Contents lists available at ScienceDirect



# Journal of Petroleum Science and Engineering

journal homepage: www.elsevier.com/locate/petrol



## Rapid bentonite-cement-oil hydration: Implications to fluid loss control



Ahmed Hafez<sup>a</sup>, Qi Liu<sup>a</sup>, Thomas Finkbeiner<sup>a</sup>, Timothy E. Moellendick<sup>b</sup>, J. Carlos Santamarina<sup>a,</sup>

<sup>a</sup> Earth Science and Engineering, KAUST, Thuwal, 23955-6900, Saudi Arabia <sup>b</sup> Saudi Aramco, Dhahran, 31 311, Saudi Arabia

| ARTICLE INFO  | A B S T R A C T   |  |  |
|---|---|--|--|
| Keywords:<br>Bentonite<br>Cement<br>Swelling<br>Lost circulation<br>Drilling fluids | Conventional particulate additives fail to control drilling fluid losses into large-aperture fractures. The separate injections of a bentonite-cement-oil suspension and water can cause rapid hydration, swelling and hardening to effectively plug fractures. This experimental study investigates underlying processes and implications in view of optimal fluid flow control in fractures. Results identify several concurrent hydro-chemo-mechanical coupled processes: capillarity-driven water invasion; cement hydration and the release of Ca <sup>2+</sup> and OH <sup>-</sup> ions; bentonite contractive aggregation and increased hydrophilicity; enlarged inter-aggregate pores that facilitate fluid flow; oil pressurization leading to the formation of oil-filled opening mode discontinuities that facilitate oil escape towards free draining boundaries, and calcium silicate hydrate formation and growth resulting in hardening. The hydration of bentonite-cement-oil suspensions proceeds several times faster than in bentonite-oil suspensions. The optimal mixture should balance competing requirements between flowability. |  |  |

swelling pressure and plug strength.

## Contributions

All authors contributed substantially to the manuscript and approved the final submission.

### 1. Introduction

Drilling muds cool the drill bit, lift and transport cuttings to the surface, maintain sufficient pressure to avoid kicks and blowouts, and ensure the mechanical integrity of the wellbore (Lavrov, 2016; Caenn and Chillingar, 1996; Sadegh Hassani et al., 2016; Abraham, 1933). Drilling fluids are typically recycled and re-injected into the well as part of the mud circulation system (White, 1956).

A filter cake forms against the borehole wall as liquids permeate into small pores and leave solids behind (Liu and Santamarina, 2018; Ferguson and Klotz, 1954). In some cases, the drilling mud flows rapidly into the formation, which results in lost circulation (Feng et al., 2016). Conditions that lead to lost circulation include high permeability formations (Lavrov, 2016; Silent, 1936), vugs and caverns which are common in carbonate rocks (Masi et al., 2011), unintentional hydraulic fractures induced in the formation (Howard and Scott, 1951) and pre-existing natural fracture zones (Lavrov, 2016).

Lost circulation accounts for 12% of the non-productive time during drilling in the Gulf of Mexico (Feng and Gray, 2017, 2018). Associated annual costs to the drilling industry exceed one billion dollars as a result of lost rig time, materials and equipment (Feng and Gray, 2018; Al Menhali et al., 2014). The impact extends beyond the oil and gas industries; for example, lost circulation adds an average 10% to the cost of a typical geothermal project in the United States (Finger and Blankenship, 2010).

Particles can be added to the drilling mud to stop uncontrolled fluid loss (Kulkarni et al., 2016; Savari et al., 2014; Ali et al., 1994; Boukadi et al., 2004; Kang et al., 2015; Sanders et al., 2010). Tried particles include wood chips, nutshells, rubber, ground marble, graphite, cellulose fibers and mica (White, 1956; Alsaba et al., 2014a, 2014b). Particles are small, typically  $d_p \leq 5$  mm, to avoid segregation and damage to pumps. Thus, conventional particulate additives usually fail to control losses into large-aperture fractures much wider than the added particle size d<sub>p</sub> (Davidson et al., 2000).

One mitigation technique sometimes employed in large-aperture fractures involves the separate injections of a bentonite-oil suspension through the drill-pipe and water through the annulus. The two fluids mix downhole and bentonite swells into a viscous plug that can seal the fracture (Lavrov, 2016; Abdulrazzaq et al., 2017; Ryan et al., 2015). Cement is added to the bentonite-oil suspension to improve the plug

\* Corresponding author. E-mail address: carlos.santamarina@kaust.edu.sa (J.C. Santamarina).

https://doi.org/10.1016/j.petrol.2022.110615

Received 14 December 2021; Received in revised form 17 April 2022; Accepted 6 May 2022 Available online 11 May 2022 0920-4105/© 2022 Elsevier B.V. All rights reserved.

| List of notations |  |  | VF[]                                  | Solids volume frac       |
|-------------------|--|--|---------------------------------------|--------------------------|
|                   |  |  | WC []                                 | Water content $= v$      |
|                   | $A [m^2]$  | Cross-sectional area normal to flow                              | $S_s [m^2/kg$                         | ] Specific surface       |
|                   | CC [ ]   | Cement content = mass of cement/solids mass                      | T [K]                                 | Temperature              |
|                   | c <sub>o</sub> [mol/n                                  | n <sup>3</sup> ] Ionic concentration of bulk fluid               | t [s]                                 | Time                     |
|                   | e [ ]  | Void ratio   | t* [s]                                | Characteristic tim       |
|                   | F [N]  | Force (cap: capillary; od: oil viscous drag; wd: water           | v [m/s]                               | Velocity (o: oil; w      |
|                   |  | viscous drag)  | <i>x(t)</i> [m]                       | Water invasion de        |
|                   | $F_c$ [C/mo  | l] Faraday's constant  | z[]                                   | Valence of exchan        |
|                   | <i>G</i> [] Specific gravity (c: cement; b: bentonite) |  | $\gamma_{ow}$ [N/m] Oil-water interfa |                          |
|                   | <i>h</i> [m]   | Interparticle separation (Note: $h = 2r$ for parallel platelets) | $\delta_t [m]$                        | Penetration depth        |
|                   | $k [m^2]$  | Permeability (o: oil-saturated; w: water-saturated               |                                       | 0 and $t = \infty$ repre |
|                   |  | bentonite)   | ε <sub>0</sub> [F/m]                  | Permittivity of free     |
|                   | m []   | Penetration rate exponent  | θ[°]                                  | Contact angle            |
|                   | MF [ ]   | Solids mass fraction = mass of solids/total mass                 | θ [m]                                 | Double layer thick       |
|                   | p <sub>swel</sub> [Pa]                                 | Swelling pressure  | κ[]                                   | Dielectric constan       |
|                   | <i>r</i> [m]   | Pore radius (Note: $r = h/2$ for parallel platelets)             | μ [Pa.s]                              | Viscosity (o: oil; v     |
|                   | R [Pa]   | Double layer repulsion force per unit area                       | $\rho  [\text{kg/m}^3]$               | ] Density; (m: mir       |
|                   | R <sub>c</sub> [J/K.n                                  | nol] Universal gas constant                                      |                                       |                          |
| L                 |  |  |                                       |                          |

strength after hydration (Messenger and McNiel, 1952; Kabir, 2001). Field results from more than 600 wells suggest that bentonite-cement-oil suspensions are more successful at controlling fluid losses than bentonite-oil suspensions across different loss zones (Alkinani et al., 2019). Bentonite and bentonite-cement suspensions have also been used as impermeable barriers for other applications such as waste disposal, wastewater treatment, landfill liners and vertical cut-off walls (Katsioti et al., 2008; Gleason et al., 1997; S á nchez et al., 2006). More recent studies investigate the partial substitution of Portland cement with metakaolin and calcined bentonite to improve concrete strength and reduce greenhouse emissions (Taylor-Lange et al., 2015; Wei and Gencturk, 2019; Taklymi et al., 2020; Laidani et al., 2020; He et al., 1996, 2000; Müller, 2005; Poon et al., 2001).

