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Analysing the Thermal Stability Performance of Fresh and Equilibrium Zeolite in Water-Based MudUsing Standard Deviationand the Effects on Rheology and Filtration

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Abstract: *In this paper, two types of nanoparticle additives, Fresh Zeolite (F-Z) and Equilibrium Zeolite (E-Z), were examined to determine which one could perform better as a thermal stability additive and at what concentration for oil and gas drilling operations. The study involved determining the stability levels of the additives at high temperatures and adding them in 0.5 g, 1.0 g, and 1.5 g quantities to WBM formulations, which were then aged at 482 °F. Rheological tests were conducted to determine fluid flow and fluid loss properties, as well as pH at temperatures ranging from 120 °F to 360 °F. The results showed that all nanoparticle-treated formulations had improved thermal stability, with 1.5 g of fresh zeolite providing the best overall thermal stability, followed by 1.5 g of equilibrium zeolite, by standard deviation. However, all formulations with nanoparticle additives showed severe fluid loss during filtration tests. The nanoparticles were also observed to act as dispersants. The study concluded that at their optimum concentrations (1.5 g F-Z, 1.5 g E-Z), these nanoparticles could function as thermal stability additives for WBMs.*

Keywords: *fresh zeolite, equilibrium zeolite, thermal stability, high temperature, water-based mud, drilling operation.*

Abbrevations: *F-Z: Fresh Zeolite; E-Z: Equilibrium Zeolite; WBM: Water-Based Mud; HPHT: High Pressure High Temperature; TGA: Thermogravimetric Analysis; API: American Petroleum Institute*

1. Introduction

With oil fields shifting to deeper depths and higher temperatures, modern drilling operations face significant challenges in High Pressure High Temperature (HPHT) zones(Mitchell, 2007). Drilling fluid systems need to be capable of providing better thermal stability in such environments, which is currently a difficult task for water-based drilling fluids due to their tendency to degrade at high temperatures (Amanullah, 2009). As a result, oil-based drilling fluids are often preferred despite being more expensive, environmentally harmful, and difficult to manage.

Various research has suggested diverse additives to enhance the heat resistance of water-based mud (WBM) systems. These additives encompass chemicals, polymers, nanoparticles, and zeolites. As a result, a wide array of chemicals and polymers are currently employed in formulating different mud systems. The objective is to fulfill specific functional criteria, including achieving suitable mud rheology, density, mud activity, and efficient fluid loss control (Anon, 2000).

Zeolites are known for their unique thermal conductivity, electrical conductivity, and optical features, and they have not yet been expensively studied in the context of drilling fluid formations(Chai et al., 2014). This research aims to investigate the effectiveness of using fresh and equilibrium zeolites in enhancing the thermal stability of water-based drilling fluids in HTHP zones. The research also seeks to conduct a performance comparison between the fresh and equilibrium zeolites under high temperature conditions.

2. Methods And Materials Used

2.1. Thermogravimetric Analysis

In this investigation, Thermogravimetric Analysis (TGA) was employed to examine the behaviour of both fresh and equilibrium zeolite nanoparticles. TGA is a technique that quantifies the mass change of a material in response to temperature or time under controlled atmospheric conditions. It involves measuring the formulation's mass while it undergoes heating or cooling in a furnace. The collected data aids in evaluating material composition and predicting thermal stability at temperatures up to 1200 oC. TGA also allows the identification of materials experiencing weight loss or gain due to processes like decomposition, oxidation, or dehydration. The results are presented as a TGA thermal curve, with time or temperature on the x-axis and weight on the y-axis.

To carry out the experiment, a thermogravimetric analyser consisting of a formulation pan supported by a precision balance was utilized. The formulation pan was placed inside a furnace and subjected to heating and cooling cycles while monitoring the formulation's mass. A continuous flow of purge gas was used to control the environment, passing over the formulation and exiting through an exhaust. The obtained TGA results offer valuable insights into the thermal characteristics and behavior of the zeolite nanoparticles under examination.

2.2. Formulation and Rheology Tests of the Water-Based Systems

A variety of tests were carried out to examine the characteristics of water-based systems during drilling operations under downhole conditions. One group of experiments focused on the rheological properties exhibited bylab-preparedWBMsinfused with both fresh and spent zeolites, with temperature variations between 120 °F and 360 °F. Another set of experiments assessed the fluid loss control and pH of these formulation systems. The mud systems were prepared with varying nanoparticles masses, as outlined in Table 1, and the standard API procedures for testing water-based fluids were adhered to.

Table1. *Additives for 10.4 ppg Water-Based Mud Formulation with Fresh and Equilibrium Zeolites*

Materials	Quantity
Fresh water	0.91 bbl
Soda Ash	0.1 ppb
Bentonite	12.5 ppb
Caustic Soda	0.3 ppb
Barazan D	0.8 ppb
PAC L	21 ppb
Barite	101.8 ppb
F-Z and E-Z	0.5, 1, 1.5 ppb

API 13B guidelines were followed to add the additives to the mud and ensure a uniform blend, with a mud mixer used to mix the formulations for 15 to 20 minutes.

2.2.1. Mud Aging

A high-temperature aging chamber was employed to mimic the mud aging process, imitating the agitation and heating that drilling fluid experiences under elevated temperatures. The formulations were aged for 16 hours at 250 °C (482 °F) before being subjected to testing.

2.2.2 Density Test

The density of the prepared formulations was determined using a standard mud balance to confirm that they were within acceptable limits.

2.2.3 Rheology Test

The rheological properties of the formulated WBMs were tested using a Model 1100 HPHT rheometer to obtain shear rate/shear stress values at different temperatures ranging from 120 °F (49 °C) to 360 °F (182 °C) at an 80 °F (27 °C) interval.

2.2.4 Fluid Loss Test

The HPHT Filter Press was used to assess theformulations’ fluid loss at the 7.5-min and 30-min marks.

