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Analysis of the Impact of Vermiculite on Hematite-Based Cement Systems

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ABSTRACT: Heavyweight oil-well cement systems are designed for isolating intervals and supporting the casing at deeper depths where high temperatures and pressures are encountered. The cement slurry should have adequate rheology to ensure efficient placement. Additionally, the hardened cement sheath should be homogeneous with lower porosity and permeability, higher strength, and sufficient flexibility. The effect of vermiculite on hematite-based cement samples has been investigated. The methodology and testing were based on the American Petroleum Institute standards and other recognized recommendations. Fluid properties were characterized by their rheology, while petrophysical and mechanical properties were used to analyze the properties



of hardened cement specimens. The vermiculite was used in concentrations of 0.25, 0.5, 1, and 2% by weight of cement (BWOC). The slurries were cured at 3000 psi and 292 °F in cubic and cylindrical molds for 24 h. The results indicate that using 1% BWOC of vermiculite yields the best cement properties. It minimizes the settling of hematite particles to a very low value compared to the base cement as shown by the method of density variation and confirmed by nuclear magnetic resonance. Compared to the base cement slurry, the slurry of 1% BWOC of vermiculite has desirable rheology in terms of plastic viscosity and gel strength. The incorporation of 1% BWOC improves the strength of the cement sheath by 50.7% for the compressive strength and 65% for the tensile strength. Adding 1% vermiculite reduces the permeability and porosity of the cement by 45.8 and 43.5% compared to the control cement. In addition, the 0.7% vermiculite cement is more flexible than the control cement in terms of the elastic properties represented by lower Young's modulus (a reduction of 33%) and higher Poisson's ratio (an increase of 2%).

1. INTRODUCTION

Since the cement sheath integrity greatly affects the overall wellbore integrity, the optimal design of cement systems through the selection of the appropriate admixtures is key in oil-well cement slurry formulation. To achieve effective zonal isolation, the density of the cement system is fine-tuned to meet the prevailing wellbore conditions. For instance, to control the wellbore fluids and remove the remaining dense drilling mud in deep HPHT wells, the density of the well cement must be increased. The heavyweight cement systems are formulated either by reducing the water-to-cement ratio or using heavyweight agents such as barite and hematite.¹ The simplest method is by reducing the water-to-cement ratio. However, there is a big challenge in maintaining good rheological properties. Furthermore, this method cannot achieve a density of 18 ppg or higher. Hence, to obtain higher density, weighting agents are mostly used.

One of the major challenges in formulating heavyweight systems is the segregation of heavyweight particles under dynamic and static wellbore conditions.² This issue results in a heterogeneous cement column that adversely affects the integrity of the cement sheath.^{3,4} Several studies have

investigated and proposed several suggestions to reduce cement segregation and enhance the cement sheath performance. Some authors proposed some anti-settling materials like perlite⁵ and laponite,⁶ while other studies modified the weighting materials either by decreasing their particles size or mixing different weighting materials.⁷

This existing work seeks to examine the influence of vermiculite on heavyweight cement systems, especially on the stability of such systems. Vermiculite is a clay mineral formed by the decomposition of micas.⁸ It is formed through the alteration of micaceous minerals, which are varieties of mixtures of several minerals such as phlogopite and hydrobiotite because of percolating groundwater, hydrothermal action, weathering, or an amalgamation of these three factors.⁹

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Vermiculites have a structured layer where the interlayer has water molecules and exchangeable cations, primarily magnesium ions. They have hydrated magnesium silicate, iron, and aluminum.¹⁰ The vermiculite has a lightweight density with a light brown or golden color.¹ When vermiculite is exposed to a high temperature between 650 and 1000 °C, its flakes expand to 8–30 times their original size as a result of the change of structure and interlayer water to steam.^{11–13} Their grain composition, shape, luster, and color mainly depend on the original raw material. Vermiculite's capacity for water absorption rises extremely when the bulk density differs from 64 to 160 kg/m³ based on the particle size.¹⁴ The world's greatest quantities of vermiculite are located in South Africa (41%), the United States (around 27%), China (21%), and Brazil (11%).^{14,15}

