

Development of Heavy-Weight Hematite-Based Geopolymers for Oil and Gas Well Cementing

Ahmed Abdelaal, Salaheldin Elkatatny,* and Ahmed Mohsen Abd El Fattah



Cite This: *ACS Omega* 2023, 8, 14025–14033



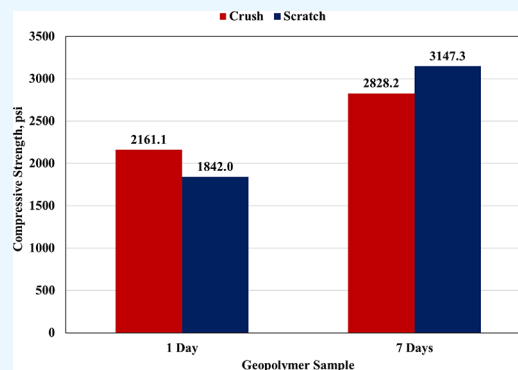
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ABSTRACT: In the petroleum industry, ordinary Portland cement (OPC) is utilized for different cementing applications. Yet, there are some technical and environmental issues for the usage of OPC in well cementing. The technical problems include gas invasion while setting, instability at corrosive environments, cement failure while perforation and fracturing due to high stiffness and brittleness, and strength reduction and thermal instability at elevated temperatures. Moreover, OPC production consumes massive energy and generates high greenhouse gas emissions. This study introduced the first hematite-based class F fly ash geopolymer formulation that can be used in oil and gas well cementing. Different properties of the designed slurry and hardened samples such as rheology, thickening time, strength, and elastic and petrophysical properties were evaluated. Moreover, mixability and pumpability challenges of heavy-weight geopolymer slurries were investigated. Unlike most of the studies in the literature, this work used 4 M NaOH solution only as an activator that can reduce the overall cost. The results showed that increasing the hematite percentage significantly decreased the thickening time. The developed formulation fell within the recommended fluid loss ranges for some cementing applications without using a fluid loss control additive. A proposed mixture of retarder and superplasticizer was introduced to enhance the thickening time by almost 5 times. The compressive strength increased by 49% and the tensile strength was enhanced by 27.4% by increasing the curing time from 1 to 7 days. The improvement in both compressive and tensile strength with curing time indicated that the geopolymerization reaction continued for extended time but with a smaller rate. The developed slurry acted more like a power law fluid at low temperatures and more like a Bingham plastic fluid at high temperatures. The elastic properties of the developed geopolymer samples proved that they are more flexible than some cement systems.



1. INTRODUCTION

1.1. OPC Challenges. Although OPC is the most extensively used substance for well cementing, it has some technical issues, especially in deep formations, and environmental concerns. The technical issues include gas invasion while setting, instability at corrosive environments, thermal instability at high temperatures, cement failure while perforation and fracturing due to high stiffness and brittleness, and strength reduction at temperatures above 230 °F.^{1,2} Gas migration may take place for different reasons such as cement channeling through mud, lost circulation during cementing, poor mud cake removal, and microannulus formation at the cement/pipe and/or cement/borehole wall interfaces.³ Stress cracking occurs in hard-set, brittle cements that are not designed for certain cyclic well loads (temperature and pressure changes).³ When thermal operations such as steam injection take place, the casing expands due to heat, and this may put extra stress on cement. This increased stress may cause cement to develop cracks around its circumference.⁴ OPC-based systems are vulnerable to acid attacks.^{5,6} Harsh environments, such as acidic or high saline conditions, can cause cement to degrade, ultimately causing

cement and well to lose their integrity. It is crucial that the cement sheath is effective at creating a barrier in these types of environments. However, when Portland cement systems are exposed to high levels of CO₂, they may deteriorate due to carbonation and the formation of unfavorable substances, which can lead to corrosion and leakage of CO₂.^{7–9} When mud contacts cement slurry, the compressive strength of OPC is significantly affected.^{10,11} Additionally, mud has adverse effects on OPC rheological properties as it increases cement slurry viscosity that affects mud pumpability.¹² Moreover, OPC introduces some environmental challenges such as high greenhouse gas (GHG) emissions as the OPC production process is considered one of the major sources of GHG. OPC

Received: January 29, 2023

Accepted: March 23, 2023

Published: April 4, 2023



production takes place through a reaction of calcium carbonate (lime) and silicon dioxide to produce calcium silicates and CO₂. Additionally, massive energy consumption is required during cement production. These challenges persuaded the researchers to search for alternative materials to overcome the technical challenges and to provide more eco-friendly cement systems. Lately, different researchers investigated the effects of various materials on cement properties such as olive waste,⁷ nano clay,¹³ granite sludge,¹⁴ polypropylene fibers,¹⁵ perlite¹⁶ and meta-kaolin.¹⁷ On the other hand, some researchers tried to totally replace OPC through exploring new materials that can form cementitious binders such as geopolymers.^{18–21}

1.2. Geopolymers. Geopolymer technology has a wide range of uses, particularly in the construction industry.²² However, full-scale deployment in well cementing has yet to be seen. Researchers in the field of cementing are currently examining the characteristics of several geopolymers under wellbore conditions.²⁰ Geopolymers are produced by the geopolymerization of aluminosilicate substances dissolved in alkali hydroxides and/or soluble silicates at room temperature or high temperatures, forming an amorphous to semicrystalline 3D silicoaluminate network structure.^{23–25} Geopolymerization refers to geosynthesis, i.e., synthesis of chemically integrated minerals.²⁶ The geopolymerization reaction forms viscous cementitious slurry that, after hardening, creates a strong, durable, and compact geopolymeric substance.²⁷

