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# Novel aspects of Bentonite assessment

## Nouveaux aspects pour l'évaluation des Bentonites

J.Kinsky – *Geotechnical Engineer, Haifa, Israel*

**ABSTRACT:** The behavior of bentonite slurries represents the determining factor in the attenuation of the negative effect of cake formation. We tested 12 different samples of bentonite powder, in suspensions of 6%, or, in 2% suspensions with additives of 0.2% polymers, prepared with tap or distilled water. The obtained cakes, on filter paper or rock samples, were tested for ease of removal with solutions of hexametaphosphate, hydrogen peroxide and enzymes, respectively. The results lead to the following recommendations: to include the consistency and power indexes into the technical specifications for bentonite assessment and to use support fluids with low solid contents, together with a polymer additive. Cleaning of the cakes was efficient with hexametaphosphate + hydrogen peroxide solutions. For suspensions with low solid contents the use of enzymes in the cleaning operation was successful.

**RESUME:** Le comportement des suspensions de bentonites constitue le facteur déterminant pour l'atténuation de l'effet négatif de la formation des tourteaux. Nous avons étudié 12 échantillons différents de bentonites, en suspension de 6% ou en suspension de 2% avec des additifs 0.2% polymères préparés avec de l'eau courante ou distillée. Nous avons examiné la facilité de détachement des tourteaux formés, sur papier filtre ou bien sur des échantillons de rocher, en utilisant des solutions de hexametaphosphate, de l'eau oxygénée ou des enzymes. Nous recommandons de tenir compte de l'indice de consistance et de l'indice de puissance dans les spécifications techniques concernant l'évaluation des bentonites et l'utilisation des suspensions avec un bas contenu de solides et avec des polymères. Le détachement des tourteaux est produit facilement avec l'hexametaphosphate et l'eau oxygénée pour les suspensions de 6%, et avec les enzymes pour les suspensions avec des polymères.

## 1 INTRODUCTION

The support fluids are usually suspensions of bentonite in water with or without additives (polymers) and are generally used for well drilling, slurry walls, impermeable diaphragm walls and in situ casting of piles.

These suspensions have to meet the required technical specifications. A number of agencies and authors have made recommendations regarding the properties of support fluids, (Edil, 1992, Medlin 1986). In civil engineering the recommendations of the Federation of Piling Specialists (FPS) are generally accepted. However, in practice suspensions with properties meeting the requirements are sometimes unsatisfactory: in some wells walls collapse, or difficulties arise at cleaning and cake removal. The purpose of this paper is to discuss ways for a more reliable assessment of the support fluids and a search towards a new approach for cleaning of boreholes. Therefore 12 bentonite samples were tested to find relevant differences even though their characteristics were similar satisfying standard requirements. In parallel the ease of removal of the formed cakes was tested using different cleaning solutions.

In order to succeed in field work the nature of the strata and of the support fluids has to be considered. In order to work with thinner cakes for the assessment of cake removal, low solid suspensions together with polymer additives were tested.

The main factors that affect the deposition of fine particles in a porous medium leading to the formation of a cake on the wall of the borehole, are:

- (1) The Geometry of the system: strata – i.e. particle and pore size distribution and borehole – diameter, depth;
- (2) Chemical & physical characteristics of the system: suspension – pH, concentration, mineralogical composition of the bentonite; fluid loss, exchangeable cations; strata – chemical composition, chemical reactions, pressure;

(3) Rheological characteristics of the suspensions – viscosity, thixotropy and gel strength.

For any given field case the geometry and a great part of the chemical and physical characteristics are predetermined. Therefore our study was aimed at the rheological and some of the chemical and physical characteristics of the suspensions. Accordingly, we attempted to find a set of characteristics that would allow a straightforward assessment of support fluids under usually encountered conditions.