Hydration mechanisms and hydration rates in bentonite-oil and bentonite-cement-oil suspensions and the parameters that govern the plug strength remain unclear. In this study, we investigate the complex hydro-chemo-mechanical interactions between bentonite, cement, oil and water in 1D and radial configurations, measure the swelling pressure and time-dependent strength changes, and gather tomographic images during hydration. Insights gathered from the different experiments help us develop a physical model relevant to bentonite-cement hydration within fractures and anticipate implications on fluid flow control to improve drilling operations.

## 2. Experimental study

We use Wyoming sodium bentonite (specific surface area  $S_s = 544$  $m^2/g$ ), Portland cement (Type I), mineral oil (oil: viscosity = 65 cP, density =  $877 \text{ kg/m}^3$ ) and tap water to prepare different suspensions and pastes defined in terms of mass fractions:

mass fraction of solids 
$$MF = \frac{M_{solid}}{M_{total}} = \frac{M_{cem} + M_{bent}}{M_{cem} + M_{bent} + M_{oil}}$$

cement content  $CC = \frac{M_{cem}}{M_{solid}} = \frac{M_{cem}}{M_{cem} + M_{bent}}$ 

and water content  $WC = \frac{M_w}{M_{solid}} = \frac{M_w}{M_{cem} + M_{bent}}$ 

where sub-indices indicate cem = cement, bent = bentonite, oil = oil and w = water. In agreement with field operations, we use tap water rather than distilled water, and natural bentonite with its own concentration of excess salt; surface conduction along bentonite particles and

| VF [ ]  | Solids volume fraction = volume of solids/total volume      |  |
|---|---|--|
| WC []   | Water content = water mass/mass of solids                   |  |
| $S_s [m^2/kg$                                     | g] Specific surface   |  |
| T [K]   | Temperature   |  |
| t [s]   | Time  |  |
| t* [s]  | Characteristic time   |  |
| v [m/s]   | Velocity (o: oil; w: water)                                 |  |
| <i>x(t)</i> [m]                                   | Water invasion depth  |  |
| z[]   | Valence of exchangeable cations                             |  |
| $\gamma_{ow}$ [N/m] Oil-water interfacial tension |   |  |
| $\delta_t [m]$                                    | Penetration depth at time t after specimen preparation (t = |  |
|   | 0 and $t = \infty$ represent asymptotes)                    |  |
| ε <sub>0</sub> [F/m]                              | Permittivity of free space                                  |  |
| θ[°]  | Contact angle   |  |
| θ [m]   | Double layer thickness                                      |  |
| κ[]   | Dielectric constant   |  |
| µ [Pa.s]  | Viscosity (o: oil: w: water)                                |  |

neral and b: bulk)

hydrated ions from excess salts and cement hydration dominate the electrical conductivity of the pore fluid.

We prepare bentonite-cement-oil suspensions by adding bentonite and cement powders to mineral oil to attain pre-defined mass fractions. Tests involve three different boundary conditions to gain complementary insights into hydration and swelling mechanisms. Test protocols and results follow.

## 2.1. Hydration of bentonite-cement-oil suspension in 1D vertical cylinders

We fill graduated cylinders with either bentonite-oil or bentonitecement-oil suspensions, add water on top and monitor the system response (Fig. 1-a). Three different cylinder diameters allow us to assess boundary effects (D = 17, 21 and 61 mm). The selected mass fractions of bentonite, oil and cement are used in common practice.

Bentonite-oil suspensions (without cement). Water hydrates the hydrophilic bentonite particles through capillary suction. Displaced oil forms droplets that escape and float towards the top of the vertically aligned cylinders (Supplementary movie 1). Some oil remains trapped and forms oil-filled opening-mode discontinuities (Supplementary Fig. S1).

The hydrated and swollen bentonite has low permeability. Reduced permeability, oil-filled gaps and increased drag distance gradually delay further water invasion, therefore, the hydration front velocity decreases with time. Fig. 2-a illustrates the hydration thickness as a function of time for different cylinder diameters selected to reflect different fracture apertures. Note: the top surface of the hydrated bentonite is uneven; thus, we measure the hydration thickness at four different positions across the cylinder and report the average value with error bars.

Hydration is faster in larger cylinder diameters (analogue to fracture apertures). Coexisting mechanisms contribute to this trend: (1) wall friction hinders expansion and leads to higher swelling pressure which more effectively seals preferential flow paths along walls in cylinders of smaller radius, i.e., narrower fracture gaps (see supplementary information & Fig. S4), (2) segregated oil droplets can flow more readily in large cylinders, (3) conversely, oil-filled gaps can easily cover the entire cross-sectional area and stop water invasion in smaller cylinders.

Hydration is faster in suspensions with lower bentonite mass fraction, probably due to the more severe reduction in permeability in suspensions with higher solids content (Fig. 2-b). Clearly, a minimum bentonite mass fraction is required for hydration, hence, there must be an optimum bentonite fraction *MF* for fastest hydration.

Bentonite-Cement-Oil Suspensions. The hydration of bentonite-cement-



**Fig. 1.** Experimental setups. (a) 1D hydration column: water placed on top of oil-based suspension invades by capillarity, hydrate minerals and displaces the oil (red). (b) Swelling pressure: The oil-based sample is placed in an oedometer cell within a rigid frame instrumented with a load cell; water invades by capillarity through the lower porous stone (sample: diameter = 50 mm, height = 10 mm). (c) Radial flow: the gap between the two transparent disks is pre-saturated with water; the injected oil-based suspension invades the gap with a quasi-circular front from the central port. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)



**Fig. 2.** Hydrated layer thickness as a function of time: <u>Bentonite-oil</u> suspensions in 1D tests. (a) Effect of tube diameter D (0.77 g bentonite/ml oil, mass fraction MF = solids mass/total mass = 0.47). (b) Effect of bentonite mass fraction MF (tube diameter D = 21 mm). Note: experimental configuration in Fig. 1-a. Note: the surface of the hydrated bentonite is uneven. Thus, we measure the hydration thickness at four different positions across the cylinder and report the average value with error bars.

oil suspensions is strikingly different from the hydration of *bentonite-oil* suspensions (Supplementary movie 2, Supplementary Fig. S1). Water invasion proceeds slowly during the initial induction period while bentonite particles at the water-oil interface control the rate of hydration. Once water contacts cement particles, water invasion and hydration suddenly accelerate and proceed several times faster than in *bentonite-oil* suspensions (Fig. 3-a). At any given time after the induction period, the invasion front is deeper in mixtures with higher cement content *CC* (Fig. 3-b-c), but there is reduced swelling with increased cement content CC (Fig. 3-d). Some studies have also reported the rapid hydration of uncalcined bentonite-cement mixtures (Wei and Gencturk, 2019; Mani  $\acute{c}$  et al., 1998); however, particle-scale mechanisms and interactions remain poorly understood in all cases, especially in the presence of oil.

