## Performance of Equilibrium Zeolite in Water-Based Mud at Elevated Temperature Conditions\*

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## Abstract

This study looks at the performance of nano equilibrium zeolite treated drilling fluids at high temperatures conditions, and their potential as alternatives for oil-based muds (OBM). Mud samples for this study were prepared and aged with the equilibrium zeolite nanoparticles concentrations of 0.0 g, 0.5 g, 1.0 g and 1.5 g. Tests were performed to determine the rheological, filtration control properties, the pH and consequently the thermal stability of the study mud samples over a temperature range of 120 °F to 360 °F. One sample without the equilibrium zeolite served as a control for the study. From the results obtained, all the nano samples had their rheological properties not exhibiting much significant variation with temperature, thus they were more thermally stable, with the optimum nanoparticle concentration being 1.5 g. It was also hypothesized that the nano equilibrium zeolite behaved as thinners, since they were able to reduce the shear stresses, yield points, plastic viscosities and gel strengths of the mud samples as temperature was varied incrementally. It was therefore concluded that the optimum concentration of aged equilibrium zeolite has the potential to act as a thermal stability additive for Water Based Mud systems (WBMs).

Keywords: Nanoparticles, Equilibrium Zeolite, Thermal Stability, Water-Based Mud

## **1** Introduction

Drilling operations are progressing into High Pressure High Temperature (HTHP) arena as the limits of the world's oil resources fields are gradually moving towards deeper depths. Studies have shown thermal stability of WBMs in HTHP arenas becomes quite challenging due to their potential degradability at higher temperatures as opposed to OBMs (Amanullah *et al.*, 2009). OBMs are consequently more desirable at such hostile depths even though comparably, they are costly and ecologically unfavourable.

Drilling HTHP formations demands the use of mud with a significant thermal robustness in order to conserve the rheology under such conditions. OBMs and other Synthetic Based Mud (SBMs) systems mostly have the intrinsic characteristic to withstand elevated temperature conditions. with а disadvantage being their environmental pollution issues. Consequently, it is necessary to advance WBM systems with robust thermal stability, since WBMs are more environmentally friendly. Some studies have proposed myriads of additives to enhance the thermal stability of WBM systems which includes chemicals, polymers, nanoparticles etc.

Tehrani *et al.* in 2007 applied synthetic polymers in a water-based fluid to enhance its fluid-loss control and thermal stability characteristics at HPHT (350 °C, 500 psi) conditions. They concluded that, a terpolymer of acrylamide, sulfonated monomer and vinylpyrrolidone achieved the best as a chrome-free additive in the laboratory with a crosslinked copolymer of acrylamide and a sulfonated monomer being the next best as both additives produce low shear strength at elevated temperature.

Jung *et al.* (2011) did some experiments to study the effects of iron oxide nanoparticles on bentonite muds at elevated temperature and pressure conditions (20 °C to 200 °C and 1 to 100 atm). They observed increments in viscosity and yield stress with increased iron oxide concentrations, temperature and pressure.

Nasser *et al.* (2013) also studied the performance of a nano-enhanced WBM over a temperature range of 25 °C to 90 °C, and observed stability of the rheological properties over the study temperature range of investigation.

Smith *et al.* (2018) probed the possibility of using nano-enhanced water-based fluids with silica and aluminium oxide as additives in advanced drilling operations. Their experiments were conducted between 23 °C and 120 °C, and their results demonstrated that nano-enhanced WBMs have enhanced rheological and filtration properties at the optimum concentration of the nanoparticle, and the fluids have improved thermal stability at heightened temperatures.

A study by Amanullah *et al.* (2009) shows that at severe HTHP conditions (above 125-130 °C), drilling fluid systems treated with macro and micro scale size materials become significantly degraded, causing reductions in their gel strength and viscous flow properties. Though these chemicals and polymers have received quite considerable attention



in drilling fluid studies, zeolites are yet to receive close attention.