2.2.5 pH Test

A pH meter equipped with a glass electrode was used to determine the degree of acidity or alkalinity of the mud formulations.

2.3. Statistical Analysis

To evaluate the thermal stabilization efficacy of nanoparticles in the aged WBM formulations, standard deviation was employed to rank their performance from best to worst. The shear stress standard deviations were calculated for each WBM formulation at four shear rates, and temperatures ranging from 120 °F to 360 °F. This process was repeated for all four shear rates and nano WBM formulations, resulting in 24 standard deviation values. To simplify the ranking process, the average standard deviations were then computed across the four shear rates for all WBM formulations, producing six 'average' standard deviation values representing each mud formulation. Based on these average standard deviation values, the ranking was determined.

3. RESULTS AND DISCUSSIONS

3.1. Thermogravimetric Analysis Plots

The thermogravimetric analysis plots in Figs. 1 and 2 depict the thermal behavior of equilibrium zeolite and fresh zeolite particles employed in this research. Observing the plots, it becomes evident that both nanoparticles possess the ability to endure high temperatures without undergoing degradation. Both F-Z and E-Z exhibited decomposition between 932 °F (500 °C) and 1,112 °F (600 °C), indicating their thermal stability up to these temperatures, which is a crucial characteristic for a thermal stability additive.

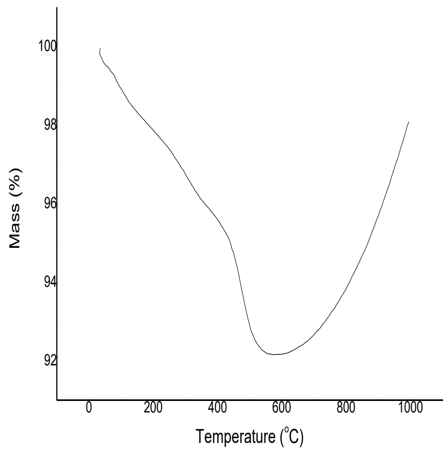

Fig1. *Thermogravimetric Analysis Plot for Equilibrium Zeolite*

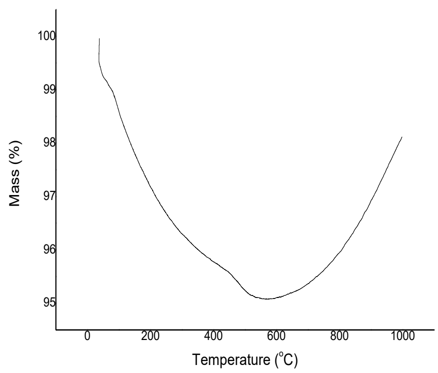

Fig2. *Thermogravimetric Analysis Plot for Fresh Zeolite*

3.2. Effects on Fluid Model

A fluid model elucidates a fluid's characteristics and flow behavior. Rheograms for all F-Z and E-Z fluid formulations across temperature ranges depictedYPs greater than zero, with n values higher than 1, as depicted in Figs. 3 to 6, implying that these fluids are not Newtonian in nature. The curves' shapes and their absence correspond to the Herschel Buckley and Bingham Plastic models. Both additives notably decreased the fluids' shear stresses. However, at 360 °F, the 0.5 g and 1.0 g F-Z mud formulations exhibited greater shear stresses than the control mud formulation, possibly indicating some fluid losses due to evaporation at that elevated temperature. Additionally, higher shear stress values were recorded for the 0.5 g E-Z mud formulation at 120 °F and 360 °F.

