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Gypsum slurries to apply in oil well: An insight into thickening time

Pastas de gesso para aplicação em poços de petróleo: Uma análise do tempo de espessamento

Pastas de yeso para pozos petrolíferos: Análisis del tiempo de espesamiento

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Abstract
Gypsum is widely used in the construction industry, especially as a hydraulic binder. Studies have indicated the use of α-HH gypsum as an alternative material to Portland Cement in oilwell cementing operations, highlighting the reduction in environmental impacts from the reduction of Portland Cement. Calcium Sulphate α-Hemihydrate ($CaSO_4$. ½ H_2O) has been shown to be a promising material to replace Portland Cement in some applications. The hydration of gypsum pastes goes through the process of saturation of the medium with Ca^{+2} and SO_4^{-2} ions, then the physical phenomenon of crystallization, and finally the phenomenon of hardening, where the crystals formed precipitate producing Dihydrate ($CaSO_4$. $2H_2O$). Gypsum pastes harden very quickly, and their pumpability is impaired with thickening times of less than 20 minutes. For applications requiring longer pumpability times, the use of retarding additives is necessary. This research studied the effects of retarding additives in α-HH gypsum paste systems by varying the water-gypsum factor (FAG 0.4; 0.5 and 0.6) using a pressurized consistometer, under conditions of 54 °C and 9500 psi, with the aim of obtaining formulations with admissible thickening times for oil well cementing applications. The results showed that it was possible to develop paste systems with varying thickening times, with intervals of more than 120 minutes. The 0.5 FAG system proved to be more stable at the same retarder concentrations when compared to the 0.4 and 0.6 FAG systems.
Keywords: Gypsum slurries; Retarding additives; Thickening times; Well cementing.

Resumo
O gesso tem sua aplicação na construção civil bastante conhecida, principalmente como aglomerante hidráulico. Estudos têm indicado o uso do gesso α-HH como material alternativo ao Cimento Portland em cimentação de poços petrolíferos, destacando a redução dos impactos ambientais oriundos da redução do Cimento Portland. O Sulfato de Cálcio α-Hemihidratado ($CaSO_4$. ½ H_2O) tem se apresentado como material promissor para substituir o Cimento Portland em algumas aplicações. A hidratação das pastas de gesso passa pelo processo de saturação do meio com os íons de Ca^{+2} e SO_4^{-2}, na sequência o fenômeno físico da cristalização, por fim o fenômeno do endurecimento, onde os cristais formados precipitam produzindo o Dihidratado ($CaSO_4$. $2H_2O$). Pastas de gesso endurecem muito rapidamente, tendo sua bombeabilidade prejudicada com tempos de espessamento inferiores a 20 minutos. Para aplicações que necessitam de tempos maiores de bombeabilidade, torna-se necessário o uso de aditivos retardadores. Esta pesquisa estudou os efeitos de aditivos retardadores em sistemas de pastas de gesso α-HH variando o fator água-gesso (FAG 0,4; 0,5 e 0,6) utilizando o consistômetro pressurizado, nas condições de 54 °C e 9500 psi, com o objetivo de obter formulações com tempo de espessamento admissível para aplicações em cimentações de poços petrolíferos. Os resultados mostraram que foi possível desenvolver sistemas de pastas com tempo de espessamento variados, com intervalos superiores a 120 minutos. O sistema de FAG 0,5 mostrou-se mais estável com as mesmas concentrações de retardadores quando comparado com os sistemas de FAG 0,4 e 0,6.
Palavras-chave: Pastas de gesso; Aditivos retardadores; Tempos de espessamento; Cimentação de poços.

Resumen
El yeso es bien conocido en la industria de la construcción, principalmente como aglutinante hidráulico. Algunos estudios han indicado el uso del yeso α-HH como material alternativo al cemento Portland en la cementación de pozos petrolíferos, destacando la reducción del impacto ambiental derivado de la reducción del cemento Portland. El sulfato de calcio α-Hemihidratado ($CaSO_4$. ½ H_2O) ha demostrado ser un material prometedor para sustituir al Cemento Portland en algunas aplicaciones. La hidratación de las pastas de yeso pasa por el proceso de saturación del medio con iones Ca^{+2} y SO_4^{-2}, luego por el fenómeno físico de la cristalización, y finalmente por el fenómeno del