In comparison to OPC, the geopolymer production process is cleaner, and the source materials do not consume that much energy.^{28–31} Geopolymers use some industrial and agro-wastes like FA, slag, silica fume, coconut ash, and rice husk that makes geopolymer eco-friendly in terms of low CO₂ manufacturing emissions and using industrial byproducts as a source of aluminosilicate materials. Moreover, clays like metakaolin or some rocks like aplite can be used to form certain types of geopolymers. The geopolymer systems exhibit superior performance to conventional cement systems. Low shrinkage, low permeability, low Young's modulus (YM), strength development, stability at high temperatures, tolerance to oil-based mud contamination, chemical corrosion resistance, and long-term durability make geopolymer systems a viable option to OPC in well cementing applications.^{6,32}

1.3. High Density Cement. Cement slurry is designed depending on the existing wellbore conditions of pressure and temperature and cement job type. High-density cement slurry with a density of 17 ppg or more should be utilized in deep wells or across high-pressure zones.³³ Decreasing water content is the cheapest approach to increase the density.³⁴ However, American Petroleum Institute (API) advises a water-to-cement ratio of 44% for primary cementing (API, 2019). The main drawback of reducing the water content is the challenge of instantaneously attaining sufficient control of fluid loss, good rheological properties, and no settling of solid particles.³⁵ Slurry bridging occurs because of uncontrollable fluid losses. Solid particle settling results in nonuniform compressive strength and bonding along the length of cemented section.³⁶ Therefore, using weighting materials is required to achieve a higher density. These substances are commonly utilized in mud to overbalance high formation pressure formations. It is necessary to use weighting agents such as hematite, Micromax, and ilmenite in cementing operations as deep wells are characterized by high pore pressures.³⁷

Limited research was conducted for using only barite in geopolymer areas that included compressive strength and

thickening time experiments. Kanesan et al.³⁸ estimated the thickening time of a barite-based fly ash geopolymer at 140 °F and 2000 psi. The authors used a combination of 8 M NaOH solution and Na₂SiO₃. They concluded that the retarder effect was significant in low- and medium-density slurries, but it slightly affected the high-density slurry thickening time. They mentioned that barite affected the retarder ability to form a layer around the reacting precursor particles. Salehi et al.³⁹ studied the effect of adding barite (15% BWOC) on the compressive strength of the FA-based geopolymer. Barite improved the strength within the first 7 days of curing. However, barite could not improve the strength after 7 days. It is clear from the literature that rare experiments were conducted using only barite as a weighting material with geopolymers for oil well cementing.

1.4. Fly Ash. Fly ash is an industrial byproduct generated from coal burning. According to ASTM C618, FA can be classified into two classes (F & C) based on the CaO content. Class F ash is distinguished by its low CaO content and produced by burning bituminous coal. Conversely, class C fly ash is characterized by its high calcium content and results from lignite and sub-bituminous coal burning. FA is a widely available byproduct that is often used in preparation of geopolymers. FA has been used since the early twentieth century, and it is typically used as a major component in concrete.⁴⁰ Using FA instead of OPC minimizes GHG emissions and lowers construction expenses. FFA has the advantages of being inexpensive, readily available, having a spherical structure, and being rich in high activity alumina and amorphous silicate.³²

This work evaluates the possibility of using hematite and fly ash to develop a new heavy-weight geopolymer formulation for oil and gas well cementing. The biggest challenge in this work was developing a flowable heavy-weight geopolymer formulation with good rheological properties and sufficient thickening time. Different properties such as rheology, filtrate loss, compressive and tensile strength, petrophysical properties, and elastic properties (Poisson's ratio (PR) and Young's modulus (YM)) were assessed for the high-density hematite-based geopolymer system. This work used only 4 M NaOH as an alkaline activator, while most of the work in the literature used a combination of a high molarity NaOH and Na₂SiO₃ to form geopolymers.

2. MATERIALS AND METHODOLOGY

2.1. Materials. The materials used in this study were FFA as an aluminosilicate material, hematite as a weighting agent, and NaOH solution as an activator, besides other chemical additives used to enhance geopolymer characteristics and facilitate using geopolymers in a wide range of wellbore conditions. The additives included retarders, deformers, and superplasticizers. The specific gravities (SG) of the FFA and hematite were 2.25 and 5.05, respectively. The particle size distributions (PSD) of the FFA and hematite were obtained using a laser diffraction particle size analyzer, as shown in Figure 1. The results showed that 50% of FFA and hematite particles had size below 19.35 and 21.54 μm, respectively.

