New Lightweight Cement Formulation for Shallow Oil and Gas Wells

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ABSTRACT: The use of lightweight pozzolanic aggregates as partial replacement of cement results in low-density cement systems. Such systems ensure effective zonal isolation in zones where low equivalent circulating densities are required. However, low pozzolanic materials, such as fly ash and ground granulated blast furnace slag (GGBFS), have poor early-age strength development and long set times, especially when used in high volume, that is, exceeding 50% by weight of cement. The objective of this study is to develop a lightweight oil- and gas-well cement recipe with enhanced properties employing the synergism that exist among fly ash, GGBFS, and silica fume. The experimental work was per laboratory procedure outlined by American Petroleum Institute. Portland class G cement and the aluminosilicate materials were admixed in water to form a 13.5 ppg slurry. Chemical admixtures were used to facilitate the dissolution of reactive components in the pozzolanic materials and the hydration process. The experimental investigations were done at 150 °F and an ambient pressure of 1500 psi. The newly developed lightweight recipe exhibited excellent rheological and mechanical properties, having a wait-on-cement time for about 4 h and a 24 h sonic strength of 3116 psi, at 150 °F and 1500 psi. The thickening time was approximately 4 h (70 Bc). This slurry will be ideal in zones that would require a low hydrostatic slurry column and rapid gel strength development.

1. INTRODUCTION

Well cementing is the most important operation during drilling and completion of oil and gas wells. A good cement job ensures efficient zonal isolation and protection of casing.1,2 Based on prevailing wellbore conditions and cementing objectives, different admixtures are incorporated into the cement slurry design to achieve an efficient cement bond.3 Cement slurries prepared with just water and cement, commonly known as neat cement, may have densities in the range of 15.6–16.4 ppg.4 In low fracture gradient formations, high hydrostatic pressure as a result of increased cement slurry density could result in formation breakdown and subsequent loss of circulation.5 Traditionally, the multistage tool is used to mechanically isolate lost circulation intervals; however, the method presents several challenges.6 To overcome this, lightweight cement systems are used to lower the equivalent circulating density.7

Lightweight cements could have thixotropic properties. Thixotropic cements possess the ability to develop static gel strength within the shortest possible time.8,9 Notwithstanding their immense role in curbing loss of circulation, thixotropic cement formulas are not without challenges. The thixotropic behavior of some cement slurries are weak and have been observed to decrease with an increasing temperature, while some thixotropic additives have a negative impact on the mechanical properties of the hardened paste.10 According to Hillery et al.,11 other thixotropic slurries tend to lose their properties as a result of shear activities during pumping. Additionally, due to their extremely viscous nature, these cement systems often require pump pressures that exceed the operating pressures of surface equipment.

This study is primarily about lightweight cement slurries. Such cement systems, with densities in the range of 4–13.5 ppg, can be designed through nitrogen gas injection,12,13 the addition of water extenders like bentonite and sodium silicate,14 or replacing proportions of cement with low-specific gravity materials, such as microspheres, fly ash, silica fume, and ground granulated blast furnace slag (GGBFS).15,16 Over the years, several studies have been conducted to investigate the properties of various lightweight cement recipes.1,17–23 However, laboratory findings and field applications have shown that many lightweight cement slurries have increased wait-on-cement (WOC) times and low early-age strength, increasing the chances of gas migration and delaying post-cementing operations, such as cement bond logging.24

The use of industrial and agro-waste in cement-based systems was initiated by the quest to mitigate carbon dioxide emissions associated with the production of Portland cement.24 Industrial byproducts, such as fly ash, GGBFS, and silica fume, possess characteristics that make them useful as
As pozzolans, they combine with the calcium hydroxide produced as a result of cement hydration to form additional cementitious materials, enhancing the mechanical properties of the hardened paste.\textsuperscript{27} Generally, cement systems formulated with fly ash or GGBFS have higher ultimate strength, improved resistance to chemical attack, and alkali–silica expansion.\textsuperscript{28} However, such systems have low early-age strength due to their low pozzolanic activity.\textsuperscript{29} Additionally, they are unsuited in highly permeable formations since they do not possess thixotropic properties.