This paper seeks to explore the thermal stability potential of equilibrium zeolite (E-Z) in WBM. Equilibrium zeolite reserves the ability to endure high temperatures without degrading, and also composes of some conductive metal elements (de Castro *et al.*, 2019). For these reasons, E-Z was considered as an additive for this study.

## 2 Resources and Methods Used

The fluid properties below were obtained after undertaking a set of laboratory tests:

The rheological properties of the prepared water-based mud samples mixed with equilibrium zeolite at a test temperature range of 120 °F – 360 °F, and the filtration control capacity and also the pH of the samples.

# 2.1 Preparation of the Water-Based Samples

Several experiments were executed to determine the performance of the rheological properties of the prepared mud samples with different mass concentrations of nanoparticle (Table 1).

The suggested API standard for field testing of WBMs was applied in the preparation of the mud samples.

Table	1	Mud	I Preparation	Additives	for a	10.4	
		ppg	Water-Based	Drilling	Fluid	with	
		Equilibrium Zeolite (E-Z)					

Materials	Quantity	
1. Fresh water	0.91 barrels	
2. Soda Ash	0.1 ppb	
3. Bentonite	12.5 ppb	
4. Caustic Soda	0.3 ppb	
5. Barazan D	0.8 ppb	
6. PAC L	21 ppb	
7. Barite	101.8 ppb	
8. E-Z (mean particle size of 100 nm)	0.5, 1, 1.5 ppb	

The addition of additives was done according to API 13B. To guarantee a homogeneous mixture, the samples were mixed between 15 and 20 minutes with the aid of a mud mixer.

## 2.2 Mud Aging

The high temperature mud aging chamber was employed to mimic the heating and shaking which the drilling fluid is likely to be exposed to at elevated temperature conditions. Samples were aged at 250  $^{\circ}\text{C}$  (482  $^{\circ}\text{F})$  for 16 hours before tests were performed.

## 2.3 Density Test

To ensure that the prepared samples for the experiments were within the acceptable density, a standard mud balance was used to determine the density as a quality assurance measure. The instrument was calibrated using fresh water to give a reading of 1000 kg/m<sup>3</sup> (8.345 lb/gal or 62.4 lb/ft<sup>3</sup>) at 70° F  $\pm$  5°F (21 °C). This was done to check for its accuracy.

## 2.4 Rheology Test

A Model 1100 HPHT rheometer was used to determine the rheological properties of the formulated WBM samples. Essentially, the shear rate/shear stress values were obtained from the rheometer at different temperatures between 120 °F (49 °C) and 360 °F (182 °C) at an 80 °F (27 °C) interval. This was to ensure any thermal changes within the fluid rheology are captured within the study temperature range.

## 2.5 Fluid Loss Test

HPHT Filter Press was employed to determine the volume of fluid loss from the samples after 7.5 and 30 minutes.

## 2.6 pH Test

A pH meter, fitted with a glass electrode was employed to determine the pH of the mud samples.

## 2.7 Properties of the Equilibrium Zeolite

The properties of the E-Z nanoparticles were determined using thermogravimetric analysis (TGA), energy-dispersive X-ray spectroscopy (EDS) and a scanning electron microscope. These test actually, provided the evidence for the absence or presence of thermal stability in the nano-WBM samples.

## Thermogravimetric Analysis (TGA) Plot

The TGA plot for the equilibrium zeolite is displayed in Fig. 1. The graph shows that the nanoparticles have the ability to endure extreme temperatures, until decomposition occurs. E-Z degraded between 500 °C and 600 °C, implying it is thermally stable up to those temperatures.





#### Fig. 1 Thermogravimetric Analysis Chart for Equilibrium Zeolite

Energy-Dispersive X-Ray Spectroscopy (EDS) Plot

The elemental composition of the equilibrium zeolite used is shown in Fig. 2. The E-Z was composed of Calcium, and Aluminium in major quantities, with elements like Silicon, Sodium and Carbon occurring in smaller quantities. This is pertinent because some elements are good conductors of heat, while others are not. Conductors will influence the thermal stability capability of the zeolites.



