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Original article

A new formulation for lightweight oil well cement slurry using a natural pozzolan

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Abstract:
Fluid loss during cementing operation in depleted reservoirs or deep wells of reservoirs with low breakdown pressure is a major concern for maintaining well integrity. This issue is usually handled by reducing the weight of cement slurry. Although various slurry formulations were proposed during the last decade, the cost and availability of required additives is still a concern, especially when the oil price is low. The objective of this study is to make lightweight cement slurry with a density of 105 lb/ft^3, using the combination of natural pozzolan and API class G cement. This study proposes a new lightweight cement formulation based on the optimum amount of a natural pozzolan and other additives. Based on the results of 24-hour compressive strength tests and free water volume, an optimum 30% of cement powder was replaced with pozzolan. Addition of more pozzolan into the slurry reduces its pumping even at room temperature. The bottom-hole condition was simulated by increasing the temperature to 150 °F, and chemical additives were used to maintain the rheological properties of this slurry. Fluid loss control agent, dispersant and retarder were used at optimum values 0.5, 0.08 and 0.05 (%bwoc), respectively. The compressive strength of the cement rock was monitored at 3, 7 and 30 days, reaching to 3528 lb/in^2 after 30 days.

1. Introduction

Cementing operation is one of the most sensitive and most important stages in the drilling and completion of oil and gas wells (Velayati et al., 2015). Failure in this operation could endanger the production from a well, and the resulting financial damage would be irreversible. Fluid loss is a common problem during drilling and cementing stages in depleted reservoirs or reservoirs with low breakdown pressure (Mehrabian and Abousleiman, 2017; Wang et al., 2017). It is advisable to reduce the hydrostatic pressure of the slurry column to prevent formation breakage and slurry loss. The reduction of the hydrostatic pressure of the slurry column can be accomplished by three approaches including multi-stage cementing, lightweight or ultra-lightweight cement slurry.

In the multi-stage cementing technique, cement job is completed in several steps, so the hydrostatic pressure of the slurry column on the formation decreases. The disadvantage of this technique is increasing in rig time, and later remedial works might also be required (Elmarsafani et al., 2007). Moreover, the multi-stage cement tool acts as a weak point in the cementing and could cause leakage in the casing (Mukhalalaty et al., 1999).

Portland cement is the main constituent of almost all drilling cement. It could be modified easily by proportionating its raw materials and changing their combination process. The type of cement produced is classified as type I, II, III, or V white cement based on American Society for Testing and Materials (ASTM) or class A, C, G or H based on American Petroleum Institute (API). The oil industry often purchases cement manufactured in accordance with API classification as published in API Spec 10A. As for class G, no additions other than calcium sulfate or water should be blended with clinker cement. Class G is available in moderate sulfate-resistant (MSR) and high sulfate-resistant (HSR) grades. A comprehensive discussion on properties of cement covered by API specification could be found in Smith (1990).

While a typical API class G cement slurry has a weight of 118 lb/ft^3, lightweight cement slurry weighs 90-110 lb/ft^3. The weight of cement slurry decreases by reducing the weight of either the liquid phase or the solid phase. The simplest way to decrease the weight of the cement slurry is to add water into it, but this excess water will dilute the slurry, causing the solid

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particles to separate and settle down. It also could reduce the compressive strength of the resulting cement rock. Therefore, this method is not reasonable, and it is suggested that other methods be used (Mata and Calabayan, 2016).

For lightening the cement slurry, oil-based fluids, water-extending additives, and lightweight additives can be used. In the first method, a liquid hydrocarbon such as kerosene is used and the light slurry is made using emulsification of hydrocarbon liquid in water and mixing it with cement. While the existence of oil in these slurries, improves the flowing property, the need for special tools limits the use of these slurries (Dumbauld et al., 1956). Gilsonite is a solid lightweight hydrocarbon, which could be used for reducing the weight of cement slurry. Since the Gilsonite slurry uses less water compared to other weighing slurries, the compressive strength of this slurry is higher. Gilsonite has a melting point in the range of 385-525 °F. Therefore, it can not be used in wells that have the potential to implement heat recovery techniques (Slagle and Carter, 1959).

