complex materials. The obtained structure will be resistant to flow at low shear rates, justifying therefore the presence of a threshold stress. When the applied stress exceeds the threshold of stress, the initial structure begins to take another shape and induces a flow. After the destruction of solid aggregates, part of the liquid phase trapped initially within these clusters of particles is released implying an increase in the liquid phase and thus a reduction of the inter-particle forces. This explains the shear thinning nature of these materials. At high shear rates, the flow fully established explains the linear behavior (Bingham-type) of the fluid and thus the constant nature of the plastic viscosity [16].
4. CONCLUSION
The experimental results show that the used materials highlight that the viscosity and the threshold stress are greater if the concentration is important and inversely. The comparison with conventional rheological models showed that, in general, that Herschel-Bulkley’s models and Bingham’s present a better correlation compared to the experimental results. It would be interesting to focus the future works on a structural study to discover the microscopic material factors which are the cause of this behavior, and to provide, by anticipation, the rheological behavior of these materials. In-situ tests, taking into account the actual conditions of the deep of the injection medium (mainly temperature and pressure) are needed to develop appropriate methodologies to achieve better control of the use of these materials required in cementing casings of oil drilling.

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