Scanning Electron Microscope Images

Fig. 3 shows an image gotten from a scanning electron microscope. The shapes of the nanoparticles are shown in this figure. From the figure, it can be seen that the nanoparticles were mostly spherical.



Fig. 3 Equilibrium Zeolite Scanning Electron Microscope Image

## **3** Results and Discussion

## 3.1 Effects of E-Z on Fluid Model

The fluid model defines the flow behaviour of a fluid. The rheograms of all the samples throughout all the temperature ranges showed yield points greater than zero, with their index factors (n values) > 1, as observed in Figs. 4 to 7. This suggests that they are non – Newtonian fluids. The Herschel Buckley and Bingham Plastic models are derived from the nature of the curves. The inclusion of E-Z greatly lowered the shear stresses of the fluids. At 360 °F though, the 0.5 g sample had a greater shear stress than the control sample.



Fig. 4 Rheogram of Equilibrium Zeolite WBM Sample at 120 °F



Fig. 5 Rheogram of Equilibrium Zeolite WBM Sample at 200 °F





Fig. 6 Rheogram of Equilibrium Zeolite WBM Sample at 280 °F



Fig. 7 Rheogram of Equilibrium Zeolite WBM Sample at 360 °F

Thermal stability is shown in the rheological properties when they vary less with temperature. A fluid is more thermally stable if its rheological properties do not change much, and are not deteriorated as temperature increases. Hence, in all subsequent graphs for rheological properties, improved thermal stability can be seen when the property of interest varies less as temperature is increased. In addition, the WBM samples containing 0.5 g, 1 g and 1.5 g of E-Z nanoparticles will be referred to as the "0.5 g sample", "1 g sample" and "1.5 g sample" in the discussion, for clarity and simplicity.

#### 3.2.1 Plastic Viscosity (PV)

As temperature rises, the plastic viscosity of drilling mud reduces because of the breakdown of the additives in the fluid, and the degradation of the molecules of the base fluid. The control mud sample for the most part shows a decline in PV values with increasing temperature, as anticipated. Addition of equilibrium zeolite in increasing quantities as seen in Fig. 8, stabilizes the PV of the samples significantly as temperature changes. This is seen in the reduced variation in the PVs of the nano samples, compared to the relatively larger variation in the PV of the control sample with temperature. Variation is at its lowest with the 1 g and 1.5 g samples.

Stabilisation appears to be better with 1 g and 1.5 g of the nanoparticle. It was also observed that the PV of the 1 g and 1.5 g fluids were less than that of the control sample at constant temperature, which was possibly due to the actions of the equilibrium zeolite. It may have functioned as a mud thinner. The 0.5 g sample had its PV less than the PV of the control sample at 200 °F and 280 °F, which also suggests that 0.5 g of E-Z acted as a thinner at those temperatures, but did not do so at 120 and 360 °F. Overall, the 1 g sample provided the best stabilisation and met the minimum PV requirement from 200 °F to 360 °F, and almost met the same requirement at 120 °F. The 1 g sample also has sufficiently low plastic viscosities to enable easy pumping during operations.

#### 3.2.2 Yield Point (YP)

The control and 0.5 g samples in Fig. 9 had their yield points reducing as temperature rose. This could be due to greater molecular separation of the base fluid as it was heated, resulting in a lessened resistance to flow. Complete stabilisation is nearly attained after adding 1 g and 1.5 g of E-Z to the mud samples. The 1.5 g concentration provides better stability than the 1 g concentration, and should be considered when selecting the optimum concentration of E-Z for this specific situation. All but the YP values of the control sample from 120 °F to 280 °F and the 0.5 g sample at 120 °F meet the API standard of a maximum YP of 50 lb/100 ft<sup>2</sup>. The potential of E-Z to perform as a thinner is seen once more in Fig. 9, mainly from 120 °F to 280 °F and partially at 360 °F.

