

## The Particle Flow Interaction Theory

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## ABSTRACT

In this work, a new material model is presented to simulate the rheological behavior of cement suspension. This material model is among others based on combined concepts by Hattori and Izumi and by Tattersall and Banfill. More precisely, coagulation, dispersion and re-coagulation of the cement particles (giving a true thixotropic behavior) in combination with the breaking of certain chemically formed linkages between the particles (giving a so-called structural breakdown behavior) are assumed to play an important role in generating the overall time-dependent behavior of the cement suspension. The model evaluation is done by comparing experimental data with model prediction.

## INTRODUCTION

The new model introduced here is designed for suspension of cement particles in water. It is based on thixotropic behavior as well as on a certain process called “structural breakdown”. The interest of the former phenomena is nearly as old as modern rheology<sup>1</sup>. The term thixotropy was originally coined to describe an isothermal reversible gel–sol (i.e. solid–liquid) transition due to mechanical agitation<sup>1</sup>. There is a comprehensive review article about thixotropic material done by Mewis<sup>1</sup> and Barnes<sup>2</sup>. Also, a shorter review is given in Mujumdar et al.<sup>3</sup> and in a textbook by Tanner and Walters<sup>4</sup>.

The term “structural breakdown” was made by Tattersall<sup>5</sup> in 1954. Because no

recovery in torque was measured in the corresponding experiment, structural breakdown was considered to be a different phenomenon than thixotropic behavior. The mechanism of structural breakdown was explained in 1983 in a textbook by Tattersall and Banfill<sup>6</sup>. There, it was attributed to the process of breaking certain linkages between the cement particles, which were assumed to be formed by the early chemical reactions of cement particles with water. The breaking of linkages was considered to be an irreversible process and thus non–thixotropic.

In the earlier work of Wallevik<sup>7,8</sup>, the use of a certain flocculation parameter was used, named “coagulated state”. This particular model was not based on the structural breakdown phenomenon, but rather only on thixotropic behavior. More precisely, it was based on several ideas proposed by Hattori and Izumi<sup>9</sup> and is called the *Modified Hattori–Izumi theory* (or the MHI–theory). However, with a purely thixotropic characteristic, the MHI–theory was only partly successful. The new model presented here, named the *Particle Flow Interaction theory* (or the PFI–theory), will contain both the structural breakdown phenomenon as well as thixotropic behavior. With these two phenomenons simultaneously present, a better outcome is attained.

The viscometer used in the experiment is the ConTec Viscometer 4<sup>10</sup>. As shown in Fig. 1, it is a coaxial cylinders viscometer that has a stationary inner cylinder (of

radius  $R_i = 85$  mm) that measures torque  $T$ , and a rotating outer cylinder (of radius  $R_o = 101$  mm). The angular velocity  $\omega_o$  of the outer cylinder during a rheological test is shown in Fig. 2. The height of the inner cylinder is  $h = 116$  mm. Shearing from the bottom part of the viscometer is filtered out by a special means<sup>11</sup>. Fig. 1 shows a numerical calculated flow inside the viscometer in question.

In this manuscript, the analysis of rheological behavior consists of measuring torque  $T = T(t)$  as a function of time  $t$ , under complicated angular velocity conditions  $\omega_o = \omega_o(t)$  shown in Fig. 2. The challenge is to create a material model that is based on the microstructural approach and can simulate the measured rheological behavior of the cement suspension under arbitrary and complex shear rate conditions. With a suitable apparent viscosity function  $\eta = \eta(\dot{\gamma}, \dots)$  used in the numerical simulation, the computed torque  $T_c$  should be able to overlap the measured torque  $T$ .

## EXPERIMENTAL

The water–cement ratio used for the cement suspension is  $w/c = 0.3$ . The amount of superplasticizer is 0.5% solids by weight of cement (sbwc). The initial phase volume of the cement suspension is  $\Phi = 0.52$  and it is always increasing due to the chemical reactions of the cement particles with water.