CT scans of a *bentonite-cement-oil* suspension 20 h after the addition of water illustrate oil segregation along diagonal opening-mode discontinuities which suggests principal stress rotation probably due to wall friction mobilized during swelling (Fig. 4, Supplementary movie 3 -

Tomographer: TESCAN CoreTOM, operated at 80 kV to gather 698 frames; post processing and 3D volume rendering using Avizo software). Apparently, these discontinuities provide pathways for enhanced water invasion and oil escape. Some elliptical gas-and-oil filled cavities with a preferential vertical orientation remain trapped in the hydrated mass.

## 2.2. Swelling and strength

The swelling pressure against fracture walls determines the differential pressure the bentonite plug may sustain. Swelling pressure measurements under 1D conditions are conducted by enforcing zero volumetric strain (Fig. 1-b). The test sequence follows: (1) place the *bentonite-oil* suspension in an oedometer cell (sample diameter = 50 mm, height = 10 mm, MF = 0.47 to 1. Note: the test at MF = 1 is under dry conditions, i.e., no oil) between porous stones covered with filter paper, (2) place the entire cell in a rigid load frame (Wille Geotechnik UL 25) and flood the cell with water, and (3) measure the stress exerted by the swelling bentonite against the load cell (deformation <0.025 mm).



**Fig. 3.** Water invasion and swell: <u>Bentonite-cement-oil</u> suspensions in 1D tests. (a) Thickness of the hydrated layer for *bentonite-oil* (0.77 g bentonite/ml oil, MF = 0.47, black), *bentonite-cement-oil* (0.77 g bentonite and 0.34 g cement/ml oil, MF = 0.56, CC = 0.31, red) and *bentonite-Ca*(OH)<sub>2</sub>-oil suspension (0.77 g bentonite and 0.16 g Ca(OH)<sub>2</sub>/ml oil, MF = 0.51, violet). Points: experimental data. Dashed lines:  $a\sqrt{t}$  trend. (b) Water invasion depth as a function of time for different cement contents *CC*. Points: experimental results. Dashed lines:  $a\sqrt{t}$ . (c) Thickness of the hydrated layer for different cement contents *CC* 1 h after induction period. The solid line captures the overall trend. (d) Ratio of swelling to invasion depth for different *CC*-values 1 h after induction period. Invasion is fast for CC = 1 and ends in less than 1 h. Note: see definitions in Fig. 1a. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)



**Fig. 4.** Bentonite-cement hydration: Ensuing internal structure. (a) Photographic image. (b&c) slices of 3D X-ray tomograms. Mixture: MF = 0.56, CC = 0.31.

Fig. 5-a illustrates the bentonite swelling pressure as a function of time for suspensions with different mass fractions MF = solids mass/ total mass. The swelling pressure increases with time as water hydrates

the bentonite platelets and gives rise to double layer repulsion. Published studies report similar trends for the swelling pressure of uncompacted sodium bentonite as a function of initial volume fraction (Liu, 2013; Yong and Warkentin, 1975). The swelling pressure for the *bentonite-cement-oil* suspension (MF = 0.56, CC = 0.31) is significantly lower than bentonite alone; apparently, hydrated ions released during cement hydration diminish double layer repulsion (further analyzed in the following section).

We use cone penetration resistance to assess the time-dependent strength of water pastes prepared with bentonite (WC = 2.0), cement (WC = 0.50) and bentonite-cement (WC = 2.0; CC = 0.31) (Hansbo, 1957; Llano-Serna and Contreras, 2020). The cone penetration protocol follows: 1) prepare the paste in the specimen cup (d = 7 cm, height = 4.5 cm); 2) lower the digital cone penetrometer (MATEST S165) until it barely touches the sample surface – see Fig. 6 inset; 3) release the stainless steel cone such that it penetrates the specimen by self-weight (cone mass = 20 g); and 4) measure the penetration depth using the attached caliper. We repeat penetration measurements at different surface locations as time progresses.

The cone penetration depth is inversely proportional to specimen shear strength (Hansbo, 1957). Initial penetration depths in Fig. 6 highlight the interplay between initial water content and specific surface on suction generation and its effect on the mixture viscosity. Penetration-time  $\delta_t$  data are fitted with an asymptotically correct model to identify asymptotic values ( $\delta_{t=0}$  and  $\delta_{t=\infty}$ ),

$$\delta_t = \delta_{t=\infty} + (\delta_{t=0} - \delta_{t=\infty}) \left[ 1 + \left(\frac{t}{t^*}\right)^m \right]^{-1} \tag{1}$$

where the characteristic time  $t^*$  corresponds to a penetration  $\delta_t = (\delta_{t=0} - \delta_t)^2$ 

0 - 0.1



10

100

**Fig. 6.** Hardening: Cone penetration depth versus time for bentonite-water paste (water content WC = 2), bentonite-cement-water paste (WC = 2, CC = 0.31), and cement-water paste (WC = 0.5). Inset: cone-penetration test set-up. Points: experimental results. Lines: equation (1). The inset table lists the asymptotic penetrations  $\delta_{t = 0}$  and  $\delta_{t = \infty}$ , the characteristic time  $t^*$  and the rate exponent m.

Time [hr]

1

 $\delta_{t=\infty}$ )/2 and the exponent *m* relates to the rate of reactions. Penetration trends in log (time/hr) scale highlight differences between the three mixtures and underlying processes (Fig. 6):

- The bentonite-water paste exhibits time-dependent thixotropic hardening (van Olphen, 1955; Rinaldi and Clariá, 2016; Diaz-Ro-driguez and Santamarina, 1999).
- The cement-water paste hardens and penetration ceases in about 10 h (as in standard Portland cement measurements with Vicat needle penetration)
- The bentonite-cement-water paste shows an extended hardening time, probably due to bentonite aggregation around cement particles and diffusion limited reactions ((Fam and Santamarina, 1996) Note differences in the exponent: m = 2.4 for the cement paste, but m = 1 for the bentonite-cement paste Fig. 6).

#### 2.3. Hydration in radial conditions – plug resistance

During typical field applications, the hydrated bentonite is squeezed radially outwards from the drill-pipe into fractures. We built an analogue fracture-like geometry to study hydration in fractures and to test the strength of the bentonite plug that forms around a well (Fig. 1-c). The device consists of two parallel transparent acrylic disks, 400 mm in diameter with a pre-fixed 5 mm thick gap. The test procedure involves four steps: (1) pre-saturate the gap between the two parallel disks with water (unless otherwise stated); (2) inject 160 ml of the selected

Journal of Petroleum Science and Engineering 215 (2022) 110615

**Fig. 5.** Bentonite swelling pressure. (a) Swelling pressure as a function of time for bentonite and bentonite-cement specimens with different mass fractions (MF = solids mass/total mass). Note: the test at MF = 1 is under dry conditions, i.e., no oil (b) Asymptotic swelling pressure as a function of solids volume fraction VF = solids volume/(solids + oil volume) for bentonite-oil (red points) and bentonite-cement-oil suspensions (black point). Solid red line: exponential trend (equation (10)). Note: tests conducted with the experimental configuration in Fig. 1-b. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

suspension into the gap from the central port; (3) allow the suspension to hydrate for 20 h (unless otherwise stated); and (4) measure the fluid pressure the plug can resist under a constant water injection flow-rate of 80 ml/min (syringe pump by Wille Geotechnik LT12700/185/CS). We measure the injection pressure (pressure transducer from Omega Engineering) and record footage of the experiment (video camera Sony Alpha 5000).