Water-extending additives are substances that absorb a lot of water, causing the slurry to become lighter. The lowest density that can be obtained by using water-extending additives, while the cement has acceptable properties, is 93.5 lb/ft^3 (Kulakofsky et al., 2011). The use of water-extending additives for making ultra-lightweight cement slurry could reduce the compressive strength of the cement rock (Slagle and Carter, 1959). Bentonite is one of the water-extending materials which has been widely used in drilling mud formulation and could also be used to lighten drilling cement slurry. Since bentonite is capable of absorbing relatively high water content, it decreases the density of the slurry and increases its volume. Low resistance to detrimental downhole conditions such as corrosive water and high temperatures, could be considered as a disadvantage of bentonite slurry (Slagle and Carter, 1959).

Pozzolanic cement, hollow glass sphere (HGS) cement, and foam cement are some other innovations for lightening cement slurry, which could reduce loss circulation in depleted fields or in filed with low fracture gradient (Liu et al., 2018). Wang and coworkers (Wang et al., 2017) developed a novel self-generating nitrogen foam cement to prevent lost circulation. The proposed formulation was tested in a field and promising results were obtained. In another study, Wang et al. (2015) used a hydrophilic fiber to reinforce an ultralow density slurry, formulated by hollow glass sphere and class G cement. As observed by these researchers, this combination solved the lost circulation in cementing coal-bed methane wells.

Pozzolans are a broad class of inorganic siliceous and aluminous materials which upon reaction with calcium hydroxide generated during the cement hydration process could produce cementitious materials (Kutchko et al., 2009). Although the use of pozzolanic materials dates back many hundreds of years ago by ancient Romans and Greeks, their commercial use as an additive to Portland cement was started 50 years ago (Smith, 1956; Smith, 1990). The benefit of Portland-amended cement is twofold. The addition of pozzolan into Portland cement can not only reduce the cement slurry density, the initiation of pozzolanic reactions under long curing time also makes a cement rock very resistant to corrosive and high-temperature conditions (Slagle and Carter, 1959; Brandle et al., 2010; Zhang et al., 2014; Bihua et al., 2018). Different pozzolanic materials have been successfully used in the concrete composition. In a study by Ahmad et al. (2008), Palm Oil Fuel Ash (POFA), fly ash and Quarry dust, as three pozzolanic materials, were used in concrete and results were compared with a control sample. Based on their observations, 15% replacement of Portland cement with pozzolanic materials showed the best results and compressive strength of 6527 lb/in^2 (45 MPa) after 28 days of curing were achieved. The application of POFA to reinforce class G cement for drilling and completion operations was recently tested by Abid et al. (2019). As observed by these researchers, POFA could improve the workability of cement without disturbing the rheology of the cement slurry.

Fly ash is a synthetic pozzolan that has been used for a long time in the oil industry to lighten cement slurry (Fasesan et al., 2005). Since this material is a by-product of the thermal coal-fired power plants, its use is limited to countries with such industries. Volcanic ash is a natural pozzolan that could be found in most parts of the world. Natural pozzolan has been widely used in the construction industry, but its use in lightweight drilling cement slurries is very limited. It is partly due to the adverse effect of pozzolan addition on the rheological properties of the cement slurry. The main objective of this study is to formulate optimized cement slurry with a weight of 105 lb/ft^3 using a natural pozzolan while maintaining required rheological properties.

2. Materials and methods

2.1 Drilling cement

In this study, API class G cement with high resistance to sulfates, produced by Kerman Cement Factory, was used. Its physical properties and chemical analysis are presented in Tables 1 and 2, respectively.

2.2 Sirjan natural pozzolan

Natural pozzolan was acquired from a mine located near Sirjan in Kerman province (29° 53’ 39.0” N, 56° 04’ 57.0” E). This mine is located on the volcanic belt of Urmia-Bazman and consists mainly of volcanic, igneous and sedimentary rocks of Tertiary age (less than 60 million years old). The length of the belt is about 1,500 kilometers and its width is from 100 to 120 kilometers, which starts from the Sultan’s mountains in Pakistan and continues to Armenia. Sirjan pozzolan is a type of crystalline tuff with porphyritic and glassy texture. Plagioclase, amphibole, biotite and quartz crystals are observed in it.

The chemical analysis of this pozzolan is given in Table 3. According to Table 3 and ASTM C618-08a (2008), this po-

Table 2. Chemical analysis of API class G cement, used in this study.