The Hobart AE120 mixer is used when mixing the cement suspension. It has three speed settings 1, 2 and 3. The fact that the test sample consisted of only pure cement suspension, resulted in a reduced reproducibility (here, *reproducibility = reproduction of rheological result of different batches, of the same mix-design*). After testing different mixing procedures to seek a better reproducibility, the following one was selected:

1. *Between 0 and 3 minutes:* Mixing of cement and water at speed 1. Most of the cement particles are more or less

moistened within the first 60 seconds.

2. *Between 3 and 6 minutes:* Hand mixing and resting.
3. *Between 6 and 10 minutes:* Mixing at speed 2.
4. *Between 10 and 11 minutes:* Check with hand mixing, if the cement suspension is homogeneous.

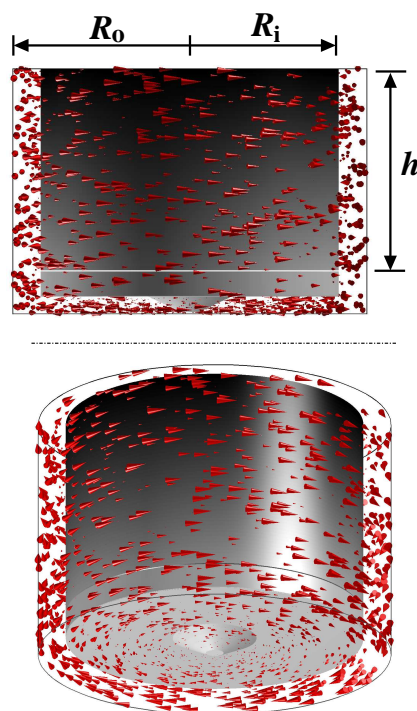


Figure 1. Computed flow inside the ConTec Viscometer 4.

At 12, 42, 72 and 102 minutes after the initial water addition, a rheological measurement with the ConTec Viscometer 4 is performed. Immediately after each measurement, a remixing by hand is done to ensure homogeneous mixture. A complete rest applies for the test material prior to any data logging in the coming measurement. The resting consisted of about 29 minutes and was considered to be sufficient for the test sample to gain a large coagulated- and linked state at the start of the next measurement. No remixing with the Hobart mixer was applied between measurements. Since the objectives

is to investigate thixotropic- and structural breakdown behavior of the cement suspension, it would be pointless to use the Hobart mixer to disperse the coagulated and linked cement particles and brake up the structure that the viscometer is supposed to measure.

As previously mentioned, a rheological measurement is made at 12, 42, 72 and 102 minutes. However, in the current article, only one of these four results is shown, analyzed and discussed. This is the measurement conducted at 72 minutes after water addition (i.e. 72 minutes after start of early chemical reactions of the cement particles with water).

The angular velocity  $\omega_o$  of the outer cylinder  $R_o$  (see Fig. 1) applied in the experiment and also used in the numerical simulation, is shown in Fig. 2. Single rheological test is made with the ConTec Viscometer 4 and begins at the (experimental) time  $t = 0$  and ends at  $t = 50$ s. This time variable is not to be confused with the time duration from mixing of water and cement (inside the Hobart mixer). The former time period spans only 50 seconds as shown in Fig. 2, while the latter spans over the whole test procedure of 102 minutes.

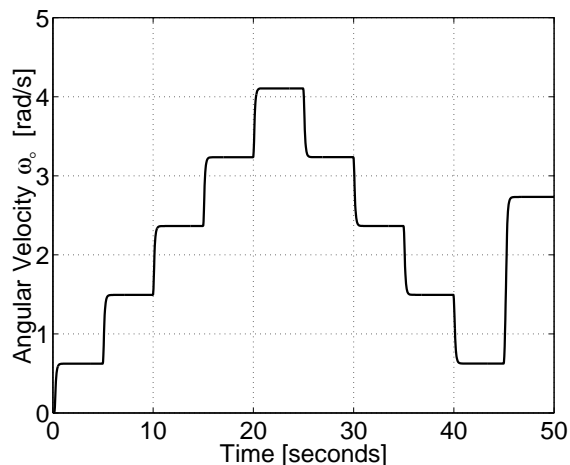


Figure 2. Angular velocity  $\omega_o$  as a function of time  $t$ .