Fig. 7 shows the pressure-time response under four different experimental conditions. Details and observations follow:

- *Non-hydrated bentonite-oil suspension* (MF = 0.47, Fig. S3-a). The gap is pre-saturated with oil. Water breaks through at a low peak pressure of 9.4 kPa.
- Oil-bentonite in impervious matrix (MF = 0.47, Fig. S3-b). We presaturate the gap with water. The injected *bentonite-oil* suspension sits within the water-saturated fracture for 20 h, yet, water hydrates



**Fig. 7.** Hydration of bentonite-oil and bentonite-cement-oil suspensions in radial flow tests. Pressure-time response curves for gaps with: non-hydrated bentonite-oil suspension (MF = 0.47, blue), hydrated bentonite-oil in impervious matrix (MF = 0.47, black), hydrated bentonite-oil in permeable matrix (MF = 0.47, green), and bentonite-cement in permeable matrix (MF = 0.56, CC = 0.31, red). Fracture aperture: 5 mm. Note: experimental configuration in Fig. 1-c. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

just the edges of the bentonite plug and breakthrough pressure reaches 34 kPa.

- Oil-bentonite in permeable matrix (MF = 0.47, Fig. S3-c). We attach filter papers to the disk surfaces to simulate a water-permeable matrix around a fracture, and repeat the previous experiment. The resulting breakthrough pressure jumps to 147 kPa as the bentonite plug hydrates more extensively and swelling pressure builds against the fracture surfaces
- Bentonite-cement in permeable matrix (MF = 0.56, CC = 0.31, Fig. S3-d). Finally, we repeat the last test but with *bentonite-cement-oil* suspension (once again, water-saturated system with filter papers on the walls). Fast hydration and the strength of hydrated cement render a plug that sustains 915 kPa inlet pressure (Fig. 7).

The Supplementary Movie 4 illustrates the hydration of *bentonite-oil* and *bentonite-cement-oil* suspensions under radial conditions. Water hydrates the suspension starting from the outer edges (note color changes). There is some oil escape towards the outer edge (counter-flow direction) followed by oil leak through the central inlet port.

The interface between the bentonite-cement plugs and the acrylic plate provides a preferential flow path for water breakthrough (Supplementary Movie 5; notice color change at the interface). After breakthrough, residual water in percolating channels augments the hydration of neighboring bentonite mixture, which swells and heals channels. Fig. 8-a shows the gain in strength for a *bentonite-cement* plug following successive water injection/channeling and healing sequences 24 h apart (Fracture aperture = 35 mm, MF = 0.56, CC = 0.31, Fig. 8-a). Plug failure in subsequent injections takes place along previous channels; hence, healing preserves memory.

The time-dependent propagation of the hydration front affects early strength gain in thick plugs. Fig. 8-b illustrates the maximum break-through pressure data gathered for different fracture apertures using *bentonite-cement-oil* suspensions 24 h after injection (MF = 0.56, CC = 0.31 – walls covered with filter paper – fracture apertures range from 5 to 35 mm). Data shows a marked decrease in breakthrough pressure  $p_b$  with increasing aperture.

#### 3. Analyses

Results highlight unique features in the concurrent hydration of bentonite-cement mixtures. We analyze these observations next to gain new insights in view of field applications.

### 3.1. Particle-level interactions

Bentonite swelling starts with inter-platelet separation at low humidity. Water hydrates the counterions that neutralize the clay surfaces at high water content. The hydrated ions remain attracted to the negatively charged clay surface to form the diffuse double layer (Madsen and Müller- Vonmoos, 1989; Santamarina et al., 2001; Delage and Tessier, 2021). At large water contents, double layer interactions dominate (Yong and Warkentin, 1975; Langmuir, 1938). The double layer thickness  $\vartheta$  is a function of temperature *T*, dielectric constant  $\kappa$  of the pore fluid, and the ionic concentration  $c_0$  and valence *z* (Gleason et al., 1997; Santamarina et al., 2001; Kolstad et al., 2004; Mitchell and Soga, 2005):

$$\vartheta = \sqrt{\frac{\varepsilon_0 R_c \kappa T}{2F_c^2 c_o z^2}} \tag{2}$$

where the universal constants are Faraday's constant  $F_c = 9.648 \times 10^4$  C/mol, the permittivity of free space  $\varepsilon_0 = 8.85 \times 10^{-12}$  F/m and the universal gas constant  $R_c = 8.314$  J/(K•mol).

The swelling pressure denotes the amount of pressure that should be applied on a clay sample to prevent volume expansion upon water addition (Sridharan et al., 1986). The swelling pressure scales with double layer repulsion R [kPa] between clay platelets separated at a distance h (Santamarina et al., 2001):

$$R = 64R_c T c_o e^{\left(-\frac{h}{\theta}\right)}$$
(3)

Non-polar fluids fail to hydrate counterions, thus, clay particles mixed with oil remain aggregated by van der Waals attraction and ionic bonds with shared cations (Sridharan and Choudhury, 2008). Bentonite experiences a significant reduction in permeability during hydration: platelets separate and swell into large pores, the controlling D85 pore size decreases, tortuosity increases, and water molecules next to bentonite surfaces experience reduced mobility (Gleason et al., 1997; Sridharan and Choudhury, 2008; Ren and Santamarina, 2018). Consequently, the permeability of bentonite to non-polar fluids can be five-to-six orders of magnitude higher than to water (Mesri and Olson, 1971).

Dry cement powder is a mixture of dicalcium silicate, tricalcium silicate, tricalcium aluminate and tetra-calcium aluminum ferrite. The early stages of cement hydration involve the dissolution of these compounds in water and the formation of calcium silicate hydrate (CSH) and calcium aluminum sulfate (ettringite). Cement hydration releases  $Ca^{2+}$  and  $OH^-$  ions, consequently, the ionic concentration and alkalinity of the pore fluid increase (Katsioti et al., 2008; Fam and Santamarina, 1996; Plee et al., 1990; Bullard et al., 2011; Kaufhold et al., 2020).

We measure the pH (Hanna instruments probe) and electrical conductivity (Metler Toledo electrode probe) of tap water and the timedependent values for bentonite (WC = 2), cement (WC = 0.5 & 2) and bentonite-cement (WC = 2) pastes during hydration to gain insight into particle-level interactions (Fig. S2). Conductivity measurements were



**Fig. 8.** Plug strength in a fracture under radial flow. (a) Self-healing after successive plug failures - The bentonite-cement plug rests for 24 h between re-injections (fracture aperture = 35 mm). (b) The effect of fracture aperture on hydration and breakthrough pressure after 24 h. All tests conducted with bentonite-cement mixtures (MF = 0.56, CC = 0.31) and fracture faces covered with filter paper. Note: experimental configuration in Fig. 1-c.

corroborated with complex permittivity spectral measurements (200 MHz–4.5 GHz). Results show that conductivity increases rapidly during the early stages of cement and bentonite-cement hydration due to the release of hydrated Ca<sup>2+</sup> ions (Fig. S2 - see (Dong et al., 2016)). Eventually, the pore fluid becomes saturated with Ca<sup>2+</sup> ions at  $\sigma \approx 10$  mS/cm (Villar-Cociña et al., 2003) and electrical conductivity reaches a maximum. Afterwards, the conductivity decreases as the cement hydration products form, such as CSH and ettringite.