Techniques such as mechanical activation have been done to improve the reactivity of such low pozzolanic materials.\textsuperscript{30} This study presents a new lightweight recipe that combines the properties of aluminosilicate materials, namely, fly ash, GGBFS, and silica fume, allowing for the incorporation of low reactive pozzolans in high amounts (70\% BWOC). The objective is to develop a lightweight cement formula with a high pozzolanic activity, thereby improving the early-age mechanical properties of the hardened paste.\textsuperscript{27} Table 1 shows the characteristics of fly ash-based cement systems.

| Sample | Fly ash, \% | Density, \textbf{PPG} | 3 Day Crush Strength, psi | 28 Day Crush Strength, psi |
|--------|-------------|------------------------|---------------------------|---------------------------|
| Cement + 0\% fly ash | 0 | 16.50 | 3244 | 5095 |
| Cement + 25\% fly ash | 25 | 15.20 | 3169 | 6118 |
| Cement + 50\% fly ash | 50 | 14.60 | 2099 | 5604 |

The density of the neat slurry system was 16.50 ppg. The density is proportional to the increasing fly ash content. There were 7.8 and 11.52\% reduction in densities for 25 and 50\% fly ash replacement, respectively. The 3-day compressive strength of the fly ash-based systems was lower than the control system designed with 0\% fly ash. The strength of the hardened cement containing 25\% fly ash was 97.69\% of the base, while that containing 50\% fly ash was 64.70\% of the base. The crush strength re-evaluated after 28 days showed higher strength for the fly ash-based systems. The percentage increase from 3 to 28 days for the base slurry, 25\% fly ash system, and 50\% fly ash system were 57, 93, and 167\% respectively. These results indicate that fly ash-based systems when used in cement replacement result in density reduction; however, such systems have low early-age strength, especially when admixed in higher replacement volumes.

Many studies have demonstrated that silica fume due to its high pozzolanic activity enhances the early-age performance of cement composites and displays a synergistic effect when combined with fly ash.\textsuperscript{32–36} Abo-El-Enein et al.\textsuperscript{36} observed that a ternary cement system composed of cement, fly ash, and silica fume showed higher strength in the long term. A study by Li and Zhao\textsuperscript{37} indicated that blended cement systems composed of both GGBFS (15\%) and fly ash (25\%) exhibited enhanced early-age and long-term performance in comparison to cement composite composed of only fly ash. Other investigations have reported that GGBFS and silica fume combine synergistically in cement systems to improve the mechanical properties of hardened cement.\textsuperscript{38–40}

The synergistic effect that exists among fly ash, silica fume, and GGBFS was therefore employed in developing the high-volume low-pozzolanic-based lightweight cement slurry.

### 2.2. Fresh Properties of the Lightweight Cement System

The objective is to design a lightweight system with enhanced early-age mechanical properties. The density and specific gravity of the slurry measured at ambient conditions using the pressurized fluid density balance is given in Table 2.

| Sample | Density, \textbf{ppg} | Specific Gravity |
|--------|------------------------|------------------|
| LWS | 13.5 | 1.62 |

The rheological behavior of the fluids was modeled after the Bingham plastic fluid model. The Bingham parameters, plastic viscosity and yield strength, and the gel strength values are presented in Table 3. The measured values indicated good slurry pumpability.

| Sample | Plastic Viscosity, \textbf{cP} | Yield Stress, \textbf{lb/100 ft}² | Gel Strength, \textbf{lb/100 ft}² | 10 s | 10 min |
|--------|-------------------------------|-----------------------------|--------------------------------|------|--------|
| LWS | 20 | 16.41 | 8.35 | 26.73 |

**Figure 1.** Effect of temperature on gel strength.
circulation formulation presented by Miranda et al. Therefore, it implies that this inherent gel behavior of the developed lightweight slurry might be due to the presence of the GGBFS.

A record of shear stress for increasing and decreasing shear rate when plotted yields a hysteresis loop, the shear-lag phenomenon, which gives a qualitative approach to study thixotropic behavior (Figure 3). The area under the loop denotes thixotropic behavior. It is a demonstration that the collapsed system takes a while to rebuild in the absence of stress. The maximum shear stress from the lowest to the highest investigated temperatures are 43.39, 39.58, 37.69, 35.19, and 34.33 lb/100 ft². It is observed that the shear stress decreased with the increasing temperature. This is because, at lower temperatures, the slurry viscosity is high, and hence high shear is required to cause deformation in the fluid.

2.2.2. Thickening Time. The thickening time is a very important parameter as it controls how long the cement slurry will remain pumpable. The key objective here is to have the slurry in a fluid state within reasonable time limits. A hasty setting can result in well abandonment, while an elongated setting promotes fluid invasion that weakens the cement bond. The results from the thickening time experiments are shown in Table 4. The times to reach the Bearden consistency units of 50 and 70 Bc were noted. The results show that the newly developed lightweight cement exhibits good thickening behavior for shallow interval applications. The times to attain 50 and 70 Bc were within the range of 3 h and 20 mins to 4 h.

2.2.3. Free Water Test. The free water test was performed at ambient conditions using a 250 mL graduated cylinder. The slurry was allowed to stand for 2 h as required by the API. The result as shown in Table 5 indicates that the newly developed slurry has minimal free water.

| sample | 50 Bc | 70 Bc |
|--------|-------|-------|
| LWS    | 3:22  | 3:55  |

2.3. Evaluation of Mechanical Properties of the Lightweight Slurry. The mechanical properties of the

Table 5. Free Water Test

| sample | volume of free water, mL |
|--------|--------------------------|
| LWS    | 0.4                      |

Figure 2. Thixotropic behavior at 175 °F.