The measured torque response attained by the test setup shown in Fig. 2 is given in Figs. 4 and 5 (apply after 72 minutes

after water addition). The difference between the two results is in the type of superplasticizer mixed with each suspension (the quantity in each case is 0.5% sbwc). These are designated as VHMW Na and HMW Na, respectively. These are very high- and high molecular weight Na-lignosulfonates, respectively and are natural polymers formed from the pulping process and are produced by Borregaard LignoTech, Norway. For further information about the overall test setup, see the author's previous work<sup>8</sup>.

## THE PARTICLE FLOW INTERACTION THEORY (PFI-THEORY)

In this section, an overview is given to the Particle Flow Interaction theory. Since this is a very comprehensive theory, its derivation and justification cannot be covered here. For such type of information, the reader must consult with the original work<sup>12</sup>. Here, the Mark II version of the PFI-theory is used, while in the previous work<sup>12</sup> the Mark I was applied. Both versions are however well explained in the mentioned work<sup>12</sup>.

### Definition of particle sizes

Between cement particles is a certain potential energy. This potential results from combined forces of van der Waals attraction, electrostatic repulsion and steric hindrance<sup>6,8,13</sup>. The polymers mentioned above (VHMW Na and HMW Na) will generally adsorb on the surface of the cement particles. Their function is to change the above mentioned total potential energy in such manner that coagulation is more difficult obtained and dispersion more easily achieved.

Cement particles are poly-dispersed in size. Some<sup>14</sup> 7 – 9% of the material (by weight) is typically finer than  $2\mu\text{m}$  in diameter and 0 – 4% coarser than  $90\mu\text{m}$ . Traditionally, the particles that are influenced by the *total potential energy* effects are considered to be colloid particles. Gen-

eral definition of such particle<sup>13</sup> is that at least one dimension is in the size range from 1 nm to 1  $\mu\text{m}$ . However, there is no clear distinction between the behavior of particles with somewhat larger dimensions than of the traditional colloidal particle<sup>13</sup>. In the earlier work done by Wallevik<sup>8</sup> (Section 2.5.2), it is demonstrated that cement particles as large as 40  $\mu\text{m}$  in diameter seem to be able to “behave”, at least to some degree, as a colloid particle, somewhat controlled by the action of the total potential energy. All cement particles (i.e. the domain of particle sizes) that can significantly be influenced by the total potential energy effects are classified in this work as “particle size number 3” (meaning that the diameter of these particles is lower than the above mentioned 40  $\mu\text{m}$ ).