#### 3.2.3 Yield Point on Plastic Viscosity (YP/PV) Ratio

YP/PV provides an idea of the thickness of the mud sample. An increasing ratio signifies increasing thickness, and vice versa. In addition, the ratio expresses how often the mud is being reused. All the equilibrium zeolite fluid samples except the 1 g and 1.5 g samples at 120 °F were lower than the API standard of a maximum ratio of 3, as shown in Fig. 10. An overall reduction in YP/PV was observed for all the mud samples as temperature rose. The reduction in the ratios was caused by the decrease in the fluids' yield points as they were being heated. The PV values also decreased at certain times, though these reductions were not sufficient to cause a rise in the YP/PV ratios. As the concentration of the nanoparticles was increased, the samples had higher ratios at lower temperatures and then became thinner at higher temperatures. However, at constant temperature, the nanoparticles caused the fluids to become thicker. This goes against the hypothesis that the E-Z acted as a thinner by reducing the PV and YP in the previous discussions.

#### 3.2.4 Gel Strength

In Fig. 11, the 10 s gel strength values of the control mud sample reduced considerably as temperature increased. This is undesirable, because a fixed value of gel strength is required while drilling. After adding E-Z, the values seemingly stabilise over the entire temperature range. The 0.5 g sample does not perform well as temperature is increased and gives a gel strength of 0 lb /100 ft<sup>2</sup> at 360 °F, indicating that solids will not be suspended at that temperature during a halt in the drilling operation. The 1 g sample performs better than the 0.5 g sample does, but does not meet the minimum API requirement half of the time. This is also not suitable for operations. The 1.5 g sample performs worse than

the 1 g sample, barely making the lower limit and steadily increasing the gel strength of the fluid. It can be seen from these results that though the 1 and 1.5 g concentrations of E-Z provide some degree of stability, E-Z generally is a poor choice for the thermal stability of the fluid in reference to its gel strength.

Just like with the 10 seconds test, the control sample and 0.5 g samples have very poor thermal stability characteristics (see Fig. 12). Both 1 g and 1.5 g samples have much better stability probably because the higher quantity of nanoparticles resulted in an increase in the binding forces between the clays of the mud, resulting in a more stable fluid requiring a little more power or force to break. Again, the E-Z acted as a thinner by gradually reducing the gel strength values of the fluid at constant temperature.



Fig. 8 Plastic Viscosity of Equilibrium Zeolite Base Mud Sample at Varying Temperatures



Fig. 9 Yield Point of Equilibrium Zeolite Base Mud Sample at Varying Temperatures



Fig. 10 YP/PV Ratio of Equilibrium Zeolite Base Mud Sample at Varying Temperatures

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Fig. 11 Ten Seconds Gel Strength of Equilibrium Zeolite Base Mud Sample at Varying Temperatures



#### Fig. 12 Ten Minutes Gel Strength of Equilibrium Zeolite Base Mud Sample at Varying Temperatures

#### 3.2.5 Fluid Loss

The fluid loss volumes for the samples are shown in Fig. 13. The API standard of less than 12 ml after 30 min (API 13B) is not met by any of the samples. The amount of filtrate acquired after the test relies partially on the viscosity of the test fluid. A more viscous fluid, particularly, one with a greater solids concentration, will more often than not have a thicker filter cake being formed quicker, thus, forestalling the continuous fluid loss. The viscosity of the samples, particularly the control sample, could explain why a lot of water was lost during the test. It was previously observed that the E-Z nanoparticles potentially acted as thinners. Once the nanoparticles were added, viscosity of the fluids was decreased, resulting in an inferior mud cake being created. Larger volumes of water were thus lost after the experiment. This is obvious after 7.5 and 30 minutes. A potential trend could likewise be obtained from the plots, since steady increases in E-Z concentration resulted in equivalent increases in filtrate volumes. Again, decrease in pH of the mud sample with increasing E-Z concentration as can be deduced in Fig. 14 can lead to acidic degradation of the filter cake resulting in the excess loss of the filtrate.

#### 3.2.6 Hydrogen Ion Concentration (pH)

The control and 0.5 g samples were basic, with the 1 g and 1.5 g samples being acidic with pH values of 6.7 and 6.4 respectively, as can be observed from Fig. 14. Issues like corrosion and embrittlement, which occur because of the fluid's acidity, might be faced with the latter samples, though they might not be severe. The samples generally became more acidic with increasing E-Z concentration. 0.5 g may be a threshold, above which the WBM turns acidic.