Figure 2 shows the elemental composition of the hematite, which was obtained using a Bruker M4 Tornado X-ray fluorescence (XRF) instrument. XRF results confirm that hematite has a high iron amount (around 95%), and X-ray diffraction (XRD) showed that it contained 100% hematite. XRF also showed that FFA has considerable amounts of silica (SiO₂) and alumina (Al₂O₃), as shown in Table 1, which play a vital role in geopolymer formation. The SEM images, as shown

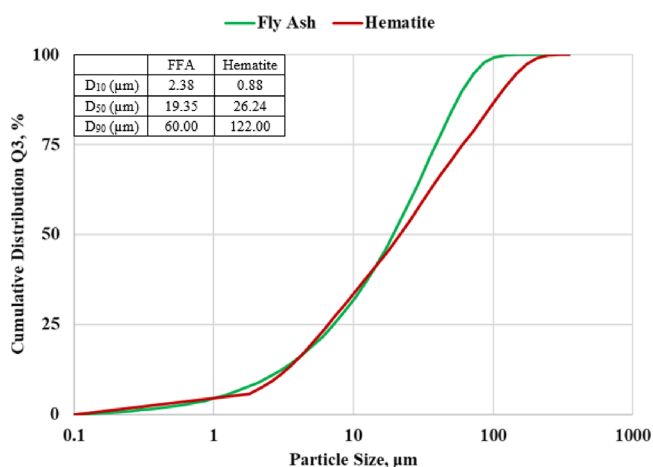


Figure 1. PSD results of FFA and hematite.

in Figure 3, confirm that the FFA particles have a spherical shape while the hematite particles have an irregular shape.

2.2. Methodology. This section discusses the methodology followed to conduct this study. It started with collection, characterization, and preparation of materials. Then, the mix design was determined based on mixability, pumpability, and rheology. After that, the cured samples were tested for mechanical and elastic properties. Figure 4 presents a summary of the methodology followed in this work.

Table 1. XRF of FFA Used in This Study

oxide	percentage, %
SiO ₂	55.92
Al ₂ O ₃	29.54
CaO	5.49
Fe ₂ O ₃	4.93
TiO ₂	1.91
K ₂ O	1.66
SO ₃	0.39
MnO	0.04
others	0.13

2.2.1. Material Preparation. NaOH solution was prepared by dissolving NaOH pellets in distilled water using a magnetic stirrer. NaOH solution was allowed to cool down to room temperature at least for one day before experiment. The specific gravity of the used powders was measured to estimate the required amounts to reach the target density (17.5 ppg). The FFA was sieved to ensure that the particles' size was lower than 100 μm, which was then confirmed by the PSD results. Two different processes (wet and dry) can be used to prepare geopolymer slurries.³⁹ In the wet process, a superplasticizer and/or retarder are mixed with activation solution for 2 min at a high shear rate (12,000 RPM) using an OFITE constant speed mixer. A binder such as FFA was then added and mixed with premixed solution at a high shear rate for another 2 min.

2.2.2. Slurry Design. Several trials were conducted to get an initial flowable slurry formulation that can be easily mixed.

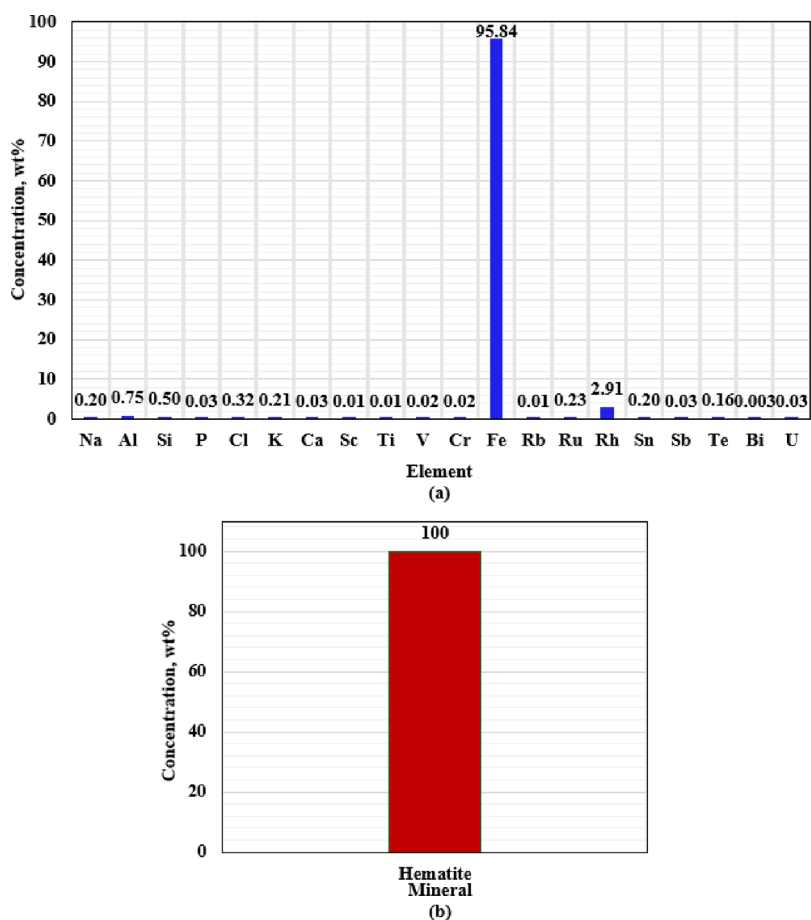


Figure 2. XRF elemental composition of hematite (a) and XRD of the sample (b).