Figure 3. Shear-lag behavior.
hardened lightweight cement composite determined were compressive strength, tensile strength, Young’s modulus, and Poisson’s ratio. The ultrasonic cement analyzer (UCA) provides a means of monitoring the strength development with time, especially at an early age. The results for the initial set time, WOC time, and 24 h compressive strength are shown in Table 6. The newly formulated lightweight slurry showed great strength development, with WOC time in about 4 h and a high 24 h compressive strength.

Table 6. Strength Development with Time

| sample | time to 50 psi (initial set time), hours:minute | time to 500 psi (wait on cement), hours:minute | 24 h sonic strength, psi |
|--------|----------------------------------|----------------------------------|-----------------|
| 1.WS   | 2:27                             | 3:48                             | 3116            |

The cylindrical samples formed after 24 h under curing conditions of 150 °F and atmospheric pressure conditions were used to assess the other mechanical properties of the hardened cement. High tensile strength and Poisson’s ratio and low Young’s modulus provide flexibility to the cement, which helps reduce the chances of cement sheath failure caused by stresses generated as a result of fluctuating pressure and temperature conditions in the wellbore. The values recorded for the mechanical properties are sufficient to ensure high early-age strength and flexibility of the cement sheath, Table 7.

Table 7. Mechanical Properties

| tensile strength, psi | Young’s modulus, psi | Poisson’s ratio |
|-----------------------|----------------------|-----------------|
| 199.43                | 1.7 × 10⁶            | 0.32            |

The compressive strength of other similar lightweight systems presented in the literature is shown in Table 8. The results show that the strength measured with the newly developed slurry outperforms these lightweight systems.

2.4. SEM and EDS Analyses of Hydrated Product. The images of the microstructure and chemical analysis using EDS are shown in Figure 4. The micrograph revealed the formation of cementitious gel and unhydrated grains. Some partially hydrated grains (P) were present. No portlandite was present in the structure. This is probably because of the low amount of Portland cement and enhanced pozzolanic activity. The results of the average weight percent of major elements and the Ca/Si, Al/Ca ratios from four separate EDS analyses are shown in Table 9. The key elements present in the hydrated product are calcium (Ca), silicon (Si), and aluminum (Al). The EDS shows a high amount of calcium and silicon, indicating the formation of C−S−H gel. However, there is also aluminum (3.4%), which would be taken up in the C−S−H structure, leading to the formation of C−A−S−H gel.

3. CONCLUSIONS

Due to the low pozzolanic reactivity of fly ash and GGBFS, their quantities in cement slurry design are limited. To use these materials in high volumes, a form of activation is often required, for example, mechanical activation through grinding. In this study, the synergistic effect that occurs among fly ash, GGBFS, and silica fume is employed. This has allowed for the design of lightweight cement slurry using low pozzolanic materials (fly ash and GGBFS) in high volume (70% BWOC). This is a low-carbon footprint slurry. Compared to other proposed lightweight systems, the developed slurry shows superior compressive strength. The laboratory investigations revealed that the proposed 13.5 ppg lightweight cement system exhibits good rheological behavior, minimal free water, reasonable thickening and WOC times, and high early-age mechanical properties. Additionally, the cement system exhibits intrinsic thixotropic behavior. These attributes make this slurry system an ideal formulation for many lost circulation conditions.

4. MATERIALS AND METHODS

4.1. Materials. The materials used in slurry preparations are class G cement, fly ash, GGBFS, and silica fume, Figure 5.

Table 8. Compressive Strength of Other Lightweight Systems

| lightweight materials | density, ppg | curing conditions, °F/psi | 24 h compressive strength, psi | 72 h compressive strength, psi | authors |
|-----------------------|--------------|----------------------------|-------------------------------|-------------------------------|---------|
| sodium metasilicate   | 13.5         | 140                        | 1278 (crush)                  | 46                           |
| water glass           | 13.6         | 140                        | 1450 (crush)                  | 46                           |
| diatomaceous Earth    | 13.5         | 140/3000                   | 710                           | 1000                          | 46      |
| cenosphere            | 13.1         | 104/1450                   | 3012 (sonic strength)         | 30                            | 21      |
| type F fly ash: cement (50:50) | 13.5 | 150                       | 1452 (crush strength)         | 30                            | 30      |
| type F fly ash: cement (50:50) | 13.5 | 150                       | 1349 (crush strength)         | 30                            | 30      |
| expanded perlite      | 13.5         | 100/3000                   | 650                           | 46                            |

“Strength type unstated.
cement contains about 67% CaO, which is the key contributor to its hydraulic activity. Other key oxides present in the cement include SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$.