According to Koksal et al.,¹⁴ the presence of vermiculite in Portland cement yielded systems with increased mechanical strength and thermal resistance. da Silva Araujo Filho et al.¹⁶ studied the effect of vermiculite on the microstructural, mechanical, and physical characteristics of lightweight well cementing. They investigated the effect of 5, 7, and 9% vermiculite in class G cement. They indicated that vermiculite had a great influence on strength development. Minaev et al.¹⁷ evaluated the influence of vermiculite on lightweight cement properties. The amount of vermiculate used was between 10 and 15%, and the water-to-cement ratio varied from 0.65 to 0.8. They reported that the consistency of the slurry decreased with increasing vermiculite content. Bubnov et al.¹⁸ analyzed the rheological properties, fluid loss, gel strength, and thickening time of the lightweight cement slurry designed with 5, 7, and 8% exfoliated vermiculite. They concluded that vermiculite was compatible with the used chemical admixtures.

Based on the conducted literature and authors' best of knowledge, there are no works that evaluated the effect of vermiculite on the strength, petrophysical, elastic, and rheological properties of heavyweight cement. Recently, Ahmed et al.¹⁹ proposed the use of vermiculite in minimizing the segregation of barite in heavyweight mud systems. This work investigates vermiculite as a novel additive to minimize cement segregation due to hematite weighting materials and improve cement properties for effective and safe cementing operations. The influence of using different amounts of vermiculite on cement segregation propensity is studied by the density variation (DV) method and nuclear magnetic resonance (NMR) technique. The compressive and tensile strengths, rheology, porosity, permeability, Poisson's ratio, and Young's modulus are used to characterize the cement systems developed with vermiculite at different concentrations.

2. MATERIALS AND METHODS

2.1. Characterization of Raw Materials. The foremost materials utilized in this work are vermiculite, hematite, and class G cement in addition to other cement admixtures. A slurry density of approximately 18 ppg was designed for this investigation. The elemental composition of the raw materials characterized using the M4 Tornado X-ray fluorescence device (Bruker) is given in Table 1. The hematite is predominantly iron (Fe), with a small amount of aluminum (Al). The vermiculite contains Si, Al, Fe, and magnesium (Mg) as key components, while calcium is the major element in the cement.

The specific gravities of the cement, hematite, and vermiculite used in this study are 3.21, 5.05, and 2.65, respectively. The particle size distribution (PSD) of the

Table 1. Elemental Composition of Raw Materials

elemental composition	hematite, %	vermiculite, %	cement, %
Na	0.2	0	0
Al	0.75	14.1	2.37
Si	0.5	34.9	12.1
Cl	0.32	0	0
K	0.21	0	0
Ca	0.03	0	72.1
Ti	0.01	0	0.39
Fe	95.84	6.24	9.08
Rh	2.91	0	0
S	0	0	2.43
Ba	0	0	0
Sr	0	0	0.15
Mg	0	20.4	1.33

vermiculite, hematite, and cement as characterized using the D_{10} , D_{50} , and D_{90} sizes is presented in Table 2. The analysis indicates that vermiculite has coarser grains while hematite is comparatively finer.

Table 2. PSD of Vermiculite, Hematite, and Cement (μm)

size	cement	hematite	vermiculite
D ₁₀	4.78	2.62	1.95
D ₅₀	23.06	16.86	23.18
D ₉₀	58.08	50.63	129

The morphology of the raw materials as seen in the secondary imaging mode using the scanning electron microscope (SEM) is shown in Figure 1. All the materials depict an irregularly shaped morphology.

2.2. Methodology. *2.2.1. Slurry Formulation.* Five cement slurries were prepared in total according to the American Petroleum Institute (API) standards.²⁰ The control slurry was prepared without vermiculite. It is composed of class G cement, 32.9% by weight of cement (BWOC) of hematite, 35% BWOC of silica flour, 0.5% BWOC of fluid loss additive, 0.25% BWOC of dispersant, 1.5% BWOC of retarder, 4.7 × 10^{-7} % BWOC of defoamer, and 44% BWOC of water. The vermiculite-based cement formulations were prepared with 0.25, 0.5, 1, and 2% BWOC of vermiculite.