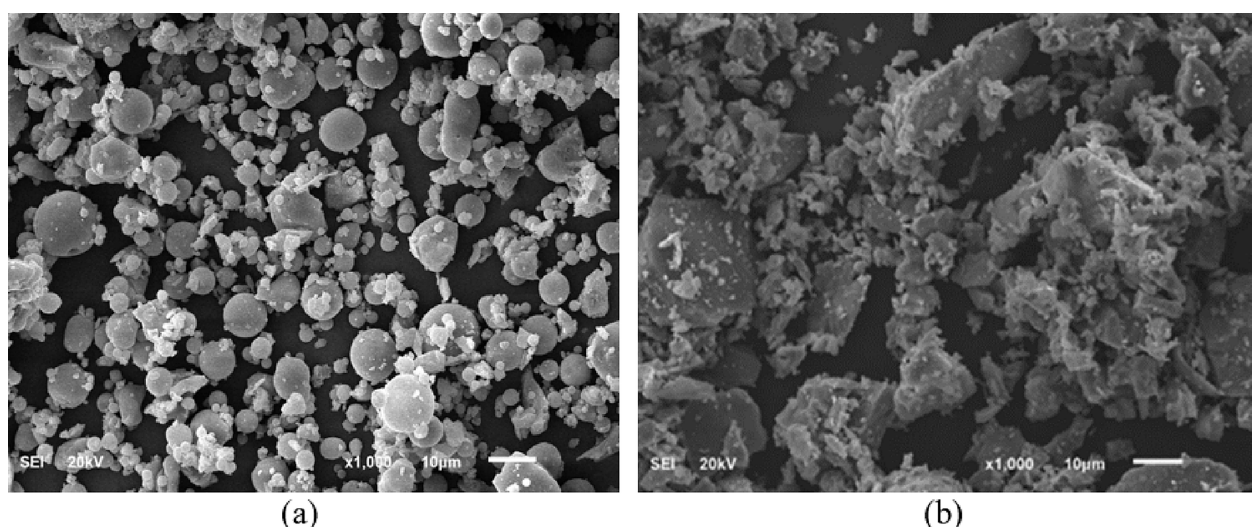


Figure 3. SEM images for FFA (a) and hematite (b).

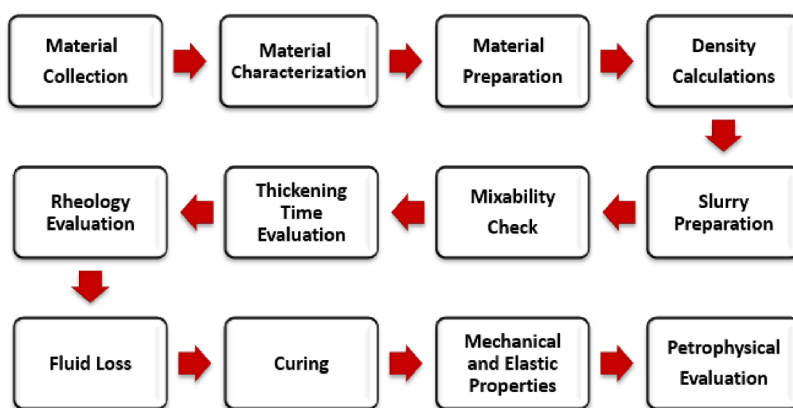


Figure 4. Summary of the methodology in this work.

Different parameters were tested such as NaOH molarity (4, 6, 8, and 10 M), liquid to binder ratio (0.5 to 0.8), and weighting agent percentage. Moreover, several additives with different concentrations were tested such as retarders and superplasticizers to enhance the slurry workability. The mix design started with testing the mixability, rheology, and thickening time. Then, the testing went through evaluation of strength and elastic and petrophysical properties.

2.2.3. Rheology, Fluid Loss, and Thickening Time. After mixing, a Grace atmospheric consistometer was used for conditioning the geopolymer slurries at 195 °F and 150 RPM for 30 min. The OFITE 900 viscometer was used to conduct rheology evaluation at an average temperature of 195 °F and atmospheric pressure. The OFITE HPHT filter press apparatus was used to measure the fluid loss. The thickening time test was conducted at 195 °F and atmospheric pressure using an atmospheric consistometer to estimate how long slurry would be pumpable. The rheology and thickening time measurements were run at least three times using different devices to ensure reliability and reproducibility of the results.

2.2.4. Mechanical and Petrophysical Properties. Evaluation of mechanical properties included unconfined compressive strength (UCS), tensile strength, and dynamic elastic properties (YM and PR), while evaluated petrophysical properties were porosity and permeability. After conditioning, the slurry was poured into cubic (2 in. in length) and cylindrical (1.5 in. in

diameter and 4 in. in length) molds, which were then placed in an HPHT curing chamber at 292 °F and 3000 psi for two periods (1 and 7 days). The Brazilian test procedure was followed to estimate the tensile strength. The UCS was measured using two techniques: a compression testing machine and scratch test. In compression testing, cubes of hardened geopolymer are crushed. The UCS is determined by the highest stress applied to a specimen that causes it to fracture.

The scratch test is designed to regulate and monitor the continual shearing action caused by the movement of a diamond cutter on a sample surface. The force operating on the cutter generates a continuous profile of rock strength along the sample. The YM and PR were then determined by getting the sonic velocities (i.e., compressional and shear waves). The ultrasonic test determines how long it takes a pressure wave to travel between two probes. The compression and flexural machine was used for compressive and tensile strength analysis, while an EPSLOG scratch testing machine with sonic mode was used for elastic properties and UCS.

For porosity measurement, helium gas was used, and Boyle's law was used in measuring porosity as discussed by Peters.⁴¹ For permeability measurement, nitrogen gas was used for measuring gas permeability at room temperature and a confining pressure of 1000 psi. The gas permeability was measured at different pressures, and a plot was constructed between gas permeability

versus $1/p_{\text{mean}}$. Then, a straight line was extrapolated to get the intercept value, which corresponds to liquid permeability.