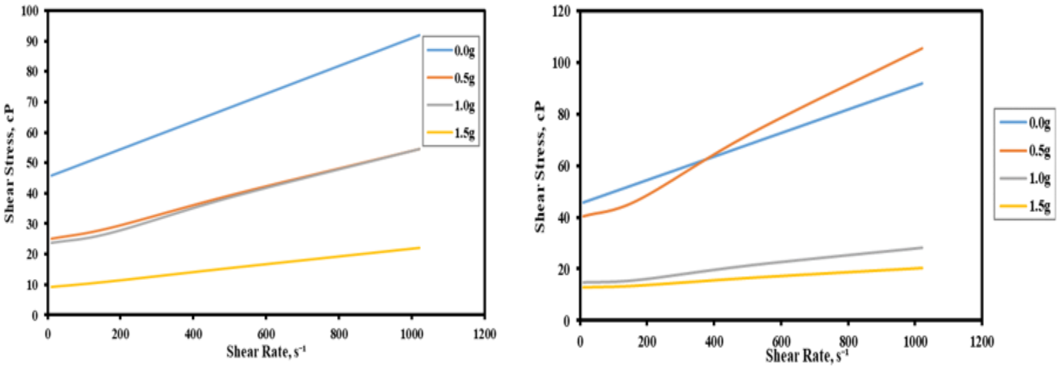

Fig3. *Rheogram of Fresh Zeolite (L) and Equilibrium Zeolite (R) WBMs at 120 °F*

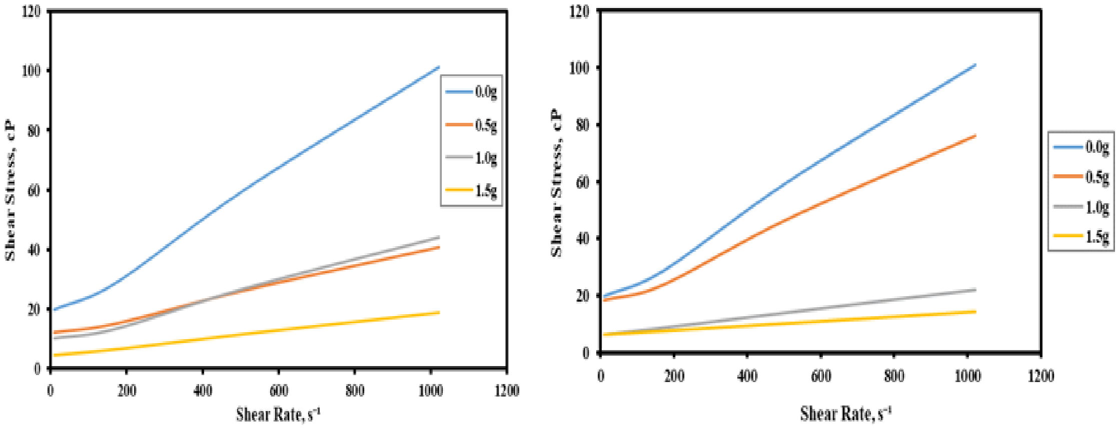

Fig4. *Rheogram of Fresh Zeolite (L) and Equilibrium Zeolite (R) WBMs at 200 °F*

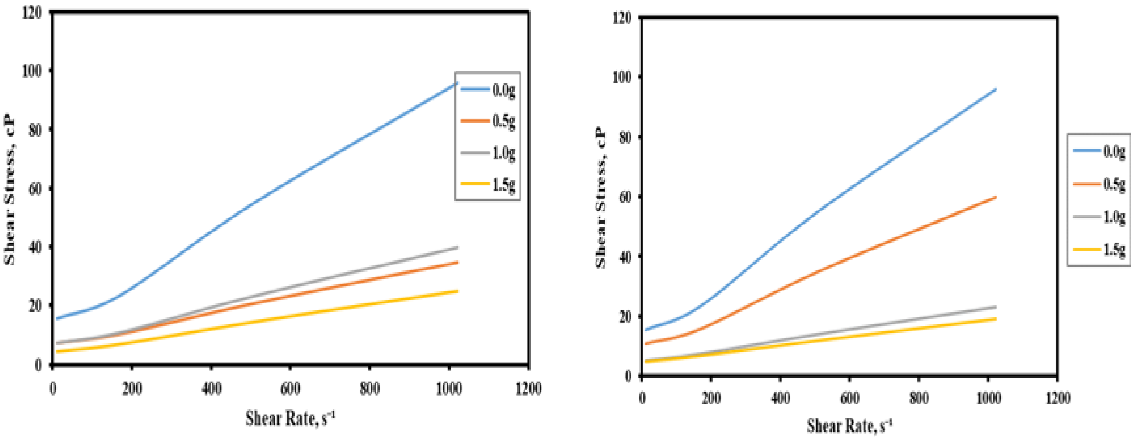

Fig5. *Rheogram of Fresh Zeolite (L) and Equilibrium Zeolite (R) WBMs at 280 °F*

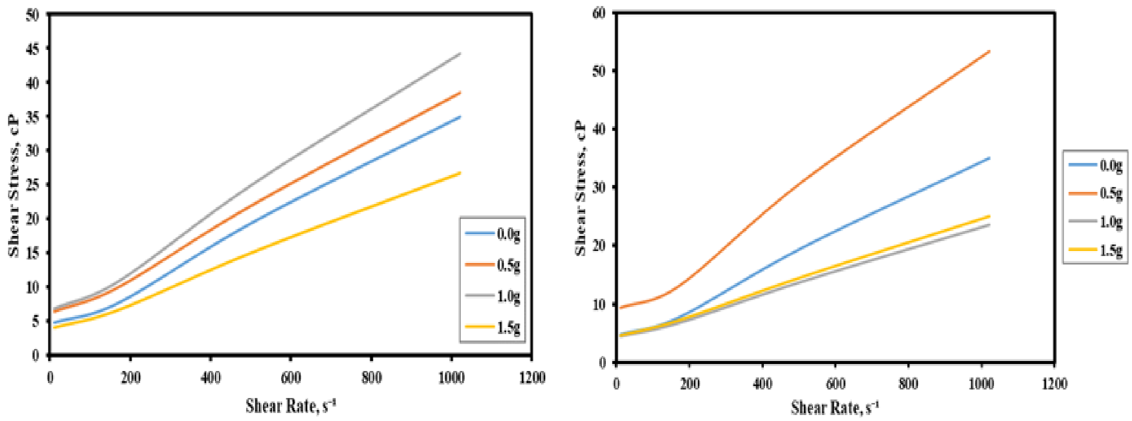

Fig6. *Rheogram of Fresh Zeolite (L) and Equilibrium Zeolite (R) WBMs at 360 °F*

3.3. Effects on Rheological Properties

3.3.1. Plastic Viscosity

The impact of nano F-Z and E-Z on the plastic viscosity (PV) of WBMs at various temperatures is visually presented in Fig. 7. As expected, the control mud formulation indicates decliningplastic viscosities as it is heated from 200 °F to 360 °F. However, upon introducing increasing quantities of fresh and equilibrium zeolite, the PV of mud formulations tends to stabilize across temperature changes. Remarkably, both F-Z and E-Z nanoparticles noticeably reduce the fluid's PV at a consistent temperature, functioning as mud thinners.

All formulations containing aged fresh zeolite meet the minimum API requirement of 7 cP, except for the 1.5 g formulation at 120 °F. The 0.5 g formulation exhibits the most desirable stability relative to the 1.0 g formulation, resulting in decreased PVs, thereby reducing the need for higher pump pressure while drilling. Moreover, it complies with the API standard, unlike the 1.5 g formulation at 120 °F.

In regard to the E-Z mud formulations, better stabilization is observed with the incorporation of 1.0 g and 1.5 g of nanoparticles. Overall, the 1.0 g F-Z mud formulation displays superior stabilization, meeting the PV requirement between 200 °F and 360 °F, and nearly reaching it at 120 °F. Furthermore, it exhibits sufficiently low plastic viscosities, which facilitates smoother pumping during operations.