% C3S		% C3A		% SO3	
Standard	Test	Standard	Test	Standard	Test
48-46	56.5	⩽ 3	2.3	⩽ 3	1.58

Table 1. Physical specifications of API class G cement, used in this study.

8-hour compressive strength (lb/in^2)				Free water (ml)		Density (g/cm^3)	
@ 100 °F		@ 140 °F					
Standard	Test	Standard	Test	Standard	Test	Standard	Test
$\geqslant$ 300	561	$\geqslant$ 1500	2018	$\leqslant$ 3.5	1.55	3.1-3.25	3.1424

Table 3. Chemical analysis of Sirjan natural pozzolan.

Components	L.O.I	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Cl
Percent (%)	7.46	59.22	16.74	4.00	6.85	0.95	0.060	3.46	1.91	0.036

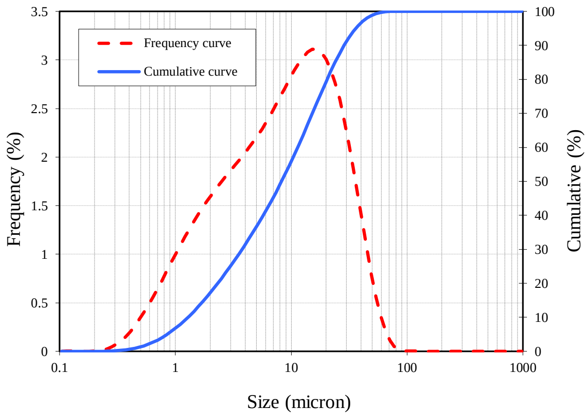

Fig. 1. Grain size distribution of Sirjan pozzolan.

zzolan is categorized as a natural pozzolan.

Also, the grain size distribution of Sirjan pozzolan, used in this study, is shown in Fig. 1. The grains have a geometric mean of 7.31 μm, with uniformity of 1.05 and specific surface area of 16906.97 cm^2/cm^3.

2.3 Chemical additives

Retarder, dispersant and fluid loss control additives were used in cement slurry formulation. Their characteristics are presented in Table 4.

2.4 Experimental procedure

2.4.1 Sampling and preparation of Sirjan pozzolan

Sampling from pozzolan depot was performed according to ASTM D75/D75M-14 (2014). The reference sample was first crushed using a jaw crusher. Since the reference sample was more than the amount required for the tests, the sample weight was reduced according to a standard procedure. There are three methods of weight reduction. Here, the quartering method was used according to ASTM C702/C702M-11 (2011). The sample obtained at the end of this method was placed in an oven at 230 °F for 24 hours to dry. It was then ground with a vibratory disc mill RS 100, made by Retsch, and was passed through a 90-micron sieve.

Table 4. Specifications of chemical additives used in this study.

Additive	COmmercial code	Density (g/cm^3)
Retarder	JR-120	1.2
Dispersant	O-CFR 4	1.4
Fluid loss control	FLC 320	1.4

2.4.2 Density measurement of solids

It is necessary to measure the density of the components of the slurry for calculating its wieght. At first, cement powder was passed through a US mesh 20, and then the density of this cement and natural pozzolan was measured using kerosene in compliance with ASTM C188-16 (2016).

2.4.3 Slurry preparation

Material balance calculations were made to determine the weight of each material in the slurry, based on the standard volume of the 600 ml. slurry (API RP 10B, 2013; API Spec 10A, 2010). The cement powder was first sieved using US mesh 20, then mixed with natural pozzolan and blended until a homogeneous solid mixture was obtained. Chemical additives were added into the water to make the liquid phase. After that, the solid phase (cement powder and Sirjan pozzolan) was added into the liquid phase while mixing at 4000 rpm in less than 15 seconds using a special mixer (Chandler Engineering). The blend was finally mixed for 35 seconds at 12000 rpm.

2.4.4 Free water test

When the slurry is left to the static state, the water may separate from the lower parts of the slurry column and migrate upward. This separation can damage the zonal isolation, which is one of the important goals of cement job (Al-Yami et al., 2008, 2010). In this test, in order to simulate the dynamic conditions of the cement replacement inside the well, the slurry is conditioned by an atmospheric pressure consistometer for 30 minutes at 80 °F. After that, in accordance with API standard, the slurry is poured into a 250 ml cylinder and the separated water from the slurry is measured after 2 hours (API RP 10B, 2013).