endurecimiento, donde los cristales formados precipitan para producir Dihidrato ($CaSO_4 . 2H_2O$). Las pastas de yeso se endurecen muy rápidamente y su bombeabilidad se ve afectada con tiempos de espesamiento inferiores a 20 minutos. Para aplicaciones que requieren tiempos de bombeo más largos, es necesario el uso de aditivos retardantes. Esta investigación estudió los efectos de los aditivos retardantes en sistemas de pastas de yeso α-HH variando el factor agua-yeso (FAG 0,4; 0,5 y 0,6) utilizando el consistómetro presurizado, en condiciones de 54 °C y 9500 psi, con el objetivo de obtener formulaciones con tiempos de espesamiento admisibles para aplicaciones de cementación de pozos petrolíferos. Los resultados mostraron que era posible desarrollar sistemas de pasta con tiempos de espesamiento variables, con intervalos de más de 120 minutos. El sistema de 0,5 FAG demostró ser más estable a las mismas concentraciones de retardante en comparación con los sistemas de 0,4 y 0,6 FAG.
Palabras clave: Pastas de yeso; Aditivos retardantes; Tiempos de espesamiento; Cementación de pozos.

1. Introduction

Gypsum has a widespread application in civil construction. Literature reports that this material has been used for making statues and molding objects since ancient times. Later, with industrial growth, gypsum was diversified, used as a covering and, mainly, as a binding material (Mota, 2022). In recent years, researchers have proposed the use of α-HH gypsum as an alternative material to ordinary Portland Cement (OPC) in oil well cementing operations, highlighting the reduction in environmental and energy impacts arising from the decrease of OPC (Meireles et al., 2019).

The production of OPC is associated with many environmental impacts, including a significant emission of Carbon Dioxide (CO_2), around 500 kg of CO_2 for each ton of clinker produced (Cao et al., 2016). Another negative point of Portland Cement production is associated with high energy consumption, representing 12 – 15% of all energy consumed by the industry. This energy is consumed for the various stages of cement production, mainly during the burning of the different materials that produce the clinker, reaching temperatures around 1450 °C (Ali et al., 2011). The most promising alternative to avoid CO_2 emissions is partially replacing clinker with other mineral materials and/or mineral waste that have less potential harm to the environment. Due to the large amount of cement produced over the years and the raw materials and energy consumed, the cement industry has become a source of environmental concern (Arruda Junior et al., 2023).

Several global sectors have aroused interest in materials with high technological efficiency, low cost, and the lowest possible environmental impact. The search for materials with good mechanical properties, which require less energy in their processing and generate less polluting waste, has become quite important. With its diverse activities, the petroleum industry has driven the development of new materials that can safely and efficiently fulfill their intended application.

In this context, α-Hemihydrate Calcium Sulfate ($CaSO_4 . ½ H_2O$ – α-HH) has been studied as a hydraulic binding material with good crystallinity and high compressive strength. Gypsum (α-Hemihydrate Calcium Sulfate) is widely used to produce noble artifacts, such as orthopedic and dental plaster, the molding industry, automotive, matrices for ceramic pieces, and architecture (Camarini & De Milito, 2011). Calcium Sulfate (Gypsum) has emerged as a promising material for replacing OPC in some applications. Some studies evaluated the application of gypsum in conjunction with cement for oilwell cementing operations (Hua et al., 2016). Two routes can obtain Calcium Sulfate Hemihydrate to produce α-HH and β-HH. The autoclave production method makes it possible to create α-HH, while the calcination production method makes it possible to obtain β-HH; both are obtained from $CaSO_4 . 2 H_2O$ (Yin & Yang, 2020).