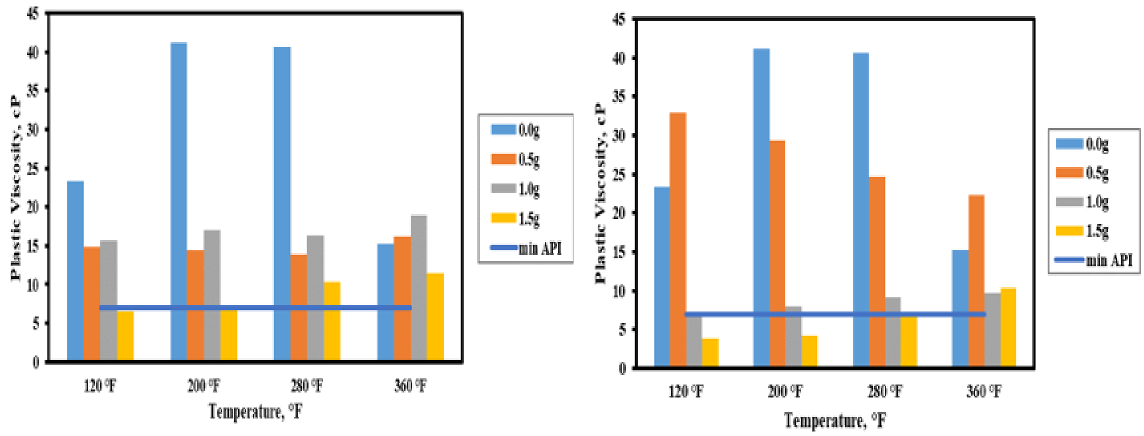

Fig7. *Plastic Viscosity of Aged Fresh Zeolite (L) and Equilibrium Zeolite (R) WBMs at Varying Temperatures*

The 0.5 g and 1.0 g mud formulations of both nanoparticles meet the API requirement for PV of a WBM. The 1.5 g F-Z and E-Z mud formulations at 360 °F both meet the minimum API requirement.

The 0.5 g and 1.0 g F-Z mud formulations are more thermally stable than the E-Z equivalents. In selecting the optimum concentration of the nanoparticles, where optimum means a trade-off between stability and API requirements, 0.5 g of F-Z and 1.0 g of E-Z should be used. The F-Z particles generally stabilised the PV of the fluid better than E-Z did.

3.3.2 Yield Point (YP)

The analysis of thermal resilience concerning the yield point (YP) of aged nano F-Z and E-Z water-based muds (WBMs) is depicted in Figure 8. The YP of the control formulation diminishes with rising temperature, potentially due to increased molecular spacing at higher temperatures. This leads to reduced resistance to fluid flow, in line with the explanation by Amani and Al-Jubouri (2012). The inclusion of 0.5 g of fresh zeolite into the drilling fluid results in early stabilization. Predictably, YP values of both 0.5 g and 1.0 g F-Z mud formulations decline between 120 °F and 280 °F. However, YP values for the 1.5 g mud formulation rise from 200 °F to 360 °F. Notably, the 1.5 g concentration yields the least YP variation with temperature changes, making it the optimal nanoparticle amount for this context. In the case of 0.5 g E-Z mud formulation, its YP decreases with increasing temperature. Notably, the addition of 1 g and 1.5 g of E-Z achieves near-complete stabilization. 1.5 g offers superior stability compared to 1 g, making it the preferable choice for the optimum E-Z concentration in this specific scenario.

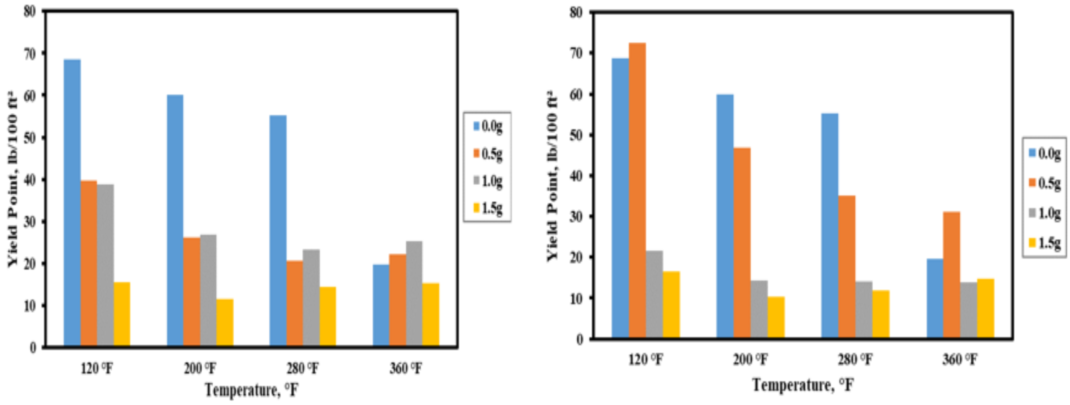

Fig8. *Yield Point of Aged Fresh (L) and Equilibrium Zeolite (R) WBMs at Varying Temperatures*

The 1.0 g and 1.5 g E-Z and F-Z mud formulations all meet the API requirement for the YP of a WBM. The 0.5 g E-Z and F-Z formulations at 280 °F and 360 °F also fall below the maximum YP allowed of WBMs. At 200 °F, the 0.5 g E-Z and F-Z formulations also meet the requirement.

The 0.5 g formulations of all the nanoparticles are not thermally stable, and the optimum amount to be used in the bid to achieve thermal stability with both nanoparticles is 1.5 g. Again, with both nanoparticles, thermal stability is better with 1.0 g than it is with 0.5 g.

At 120 °F, there is an initial rise in YP after 0.5 g of E-Z is added, and then subsequent drops when the concentration of the nanoparticle is increased from 0.5 g to 1.5 g. This is different from the behaviour of the F-Z formulations at the same temperature. The F-Z formulation drops gradually with increments in the concentration of the nanoparticle right from 0.5 g.The F-Z particles generally stabilised the YP of the fluid better than E-Z did.