2.4.5 Rheology test

The slurry was conditioned in an atmospheric pressure consistometer at 150 °F, before measuring rheological parameters using a standard viscometer (model 3500, Chandler Engineering). In accordance with API standard, the plastic viscosity and yield point values were calculated using relations 1 and 2, respectively (API RP 10B, 2013):

$$\mu_p = 1.5 \cdot F \cdot (\theta_{300} - \theta_{100}) \tag{1}$$

where μ_p is plastic viscosity, F is spring factor (dimensionless), θ_{300} is dial reading at the rotational speed of 300 rpm (degree) and 100 is dial reading at the rotational speed of θ_{100} rpm (degree).

$$\tau_0 = F \cdot \theta_{300} - \mu_p \tag{2}$$

here, τ_0 is yield point (lb/100 ft^2).

2.4.6 Compressive strength

The prepared slurry was poured into standard 2×2×2 inch cubic molds and was cured at 140 °F in accordance with API standard (API Spec 10A, 2010). After a specified time, which depends on the aim of the experiment, cement cubes were crushed under a hydraulic press and the uniaxial compressive strength for these cubes was calculated. A Toni Technik (model Tonizem) hydraulic press machine was used for crushing cement rock cubes. In this study, 8-hour, 24-hour, 3-day, 7-day and 30-day compressive strength was measured on different cement blocks.

3. Results and discussions

3.1 The density of cement powder and pozzolan

The density test results for cement powder and Sirjan pozzolan are given in Table 5. According to Table 5, the pozzolan density is less than that for cement powder. Therefore, it is expected that slurry density decreases when replacing a part of cement powder with Sirjan pozzolan.

Based on the results obtained, the density of the Sirjan natural pozzolan (2.43 g/cm^3) is almost the same as the density of fly ash (2.5 g/cm^3) used by Purvis and Merritt (2003).

3.2 The optimum amount of Sirjan pozzolan

The amounts of 10, 30, 50 and 77 %bwoc (based on the weight of cement) from pozzolan were mixed with drilling cement powder for making slurry at 105 lb/ft^3. When the amount of pozzolan was 77 %bwoc or 50 %bvob (based on the volume of bulk solid), the slurry was gelled, implying that adding this amount of pozzolan or more will damage the rheology of the cement slurry. Purvis and Merritt (2003) used more than 50 %bvob of fly ash to make slurry with a density of nearly 105 lb/ft^3. This difference could be mainly attributed to the difference in the shape of the particles. Fly ash particles are spherical, which helps to facilitate slurry flow and improve rheology. Therefore, using its large amounts in the slurry does not cause much damage to rheology. However, the particles of natural pozzolan, used in this study, have irregular shapes and that is might be the reason why slurry containing 77 %bwoc of pozzolan was rejected.

Table 5. Density of solid materials.

Solid Materials	Density (g/cm^3)
API class G cement powder	3.14
Sirjan natural pozzolan	2.43

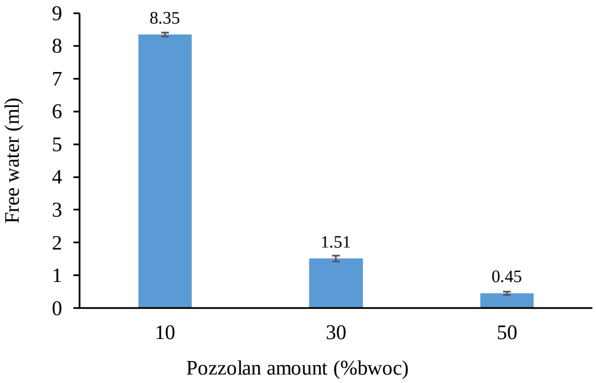

Fig. 2. Free water volume of pozzolanic slurries.