α-HH has garnered attention for years since its manufacturing process is less environmentally harmful and requires lower energy consumption than obtaining ordinary Portland cement (OPC). α-HH is obtained in an autoclave with a temperature of around 150 °C and controlled pressure (Eq. (1)) with the release of water vapor (Hao et al., 2021). Meanwhile, for OPC, clinker is obtained by calcining $CaCO_3$ at temperatures ranging from 900 to 1450 °C, producing CaO and releasing CO_2 into the atmosphere (Eq. (2)) (Ludwig & Zhang, 2015).

$$CaSO_4.2\,H_2O \xrightarrow{150\ °C} CaSO_4.{}^1/_2\,0{,}5H_2O + 1{,}5\,H_2O$$
Eq. (1)

$$CaCO_3 \xrightarrow{900\ to\ 1450\ °C} CaO + CO_2\uparrow$$
Eq. (2)

The mixture of water with α-HH powder in specific quantities produces gypsum pastes, which go through the hydration process where the medium is first saturated with Ca^{+2} and $SO4^{-2}$ ions, followed by the physical phenomenon of crystallization, where the medium becomes supersaturated in those ions. Finally, the hardening phenomenon occurs, where the crystals formed in the previous phase tend to precipitate, forming Dihydrate crystals ($CaSO_4$. 2 H_2O). Gypsum pastes harden very quickly; naturally, hardening depends on several factors, mainly the water/gypsum ratio (C. Liu et al., 2021). Typically, gypsum pastes have their pumpability impaired as thickening times are less than 20 minutes. For applications that require longer pumping times, retardant additives must be added to gypsum paste systems, and thickening time tests must be carried out (Panpa & Jinawath, 2006).

The present research studied the effects of retardant additives in gypsum paste systems using a pressurized consistometer under 54° and 9500 psi conditions, varying the gypsum/water factor in ratios of 0.4, 0.5, and 0.6, to obtain paste systems with allowable thickening time for oil well cementing operations applications. Therefore, it was possible to develop paste systems with varying thickening times, with intervals exceeding 120 minutes.

2. Methodology

This section addresses the items that make up the methodology used, from the selection of materials, the preparation of the pastes, and the execution of thickening time tests with the water-gypsum factor (FAG) of 0.4, 0.5, and 0.6 for evaluation.

2.1 Materials and Methods

Table 1 shows the materials used in the pastes' mix design. The pastes were formulated based on Standard ABNT NBR 9831, replacing Portland Cement with α-Hemihydrate Calcium Sulfate. Two retarders (Inorganic and Organic) were added to the paste systems to alter Hemihydrate Calcium Sulfate's hydration reactions.

Table 1 - Materials used in preparing the pastes.

Material	Supplier
Calcium Sulfate (α-HH)	Mineradora São Jorge
Water	CAERN/Natal
Inorganic Retarder	Neon
Organic Retarder	Synth

Source: Authors.

The gypsum pastes were formulated according to ABNT NBR 9831, where it was possible to calculate the respective weight of each component of the formulations from mass balance calculations taking into account the water/cement ratio, in this case, replacing the cement by Hemihydrate Calcium Sulfate. The work also took into account a variation in the water/gypsum factor (FAG) of 0.4, 0.5, and 0.6 in the paste compositions. Where the FAG 0.4 paste system represents a water/gypsum ratio = 0.4, the same was done for the FAG 0.5 and 0.6 systems. Some of these water/gypsum relationships were studied in the works of Duarte (2014) and Yu and Brouwers (2011). Table 2 below presents the constituents and their concentrations due to the formulation process.

Table 2 - Formulation of the studied paste systems.

Material	Density (g/cm³)	Density (lb/gal)	Concentration	(%) or gpc
α-HH Calcium Sulfate	2.78	23.2	QSP	%
Water	0.9969	8.34	QSP	%
Inorganic Retarder	1.71	14.32	0.7	%
Organic Retarder	1.6	13.4	0.15	%

Source: Authors.

2.2 Paste preparation

The pastes were prepared in a Chandler Engineering 3260 mixer (Figure 1). The α-HH gypsum was added to the water (with retarders) for 15 seconds at a mixing speed of 4000 rpm, ending with 12,000 rpm for 35 seconds to homogenize the paste, according to API RP 10B-2 effectively.

Figure 1 – Paste mixer.

Source: Authors.

2.3 Thickening time test

Thickening time tests were performed based on API RP 10B-2, conducted on an HTHP Chandler Engineering model 8040 consistometer (Figure 2).

The test conditions were based on adaptations from the work of (Fagundes et al., 2013). The tests followed a circulation temperature (BHCT) of 54 °C and a pressure of 9500 psi, with a 45-minute ramp. The proposed test conditions are characteristic of Brazilian pre-salt wells with depths ranging from 5000 to 7000 meters. The fresh paste was placed in the HTHP consistometer cell, and then the test conditions were set (54 °C and 9500 psi). Finally, the test completion condition was awaited when the fresh paste gained consistency, reaching 100 Bc, leaving its state from fluid paste to solid state.