3. RESULTS AND DISCUSSION

3.1. Mix Design. In the early stage, the FFA, hematite, and NaOH solution were mixed without any chemical additives. Different parameters were changed such as NaOH molarity, hematite percentage, and liquid to binder ratio. Initially, the mixing of the tested geopolymer slurries was difficult and the mixed slurries were thick and cannot be taken out of the mixer. By adding hematite, the slurry became thicker until a nonflowable state at high density was reached. Then, different superplasticizers and retarders were tested to enhance the workability and flowability of the geopolymer slurries. High NaOH molarity solutions (6, 8, and 10 M) developed mixable and flowable slurries, but the challenges were thickening time and cost optimization. It was observed that thickening time decreases as NaOH molarity increases. Moreover, increasing NaOH molarity requires higher amounts of NaOH, which in turn increases overall costs. As a result, a 4 M NaOH solution was used as an activator solution for the next stage. The liquid to binder ratio was changed until good results around 0.56 were obtained. The mix design used in this study is shown in Table 2 and expressed as by weight of binder (BWOB).

Table 2. Mix Design Used in This Study

component	BWOB (%)	weight (g)	SG
FFA	100	400	2.25
hematite	80	320	5.05
defoamer	0.0164	0.0656	1.10
superplasticizer	5	20	1.28
retarder	5	20	1.22
4 M NaOH solution	56	224	1.15
overall density (ppg)	17.5		

3.2. Thickening Time. The effect of hematite on thickening time was studied by increasing the hematite percentage from 25 to 75% BWOB at 195 °F without using any chemical additives. It

is found that increasing the hematite percentage significantly decreased the thickening time, as shown in Figure 5. Decreasing the hematite percentage from 75 to 25% increased the thickening time by almost 6 times. The reduction in thickening time can be attributed to the explanation stating that Si-O-Al or Si-O-K bonds form with a faster rate in the consolidated material when kaolin is calcined and/or iron oxide is present in metakaolin-based geopolymers.⁴² Then, different retarders and superplasticizers were tested to solve the thickening time challenge when increasing the hematite percentage in geopolymer slurries to achieve the required density. By doing so, a new mixture of a superplasticizer and a retarder succeeded in increasing the thickening time (100 BC) from 50 to 392 min at a BHCT of 195 °F, as shown in Figure 6. The addition of only

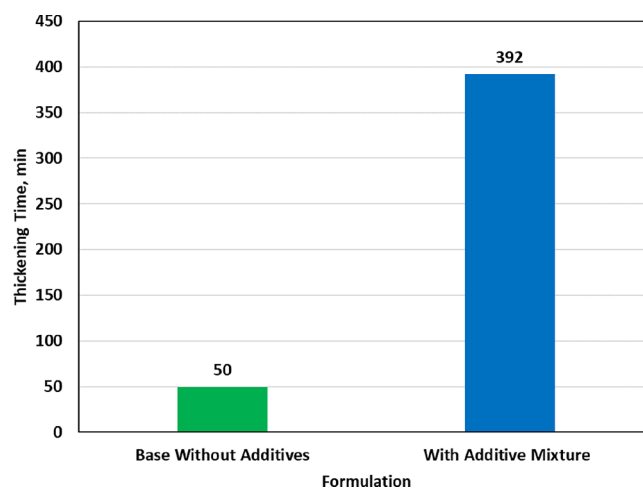


Figure 6. Effect of the developed mixture of additives on thickening time at 195 °F.

superplasticizer (5% BWOB) slightly increased the thickening time and the same for the retarder. When both were added together (5% BWOB each) to the mix design, the thickening time was greatly improved. The flocculated cement particles are dispersed by some superplasticizers via an electrostatic repulsion

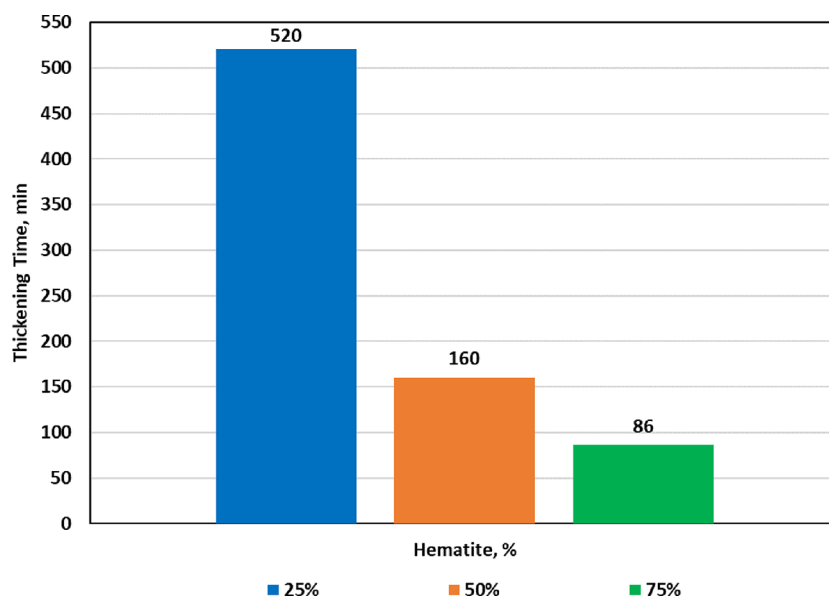


Figure 5. Effect of hematite without any additives on thickening time at 195 °F.

mechanism. When the active ingredients are adsorbed onto cement particles, they give them a negative charge, which causes them to repel one another. The cement particles are encircled by the lengthy molecules, which give them a strong negative charge that makes them repel each other.