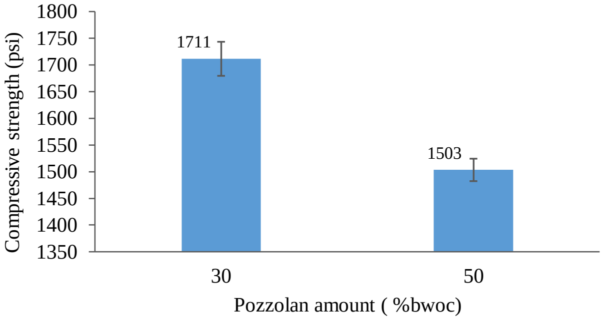

Fig. 3. 24-hour compressive strength of pozzolanic slurries at 100 °F.

The free water test was then carried out for pozzolanic slurry at 80 °F and the results are shown in Fig. 2. As shown, the free water volume for slurry containing 10% pozzolan was 8.35 ml, while the acceptable free water volume for API class G cement is less than 3.5 ml, so this composition was also rejected. Based on different applications, the acceptable free fluid could change; however, excessive free water damages the quality of the cement sheath. In the following, a uniaxial compressive strength test was performed for the two remaining compounds.

Pozzolanic slurries were cured for 24 hours at 140 °F and the resulting cement rock was crushed by a hydraulic press. The compressive strength results of these slurries are shown in Fig. 3. Slurry containing 30% of pozzolan has higher compressive strength, so it was selected as an optimal amount.

The cement sheath should be strong enough to hold the casing. While a compressive strength of 100 lb/in^2 is usually enough to keep casings in place, 500 lb/in^2 is mostly considered as a criterion for casing support (Al-Yami et

Table 6. Rheology results of cement slurry at 150 °F.

Pozzolan (%bwoc)	Cement (%bwoc)	Fluid loss control (%bwoc)	Retarder (%bwoc)	Dispersant (%bwoc)	Plastic viscosity (cp)	Yield point (lb/100ft^2)
30	100	0.5	0.05	0.08	30	6

Table 7. Weight percent of solids in pozzolanic and neat slurries.

Slurry number	API class G cement (%bwoc)	Sirjan pozzolan (%bwoc)	Dispersant (%bwoc)	Retarder (%bwoc)	Fluid loss control (%bwoc)
1	100	0	0	0	0
2	100	30	0	0	0
3	100	30	0.08	0.05	0.5

al., 2008) and a permission to continue drilling operations. Based on Fig. 3, the 24-hour compressive strength of the Pozzolanic slurry exceeds the proposed values of 100 and 500 lb/in^2. This indicates that the pozzolanic slurry has a sufficient compressive strength to hold the casing in place.

Purvis and Merritt (2003) and Fasesan et al. (2005) developed cement slurries with densities of about 105 lb/ft^3 using 50 %bvob or more of fly ash. In this study, the cement slurry with the same density was made using 30 %bwoc (or 28 %bvob) of natural pozzolan. Since the density of natural pozzolan and fly ash are almost equal, the reason for the difference in the used volume of these materials is the higher water absorption of natural pozzolan compared to fly ash.

The slurry weight and its rheology are two of the most crucial parameters in well cementing job. Since rheology of the slurry is sensitive to the interaction of the additives, the rheological control could be more difficult than weight control. The lead cement slurry should have enough viscosity to move the mud from the annulus upward. For cement slurry with specified viscosity, the additives and size of cement particles are effective.

Based on our observations, the slurry containing 30% of the pozzolan was gelled at 150 °F, so it was not possible to measure its rheological properties. This problem was resolved with the help of chemical additives. The results of the slurry rheology along with the chemical additive used are given in Table 6. According to Table 6, the values of 0.5, 0.05 and 0.08 %bwoc from fluid loss control, retarder and dispersant agent were added to slurry, respectively. These amounts are optimum and were obtained through intensive laboratory measurements. Plastic viscosity and yield point for this slurry were measured 30 cp and 6 lb/100ft^2, respectively.

Three samples were prepared to compare free water and compressive strength of pozzolanic slurry with that of neat slurry. The details of these three slurries are reported in Table 7.

The results of free water content and 24-hour compressive strength at 140 °F for these samples are shown in Fig. 4 and Fig. 5, respectively. According to Fig. 4, the free water volume of the pozzolanic slurry is similar to that of neat cement slurry, but the amount of free water was increased with the addition of chemical additives in slurry number 3. This is mainly due to the lower water absorption of chemical additives compared to the natural pozzolan. Although the amount of free water increased, the slurry is still in the standard range of free water.