3.3.3 Gel Strength

Figure 9 displays the 10-second gel strength of F-Z and E-Z water-based muds (WBMs) across different temperatures. In this graph, the gel strength of the control formulation noticeably decreased with rising mud temperature, which is suboptimal since stable gel strength is crucial during operations. After F-Z is added, the gel strength becomes stable within the experimental temperature range. Both 0.5 g and 1.0 g mud formulations exhibited comparable stability levels, falling within the API-recommended range (3 – 20 lb /100 ft²). Thus, both concentrations can effectively maintain consistent 10-second gel strength as temperatures vary with depth. Opting for the 1.0 g concentration might be preferred, given its higher gel strength values than the 0.5 g mass at 200 °F and 280 °F, enhancing solids-suspension properties and remaining comfortably above the API lower limit. However, the 1.5 g mud formulation displayed inadequate performance, falling below the lower limit at 120 °F, 200 °F, and 360 °F, rendering it unsuitable for use. F-Z proved more effective than E-Z as a thermal stability enhancer for gel strength.

Upon introducing E-Z to the mud formulations, gel strength values seemed to stabilize within the experimental temperature range. The 0.5 g mud formulation exhibited poor performance as temperature increased, resulting in a 10-second gel strength of 0 lb /100 ft² at 360 °F, indicating inadequate solids suspension during drilling operations halts. The 1.0 g mud formulation outperformed the 0.5 g formulation, while the 1.5 g mud formulation fared worse than the 1.0 g version, barely reaching the lower limit and progressively increasing gel strength. These results highlight that although the 1.0 g and 1.5 g concentrations of aged E-Z WBMs offered some level of stability, E-Z is generally an unsuitable choice for enhancing the thermal stability of the fluid in relation to its 10-second gel strength.

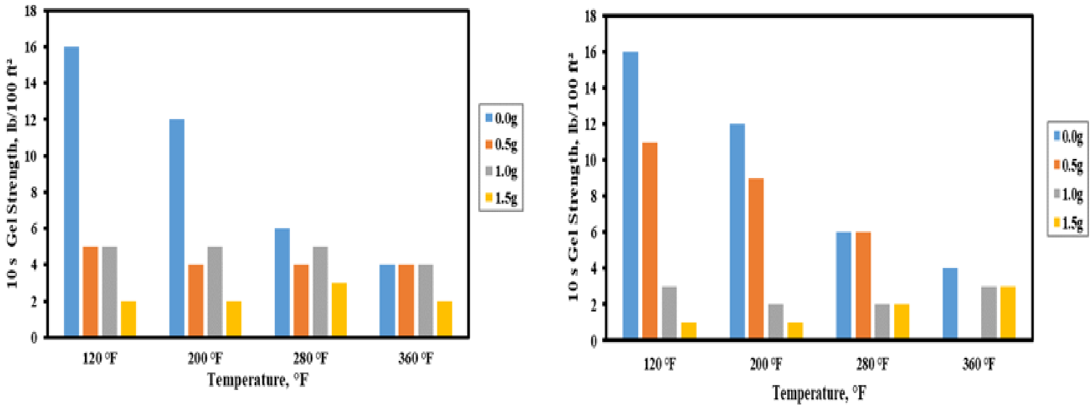

Fig9. *Ten Seconds Gel Strength of Aged Fresh (L) and Equilibrium (R) Zeolite WBMs at varying Temperatures*

Just like the 10-second gel experiment, the gel strength of the control mud formulation displayed the anticipated decrease with rising temperature in Figure 10. Upon the introduction of F-Z particles to the mud formulations, stabilization was observed, with the 0.5 g formulation achieving the most effective stabilization. Just like in the 10-second gel test, the 1.5 g F-Z mud formulation experienced values below the 3 lb /100 ft² lower limit at 120 °F and 200 °F. Similar to the 10-second test, the 0.5 g E-Z mud formulation exhibited unfavorable thermal stability characteristics in Figure 10. Both the 1 g and 1.5 g E-Z formulations demonstrated improved stability, likely because the higher nanoparticle quantity led to heightened binding forces among the mud's clays. PerBaroids, Halliburton, a bentonite pad mud should possess a maximum 10-minute gel strength of 57 lb/100 ft². Although all 10-minute values fell below the threshold, the most stable formulations had considerably low gel strengths, akin to the 10-second test, which might not effectively support solids suspension.

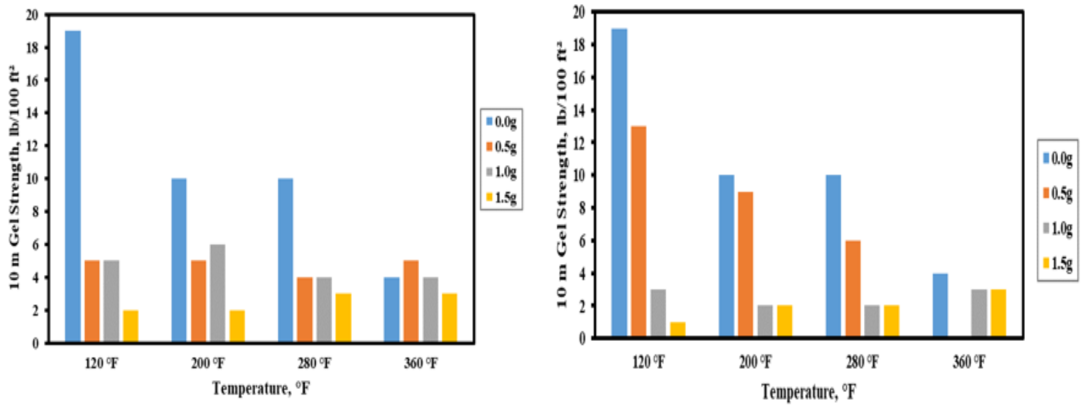

Fig10. *Ten Minutes Gel Strength of Aged Fresh (L) and Equilibrium (R) Zeolite WBMs at Varying Temperatures*

The optimum concentration of F-Z for the 10 s gel strength was 0.5 g. E-Z provided some thermal stability at 1 g and 1.5 g, but they hardly met the API standard across the range of temperatures. For both 10 s and 10 m gel strengths, fresh zeolite provided better thermal stability than the equilibrium zeolite catalyst.