The process of cake formation and clogging of productive strata was studied by many researchers (Gruesbeck, 1988; Majano & al. 1993; Hofsaess 1995). The most common model, the hyperbolic, is used in the API 13A specification: this implies a linear relationship between the volume of leak and the square root of time. The second model, the parabolic, separates the process into two parts: a) an initial invasive phase and b) the subsequent wall building phase. The mathematical expression describing the latter model can be compared with the equation of a conical section. The coefficients of the equation reflect the influence of rheological parameters ( $k$ , consistency index;  $n$ , the power index, etc). A third, probabilistic model is based on the solid concentration at a given point and the deposition of particles in the porous medium. Particles trapped between pore openings form the internal filter cake. Once the formation of the internal cake is completed, the external cake starts to build up.

## 2 EXPERIMENTAL

We tested 12 samples of different bentonite powders, for characteristic behavior and cake removal.

### 2.1 Methods

**Suspension preparation:** The bentonite powder was slowly poured into a high speed mixer containing tap water or

distilled water, then stirred for 2 hours at 17000 rpm. The suspensions were tested after a swelling time of minimum 24 hours at room temperature followed by high speed mixing for 15 minutes. The concentration of the suspensions was 6% or 2%.

**2.2 Tests:**

The rheological characteristics were determined with a Fann type rotative viscometer (8 speeds). The readings are expressed in centiPoise (cP), and represent the basic values used furtheron. We define the thixotropy increase ratio (TIR) as the ratio between the gel strength, in N/m<sup>2</sup>, as determined after 10 seconds and again after 10 minutes.

The following physical and chemical characteristics were determined:

Stability: this is the amount of water separated after 24 h, expressed as the percentage by volume of the remaining suspension.

Water loss: the filtrate obtained from the suspension in a Baroid type pressure cell, under 7 bars in 30 min., expressed in ml. (if not mentioned otherwise).

pH: as determined with a pHmeter,

calcium: estimated by means of hardness indicator paper.

Cake thickness: as obtained on a standard filter paper in a Baroid type pressure cell, expressed in mm. In some tests we used sandstone, chalk or Nubian sand cores instead of filter paper. Since there were no obvious differences between the tests run on filter paper and with the cores, the results regarding the latter tests are omitted from the tables.

The ease of cake removal from the filter paper was determined by introducing the obtained cake together with the filter paper into a cleaning solution in a Berzelius flask.

The detachment of the cake from the filter paper was assessed visually at different time intervals and recorded. If the filter paper remained totally clean we recorded (+++), if the cake did not detached at all we assigned (-). Several intermediary degrees of removal are represented by (++) , (+) , (±).

**2.3 Materials**

Bentonite: we used 12 different samples, numbered from 1 to 12, obtained from several manufacturers.

Water: Tap water or distilled water were used for the preparation of the bentonite suspensions.

Additives: Two types of polymers were used as additives - anionic high molecular weight polyacrylamidic and polyanionic cellulosic. The suspensions with polymer additives had a low solid content, (2% bentonite and 0.2% additive, if not otherwise mentioned). We used the following polymers: CELPOL R , FINPOL 215, DF25, STAFLO , ANTISOL 1000 and 30000 , SUPERMUD, designated in the tables as PC1, PC2, PC3, PA1, PA2, PA3, PA4 respectively. Cleaning materials: sodium hexametaphosphate (calgon) 7% solution (by weight), hydrogen peroxide 5% and 10 % solutions, calgon 7%+ H2O2 5%, sodium peroxide 5%, enzymes "10" or "11" or "19", 5% and calgon 2% + enzymes 5%.

**3 RESULTS**

The results are analysed by comparing at first the behavior of different bentonite suspensions among themselves, and then with similar low-solid suspensions, prepared under the same

Table 1 Comparison between bentonite suspensions prepared with tap water (TW) or distilled water (DW)