3.3. Rheology Evaluation. Rheology evaluation of cement slurries is significant because it influences slurry mixing, pumping requirements, and drilling fluid displacement. The geopolymer rheology was evaluated at atmospheric (82.7 °F) and downhole (186.6 °F) temperatures. The shear stress and viscosity versus shear rate are presented in Figure 7 and Figure 8,

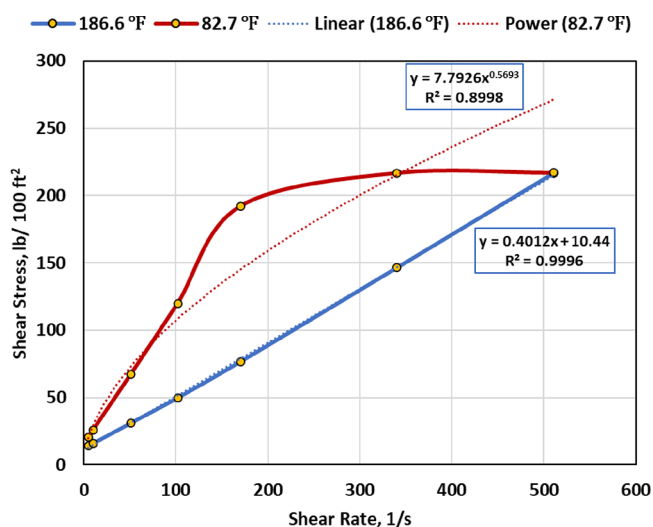


Figure 7. Shear stress vs shear rate of the developed geopolymer slurry.

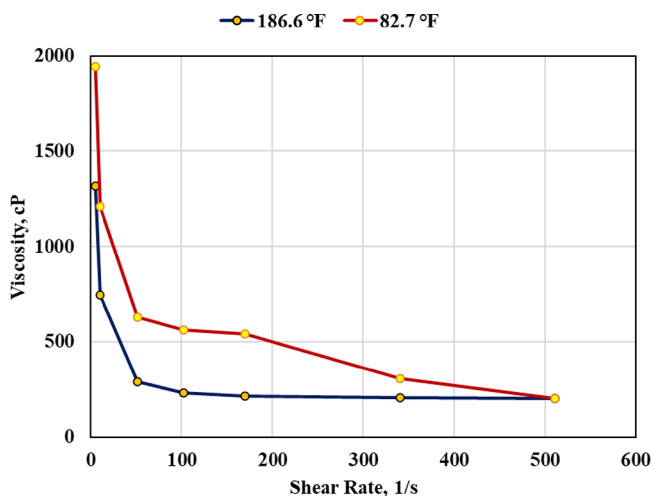


Figure 8. Viscosity vs shear rate of the developed geopolymer slurry.

respectively. The Bingham plastic flow model provided the best fit for the rheology evaluated at 186.6 °F with an R^2 of 0.999. The plastic viscosity and the yield point were 192.49 cP and 10.45 lb/100 ft², respectively. For atmospheric temperature, the flow curve did not follow the Bingham plastic model but the power law model with an R^2 of 0.90. The consistency index (K) and power law index (n) were 7.79 lb \cdot s ^{n} /100 ft² and 0.57, respectively. The power law index is lower than 1, indicating shear thinning behavior, and this can be confirmed by Figure 8 in which the viscosity reduces with increasing shear rate. The gel strength of the developed geopolymer was also investigated at 10

s and 10 min. The gel strength determines the slurries' carrying ability in a static condition, and its growth helps resist gas invasion. The developed slurry acted more like a power law fluid at low temperatures and more like a Bingham plastic fluid at high temperatures. The summary of the rheological properties of the developed geopolymer slurry is presented in Table 3.

Table 3. Rheological Properties of the Developed Slurry

property	unit	186.6 °F	82.7 °F
PV	cP	192.49	
YP	lb/100 ft ²	10.45	
n	dimensionless		0.56
K	lb \cdot s ^{n} /100 ft ²		7.79
10 s gel	lb/100 ft ²	16.00	23
10 min gel	lb/100 ft ²	171.00	204

3.4. Filtration. Water naturally leaks into subterranean formations from cement slurries. Fluid loss additives reduce loss by preventing cement slurry dehydration. If excessive fluid loss happens, a dry filter cake is formed. The filter cake may serve as a pathway for gas migration, impacting the whole zonal isolation. It also takes up space along the borehole wall, narrowing the annular gap, limiting the flow area, and raising the pressure required to pump the slurries. The estimated fluid loss indicates the slurries' impedance to gas migration, where a high filtrate loss refers to a high gas migration possibility.⁴³ For high-performance slurries, the slurry should have a fluid loss less than 50 mL/30 min as specified by API standards. The developed geopolymer slurry had a fluid loss of 38 mL/30 min, as shown in Figure 9. It is worth mentioning that the developed formulation