3.3.4. Fluid Loss

Figure 11 illustrates the filtration loss observed in mud formulations incorporating fresh and equilibrium zeolite nanoparticles. The quantity of filtrate collected in the test is influenced by the viscosity of the tested fluid. Fluids possessing higher viscosity, particularly those with elevated solids content, tend to form thicker mud cakes more quickly (arising from the fluid's solids concentration residue), thereby impeding continuous fluid loss. Notably, both F-Z and E-Z particles have demonstrated potential dispersant properties, contributing to a reduction in drilling fluid viscosity. Given this context, a plausible explanation exists for the higher filtrate volumes in the 0.5 g, 1 g, and 1.5 g formulations compared to the control. The introduction of nanoparticles might have diminished fluid viscosity, resulting in the creation of a less effective filter cake. Consequently, greater amounts of water were lost during the test, evident both at 7.5 and 30 minutes. The pronounced filtrate losses from zeolite-containing mud formulations could be attributed to the pseudo-thinning capacity of nanoparticles due to their miniature dimensions. Furthermore, Majid et al. (2018) proposed an indirect relationship between pH and filtration loss in WBMs. Their observations indicated that lowering the base fluid's pH led to heightened filtration losses, potentially stemming from filter cake modification or damage caused by the acidic environment.

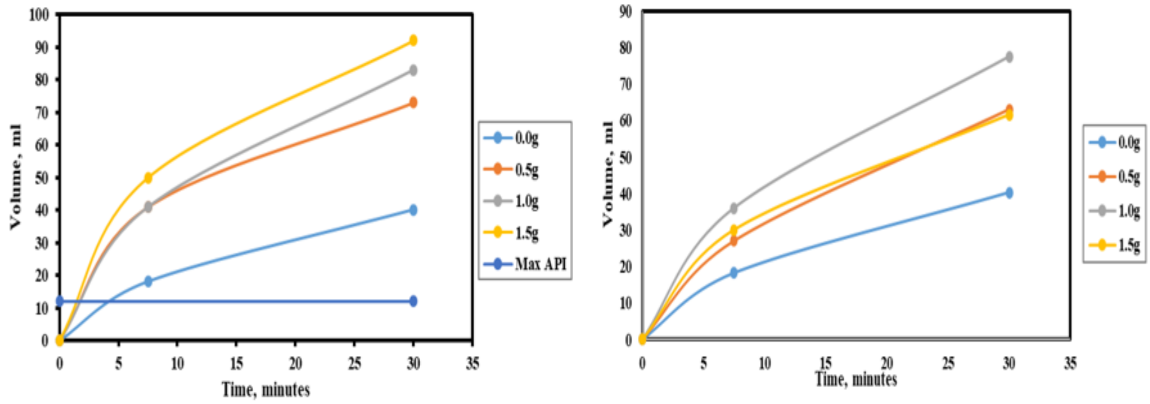

Fig11. *Filtration Loss of Aged Fresh (L) and Equilibrium (R) Zeolite WBMs after 7.5 and 30 Minutes*

3.3.5. Hydrogen Ion Concentration (pH)

The impact of nano F-Z and E-Z on the pH of the aged WBMs is depicted in Figure 12. It can be observed that the majority of F-Z formulations exhibited alkaline properties, except for the 1.5 g mud formulation, which displayed slight acidity with a pH of 6.53. Other formulations also exhibited decreasing alkalinity with rising nanoparticle concentrations. This alteration can be attributed to fresh zeolite acting as a Brønsted–Lowry acid, as explained by Amarfio et al. (2020), resulting in pH reduction within the fluids. Regarding E-Z, the control and 0.5 g formulations showcased alkaline characteristics, whereas the 1.0 g and 1.5 g mud formulations demonstrated slight acidity, having pH values of 6.7 and 6.4 respectively.

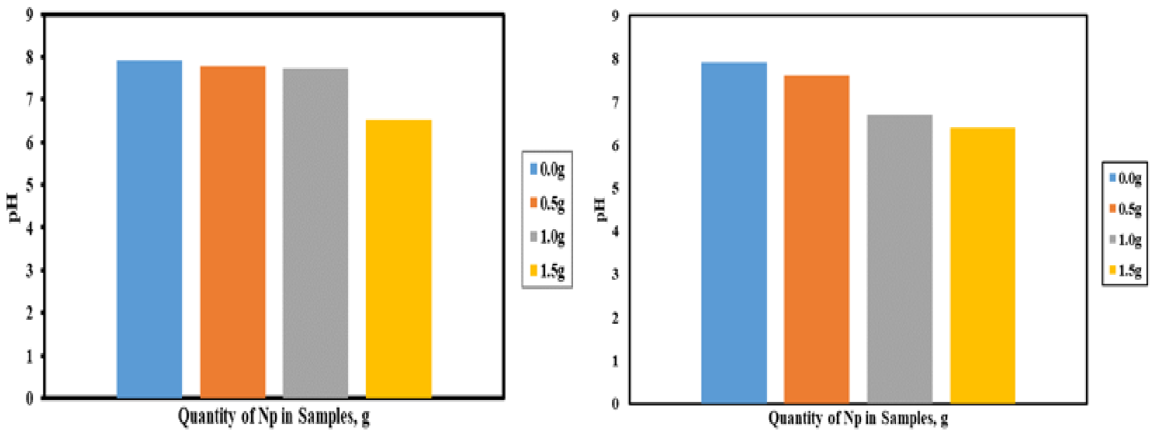

Fig12. *pH of Varying Quantities of Aged Fresh (L) and Equilibrium (R) Zeolite in WBM Samples*

3.3.6. Thermal Stability

Figures 13 to 16 present the relationships between shear rate and shear stress for the control, 0.5 g, 1.0 g, and 1.5 g F-Z mud formulations, each measured at various temperatures. Across all four shear rates, the shear stress values of the formulations exhibited a consistent decline with increasing temperature. This outcome aligns with expectations, as higher temperatures lead to a thinner drilling fluid consistency and subsequent reduction in viscosity.