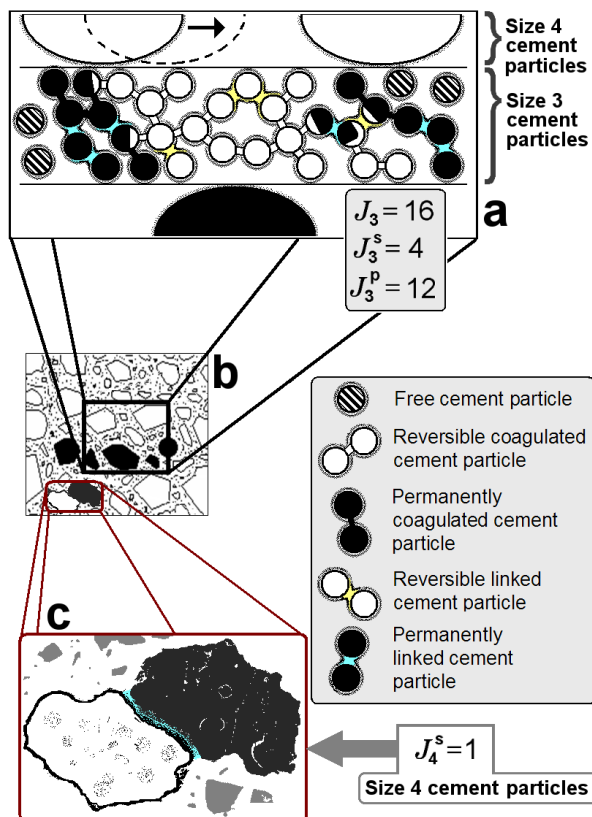


Figure 3. Example of size 3 and 4 cement particles and their number of connections.

When considering a pair of cement particles somewhat larger than of “particle size number 3”, their kinetic energy (or inertia) starts to be sufficiently large to overcome

their mutual energy barrier against coagulation and dispersion. When this condition applies, coagulation does not occur and the cement particles rather interact with each other by a pure hard sphere collision factor. Such cement particles that are not influenced by the total potential energy effects, are classified here as “particle size number 4”. Example of size 3 and 4 cement particles and their number of connections is shown in Fig. 3.

### Reversible and permanent junctions

The connections (or contacts) between the cement particles, formed by the total potential energy interaction, are named *junctions*. The number of *reversible junctions* (from the process of reversible coagulation) is represented with the term  $J_3$ . Two coagulated cement particles by such junction can be separated (i.e. dispersed) again for the given rate of work available to the suspension (the rate of work is provided by the engine of the viscometer).

The number of *permanent junctions* (from permanent coagulation) is designated with  $J_3^p$ . Two coagulated cement particles by such junction cannot be separated (i.e. dispersed) again for the given rate of work available to the suspension. The superscript “p” is an acronym for “permanent”, or more precisely “permanent coagulation” (and “permanent linking”, as explained below).

### Breakable and permanent linkages

Within a few seconds of the initial contact between cement and water, the surfaces of the cement particles are covered by a membrane of gelatinous calcium silicate/sulphoaluminate hydrate<sup>6</sup>. Hence, it is suggested<sup>6,15</sup> that when a pair or more, of cement particles come into contact with water, this hydrate membrane forms around both of them. As soon as the cement suspension is agitated, the linkages between the cement particles may be broken<sup>6</sup>. That is, the bridging mem-

brane ruptures and the cement particles separate. As no recovery of structure was measured for such cases (i.e. is an irreversible process), the term “structural breakdown” was preferred over “thixotropic behavior”<sup>6,5,15,16,17</sup>. The notion of linkages between cement particles was first proposed by Tattersall<sup>17</sup>.

In this work, it is assumed that there are basically two kinds of bridging membranes. The first type is the *breakable membrane*, where two linked cement particles can be broken apart (i.e. dispersed) for the given rate of work available to the suspension. The second type of bridging membrane is the *permanent membrane*, where the two linked cement particles cannot be broken apart with the given power available. The strength of the link between two (or more) cement particles depends on the thickness of the membrane around them and contact geometry<sup>6</sup>. Hence, with different conditions between different cement particles, it is not hard to imagine a simultaneous presence of both permanent- and breakable linked cement particles in the suspension.

As mentioned above, the connections (or contacts) between the cement particles, formed by the hydrate membrane, are named *linkages*. The number of *breakable linkages* (by the formation of breakable weak/thin hydrate membrane) is represented with the term  $J_3^s$  (the superscript “s” is an acronym for “structural breakdown”). Likewise, the number of *permanent linkages* (by the formation of permanent strong/thick hydrate membrane) is designated with  $J_3^p$ .

As cement particle of size 4 do not coagulate, the corresponding (reversible) junction number is always zero, meaning  $J_4 = 0 \text{ m}^{-3}$ . However, as it is fair to assume that the cement particles of size 4 are covered by a hydrate membrane, just as for size 3, the values of  $J_4^s$  (breakable linkages) and  $J_4^p$  (permanent linkages) do not have to be zero.