The XRD patterns of the raw materials are from 2-theta angles from 4$°$ to 80$°$, Figure 8. The presence of a broad hump at 2-theta angles of 25$°$–37$°$ for GGBFS and 19$°$–25$°$ for silica fume, and the absence of clearly defined peaks in their XRD patterns indicate that the materials have an amorphous structure; however, certain crystalline phases like quartz, moissanite 3C, hematite, and andalusite were present. The fly ash shows crystalline peaks attributed to mullite and quartz phases. Hematite, hatrurite (alite), portlandite, and calcite are the main minerals in the class G cement.

4.2. Method. The experimental procedure was according to API specifications. The slurries were prepared using the 15 s/4000 rpm and subsequent 35 s/12,000 rpm mixing rule. A preliminary study was conducted, replacing Portland cement with 25 and 50% fly ash. The samples were cured in a water bath for 3 and 28 days at 105 $°$F and ambient pressure. The 28 day strength is due to the hydration of the tricalcium silicate.

Table 9. Elemental Composition of Hydrated Gel

| element | Si | Al | Ca | Al/Si | Ca/Si | Al/Ca |
|---------|----|----|----|-------|-------|-------|
| weight percent | 8.2 | 3.4 | 14.7 | 0.4 | 1.8 | 0.23 |

Table 10. Summary of the PSD of Raw Materials

| powder      | $D_{10}$, μm | $D_{50}$, μm | $D_{90}$, μm |
|-------------|--------------|--------------|--------------|
| silica fume | 3.70         | 18.59        | 36.64        |
| GGBFS       | 2.46         | 16.85        | 45.56        |
| fly ash     | 2.61         | 17.91        | 60.05        |
| cement      | 4.86         | 25.45        | 63.36        |
phase present in the cement. The 3 day strength was used to assess strength development at a much earlier age. The mix proportions are listed in Table 12. Different combinations of aluminosilicate materials, namely, fly ash, GGBFS, and silica fume, were dry-mixed with cement and mixed in water to obtain a final slurry density of 13.5 ppg, Table 13. Chemical admixtures were used in the final recipe to design slurry (lightweight slurry, LWS). The final proportion of materials and additives used in preparing the LWS is listed in Table 14. The fly ash and GGBFS, low pozzolanic materials, were used in high volume, a total amount of 70% by weight of blend (BWOB), that is, 45% fly ash and 25% GGBFS.

The analytical characterization of the raw materials and hydrated products was done using X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), and electron dispersive X-ray spectroscopy (EDS). The particle size distributions of the raw grains were studied with the Helos particle size analyzer. The thickening time was measured with the atmospheric consistometer supplied by Grace Instrument. The Grace M3600 viscometer and M3600DAQ software were used to determine the rheological

### Table 11. Chemical Composition of Raw Materials

| chemical oxides | weight percent |
|-----------------|----------------|
| SiO₂            | 19.84          |
| Al₂O₃           | 3.31           |
| Fe₂O₃           | 8.08           |
| CaO             | 67.25          |
| MgO             | 0.82           |
| P₂O₅            | 0.06           |
| MnO             | 0.06           |
| ZnO             | 0.06           |
| TiO₂            | 0.31           |
| Cr₂O₃           | 0.02           |
| NiO             | 0.19           |
| SrO             | 0.19           |

### Table 12. Mix Proportions for the Preliminary Study

| sample          | fly ash, % BWOB | water, % BWOB | antifoam, % BWOB |
|-----------------|-----------------|---------------|-------------------|
| cement + 0% fly ash | 0               | 44            | 0.05              |
| cement + 25% fly ash | 25              | 44            | 0.05              |
| cement + 50% fly ash | 50              | 44            | 0.05              |

### Table 13. Density Formulations

| fly ash, % BWOB | GGBFS, % BWOB | silica fume, % BWOB | cement, % BWOB | water, % BWOB | density, ppg |
|-----------------|---------------|---------------------|----------------|---------------|--------------|
| 25              | 50            | 50                  | 25             | 44            | 14.60        |
| 25              | 50            | 25                  | 25             | 48            | 14.40        |
| 50              | 25            | 25                  | 25             | 48            | 13.80        |
| 50              | 25            | 50                  | 25             | 50            | 13.75        |
| 45              | 25            | 5                   | 25             | 55            | 13.50        |

Figure 8. XRD pattern of raw materials.
behavior of the slurry. The ultrasonic cement analyzer (UCA) was used to monitor the strength development with time under curing conditions of 150 °F/1500 psi. The slurry was also cured in a water bath for 24 h at 150 °F/ambient pressure conditions for analyses of the tensile strength and elastic properties. The Brazilian test was used for the tensile strength measurement. The elastic properties (Young’s modulus and Poisson’s ratio) were evaluated using ultrasonic waves.

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