| Bentonite Sample #                     | with        | 1     | 1     | 4     | 4     | 5     | 5     | 7     | 7     | 8     | 8     |
|--|-------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|  |             | TW    | DW    | TW    | DW    | TW    | DW    | TW    | DW    | TW    | DW    |
| Tests                                  | Spec. *     |       |       |       |       |       |       |       |       |       |       |
| Density g/ml                           | <1.1        | 1.04  | 1.04  | 1.04  | 1.03  | 1.05  | 1.04  | 1.03  | 1.03  | 1.05  | 1.05  |
| Apparent viscosity cP                  |             | 15.00 | 19.00 | 17.00 | 18.50 | 17.50 | 19.00 | 16.00 | 18.00 | 16.00 | 19.00 |
| Plastic viscosity cP                   |             | 6.00  | 8.00  | 7.00  | 6.00  | 9.00  | 8.00  | 8.00  | 9.00  | 15.00 | 11.00 |
| Yield point N/m <sup>2</sup>           |             | 20.00 | 22.00 | 23.00 | 33.00 | 21.00 | 26.00 | 15.00 | 18.00 | 28.00 | 25.00 |
| Rheologic power n                      |             | 0.12  | 0.13  | 0.12  | 0.16  | 0.19  | 0.21  | 0.31  | 1.85  | 0.20  | 1.91  |
| Consistency index k                    |             | 6.73  | 1.11  | 10.67 | 1.25  | 9.61  | 1.37  | 5.31  | 0.92  | 1.57  | 0.73  |
| Gel strength N/m <sup>2</sup> , 10 s   |             | 10.00 | 27.00 | 17.00 | 15.00 | 11.00 | 9.00  | 12.00 | 18.00 | 8.00  | 9.00  |
| Gel strength N/m <sup>2</sup> , 10 min | 4 to 40     | 20.00 | 37.00 | 23.00 | 20.00 | 22.00 | 18.00 | 18.00 | 21.00 | 14.00 | 15.00 |
| TIR                                    |             | 2.00  | 1.37  | 4.35  | 1.33  | 2.00  | 2.00  | 1.50  | 1.16  | 1.75  | 1.66  |
| Water loss 30 min. ml                  | <40 **)     | 14.00 | 14.00 | 15.00 | 13.00 | 13.00 | 12.00 | 15.00 | 12.00 | 17.00 | 16.00 |
| Cake thickness mm                      |             | 1.10  | 1.20  | 1.10  | 1.00  | 1.10  | 1.10  | 1.20  | 1.00  | 1.80  | 1.80  |
| Marsh visc. S                          | 30 to 70    | 47.00 | 56.00 | 48.00 | 58.00 | 46.00 | 55.00 | 47.00 | 58.00 | 40.00 | 52.00 |
| PH                                     | 9.5 to 10.8 | 9.00  | 8.50  | 9.00  | 9.00  | 9.00  | 8.50  | 9.50  | 9.00  | 8.50  | 8.50  |

Visual assessment of cake removal with solutions of:

|            | 1  | 1  | 4  | 4  | 5  | 5  | 7  | 7  | 8  | 8  |
|------------|----|----|----|----|----|----|----|----|----|----|
|            | TW | DW | TW | DW | TW | DW | TW | DW | TW | DW |
| Calgon (C) | +  | +  | ++ | +  | +  | +  | +  | +  | +  | +  |
| Enzyme (E) | -  | -  | -  | -  | -  | -  | -  | -  | -  | -  |
| H2O2       | ++ | +  | +  | +  | ++ | +  | +  | +  | ++ | +  |
| C+ H2O2    | +  | +  | ++ | +  | +  | +  | +  | +  | +  | +  |
| C+Enzyme   | +  | ±  | +  | +  | ±  | ±  | +  | ±  | +  | ±  |
| H2O2+ E    | ++ | +  | +  | +  | ++ | +  | +  | +  | ++ | +  |

\*) Spec. = Federation of Piling Specialists (FPS) specification 1996, in collaboration with The Institution of Civil Engineering, The Highways Agency and Ove Arup & Partners

\*\*\*) More stringent specifications (API) recommend ? 15

conditions (tap water and distilled water). Subsequently the influence of the fluid medium on the behavior of a bentonite is considered. The performances of different kinds of materials used for cake detachment were also assessed. Regarding the behavior of the different bentonite suspensions in tap water (TW) or distilled water (DW), as presented in Table 1, we found:

All the suspensions meet the requirements of the FPS specifications. As mentioned, it happens sometimes, that despite the fact that the bentonite complies with the requirements, in practice, walls collapse, bentonite is included in the piles, damaged formation, etc.