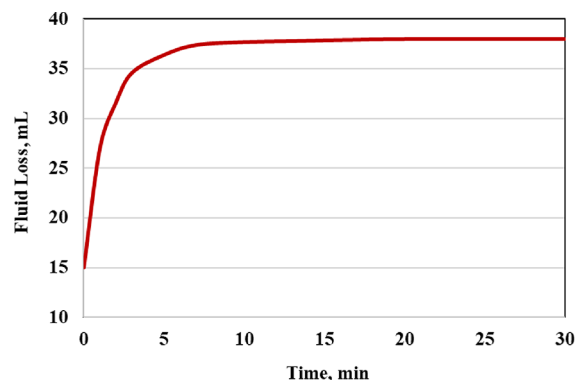


Figure 9. Effect of hematite on fluid loss for 30 min at 195 °F.

fell within the acceptable fluid loss ranges for different cementing applications, as shown in Table 4.⁴³ The developed geopolymer formulation provided this fluid loss behavior without adding any fluid loss control additive.

Table 4. Fluid Loss Ranges for Different Cementing Applications⁴³

application	fluid loss
horizontal well	<50 mL/30 min
risk of gas migration	30–50 mL/30 min
casing cementing	100–300 mL/30 min
low water content slurry (e.g., high-density slurry)	<50 mL/30 min
liner cementing	<50 mL/30 min

3.5. Mechanical and Petrophysical Properties. Mechanical properties of the set geopolymers are considered an important part in well design. Two techniques were used to evaluate the compressive strength of the developed geopolymer hardened samples. Three cubes were crushed for each curing period (1 and 7 days), and the average was reported as the crush strength. For the scratch test, cylindrical samples were used for scratch strength evaluation. The compressive strength values are presented in Figure 10 for both techniques. The compressive

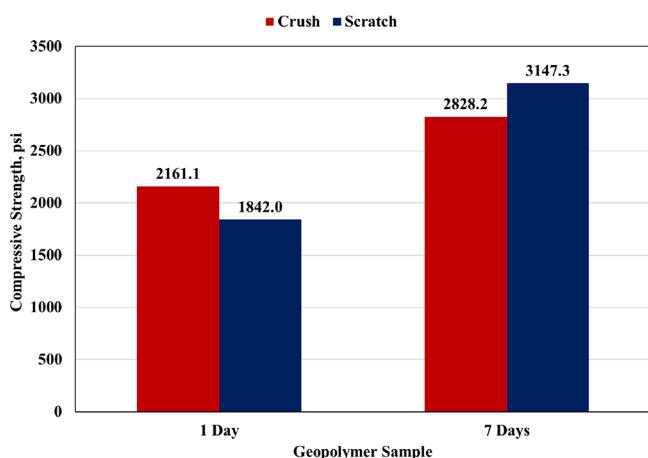


Figure 10. Compressive strength for different curing times at 292 °F using scratch and crush tests.

strength increased by 49% as calculated by the average values of both techniques for 1 and 7 days. The tensile strength was enhanced by 27.4% as the curing time increased from 1 to 7 days, as shown in Figure 11. The improvement in both compressive

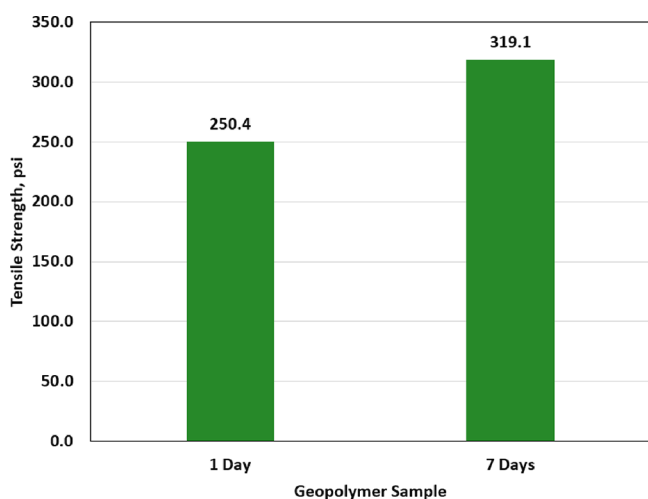


Figure 11. Tensile strength for different curing times at 292 °F using the Brazilian test.

and tensile strength with curing time indicated that the geopolymerization reaction continued for extended time but with a smaller rate. When the slurry gets 50 psi compressive strength, it can serve as a barrier to fluid and gas flow. At 500 psi compressive strength, it is strong enough to be drilled or tagged. Although it is possible to drill cement with less than 500 psi, the industry utilizes this as a rule of thumb.⁴³ The developed slurry got a 24 h compressive strength of at least 1842 psi, which is

larger than some reported values in high density cementing programs in the Middle East.

Cement sheath flexibility is important in wells where cement is subjected to large stresses, e.g., steam injection, geothermal, and hydraulic fracturing. Long-term well integrity is provided by more flexible cements with lower YM and higher PR. Set cements should have a YM that is smaller than that of the surrounding formations. Smaller YM set cements may be preferable for unconsolidated strata than higher YM set cements.⁴³ The developed geopolymer YM increased from 5.18 to 8.2 GPa and the PR decreased from 0.26 to 0.20 as curing time increased from 1 to 7 days, as shown in Figures 12 and 13.