The electrical conductivity  $\sigma$  is a volumetric measure of charge carriers and their mobility; therefore, the paste conductivity depends on the mixture porosity, the pore fluid conductivity which is a function of ionic concentration and mobility, and surface conduction. In bentonitecement-water mixtures, the pore fluid conductivity is controlled by hydrated excess salts in the bentonite and hydrated ions released during cement hydration. On the other hand, surface conduction depends on the volumetric fraction of bentonite. Previous experimental studies confirm that the electrical conductivity of water-saturated cement pates during the early stages of hydration increases with water cement ratio (see Fig. S2 - cement: WC = 0.5,  $\sigma$  = 4.5 to 6.5 mS/cm; WC = 2,  $\sigma$  = 5 to 9.5 mS/cm (Liu et al., 2008; Dong et al., 2016; Wei and Li, 2006);). Porosity n depends on the mass ratios for water-cement wc, and bentonite-cement bc, and the specific gravity of cement and bentonite Gc and Gb (Note: the gravimetric/volumetric analysis assumes water saturation):

$$n = \frac{Gc \bullet wc}{1 + Gc \bullet wc + \frac{Gc}{Gb} \bullet bc}$$
(4)

Changes in pore fluid pH and ionic strength affect clay aggregation. While surface charge due to isomorphic substitution is pH independent, protonation/deprotonation and surface complexation are both pH and valence dependent. In fact, both edges and faces become negatively charged at high pH (above the edge isoelectric point), prompting a dispersed fabric made of parallel platelets (rather than edge-to-face aggregation (Santamarina et al., 2001; Pecini and Avena, 2013);). The dispersed fabric is a transient state: soon, the high ionic concentration in the pore fluid diffuses between platelets, hinders platelet repulsion and causes aggregation (Lagaly et al., 2006; Tombácz and Szekeres, 2004) (Equations (2) and (3)). Published studies disagree on the mode of bentonite particle aggregation at high pH and ionic concentration, whether it is edge-to-face, edge-to-edge or face-to-face (Santamarina et al., 2001; Rand et al., 1980; Lagaly and Ziesmer, 2003). Nevertheless, macro-scale experimental observations show volume contraction leaving cavities and pathways between them that enhance fluid permeability.

Other concurrent processes may play important roles as well. In particular, there is evidence that montmorillonite hydrophilicity increases in alkaline conditions (Pan et al., 2020; Yi et al., 2018), and may result in capillary-enhanced contraction. We run complementary hydration tests to corroborate the role of pH on bentonite hydration. The tested bentonite mixtures involve either CaCl<sub>2</sub> or Ca(OH)<sub>2</sub> powders, so that Ca<sup>2+</sup> is a common cation. The bentonite-Ca(OH)<sub>2</sub>-oil suspension shows a high rate of hydration similar to bentonite-cement-oil (Fig. 3-a), however, bentonite-CaCl<sub>2</sub>-oil exhibits hydration rates similar to bentonite-oil (not shown). Clearly, surface charge changes in alkaline conditions plays a critical role in the fast hydration of bentonite-cement-oil mixtures. Finally, montmorillonite particles experience dissolution at high pH (Sánchez et al., 2006; Santamarina et al., 2001).

In summary, the hydration of *bentonite-oil* mixtures causes swelling and a pronounced decrease in permeability. On the other hand, cement hydration in *bentonite-cement-oil* mixtures counteracts swelling tendencies, prompts the contractive aggregation of clay particles and the formation of oil-filled pathways for oil scape so that very high hydration rates take place.

## 3.2. Bentonite-oil hydration

Let's analyze 1D water invasion into a saturated bentonite-oil mixture driven by the capillary force  $F_{cap}$  that water generates in contact with hydrophilic clay platelets (Fig. 9 inset - Note: the buoyant force  $F_b=gV_{droplet}(\rho_o - \rho_w)$  is orders of magnitude smaller and is ignored in this analysis); for a cross sectional area A:

$$F_{\rm cap} = \frac{2\gamma_{ov} \cos\theta}{r} A \tag{5}$$

where the oil-water interfacial tension is  $\gamma_{ow}$ =0.052 N/m and the assumed contact angle is the limiting value  $\theta = 0^{\circ}$  for perfectly hydrophilic clay platelets. The mean pore radius *r* can be estimated from the void ratio *e*, the bentonite specific surface *S*<sub>s</sub> and mineral density  $\rho_m$ ; for thin parallel platelets (Phadnis and Santamarina, 2011):

$$r = \frac{e}{S_s \rho_m} = \frac{1 - VF}{S_s \rho_m VF} \tag{6}$$

where the second equality is in terms of the solids volume fraction VF = volume solids/total volume which is related to the mass fraction as MF= ( $\rho_s/\rho_b$ )•VF. Darcy's law allows us to estimate the resisting viscous forces in the water-saturated segment (Fig. 9 inset) as a function of the water viscosity  $\mu_w$ , the hydrated bentonite permeability  $k_w$  and flow velocity v:

$$F_{wd} = A\left(v \frac{\mu_w}{k_w}\right) x(t) \tag{7}$$

where x(t) is the position of the water front at time t. Measurements conducted as part of this study using 1D consolidation tests reveal that the permeability in oil saturated bentonite  $k_o$  is six orders of magnitude higher than  $k_w$ , thus, the oil drag force tends to vanish for geometrical conditions relevant to this study (whether oil is flowing in the same direction to water invasion, or in counterflow direction along oil-filled discontinuities).

The flow velocity is the front velocity, v = dx/dt. Then, equations (5)–(7) combine to predict the invasion process (Note: it disregards inertial effects):

$$\frac{dx}{dt}\frac{\mu_w}{k_w}x(t) - \frac{2\gamma_{ow}}{s}\frac{S_s\rho_m}{e} = 0$$
(8)

The solution of the differential equation predicts the position of the



**Fig. 9.** Hydration of *bentonite-oil* suspensions with a free-flow boundary. Analytical model (geometry and parameters), experimental results (data points for MF = 0.47) and fitted equation (9) for  $k_w = 1.1 \times 10^{-20}$  m<sup>2</sup>. Note: see definitions in Fig. 1-a.

#### A. Hafez et al.

invasion front at time *t* (initial condition  $x_{t=0}=0$ )

$$x(t) = \sqrt{4\frac{k_w}{\mu_w}\frac{\gamma_{ow} S_s \rho_m}{e}t} = \alpha \sqrt{t}$$
(9)

Thus, invasion follows a square root of time function. Model predictions agree well with the experimental data gathered with a free-flow boundary to allow for co-linear flow where water and oil move in the same direction (Fig. 9 for  $k_w = 1.1 \times 10^{-20} \text{ m}^2$ ).