**Rheological characteristics:** The Marsh viscosity values are close for all but one sample: (in TW 46-48 s. and for DW 55-58 s.). The apparent viscosity values are close for all samples in TW (15-17 cP) and for 1, 4 in DW (18.5-19 cP). The plastic viscosity values for samples 1, 4 in both TW and DW are close (6-8 cP) but is high for sample 8 (>11 cP). The yield point values are close for samples 1, 4, 5 in TW (20-23 N/m<sup>2</sup>) but in DW they are different (sample 1: 22, sample 4: 33 N/m<sup>2</sup>). The water loss values were similar for samples 1, 4, 5, 7, (12 – 15 ml) in both TW and DW. The respective values for cake thickness were similar (0.9-1.1 mm). The water loss and cake thickness values shared little variation and so their tests is a poor indicators. The n and k values, are calculated from the rheological equation (Technip. 1979)  $y = \log k + nx$  where  $y = \log \tau$ ,  $x = \log \dot{\gamma}$  ( $\tau$ =shear stress,  $\dot{\gamma}$ =deformation rate). The n values are very close for all the suspensions in TW, but the DW values for samples 7,8 are significantly higher, 0.31, almost tenfold than for samples 1, 4, 5. The k values are lower for samples 7, 8 in both TW and DW than the corresponding values for samples 1, 4, and 5, and the differences between the TW and DW values for both 7 and 8 are smaller than the corresponding differences for suspensions 1, 4 and 5. The above remarks regarding k and n

enable to distinguish between two groups of bentonite: (a) 1, 4, 5 and similar, and (b) 7, 8 and similar.

The detachment of the cake was examined as a function of solution type. Cakes formed from tap water suspensions could be efficiently removed by hydrogen peroxide (10%) by itself or together with hexametaphosphate (calgon). The efficiency of hexametaphosphate alone is limited and depends on the bentonite type used in the suspensions. Bentonite samples 1, 4 and 7 respond well to the majority of the cleaning solutions used. The enzymes alone were found ineffective.

The results of the tests for suspensions with low solid contents and additives prepared with tap water and distilled water are summarized in Table 2.

The density of all the low-solid suspensions is about 1 g/ml. The Marsh viscosity of these suspensions is between 29 and 31 s., practically the same. pH values are close to neutral, only the suspension PA4 is markedly basic.

The rheological characteristics and stability tests of the suspensions with cellulosic polymers (PC) are significantly different from those of the suspensions with acrylamide polymers (PA). The PC3 cellulosic suspension is an exception, the molecular weight of the polymer is half that of the other cellulosic polymers tested.

The apparent viscosity is ~22 cP for PC polymer and half that for the PA polymer. The same tendency is observed for plastic viscosity and yield point (13 vs. 6, 7 cP; 21 vs. 10, 12 N/m<sup>2</sup>). PC3 is again exceptional in these tests.

It is important to mention that the differentiation of the two types of polymers, without exception, can be observed for consistency index and rheological power values (n= ~ 0.4 for cellulosic and ~ 0.5 for acrylamidic, k = ~ 3 for cellulosic and ~1 for acrylamidic).

The water loss and cake thickness have close values for the

Table 2. Bentonite suspension 4 with low solid (LS) content prepared with tap water or distilled water  
Polymers added (0.2%) to the suspensions:

| LS with polymer<br>Tests               | PC1   | PC1   | PA1   | PA1   | PC3   | PA3   | PA4   | PA4   |
|--|-------|-------|-------|-------|-------|-------|-------|-------|
|  | TW    | DW    | TW    | DW    | TW    | TW    | TW    | DW    |
| Density g/ml                           | 1.00  | 1.00  | 1.00  | 1.00  | 1.01  | 1.00  | 1.02  | 1.00  |
| Apparent viscosity cP                  | 22.00 | 23.00 | 14.00 | 10.50 | 16.00 | 10.50 | 12.00 | 11.00 |
| Plastic viscosity cP                   | 12.00 | 13.00 | 8.00  | 7.00  | 14.00 | 10.00 | 6.00  | 6.00  |
| Yield point N/m <sup>2</sup>           | 20.00 | 21.00 | 14.00 | 11.00 | 4.00  | 12.00 | 10.00 | 12.00 |
| Rheologic power n                      | 0.44  | 0.47  | 0.59  | 0.55  | 0.41  | 0.55  | 0.53  | 0.61  |
| Consistency index k                    | 3.26  | 2.81  | 1.42  | 1.14  | 1.24  | 1.37  | 1.42  | 1.46  |
| Gel strength N/m <sup>2</sup> , 10 s   | 10.00 | 4.00  | 4.00  | 2.00  | 5.00  | 4.00  | 2.00  | 1.00  |
| Gel strength N/m <sup>2</sup> , 10 min | 23.00 | 18.00 | 8.00  | 9.00  | 8.00  | 17.00 | 8.00  | 11.00 |
| TIR                                    | 2.30  | 4.50  | 2.00  | 4.50  | 1.60  | 4.20  | 4.00  | 11.00 |
| Water loss 30 min. ml                  | 13.00 | 11.00 | 13.00 | 11.00 | 15.00 | 13.00 | 14.00 | 14.00 |
| Cake thickness mm                      | 0.20  | 0.10  | 0.30  | 0.40  | 0.50  | 0.30  | 0.40  | 0.40  |
| Marsh visc. S                          | 32.00 | 29.00 | 31.00 | 30.00 | 29.00 | 30.00 | 31.00 | 31.00 |
| PH                                     | 8.00  | 7.00  | 8.00  | 7.00  | 8.00  | 8.00  | 8.50  | 9.00  |

Visual assessment of cake removal with solution of:

|            | PC1 | PC1 | PA1 | PA1 | PC3 | PA3 | PA4 | PA4 |
|------------|-----|-----|-----|-----|-----|-----|-----|-----|
|            | TW  | DW  | TW  | DW  | TW  | TW  | TW  | DW  |
| Calgon (C) | ±   | -   | -   | -   | -   | -   | -   | ±   |
| Enzyme (E) | +++ | +++ | ++  | +++ | ++  | ++  | ++  | +   |
| H2O2       | ++  | ++  | ++  | ++  | ++  | ++  | +   | +   |
| C+ H2O2    | ++  | +++ | ++  | +++ | ++  | ++  | ++  | ++  |
| C+Enzyme   | +++ | +++ | +++ | +++ | ++  | ++  | ++  | ++  |
| H2O2+ E    | +++ | +++ | +++ | +++ | ++  | ++  | ++  | ++  |

two types of polymers. The cakes are thin ( $\sim 0.2$ ) and homogeneous.

The detachment of the formed cake was more easily realized than with the suspensions with bentonite alone. The detachment time was found to be half that of the bentonite suspensions themselves for low solid suspensions with polymer.

The hydrogen peroxide and the enzyme solutions by themselves or together with calgon are efficient for the removal of cakes from the filter paper or core, regardless of the polymer type. The hexametefosphate was found inefficient

#### 4 CONCLUSIONS

From the above data and discussion the following conclusions were reached:

The consistency index "k" and rheological power value "n" allow a substantial differentiation between different samples of bentonite.

Bentonite suspensions prepared with distilled water exhibited better rheological characteristics than their analogs prepared with tap water. In the suspensions prepared with tap water, Bentonite suspensions prepared with distilled water exhibited better rheological characteristics than their analogs prepared with tap water. In the suspensions prepared with tap water, the Na from the bentonite is at least partly exchanged by Ca ions affecting the characteristics of the suspensions. Low solid suspensions prepared either with distilled or tap water with additives have very similar properties. This indicates that the polymer forms a protective layer on the bentonite particles concealing differences between the bentonites, and resulting in easier work in practice. The 5% hydrogen peroxide solutions were found efficient for the removal of the cakes formed from low solid or bentonite suspensions, but for the latter case they worked slowly.

Cakes originating from bentonite suspensions could be easily removed with hydrogen peroxide combined with hexametefosphate.

The enzymes were found effective only for the low solid suspensions. The enzymes promote degradation of the polymers.

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