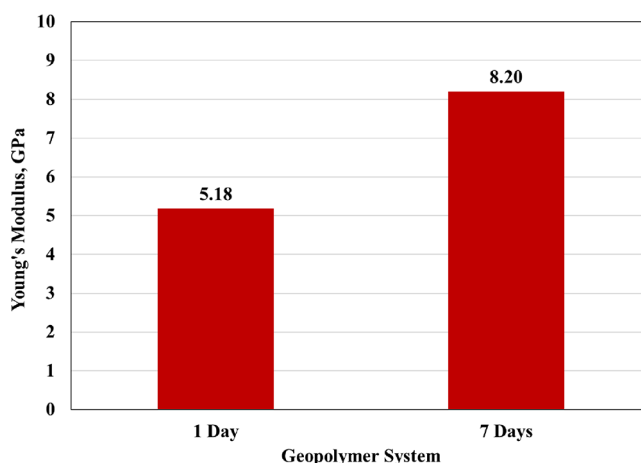


Figure 12. Effect of curing time on YM.

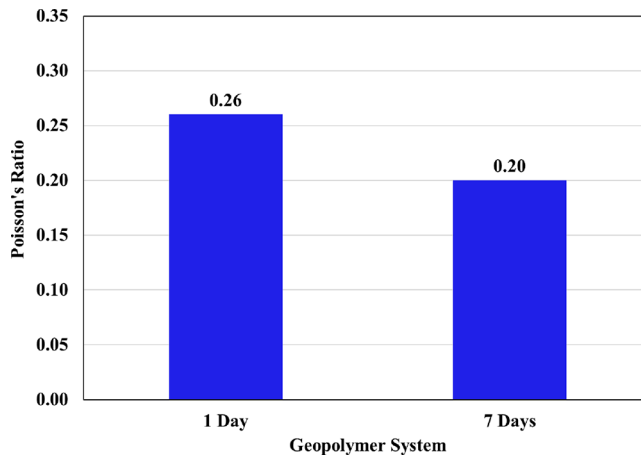


Figure 13. Effect of curing time on PR.

The developed geopolymer is more flexible than class G cement, as presented in Table 5. Moreover, it possessed a YM that is lower than shale and consolidated formations and thus can be used adjacent to these formations as stated earlier by Liu. Generally, more flexible cements, those with lower YM and higher tensile strength, with enough compressive strength, tend to perform well when simulating sheath stresses. The geopolymer YM increased with increasing strength that proved agreement between strength results and the dynamic elastic properties of the developed system in this study. Petrophysical measurements such as porosity and permeability were conducted for the developed geopolymer system after drying the samples at 221 °F for 24 h in an oven. The geopolymer system had a permeability of 0.01 mD and a porosity of 0.38.

Table 5. Young's Modulus and Poisson's Ratio for Some Materials⁴³

material	YM (GPa)	PR
shale	30.95	0.35
consolidated sandstone	24.06	0.25
unconsolidated sandstone	5.17	0.30
class G cement	11.03	0.17
extended cement systems	2.27	0.17
flexible cement systems	1.99	0.22
developed geopolymer	5.18–8.20	0.20–0.26

4. CONCLUSIONS

This work introduced the first hematite-based class F fly ash geopolymer formulation for oil and gas well cementing. Unlike the other studies in the literature, this work used 4 M NaOH solution only as an activator that can reduce the overall cost. This work used fly ash (waste material) as an aluminosilicate source, which reduces the cementing cost, cement carbon emissions, and massive energy consumption and tackles the environmental issue related to fly ash disposal. Various properties of the designed slurry and hardened samples were evaluated using recommended practices with some modifications. The outcomes of this study can be summarized as follows:

- Increasing the hematite percentage significantly decreased the thickening time of geopolymers.
- A developed mixture of a retarder and a superplasticizer was introduced that enhanced the workability and prolonged the thickening time by 500%.
- The developed formulation achieved a low fluid loss without any fluid control additive (38 mL/30 min) that made it a good candidate for a wide range of cementing applications such as horizontal drilling, gas migration risk, and casing cementing that require fluid loss <50 mL/30 min.
- The developed geopolymer slurry had a density of 17.5 ppg, which can be used in high pressure well cementing, and the developed slurry acted more like a power law fluid at low temperatures and more like a Bingham plastic fluid at high temperatures.
- The compressive and tensile strength were enhanced by increasing the curing time, confirming the continuation of geopolymerization and the elastic properties of the developed geopolymer proved that it is more flexible than some cement systems.

■ AUTHOR INFORMATION

Corresponding Author

Salaheldin Elkatatny – College of Petroleum Engineering and Geosciences, King Fahd University of Petroleum & Minerals, Dhahran, Eastern Province 31261, Saudi Arabia;
 ● orcid.org/0000-0002-7209-3715; Email: elkatatny@kfupm.edu.sa

Authors

Ahmed Abdelaal – College of Petroleum Engineering and Geosciences, King Fahd University of Petroleum & Minerals, Dhahran, Eastern Province 31261, Saudi Arabia

Ahmed Mohsen Abd El Fattah – Department of Architecture, King Fahd University of Petroleum & Minerals, Dhahran, Eastern Province 31261, Saudi Arabia; ● orcid.org/0000-0002-0553-8817

Complete contact information is available at:
<https://pubs.acs.org/10.1021/acsomega.3c00590>

Author Contributions

The manuscript was written by the contributions of all authors [A.A., S.E., and A.M.]. Authors have approved the final version of the manuscript.

Funding

This research received no external funding.

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

The authors wish to acknowledge King Fahd University of Petroleum & Minerals (KFUPM) for permitting the publication of this work.

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