The hydration rate of *bentonite-cement-oil* suspensions follows a similar square-root of time function for the different cement contents (CC = 10%–50%, Fig. 3); this confirms the dominant role of capillary forces regardless of cement content. However, the  $\alpha$ -factor in Equation (9) is higher than in bentonite-oil due to the higher permeability that results from cement hydration.

More complex flow patterns include counterflow where the displaced oil exits against the invading water or when transverse oil-filled gaps emerge and stop further capillary-driven flow as observed in Figs. 2 and 3 (hydration experiments in vertical column - Fig. 1-a).

### 3.3. Bentonite swelling pressure

Consider dispersed bentonite platelets with parallel face-to-face configuration. The inter-particle distance is h = 2r. Then, equations (3) and (6) suggest that the swelling pressure is an exponential function of the volume fraction, for a given clay, ionic concentration and double layer thickness

$$p_{swel} = \beta e^{-\left(\frac{1-VF}{VF}\right)}$$
(10)

Indeed, the maximum swelling pressures measured for various volume fractions as part of this study follow an exponential trend with volume fraction (Fig. 5-b). The fitted factor  $\beta = 650$  kPa applies to low solid fractions and it is not the asymptotic swelling pressure as the volume fraction of solids VF $\rightarrow$ 1.0 because Equation (3) applies to large interparticle separations (Note: other forces gain relevance at short distances, such as van der Waals attraction and hydration forces). Admittedly, there is a tradeoff between flowability (decreases with VF) and swell and plug resistance (increase with VF); consequently, field applications should seek a compromise volume fraction for optimal effects.

## 3.4. Bentonite-cement hydration - summary of physical processes

Previous sections identified processes related to bentonite hydration, swelling, evolution of permeability, fluid migration, and the effect of concurrent cement hydration. These physics foundations and experimental observations help us identify the sequence of coupled hydrochemo-mechanical processes that accompany the hydration of *bentonite-cement-oil* suspensions (Fig. 10):

- <u>Capillary invasion</u>: Water hydrates bentonite particles near the oilwater interface. Capillary-driven water invasion will increase the pressure in the non-wetting oil forcing it to drain out of the bentonite paste towards free-draining boundaries (even in counterflow direction to water invasion).
- <u>Chemo-hydro coupling</u>: Cement hydration releases Ca<sup>2+</sup> and OH<sup>-</sup> ions. Negatively charged hydrated bentonite platelets at high pH aggregate and contract due to the high Ca-concentration and leave open pathways for enhanced permeability (Fig. 3-a & 4). Furthermore, hydrophilicity increases in alkaline conditions causing capillary-enhanced contraction at high pH. The rate of water invasion increases with cement content (within the range of mass fractions explored in this study - Fig. 3-b).
- <u>Hardening</u>: The hydrated bentonite-cement mixture gains strength as calcium-silicate hydrate needles grow, bond and interlock. The hardening rate is lower in bentonite-cement than in cement pastes alone, as shown in cone penetration results (Fig. 6).
- <u>Hydro-mechanical:</u> Displaced oil experiences high pressure, forms opening-mode discontinuities and can eventually escape to free boundaries. Oil trapped in oil-filled lenses hinders capillary-driven water invasion.

Field conditions will affect hydration and hardening rates and the ultimate plug strength. For example, the hydration rate benefits from readily available subsurface water and permeable formations, yet, saline formation water hinders bentonite swelling. Finally, swelling increases (Bag and Rabbani, 2017; Akinwunmi et al., 2019) and the oil viscosity decreases under the higher formation temperature allowing for faster oil displacement.



Fig. 10. Hydration of bentonite-cement-oil suspensions: Underlying processes. The sketch captures an instant in the hydration process (geometry similar to vertical cylinders in Fig. 1-a). The lower layer shows the bentonite-cement-oil, capillary-driven water invasion along small inter-particle pore spaces between hydrophilic bentonite particles, and oil droplets escaping from the larger pores. The intermediate layer shows cement hydration and the release of OH- and Ca2+ ions. Bentonite particles gain a prevalent negative charge and initially disperse but readily form aggregates as the high concentration of Ca<sup>2+</sup> ions collapses double layers. Clay aggregation leaves pathways that enhance water invasion and oil escape (see Fig. 4). Calcium silicate hydrate needles grow, interlock-and-bond, and cause hardening. The upper layer shows buoyant oil migrating towards the top of the water column.

## 4. Conclusions and implications

Effective fluid control is needed to advance drilling operations. Consequently, lost-circulation materials should rapidly set into a strong plug that can sustain the differential fluid pressure between the borehole and the formation. *Bentonite-oil* suspensions are unsuitable for controlling fluid loss into large-aperture fractures due to limited strength and very slow hydration rate. On the other hand, *bentonite-cement-oil* suspensions hydrate faster than *bentonite-oil* suspensions and build considerable strength in hours.

The fast hydration of *bentonite-cement-oil* mixtures is a consequence of several concurrent events: capillarity-driven water invasion; cement hydration and the release of  $Ca^{2+}$  and  $OH^-$  ions; bentonite contractive aggregation and increased hydrophilicity; enlarged inter-aggregate pores that facilitate fluid flow; and oil pressurization leading to the formation of oil-filled opening mode discontinuities that facilitate oil escape towards free draining boundaries.

The plug strength is determined by the cement content; yet, the rate of hardening is delayed by the presence of bentonite. The resulting bentonite-cement plugs are self-healing as injected water contributes to bentonite and cement hydration along the percolating channel.

The optimum bentonite mass fraction *MF* and cement content *CC* for field applications should balance competing requirements between flowability, water invasion speed, swelling pressure and plug strength.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Acknowledgements

This work was supported by the KAUST Endowment and Saudi Aramco. Gabrielle E Abelskamp edited the manuscript.

## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.petrol.2022.110615.

#### References

- Abdulrazzaq, W., AlBuraikan, R., Savari, S., Whitfill, D.L., 2017. New methodology to evaluate the performance of chemical sealant loss circulation materials. In: SPE Kuwait Oil & Gas Show and Conference. Society of Petroleum Engineers, Kuwait City, Kuwait, p. 7.
- Abraham, W.E.V., 1933. The Functions of Mud Fluids Used in Rotary Drilling, 1st World Petroleum Congress. World Petroleum Congress, London, UK, p. 5.
- Akinwunmi, B., Sun, L., Hirvi, J.T., Kasa, S., Pakkanen, T.A., 2019. Influence of temperature on the swelling pressure of bentonite clay. Chem. Phys. 516, 177–181.
- Al Menhali, S., Abdul Halim, A.O., Al Menhali, S., 2014. Curing Losses while Drilling & Cementing, Abu Dhabi International Petroleum Exhibition and Conference. Society of Petroleum Engineers, Abu Dhabi, United Arab Emirates.
- Ali, A., Kalloo, C.L., Singh, U.B., 1994. A practical approach for preventing lost circulation in severely depleted unconsolidated sandstone reservoirs. SPE Drill. Complet. 9, 32–38.
- Alkinani, H.H., Al-Hameedi, A.T., Dunn-Norman, S., Al-Alwani, M.A., Mutar, R.A., Al-Bazzaz, W.H., 2019. State-of-the-Art review of lost circulation materials and treatments Part I: general trends and uses. In: Abu Dhabi International Petroleum Exhibition & Conference.
- Alsaba, M., Nygaard, R., Hareland, G., Contreras, O., 2014a. Review of Lost Circulation Materials and Treatments with an Updated Classification. American Association of drilling engineers Fluids technical conference and exhibitionHouston, Texas, United States of America.
- Alsaba, M.T., Nygaard, R., Saasen, A., Nes, O.-M., 2014b. Lost circulation materials capability of sealing wide fractures. In: SPE Deepwater Drilling and Completions Conference. Society of Petroleum Engineers, Galveston, Texas, USA.
- Bag, R., Rabbani, A., 2017. Effect of temperature on swelling pressure and compressibility characteristics of soil. Appl. Clay Sci. 136, 1–7.
   Boukadi, F., Vachi, B., Al-Hadrami, H., Bemani, A. L., Babadadi, T., De Mestre
- Boukadi, F., Yaghi, B., Al-Hadrami, H., Bemani, A.L.I., Babadagli, T., De Mestre, P., 2004. A comparative study of lost circulation materials. Energy Sources 26, 1043–1051.