### Indirect microstructural parameters

For size 3 cement particles, two types of primary (cement) particles are defined. One is primary particles that will undergo a reversible coagulation and linking and is designated with  $n_3$ . The other type is primary particles that will undergo a permanent coagulation and linking and is represented with  $n_3^p$ . The physical unit of  $n_3$  and  $n_3^p$  is in  $[\text{m}^{-3}]$ , meaning number of primary particles per unit volume. As discussed in earlier work by Wallevik<sup>7</sup> (Section 3.4.2), when considering cement based materials, the number of primary particles is slowly changing with time. However, relative to a single experiment of 50 seconds  $n_3$  and  $n_3^p$  (as well as  $n_4$  and  $n_4^p$ ) can safely be considered as constants.

From the above text, an *indirect microstructural parameter* can be defined as  $U_3 = J_3/n_3$ ,  $U_3^s = J_3^s/n_3$  and  $U_3^p = J_3^p/n_3^p$ . In the same fashion, for size 4 cement particles, then  $U_4^s = J_4^s/n_4$  and  $U_4^p = J_4^p/n_4^p$ . The indirect microstructural parameter can generally have values ranging from<sup>7</sup> 0 to 1. There is however now a specific restriction to this consisting of  $0 \leq U_3 + U_3^s \leq 1$ .

The term  $U_3$  are referred to as the (reversible) coagulated state. Likewise, the two terms  $U_3^s$  and  $U_4^s$  are referred to as (reversible) linked state.

### System of equations

The PFI-theory (Mark II) consists of the following system of equations

$$\frac{dU_3^s}{dt} = -I_3^s U_3^s \quad (1)$$

$$\frac{dU_3}{dt} = H_3 ([1 - U_3^s] - U_3)^2 - I_3 U_3^2 \quad (2)$$

$$\frac{dU_4^s}{dt} = -I_4^s U_4^s \quad (3)$$

$$\rho \frac{d\mathbf{v}}{dt} = \nabla \cdot \boldsymbol{\sigma} + \rho \mathbf{g} \quad (4)$$

Where  $d/dt = \partial/\partial t + \mathbf{v} \cdot \nabla$  (see for example Section<sup>8</sup> 2.2). The terms  $\rho$  and  $\mathbf{g}$  used

in Eq. (4) are the density of the cement suspension and gravity, respectively. Evolution equations for  $U_3^p$  and  $U_4^p$  need not to be included in the above set of equations, since these terms are constants during a single rheological test of 50 seconds.

In the above list, the equation of motion, Eq. (4), is included to take into account the effects of inertia. Also, in order to calculate the shear rate  $\dot{\gamma}$  in correct manner, Eq. (4) has to be solved simultaneously with Eqs. (1), (2) and (3). The shear rate is calculated by<sup>18,19,20</sup>

$$\dot{\gamma} = \sqrt{2 \dot{\boldsymbol{\varepsilon}} : \dot{\boldsymbol{\varepsilon}}} \quad (5)$$

where  $\dot{\boldsymbol{\varepsilon}} = \frac{1}{2}(\nabla \mathbf{v} + (\nabla \mathbf{v})^T)$  and  $\mathbf{v}$  are the rate-of-deformation tensor and velocity, respectively<sup>21,22</sup>. This type of shear rate calculation is well known in theoretical rheology and dates back at least to 1947<sup>19</sup>.