In Figure 14, a notable stabilization is observed between 200 °F and 360 °F upon the introduction of 0.5 g of F-Z into the mud formulation. As nanoparticle concentration increases, the majority of shear stress values decrease, a trend evident across most of the dataset. This trend is further highlighted in Figure 15, where the 1.0 g F-Z mud formulation demonstrates significantly improved stability compared to the 0.5 g formulation.

Similarly, Figure 16 showcases the enhanced stability of the mud formulation at the 1.5 g F-Z concentration when compared to other concentrations. At shear rates of 10.2204 s-1 and 170.34 s-1, the stresses from 200 °F to 360 °F remain nearly equivalent. Remarkably, at 511.02 s-1, the shear stress at 360 °F closely matches that at 120 °F, signifying a strong indicator of thermal stability. However, at shear rate 1022.04 s-1, the shear stress values at higher temperatures start to surpass those at 120 °F. These findings suggest that 1.5 g of F-Z could be considered the optimal amount for achieving thermal stability.

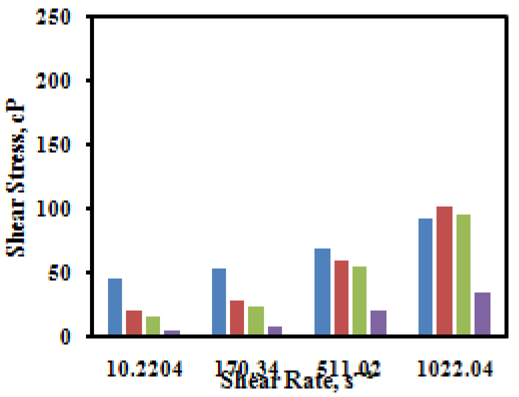

Fig13. *Rheogram of 0 g of Fresh Zeolite WBMs*

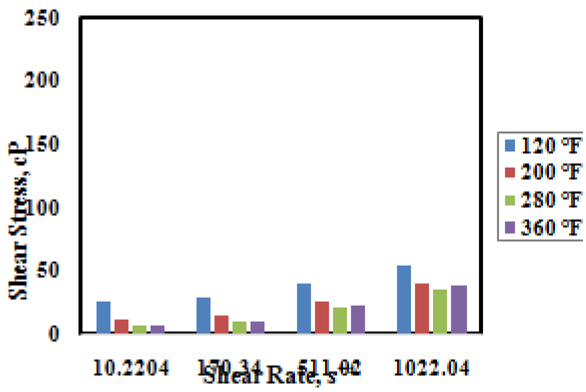

Fig14. *Rheogram of 0.5 g of Fresh Zeolite WBMs*

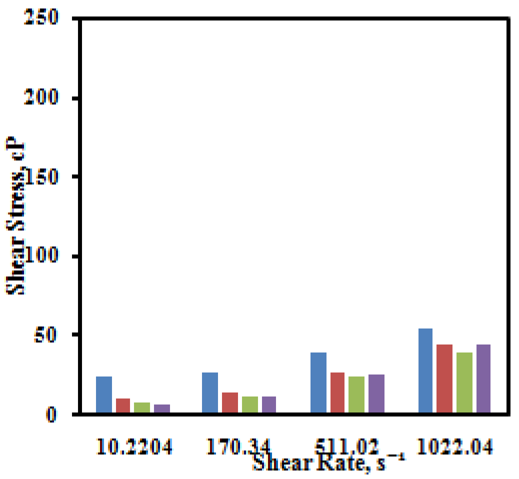

Fig15. *Rheogram of 1.0 g of Fresh Zeolite WBMs*

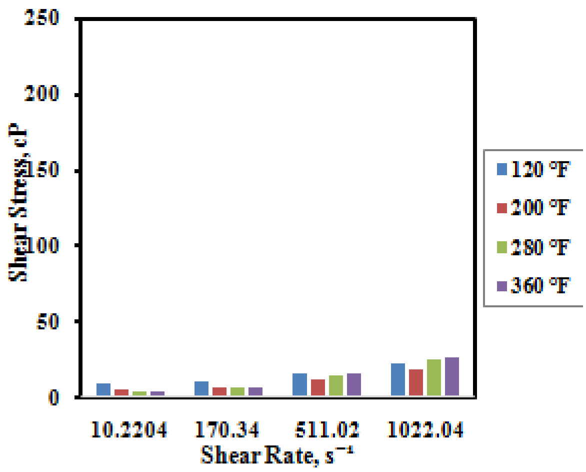

Fig16. *Rheogram of 1.5 g of Fresh Zeolite WBMs*

Figures 17 to 20 depict the shear rate – shear stress relationships for the control and modified mudformulations, each measured at various temperatures. Upon the introduction of 0.5 g of E-Z, stabilization was not evident, as shown in Figure 18. It appears that higher concentrations will likely be necessary to achieve the desired stability. However, with the addition of 1.0 g of E-Z, the fluid seemed to stabilize across the temperature range of 200 °F to 360 °F at all shear rates. This stabilizing effect is most pronounced at 511.02 s-1, as illustrated in Figure 19.

Upon incorporating 1.5 g of E-Z, stabilization became apparent at shear rates of 10.2204 s-1 and 170.34 s-1, as presented in Figure 20. However, at shear rates of 511.02 s-1 and 1022.04 s-1, the shear stress values began to rise with increasing temperature. It seems that 1.0 g of E-Z could mark a threshold beyond which the nanoparticle's stabilization ability becomes less predictable.