- Bullard, J.W., Jennings, H.M., Livingston, R.A., Nonat, A., Scherer, G.W., Schweitzer, J. S., Scrivener, K.L., Thomas, J.J., 2011. Mechanisms of cement hydration. Cement Concr. Res. 41, 1208–1223.
- Caenn, R., Chillingar, G.V., 1996. Drilling fluids: state of the art. J. Petrol. Sci. Eng. 14, 221–230.
- Davidson, E., Richardson, L., Zoller, S., 2000. Control of Lost Circulation in Fractured Limestone Reservoirs. IADC/SPE Asia Pacific Drilling Technology, Society of Petroleum Engineers, Kuala Lampur, Malaysia.
- Delage, P., Tessier, D., 2021. Macroscopic effects of nano and microscopic phenomena in clayey soils and clay rocks. Geomech. Energy Environ. 27, 100177.
- Diaz-Rodriguez, J.A., Santamarina, J.C., 1999. Thixotropy: the case of Mexico city soils. In: XI Panamerican Conference on Soil Mechanics and Geotechnical EngineeringIguaza Falls, Brazil, pp. 441–448.
- Dong, S., Zhang, J., Wang, Y., Fang, G., Liu, Y., Xing, F., 2016. Evolutionary trace for early hydration of cement paste using electrical resistivity method. Construct. Build. Mater. 119, 16–20.
- Fam, M.A., Santamarina, J.C., 1996. Study of clay-cement slurries with mechanical and electromagnetic waves. J. Geotech. Eng. 122, 365–373.
- Feng, Y., Gray, K.E., 2017. Review of fundamental studies on lost circulation and wellbore strengthening. J. Petrol. Sci. Eng. 152, 511–522.
- Feng, Y., Gray, K.E., 2018. Introduction, Lost Circulation and Wellbore Strengthening. Springer International Publishing, Cham, pp. 1–8.
- Feng, Y., Jones, J.F., Gray, K.E., 2016. A review on fracture-initiation and -propagation pressures for lost circulation and wellbore strengthening. SPE Drill. Complet. 31, 134–144.
- Ferguson, C.K., Klotz, J.A., 1954. Filtration from mud during drilling. J. Petrol. Technol. 6, 30–43.
- Finger, J., Blankenship, D., 2010. Handbook for Best Practices for Geothermal Drilling. Sandia National Laboratories, Albuquerque, New Mexico, United States.
- Gleason, M.H., Daniel, D.E., Eykholt, G.R., 1997. Calcium and sodium bentonite for hydraulic containment applications. J. Geotech. Geoenviron. Eng. 123, 438–445.
- Hansbo, S., 1957. New Approach to the Determination of the Shear Strength of Clay by the Fall-Cone Test. Royal Swedish Geotechnical Institute, Stockholm.
- He, C., Makovicky, E., Osbaeck, B., 1996. Thermal treatment and pozzolanic activity of Na- and Ca-montmorillonite. Appl. Clay Sci. 10, 351–368.
- He, C., Makovicky, E., Osbæck, B., 2000. Thermal stability and pozzolanic activity of raw and calcined mixed-layer mica/smectite. Appl. Clay Sci. 17, 141–161.
- Howard, G.C., Scott Jr., P.P., 1951. An analysis and the control of lost circulation. J. Petrol. Technol. 3, 171–182.
- Kabir, A.H., 2001. Chemical water & gas shutoff technology an overview. In: SPE Asia Pacific Improved Oil Recovery Conference. Society of Petroleum Engineers, Kuala Lumpur, Malaysia, p. 14.
- Kang, Y., Xu, C., Yu, H., Tang, L., 2015. An optimal design for millimeter-wide facture plugging zone. Nat. Gas. Ind. B 2, 113–119.
- Katsioti, M., Katsiotis, N., Rouni, G., Bakirtzis, D., Loizidou, M., 2008. The effect of bentonite/cement mortar for the stabilization/solidification of sewage sludge containing heavy metals. Cement Concr. Compos. 30, 1013–1019.
- Kaufhold, S., Dohrmann, R., Ufer, K., 2020. Determining the extent of bentonite alteration at the bentonite/cement interface. Appl. Clay Sci. 186, 105446.
- Kolstad, D.C., Benson, C.H., Edil, T.B., 2004. Hydraulic conductivity and swell of nonprehydrated geosynthetic clay liners permeated with multispecies inorganic solutions. J. Geotech. Geoenviron. Eng. 130, 1236–1249.
- Kulkarni, S.D., Jamison, D.E., Teke, K.D., Savari, S., 2016. Managing suspension characteristics of lost-circulation materials in a drilling fluid. SPE Drill. Complet. 30, 310–315.
- Lagaly, G., Ziesmer, S., 2003. Colloid chemistry of clay minerals: the coagulation of montmorillonite dispersions. Adv. Colloid Interface Sci. 100–102, 105–128.
- Lagaly, G., 2006. Chapter 5 colloid clay science. In: Bergaya, F., Theng, B.K.G., Lagaly, G. (Eds.), Developments in Clay Science. Elsevier, pp. 141–245.
- Laidani, Z.E.-A., Benabed, B., Abousnina, R., Gueddouda, M.K., Khatib, M.J., 2020. Potential pozzolanicity of Algerian calcined bentonite used as cement replacement: optimisation of calcination temperature and effect on strength of self-compacting mortars. Eur. J. Environ. Civil Eng. 1–23.
- Langmuir, I., 1938. Repulsive forces between charged surfaces in water, and the cause of the Jones-Ray effect. Science 88, 430–432.
- Lavrov, A., 2016. Lost Circulation: Mechanisms and Solutions. Gulf Publishing Company, US.
- Liu, L., 2013. Prediction of swelling pressures of different types of bentonite in dilute solutions. Colloids Surf. A Physicochem. Eng. Asp. 434, 303–318.
- Liu, Q., Santamarina, J.C., 2018. Mudcake growth: model and implications. J. Petrol. Sci. Eng. 162, 251–259.
- Liu, S.Y., Du, Y.J., Han, L.H., Gu, M.F., 2008. Experimental study on the electrical resistivity of soil-cement admixtures. Environ. Geol. 54, 1227–1233.
- Llano-Serna, M.A., Contreras, L.F., 2020. The effect of surface roughness and shear rate during fall-cone calibration. Geotechnique 70, 332–342.
- Müller, C., 2005. Pozzolanic Activity of Natural Clay Minerals with Respect to Environmental Geotechnics, Technical Science. Swiss Federal Institute of Technology Zurich, Zurich.
- Madsen, F.T., Müller-Vonmoos, M., 1989. The swelling behaviour of clays. Appl. Clay Sci. 4, 143–156.
- Manić, V., Miljković, L., Stefanović, M., Kovačević, R., 1998. A Study of Na-Montmorillonite Additions Influence on Portland Cement Hydration by 1H T1 Relaxation. Springer Berlin Heidelberg, Berlin, Heidelberg, pp. 363–367.
- Masi, S., Molaschi, C., Zausa, F., Michelez, J., 2011. Managing Circulation Losses in a Harsh Drilling Environment: Conventional Solution vs. CHCD through a Risk Assessment. SPE Drilling and Completion.