The slurries were conditioned in an atmospheric consistometer for approximately 30 min at 194 °F. Rheological characterization was performed immediately at the end of the conditioning period. Subsequently, the fresh slurries were poured into cubical and cylindrical molds and cured at 3000 psi and 292 °F. The cubical molds have dimensions of 50 mm \times 50 mm \times 50 mm, while the cylindrical molds have a diameter of 1.5 in. and a length of 4.0 in. All samples were cured for 24 h.

2.2.2. Density Variation. The density discrepancy across the cement column due to hematite was evaluated to study the influence of vermiculite particles on the settling of hematite particles. To analyze the homogeneity of the hardened cement samples, cylindrical cement cores were used for this test. The cores were cut into three sections (upper, lower, and middle), and the density of each section was computed. Then, the ratio of the density difference between the upper and lower sections of the sample to the density at the lower section was calculated to represent the DV along the cement sample. Moreover, the



Figure 1. Morphology of (a) hematite, (b) vermiculite, and (c) cement.



Figure 2. Effect of vermiculite on the DV of the cylindrical cement samples.





NMR equipment was used to investigate the pore size distribution of the three cut sections.

2.2.3. Strength Measurement. The compressive and tensile strengths were used to characterize the mechanical performance of the hardened cement specimen. The samples were crushed uni-axially based on the American Society for Testing and Materials (ASTM) standard.²¹ The Brazilian test was used for the tensile strength investigation on 1.5 in. diameter by 0.75 in. thickness of cylindrical cement cores.²²

2.2.4. Petrophysical Measurement. To determine how the vermiculite affected the petrophysical characteristics of the hematite-weighted cement specimens, the porosity and permeability of all the specimens taken into consideration in this work were assessed. For the evaluation of petrophysical parameters, samples with dimensions of 1.5 in. in diameter and

1.0 in. in thickness were employed, as explained by Ahmed et al. $^{\rm 23}$

2.2.5. Elastic Measurement. Young's modulus and Poisson's ratio were used to characterize the flexibility/ elasticity of all cement samples using cylindrical samples with a length of 4.0 in. and a diameter of 1.5 in. They were determined through ultrasonic analysis, as explained by Ahmed et al.²⁴

3. RESULTS AND DISCUSSION

3.1. Density Variation. The results of the DV along the vertical column of the cylindrical cement samples are shown in Figure 2. The figure compares the top and bottom samples of the same specimen. It is observed that there is great variation in the density of all samples, except that developed with 1%

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Figure 4. Effect of vermiculite on plastic viscosity.



Figure 5. Impacts of vermiculite on yield point and 10 s gel strength.



Figure 6. Influence of vermiculite on compressive and tensile strengths.

vermiculite. The inclusion of 1% vermiculite shows an insignificant DV between the sections, indicating a homogeneous system. The pore size distribution of the control specimen and that composed of 1% vermiculite was investigated using the NMR equipment to obtain a microscopic understanding of the observed phenomenon. The results are shown in Figure 3. The differences between the NMR pore size distribution function (PDF) and cumulative distribution function (CDF) curves for the two sections of the base cement sample (0% BWOC of vermiculite) are conspicuous, and it confirms the remarkable DV between the top and bottom sections of the base cement sample. Contrarily, it confirms that the addition of vermiculite yields a homogeneous system, indicated by the uniform porosity profile across the different sections. This shows that with a 1% vermiculite concentration, stable heavyweight cement systems can be designed. It should be mentioned that the vermiculite particles have a large effect on the density due to the high surface colloidal particles of vermiculite, which may be responsible for the resulting reduction in DV. The primary factor influencing this colloidal activity is water absorbability, and vermiculite clay is known for being exceptionally water absorbing.

3.2. Rheology Measurement. Rheological properties (plastic viscosity, yield point, and gel strength) of the five cement slurries were measured to evaluate the effect of vermiculite on the flow properties of the cement. The Bingham plastic model was used to analyze the rheology of the slurries. The plastic viscosity investigations are reported in Figure 4. The plastic viscosity increases with increasing vermiculite



Figure 7. Effect of vermiculite on the porosity and permeability.