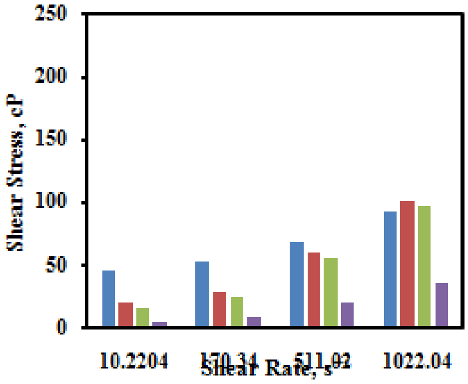

Fig17. *Rheogram of 0.0 g of Equilibrium Zeolite WBM*

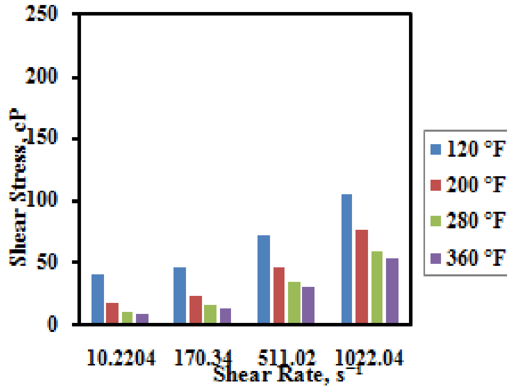

Fig18. *Rheogram of 0.5 g of Equilibrium Zeolite WBM*

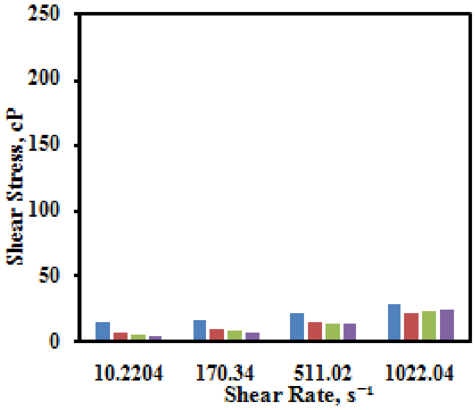

Fig19. *Rheogram of 1.0 g of Equilibrium Zeolite WBM*

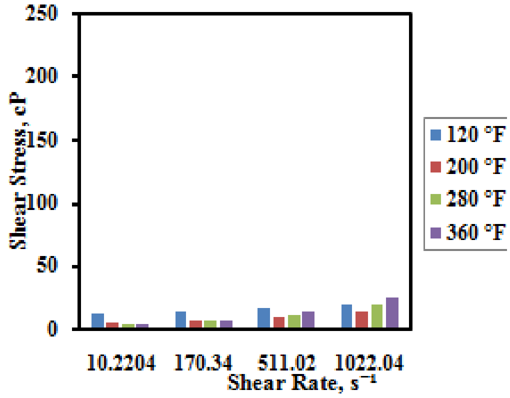

Fig20. *Rheogram of 1.0 g of Equilibrium Zeolite WBM*

3.4. Thermal Stability Performance Ranks

Table 2 displays the computed standard deviations of shear stress along with the corresponding mean values. As can be seen, the 1.5 g F-Z mud formulation had the least average standard deviation for its shear stress values over the experimental temperature range. The 0.5 g E-Z formulation had the highest average. This implies that the 1.5 g of F-Z, overall, was the best-performing additive for thermal stability. The worst-performing was 0.5 g of E-Z.

Table2. *Calculated Shear Stress Standard Deviations for Aged Mud Samples*

Shear Rate (s^{-1})	**Standard Deviation of Shear Stresses (120 °F – 360 °F), cP**					
	Fresh Zeolite			**Equilibrium Zeolite**		
	0.5 g	**1 g**	**1.5 g**	**0.5 g**	**1 g**	**1.5 g**
10.2204	7.544	6.794	2.144	12.333	4.218	3.312
170.34	7.615	6.586	1.190	13.061	3.616	2.722
511.02	7.475	6.047	1.582	16.150	3.235	2.438
1022.04	7.527	5.445	3.021	20.147	2.353	3.800
Average	**7.540**	**6.218**	**2.164**	**15.422**	**3.356**	**3.068**

Table 3 shows the overall ranks of the six aged nano WBM formulations, from best-performing to worst-performing

Table3. *Sample Ranks*

Rank	Mud Sample(g)
1	1.5 F-Z
2	1.5 E-Z
3	1.0 E-Z
4	1.0 F-Z
5	0.5 F-Z
6	**0.5 E-Z**

The thermal durability conferred by the fresh and equilibrium zeolite catalysts could stem from factors such as the nanoparticle's surface area, size, and the presence of thermally conductive metals. Studies conducted by researchers like Smith et al. (2018) have linked thermal stability to the thermal conductivity of metallic components, enabling effective heat dissipation from fluids through Brownian motion. Consequently, the fluid's susceptibility to temperature fluctuations is diminished.

4. Conclusions

The study yielded the following noteworthy conclusions:

Fresh zeolite and equilibrium zeolite exhibited comparable impacts on the rheological characteristics of the WBM formulations. Both additives generally lowered plastic viscosities, yield points, and gel strengths of the fluids, while leading to increased fluid loss in filtration control experiments.

Both additives demonstrated thermal stabilization of the WBM formulations across various temperature ranges, with fresh zeolite proving more effective. Optimal results for shear stress and rheological properties were achieved with 1.5 g of F-Z, 1.5 g of E-Z, and 1.0 g of E-Z.

Both fresh zeolite and equilibrium zeolite displayed potential as fluid dispersants, evidenced by the consistent reduction in PV, YP, and gel strength reductions.

Introduction of F-Z and E-Z made the fluids to be less basic.

The fluid behavior models in all aged systems adhered to the Bingham Plastic and Herschel Buckley models.

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