#### A. Hafez et al.

#### Journal of Petroleum Science and Engineering 215 (2022) 110615

- Mesri, G., Olson, R.E., 1971. Mechanisms controlling the permeability of clays. Clay Miner. 19, 151–158.
- Messenger, J.U., McNiel Jr., J.S., 1952. Lost circulation corrective: time-setting clay cement. J. Petrol. Technol. 4, 59–64.
- Mitchell, J.K., Soga, K., 2005. Fundamentals of Soil Behavior, third ed. John Wiley & Sons, Hoboken, N.J.
- Pan, B., Yin, X., Iglauer, S., 2020. A review on clay wettability: from experimental investigations to molecular dynamics simulations. Adv. Colloid Interface Sci. 285, 102266.
- Pecini, E.M., Avena, M.J., 2013. Measuring the isoelectric point of the edges of clay mineral particles: the case of montmorillonite. Langmuir 29, 14926–14934.
- Phadnis, H.S., Santamarina, J.C., 2011. Bacteria in sediments: pore size effects. Géotech. Lett. 1, 91–93.
  Plee, D., Lebedenko, F., Obrecht, F., Letellier, M., Van Damme, H., 1990. Microstructure,
- permeability and rheology of bentonite cement slurries. Cement Concr. Res. 20, 45–61.
- Poon, C.S., Lam, L., Kou, S.C., Wong, Y.L., Wong, R., 2001. Rate of pozzolanic reaction of metakaolin in high-performance cement pastes. Cement Concr. Res. 31, 1301–1306.
- Rand, B., Pekenć, E., Goodwin, J.W., Smith, R.W., 1980. Investigation into the existence of edge—face coagulated structures in Na-montmorillonite suspensions. J. Chem. Soc., Faraday Trans. 1: Phys. Chem. Condens. Phases 76, 225–235.
- Ren, X.W., Santamarina, J.C., 2018. The hydraulic conductivity of sediments: a pore size perspective. Eng. Geol. 233, 48–54.
- Rinaldi, V.A., Clariá, J.J., 2016. Time dependent stress-strain behavior of bentonite slurries; effect of thixotropy. Powder Technol. 291, 311–321.
- Ryan, J., Akyabi, K., Saleh, M., Omgbu, J., Kustanto, S., Ivan, C., Mihalic, B., 2015. Successful application of reverse gunk pill to cure losses in basinal limestone with interbedded marl and shale formation drilling offshore Abu Dhabi. In: Abu Dhabi International Petroleum Exhibition and Conference. Society of Petroleum Engineers, Abu Dhabi, UAE, p. 9.
- Sánchez, L., Cuevas, J., Ramírez, S., Riuiz De León, D., Fernández, R., Vigil Dela Villa, R., Leguey, S., 2006. Reaction kinetics of FEBEX bentonite in hyperalkaline conditions resembling the cement–bentonite interface. Appl. Clav Sci. 33, 125–141.
- Sadegh Hassani, S., Amrollahi, A., Rashidi, A., Soleymani, M., Rayatdoost, S., 2016. The effect of nanoparticles on the heat transfer properties of drilling fluids. J. Petrol. Sci. Eng. 146, 183–190.
- Sanders, M.W., Scorsone, J.T., Friedheim, J.E., 2010. High-fluid-loss, high-strength lost circulation treatments. In: SPE Deepwater Drilling and Completion Conference. Society of Petroleum Engineers, Galveston, Texas.

- Santamarina, J.C., Klein, K., Fam, M., 2001. Soils and Waves: Particulate Materials Behavior, Characterization and Process Monitoring. Wiley, United States.
- Savari, S., Whiftill, D.L., Jamison, D.E., Kumar, A., 2014. A method to evaluate lostcirculation materials - investigation of effective wellbore-strengthening applications. SPE Drill. Complet. 29, 329–333.
- Silent, R.A., 1936. Circulation Losses, Drilling and Production Practice. American Petroleum Institute, New York.
- Sridharan, A., Choudhury, D., 2008. Computation of hydraulic conductivity of montmorillonitic clays by diffuse double layer theory. Int. J. Geotech. Eng. 2, 1–10. Sridharan, A., Rao, A.S., Sivapullaiah, P.V., 1986. Swelling pressure of clays. Geotech. Test J. 9, 24–33.
- Taklymi, S.M.Q., Rezaifar, O., Gholhaki, M., 2020. Investigating the properties of bentonite and kaolin modified concrete as a partial substitute to cement. SN Appl. Sci. 2, 2023.
- Taylor-Lange, S.C., Lamon, E.L., Riding, K.A., Juenger, M.C.G., 2015. Calcined kaolinite–bentonite clay blends as supplementary cementitious materials. Appl. Clay Sci. 108, 84–93.
- Tombácz, E., Szekeres, M., 2004. Colloidal behavior of aqueous montmorillonite suspensions: the specific role of pH in the presence of indifferent electrolytes. Appl. Clay Sci. 27, 75–94.
- van Olphen, H., 1955. Forces between suspended bentonite particles. Clay Clay Miner. 4, 204–224.
- Villar-Cociña, E., Valencia-Morales, E., González-Rodriguez, R., Hernández-Ruiz, J., 2003. Kinetics of the pozzolanic reaction between lime and sugar cane straw ash by electrical conductivity measurement: a kinetic–diffusive model. Cement Concr. Res. 33, 517–524.
- Wei, J., Gencturk, B., 2019. Hydration of ternary Portland cement blends containing metakaolin and sodium bentonite. Cement Concr. Res. 123, 105772.
- Wei, X., Li, Z., 2006. Early hydration process of Portland cement paste by electrical measurement. J. Mater. Civ. Eng. 18, 99–105.
- White, R.J., 1956. Lost-circulation Materials and Their Evaluation, Drilling and Production Practice. American Petroleum Institute, New York, New York, p. 8.
- Yi, H., Jia, F., Zhao, Y., Wang, W., Song, S., Li, H., Liu, C., 2018. Surface wettability of montmorillonite (0 0 1) surface as affected by surface charge and exchangeable cations: a molecular dynamic study. Appl. Surf. Sci. 459, 148–154.
- Yong, R., Warkentin, B., 1975. Soil Properties and Behviour. ElSevier, Amsterdam, The Netherlands.