The term  $\boldsymbol{\sigma}$  in Eq. (4) is the constitutive equation given by<sup>21,22</sup>

$$\boldsymbol{\sigma} = -p \mathbf{I} + 2 \eta \dot{\boldsymbol{\varepsilon}} \quad (6)$$

where the terms  $p$  and  $\mathbf{I}$  are the pressure and the unit dyadic, respectively. Torque is calculated as<sup>7,8</sup>

$$T_c = 2 \pi R_i^2 h \eta \dot{\gamma} \quad (7)$$

where  $\eta$  is the apparent viscosity given by

$$\eta = \mu_{[t]} + \frac{\tau_{0[t]}}{\dot{\gamma}}, \quad \tau \geq \tau_{0[t]} \quad (8)$$

$$\eta \rightarrow \infty, \quad \tau < \tau_{0[t]} \quad (9)$$

The terms  $\mu_{[t]}$  and  $\tau_{0[t]}$  are the so-called total plastic viscosity and the total yield stress, and are defined by<sup>12</sup> Eqs. 10 and 11.

$$\mu_{[t]} = \mu + \tilde{\mu}(U_3) + \hat{\mu}(U_3^s, U_4^s) \quad (10)$$

$$\tau_{0[t]} = \tau_0 + \tilde{\tau}_0(U_3) + \hat{\tau}_0(U_3^s, U_4^s) \quad (11)$$

The value of  $\tau_{0[t]}$  at  $t = 0$  can be designated as static yield stress (see Fig. 6a).

The term  $I_3^s$  used in Eq. (1) represents the rate of “break-apart” function. That is, it describes the rate which the linkages

between the size 3 cement particles are broken apart and has the physical unit of [1/s]. It is given by

$$I_3^s = \lambda \dot{\gamma}^{0.4} \quad (12)$$

The term  $H_3$  used in Eq. (2), represents the coagulation rate for cement particles of size 3, and has the physical unit of [1/s]. It is given by

$$H_3 = \frac{K}{\dot{\gamma}^2 + l} \quad (13)$$

where  $K = (\alpha \dot{\gamma}^{0.01} + \beta \dot{\gamma}^{0.1}) e^{-\zeta(\partial \dot{\gamma} / \partial t)^2} + K_0$ . The terms  $l$ ,  $\zeta$  and  $K_0$  are empirical constants, kept equal to  $l = 10 \text{ s}^{-2}$ ,  $\zeta = 4 \cdot 10^{-4} \text{ s}^4$  and  $K_0 = 10^{-4} \text{ s}^{-3}$  at all times.

The term  $I_3$  used in Eq. (2), is the dispersion rate for cement particle of size 3, and has the physical unit of [1/s]. It is given by

$$I_3 = \kappa \dot{\gamma}^{2.0} \quad (14)$$

The term  $I_4^s$  used in Eq. (3) represents the rate of “break-apart” function. That is, it describes the rate which the linkages between the size 4 cement particles are broken apart and has the physical unit of [1/s]. It is given by

$$I_4^s = \chi \dot{\gamma}^{0.1} \quad (15)$$

The main Eqs. (1) to (4) are interlinked by Eqs. (5) to (15). For each computed time step, all equations have to be (and are) solved simultaneously together.

## AUTOMATIC PARAMETER IDENTIFICATION

A drawback of the comprehensive PFI-theory is that its material parameters are not directly observable. For finding the parameters of this complex model, certain automatic parameter identification (API) algorithm is applied. The algorithm solves an optimization problem, in which the difference between the measured data and the output of the PFI-theory is minimized.

During the API search, a certain parametric restriction is needed. For example

a negative parameter would be unphysical leading to a computer crash during the PFI simulation.

Although determined by the API-algorithm, the material parameters in Eqs. (12) to (15) depends, among other factors, on the total potential energy interaction between the cement particles and the chemical reactivity of the cement particles with water. Other material parameters depends on different factors like the surface roughness of the cement particles, solid concentration  $\Phi$ , particle shape and hydrodynamic effects (if present at relevant magnitude).

### THE SOFTWARE – VVPF 3.2

The numerical software used in solving Eqs. 1 to 15 is named Viscometric-ViscoPlastic-Flow 3.2 (or VVPF 3.2). It will be GNU GPL licensed (and thus free of charge) and will be available from <http://www.vvpf.net>. Versions 1.0 and 2.0 are already available. Version 3.2 is basically the same as version 2.0, however without an interface to an API algorithm. The last mentioned version is a finite difference model and is designed for time-dependent (transient) and time-independent (steady state) viscoplastic materials. It was used for the Mark I version of the PFI-theory<sup>12</sup>. The outcome of version 2.0 has been compared to version 1.0 (the latter version was used for the MHI-theory<sup>7</sup>).