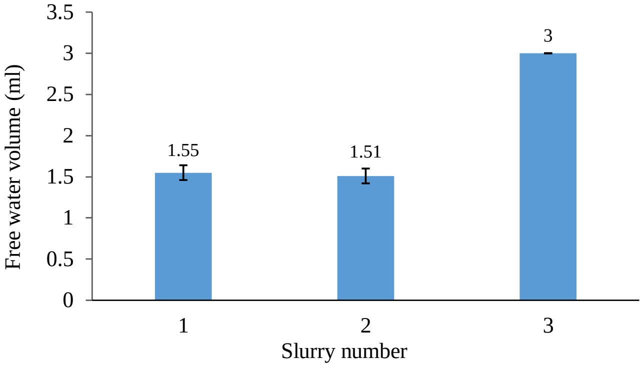

Fig. 4. Comparison of free water content between pozzolanic slurry 2 (30 %bowc pozzolan without rheology-control additives) and slurry 3 (30 %bwoc pozzolan with rheology-control additives) and neat slurry 1.

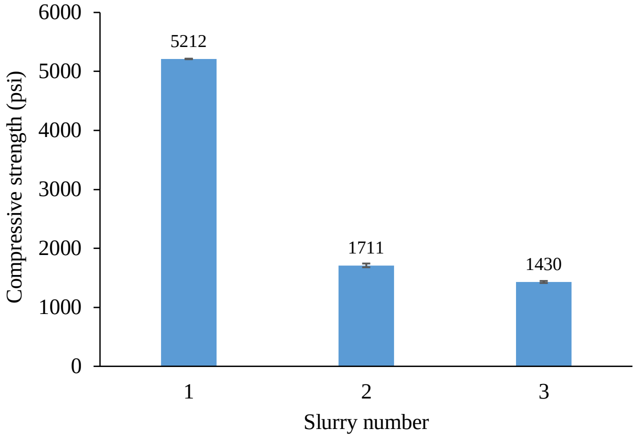

Fig. 5. Comparison of compressive strength between pozzolanic slurry 2 (30 %bowc pozzolan without rheology-control additives) and slurry 3 (30 %bwoc pozzolan with rheology-control additives) and neat slurry 1.

According to Fig. 5, the compressive strength of a pozzolanic slurry with a weight of 105 lb/ft^3 is lower than neat

API class G cement slurry. In pozzolanic slurry, the amount of drilling cement is about half of that in the neat slurry, making the reduction of compressive strength predictable. This conclusion is in agreement with the study of Brandl et al. (2010), who reported reduction in the compressive strength of fly ash contained cement slurry. Moreover, by adding chemical additives, the compressive strength of the pozzlanic slurry was decreased more, which is due to the retarding effect of the chemical additives.

3.3 Long-term compressive strength of pozzolanic and neat slurry

The strength development of the cement rock will not end with thickening of the slurry, but will continue for months, even years after that. In order to better understand the behavior of compressive strength at more than 24 hours, it was decided to examine the strength of the cement rock after 3, 7, and 30 days in compliance with studies performed by (Benge et al., 1982; Harms and Sutton, 1983; De Rozieres and Ferriere, 1991; Al-Yami et al., 2010, 2012).

The hydrated calcium silicate and the portlandite (Calcium hydroxide) are mainly formed during the cement hydration period (reaction 3 and 4).

$$2\,C_3S + 6\,H \rightarrow C_3S_2H_3 + 3\,CH \tag{3}$$

$$2\,C_3S + 4\,H \rightarrow C_3S_2H_3 + CH \tag{4}$$

Hydrated calcium silicate (C-S-H) is responsible for the strength of the cement rock. Portlandite, on the other hand, not only does not contribute to the strength of the cement rock, it is also a weak point in the cement matrix. Pozzolanic substances have SiO_2 (and Al_2O_3) compounds that react with portlandite and form further C-S-H phases (or C-A-S-H phases), which increases the strength of the cement rock (reaction 5).

$$Ca(OH)_2 + SiO_2(Al_2O_3) \rightarrow C{-}S{-}H(C{-}A{-}S{-}H)\ \text{phases} \tag{5}$$

Based on reactions 3, 4 and 5, while cementitious reactions are first initiated, pozzolanic reactions will occur later. In other words, the pozzolanic reactions show their effects better over time (Brandle et al., 2011).