Figure 2 - Preparation sequence for the thickening time test. (a) Paste in the fresh state. (b) Consistometer adjusted with pressure and temperature. (c) Sample after thickening test.

(a) (b) (c)

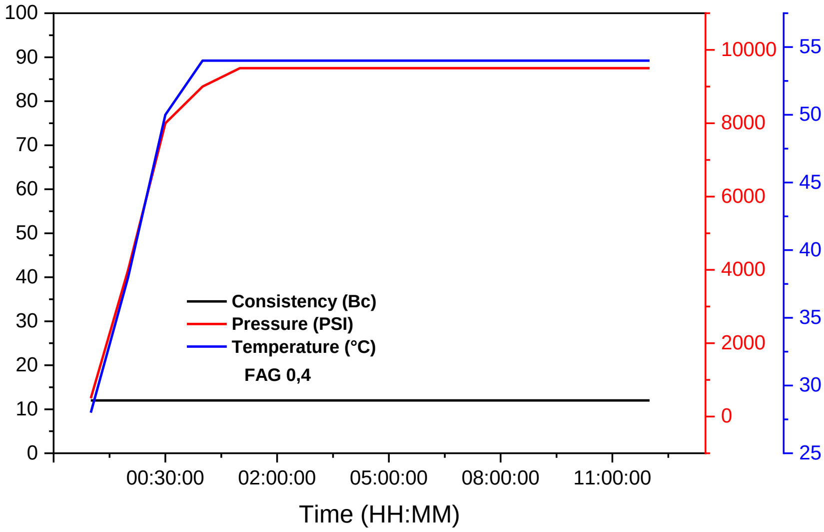
Source: Authors.

3. Results and Discussion

This section presents the results of the studied paste systems with FAG of 0.4, 0.5, and 0.6, with the presence of retarders (inorganic and organic) in the composition of the pastes under conditions of 54 °C and 9500 psi.

Figure 3 shows the thickening times for FAG 0.4. the test lasted more than 11 hours, and the system in question did not show an increase in consistency, remaining at 12 Bc. The system with FAG 0.4 has in its formulation the smallest amount of water and the most significant amount of α-HH gypsum when compared to the systems with FAG 0.5 and 0.6.

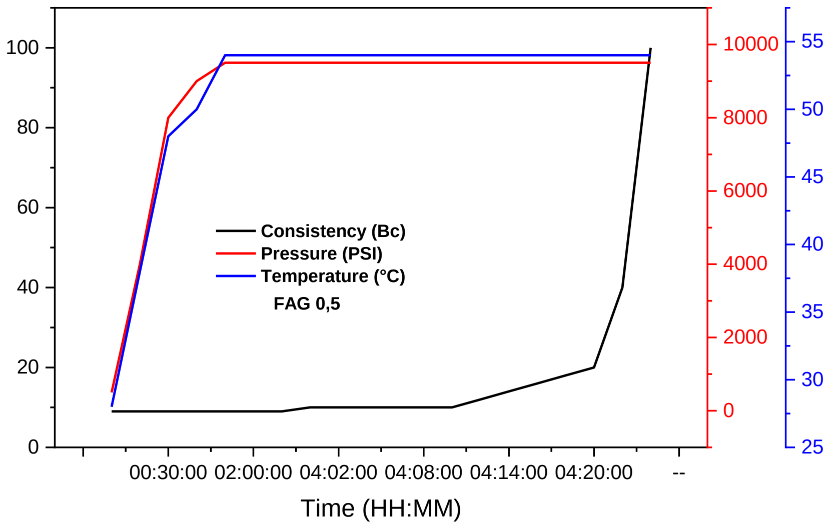
Figure 3 - Consistency test of the FAG 0.4 system under conditions of 9500 psi and 54°C.



Source: Authors.

Figure 4, referring to the system formulated with FAG 0.5, presented a thickening time of 262 minutes (4 hours and 22 minutes) under conditions of 9500 psi and 54 °C, highlighting the right angle of growth in the paste consistency, reaching 100 Bc abruptly. This behavior is interesting for systems intended for wells with gas migration, where the slurry must present a short transition from liquid to solid state.