### RESULTS

The experimental results  $T$  given by the viscometer (solid lines) and the computed results  $T_c$  given by the software (white lines) are shown in Figs. 4 and 5. As shown in these figures, there is a good correspondence between the measured torque  $T$  and its computed counterpart  $T_c$ . This applies for both cases VHMW Na and HMW Na.

When using the HMW Na polymer in the cement suspension (Fig. 5), a somewhat larger rebuild in torque is measured relative to the VHMW Na-case (Fig. 4).

This is apparent when comparing the two figures. The MHI-theory<sup>7</sup> could not simulate this type of recovery in torque, while the PFI-theory can. Here, the benefit of the new theory is most clear.

The number of material parameters in the PFI-theory is quite many. The numerical value of each parameter is perhaps not so interesting and will not be addressed here. However, from these attained parameters (attained by the API search), one can calculate quantities of important relevance. In this manuscript, both the static yield stress as well as work are calculated, and the results are shown in Fig. 6.

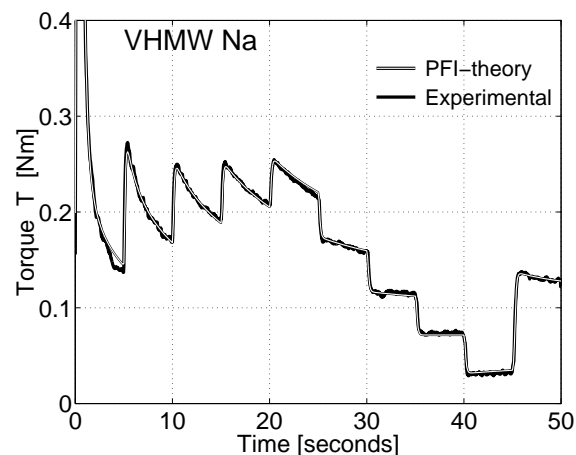


Figure 4. Measured and computed torque  $T$  as a function of time  $t$  (at 72 minutes).

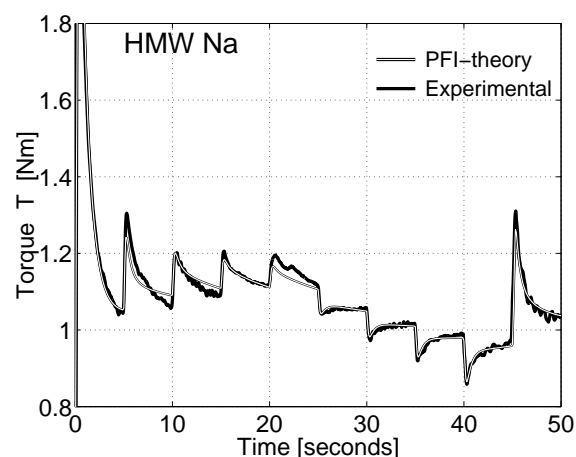


Figure 5. Measured and computed torque  $T$  as a function of time  $t$  (at 72 minutes).

In Fig. 6a is the (PFI) calculated yield stress. Its value is calculated by Eq. 11,

where the time  $t$  is put equal to zero. That is, this is the initial yield stress that is valid for the cement suspension after about 30 minutes resting. For the VHMW Na-case, its value is equal to 119.7 Pa. Roughly 67% of this value is attributed to linked cement particles (structural breakdown behavior), while 33% is attributed to coagulated cement particles (thixotropy). The yield stress  $\tau_0$  for the VHMW Na-case is zero.