Figure 8. Impact of vermiculite on Young's modulus and Poisson's ratio.

concentration up to 0.5%. An increase in the vermiculite concentration by 1% results in a decrease in the plastic viscosity to a value of 148 cP, representing a 10.80% reduction in the viscosity compared to the control. However, the plastic viscosity increases to 153.8 cP at 2% vermiculite. A lower plastic viscosity of the cement slurry is recommended because its resistance to flow and pumping requirements are lower.²⁵

The yield point and gel strength evaluations are shown in Figure 5. The gel strength is an important parameter under static conditions as it characterizes the interparticle attraction under non-flow conditions, while the yield stress defines the attractive forces between the particles under flow conditions.²⁶ Additionally, yield stress defines the initial resistance that must be overcome to initiate flow.²⁷ Even though the 1 and 2% vermiculite improves the gel strength in comparison to the control sample, the yield stress is relatively higher. However, the yield stress can be improved with the addition of a dispersant if it is beyond an unacceptable threshold. The improvement in slurry rheology contributed to the improvement in cement stability. For instance, the velocity of solids settlement was reduced, while the plastic viscosity was kept as low as possible. The improved yield point and gel strengths provided sufficient solids suspension capability at both dynamic and static conditions, emphasizing the effects of DV.

3.3. Strength Measurement. The strength of the control and vermiculite-based cement samples is characterized by both compressive and tensile strengths, as shown in Figure 6. Both the compressive and tensile strengths are highly improved by the vermiculite, indicated by the comparatively higher compressive and tensile strengths of all the vermiculite-based samples. The concentration of 1% BWOC of vermiculite yields the highest compressive strength, with a compressive strength of 79.4 MPa (50.7% higher than the base cement) and a tensile strength of 11.5 MPa (65% higher than the base cement). It is

recommended to have high compressive strength as it ensures more support to the wellbore and casing. Also, high tensile strength is recommended to resist the tension forces and to carry the casing weight.

3.4. Petrophysical Measurement. The petrophysical properties characterized by porosity and permeability (Figure 7) were examined to determine the impact of vermiculite on these properties. Figure 7 reveals that both porosity and permeability of the cement samples reduce as the amount of vermiculite increases. However, the porosity and permeability values in the 1% vermiculite sample are lower than those in the specimen prepared with 2% BWOC of vermiculite. Comparing the values from the 1% vermiculite sample to the base sample, the porosity decreases by 25.5% while the permeability is lowered by 43.5%. Low petrophysical properties ensure effective zonal isolation.

3.5. Elastic Measurement. The elastic properties such as Young's modulus and Poisson's ratio were measured to determine the effect of vermiculite addition on the elasticity of the cement samples. The results are shown in Figure 8. Increasing the amount of vermiculite decreases Young's modulus of the cement sample to a value of 15 GPa at 1% BWOC of vermiculite and then increases dramatically to 22.6 GPa at 2% BWOC of vermiculite. Poisson's ratio of the cement samples increases with the increase of vermiculite concentrations, obtaining a value of 0.263 at 1% BWOC of vermiculite, and drops to 0.251 when 2% BWOC of vermiculite is used. Adding 1% BWOC of vermiculite reduces Young's modulus of the cement by 33% compared to the 0% vermiculite and improves Poisson's ratio by 2% compared to the base cement. A lower Young's modulus and higher Poisson's ratio are recommended to provide long-term cement integrity by improving its resistance against the high stresses in the wellbore.

4. CONCLUSIONS

The effect of vermiculite inclusion in hematite heavyweight cement systems designed at 18 ppg has been investigated. The parameters investigated include stability (DV), rheology, porosity, permeability, strength, and elastic properties. The study indicates that the optimum concentration of vermiculite (1% BWOC) has better cement properties than the base cement as follows:

- Vermiculite improves the stability of the hematite heavyweight cement through a 68.5% reduction in the settling of heavyweight particles.
- Vermiculite enhances the rheology of the cement slurry through a 10.8% decrease in the plastic viscosity, a 19.1% increase in the yield point, and a 25% increase in the gel strength.
- Vermiculite increases the strength of the cement sheath represented by 50.7 and 65% increases in compressive and tensile strengths, respectively.
- Vermiculite reduces the petrophysical properties of the cement represented by lower permeability and porosity (reduction of 43.5 and 25.5%).
- Vermiculite has better elasticity in terms of lower Young's modulus (33% reduction) and higher Poisson's ratio (2% increase).

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