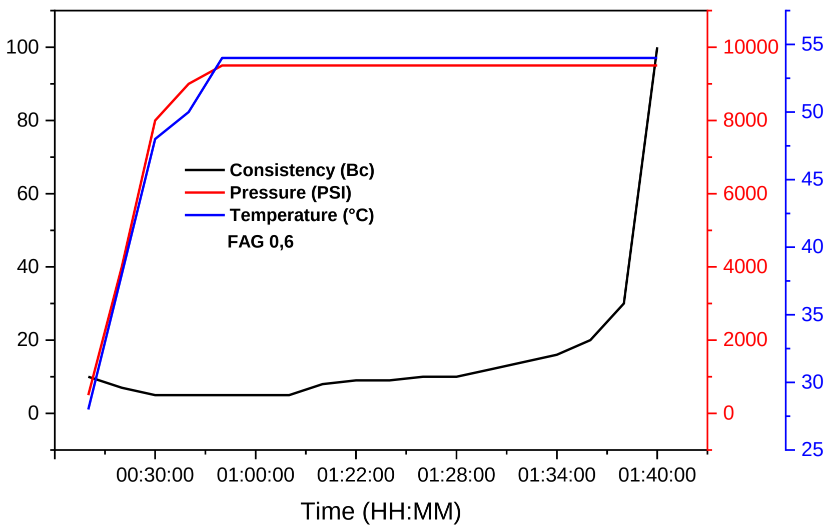
Figure 4 - Consistency test of the FAG 0.5 system under conditions of 9500 psi and 54°C.



Source: Authors.

The graph presented in Figure 5, referring to the system formulated with FAG 0.6, showed a thickening time of 90 minutes (1 hour and 30 minutes), shorter than the system developed with FAG 0.5. The system composed of FAG 0.6 has a more significant amount of water and a smaller amount of gypsum when compared to FAG 0.5.

Figure 5 - Consistency test of the FAG 0.6 system under conditions of 9500 psi and 54°C.



Source: Authors.

When analyzing Figures 3, 4, and 5, it was observed that for different FAGs, different thickening times were obtained, even knowing that the water/gypsum ratios (0.4, 0.5, and 0.6) have the same concentration of its additives, varying only the amount of water and α-HH gypsum.

In addition to the fact that FAG 0.4 is the paste system with the least amount of water, there is the presence of retarders (organic and inorganic) that interact strongly with water and hinder the hydration of calcium sulfate hemihydrate (Mróz & Mucha, 2018).

The inorganic additive used in the FAG 0.4 paste system results in poorly soluble or insoluble products around the calcium sulfate dihydrate crystals, delaying their development and, consequently, their precipitation, which causes the system to harden or set (Duarte., 2014).

In the case of organic retarder, part of the mixing water interacts with the hydroxyls present in its structure; in addition, this retarder has the potential to form a gel around the hemihydrate crystal, preventing its contact with water (Pourchez et al., 2006).

Even with the presence of two retarders in its formulation, the FAG 0.6 system has a faster setting because there is an "excess" of water that facilitates the hydration of calcium sulfate hemihydrate to dihydrate with consequent saturation and subsequent precipitation and curing or hardening of the paste (Yu & Brouwers, 2011). According to (Kanno, 2010), it is worth highlighting that excess water in gypsum pastes causes a decrease in mechanical properties due to the presence of free water between the crystals.

The system with FAG of 0.4, which did not show signs of consistency gaining up to 11 hours of the test, underwent a change in its formulation to reduce the concentration of retarders to evaluate whether the smaller amount of water in the FAG system 0.4, added to the concentration of the two retardants, significantly reduced the possibility of formation and saturation of the dihydrate. Therefore, the formulation was changed by reducing the concentration of organic and inorganic retardants, called FAG 0.4b, as shown in Table 3 below:

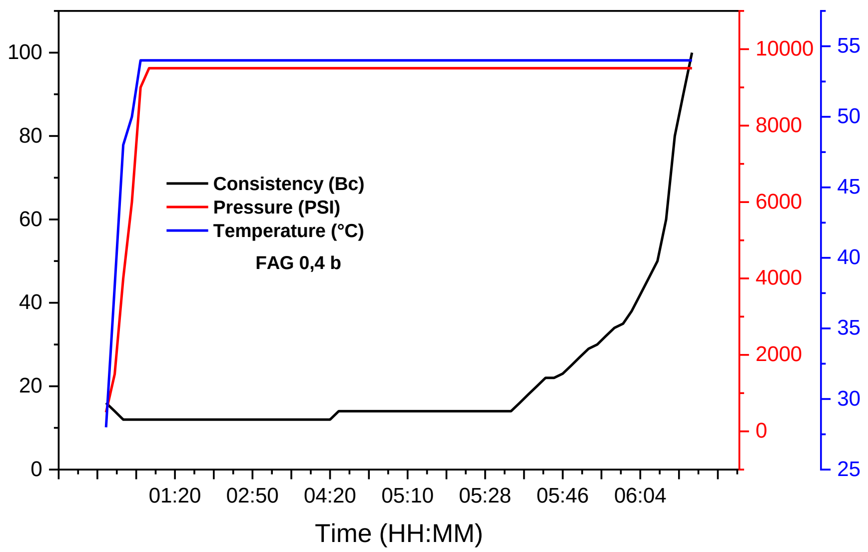
Table 3 - New formulation with change in the retarder concentration for the FAG 0.4b paste system.

Material	Density (g/cm³)	Density (lb/gal)	Concentration	(%) ou gpc
α-HH Calcium Sulfate	2.78	23.2	QSP	%
Water	0.9969	8.34	QSP	%
Inorganic Retarder	1.71	14.32	0.64	%
Organic Retarder	1.6	13.4	0.093	%

Source: Authors.

Figure 6 below refers to the thickening time test for the formulation system with FAG 0.4 with modification in retarder concentrations, as shown in Table 3 above.

Figure 6 - Consistency test of the FAG 0.4b system under conditions of 9500 psi and 54°C.



Source: Authors.

Figure 6 shows that the paste system with FAG 0.4b formulation is strongly dependent on the amount of water present in its formulation, as a slight decrease in retarder concentration enabled the formation and saturation of the dihydrate so that there was a gain in consistency with subsequent hardening of the paste (J. Liu et al., 2024), with a thickening time of 375 minutes (6 hours and 15 minutes).

Table 4 compiles thickening time data for FAG systems 0.4, 0.5, 0.6, and 0.4b when the paste reached 100 Bc.

Table 4 - Thickening times for FAG systems 0.4, 0.5, 0.6, and 0.4b.

Slurry thickening	
	Thickening time (100 Bc)
FAG 0.4	> 11 h
FAG 0.5	4:36 h
FAG 0.6	1:66 h
FAG 0.4b	6:25 h

Source: Authors.

Thickening times exceeding 11 hours, as observed in the FAG system 0.4, are considered undesirable due to their prolonged duration. Conversely, for FAG 0.5, a thickening time of 4 hours and 22 minutes is deemed reasonable for various applications. However, in the case of FAG 0.6, with a thickening time of 1 hour and 40 minutes, it proves insufficient for the majority of applications. Notably, FAG 0.4b exhibited a thickening time of 6 hours and 15 minutes, allowing the paste to remain pumpable for over 6 hours, thereby ensuring fluidity and workability of the paste system.

4. Conclusion

The main conclusions we highlight for this research are:

- The paste system added with retarders (inorganic and organic) made it possible to obtain thickening times greater than 4 hours and 20 minutes under conditions of 9500 psi and 54 °C. It is noteworthy that, with the adjustment of the formulation, the proposed paste system (FAG 0.4b) can be adjusted for thickening times of approximately 6 hours, which are the times practiced in offshore applications;
- The FAG 0.5 paste system can be classified as the best system studied, corroborated by studies by (Guan et al., 2010). The FAG 0.5 system proved to be more stable when compared to FAG 0.4 and 0.6. With the same concentrations of additives and varying only the FAG, the system with a ratio of 0.4 needs more than 11 hours to induce a gain in consistency, while the system with a ratio of 0.6 has an earlier setting, around 90 minutes, while the FAG 0.5 system has a time of approximately 260 minutes;
- The paste systems studied have the potential to be applied as hydraulic binding material under test conditions (54 °C and 9500 psi).

For future work, the authors recommend evaluating the effect of retarders on the compressive strength of specimens cured under conditions of 54° C and 9500 psi, along with microstructural and morphological analysis.

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