To better understand the behavior of pozzolan-amended cement over time, a comparison on compressive strength of neat and pozzolanic cement was performed and the result is shown in Fig. 6. The amount of cement used in the neat slurry is greater than the pozzolanic slurry. Therefore, cementitious reactions will be higher in the neat slurry. For the same reasons and according to Fig. 6, in the first 24 hours, the strength development rate of neat slurry is greater than that of the pozzolanic slurry. After that, due to the activation of the pozzolanic reactions, the difference in the development rate between the two cement slurries is decreased.

More detailed results of long-term compressive strength are reported in Table 8. As shown, after three days, the compressive strength of the pozzolanic slurry reaches 2589 lb/in^2, which is higher than what standard requires.

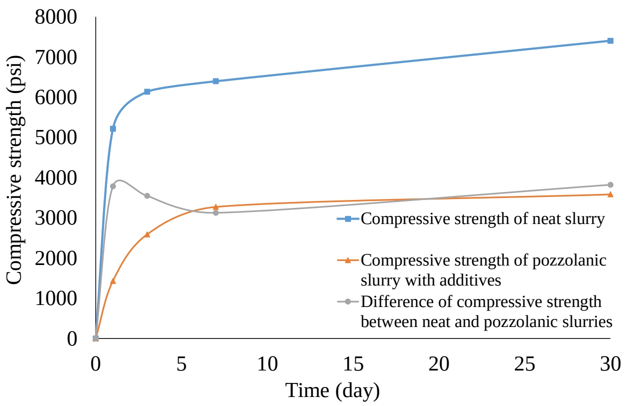

Fig. 6. Long-term compressive strength of pozzolanic and neat slurries.

Table 8. The results of long-term compressive strength (lb/in^2).

Slurry number	1 day	3 days	7 days	30 days
1	5212	6135	6396	7404
3	1430	2589	3271	3582

Brandl et al. (2010) reported a three-day compressive strength of 2529 lb/in^2 for a slurry containing fly ash with the same density to the pozzolan we used here. Curing temperature is one of the important parameters in the compressive strength of cement slurry so that, with other parameters remaining constant, the compressive strength of the slurry increases with increasing temperature. Although the curing temperature for pozzolanic slurry was 40 °F less than the slurry containing the fly ash made by Brandl et al. (2010), the compressive strength of the two slurries is similar. Safe production of wells after perforation requires the final compressive strength of at least 1500 lb/in^2 under bottomhole condition (Putra et al., 2016). As reported in Table 8, the proposed pozzolanic slurry has reached to this required strength.

4. Conclusions

In this study, we tried to design a lightweight cement slurry with a weight of 105 lb/ft^3 using a natural pozzolan. The main findings obtained are as follow:

- The optimal amount of Sirjan natural pozzolan for making lightweight cement slurry with a weight of 105 lb/ft^3 is 30 %bwoc (or 28 %bvob).
- The excessive addition of natural pozzolan to slurry with specified weight results in more water absorption and will reduce pumping of the slurry.
- The lightweight pozzolanic slurry has a lower compressive strength compared to the normal API class G cement slurry.
- When using pozzolanic slurry at the circulating temperatures of 150 or more, it is essential to use chemical additives to improve rheology.
- In the short term, the compressive strength development

for pozzolanic slurry is less than the neat API class G cement slurry, but over time, due to the activation of pozzolanic reactions, this difference is reduced.

- The replacement of a part of cement powder with a natural pozzolan in slurry composition could be considered as an important step in reducing costs and more importantly in protecting the environment by reducing the CO_2 emission.

Nomenclature

Abbreviations

API = American Petroleum Institute
ASTM = American Society for Testing and Materials
bvob = based on volume of bulk
bwoc = based on weight of cement
HGS = Hollow Glass Sphere
HSR = High Sulfate-Resistant
MRS = Moderate Sulfate-Resistant
POFA = Palm Oil Fuel Ash

Variables

μ_p = Plastic viscosity, cp
θ_{300} = Dial reading at the rotational speed of 300 rpm, degree
θ_{100} = Dial reading at the rotational speed of 100 rpm, degree
F = Spring factor, dimensionless
τ_0 = Yield point, lb/100 ft^2

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