For the HMW Na-case, the static yield stress is 266.4 Pa. Roughly 30% of this value is attributed to linked cement particles, while 17% is attributed to coagulated cement particles. The yield stress  $\tau_0$  for the HMW Na-case is 140.0 Pa and thus contributes 53% to the static yield stress (“irreversible contribution”). Therefore, by re-agitation (leading to breaking of linkages and junctions), the static yield stress can only be reduced down to 140.0 Pa for the HMW Na-case, while down to zero for the VHMW Na-case. Such reduction is important relative to a so-called Self-Compacting Concrete.

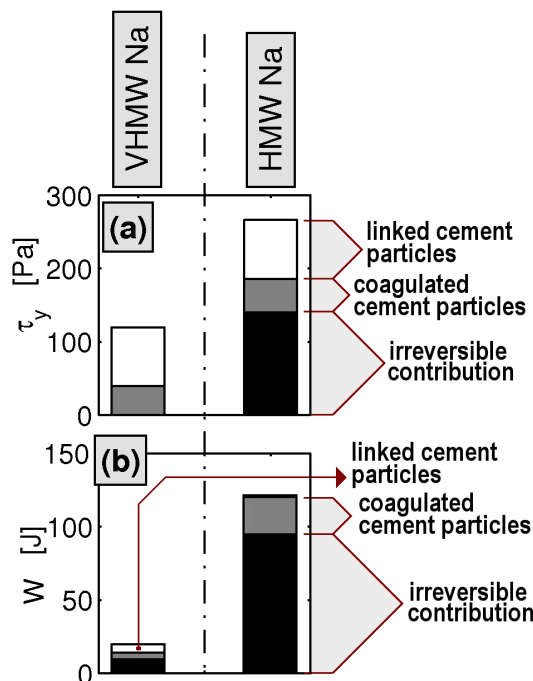


Figure 6. Static yield stress and work for VHMW Na- and HMW Na-cases.

In Fig. 6b is the calculated work  $W$  and

it represents the effort made by the (rotating) outer cylinder of the viscometer in deforming the cement suspension during a single rheological test of 50 seconds. The calculation of work is a rather complicated procedure and will not be discussed in the current manuscript. For the VHMW Na-case, its value is equal to 19.9 J. Roughly 28% of this value is attributed to linked cement particles (structural breakdown behavior), while 24% is attributed to coagulated cement particles (thixotropy). Irreversible contribution is (i.e. from  $\mu$  and  $\tau_0$ ) consists of 47%. For the HMW Na-case, the work is equal to 121.1 J. Only 1% of this value is attributed to linked cement particles, while 21% is attributed to coagulated cement particles. Irreversible contribution is consists of 78%. The relatively low work contribution  $W$  from the linked cement particles for the HMW Na-case in combination with high static yield stress contribution  $\tau_y$  gives that the chemical formed linkages between the cement particles are easily broken apart, right after a rheological test is started (i.e. the chemical bondings are weak). This applies also for the VHMW Na-case.

## CONCLUSIONS

In this work, the effects of two superplasticizers have been presented, designated as VHMW Na and HMW Na. The former product shows a better outcome in terms of static yield value, since by re-agitation it can be significantly reduced. In both cases of VHMW Na and HMW Na a substantial number of linkages are formed during the resting time of about 30 minutes. This is clear with the relative large contribution to the static yield stress  $\tau_y$ .

However, because of its low contribution to work  $W$ , the chemical bonding is weak, meaning that linkages between the cement particles are easily broken apart. For the VHMW Na-case, the same applies for the coagulated cement particles, meaning that coagulation is more difficult ob-

tained and dispersion more easily achieved.

The PFI-theory<sup>12</sup> was applied to attain the information about the rheological properties of the cement suspension mixed with VHMW Na and HMW Na polymers. The software used in this work is named VVPF 3.2 and will be freely available at [www.vvpf.net](http://www.vvpf.net) in the near future. The primary reason for making the code public, is so that a third party can utilize and enhance the PFI-theory further, make his/her own version of it in a separate research project. Time will most likely show that the PFI-theory is far from being perfect. Still, in its current state, it is an improvement relative to many other material models available.

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