Fig. 13 Filtration Loss of Equilibrium Zeolite Base Mud after 7.5 and 30 Minutes



Fig. 14 pH of Varying Quantities of Equilibrium Zeolite in Mud Samples

## 3.3 Thermal Stability

Thermal stability occurs when the shear stresses of a fluid remain constant with a rise in temperature. A large number of the rheological properties of a fluid, including plastic viscosity and yield point, are influenced by its shear stress. As long as the shear stress of a fluid remains consistent, its rheological properties will not vary much with temperature. The shear stresses of the control sample reduced with increasing temperature at all four shear rates. This was anticipated since at elevated temperatures, the drilling mud becomes less viscous. A fluid's viscosity and shear stress have a directly proportional relationship. Similarly, shear stresses (internal reactions to external shearing forces) and rates are directly proportional.

Upon addition of 0.5 g of E-Z (See Fig. 16), the fluid did not show signs of stabilisation. After 1 g of E-Z (See Fig. 17) was added, the fluid seemed to stabilise from 200 °F to 360 °F at all shear rates. This was most prominent at 300 rpm. After 1.5 g of E-Z was added as shown in Fig 18, stabilisation was observed at 6 and 100 rpm from 200 °F to 360 °F. At 300 and 600 rpm, the shear stress values began to rise with temperature.

1 g of E-Z may be a threshold beyond which the stabilisation ability of the E-Z nanoparticle is unpredictable. It also appears that E-Z stabilises the fluid by increasing the shear stress at higher temperatures in order to get them to be equal to or greater than the shear stress at lower temperatures.

This is seen from the rheograms of the 0.5, 1 and 1.5 g samples at 300 and 600 rpm. The right amount of the nanoparticle should therefore be selected carefully to prevent higher shear stresses than required at higher temperatures.



Fig. 15 Rheogram of 0 g of Equilibrium Zeolite Base Mud







Fig. 17 Rheogram of 1.0 g of Equilibrium Zeolite Base Mud

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Fig. 18 Rheogram of 1.5 g of Equilibrium Zeolite Base Mud

In order to rank the samples from best-performing to worst-performing, and further support the thermal stability discussion, standard deviation was used. The standard deviations of the shear stresses of each of the samples at the various shear rates was calculated. For example, at 10.2204 s<sup>-1</sup>, for 0.5 g E-Z, the standard deviation of the shear stresses recorded at each of the temperatures (from 120 °F to 360 °F) was calculated. This was done at all four shear rates, for all the WBM samples, producing a total of 16 standard deviation values. Averages of the standard deviations across the four shear rates for each sample were then calculated to help simplify the ranking. Essentially, four 'average' standard deviations were obtained, each representing one mud sample. The ranking was then made based on these average standard deviations. Table 2 shows the calculated shear stress standard deviations and the average values obtained. From the table, it can be seen that the shear stresses of the 1.5 g sample deviated the least from the mean value over the temperature range of investigation. The standard deviations were highest in the control sample, signifying poor thermal stability, and reduced with increasing E-Z concentration, signifying improving thermal stability.

## **3.4 Thermal Stability Explained**

The thermal stability obtained from E-Z might have stemmed from one or both of the following:

- i. The metals composition that conducts and disperses heat
- ii. The surface area and morphology of the nanoparticles

The metal elements in the nanoparticle samples aided in heat dispersion. The nanoparticles contained elements such as aluminium, calcium and sodium (in substantial amounts), which are all heat conductors. In addition, it contained carbon and silicon, which are semiconductors (Fig. 2). Heat dispersion was assisted with the presence of these components, reducing the effect of rising temperature on the WBM sample.

In addition, the surface area to volume ratio of a material made of nanoparticles has a considerable impact on the characteristics of the substance.

Generally, a substance made up of nanoparticles will have a relatively bigger surface area than a similar substance formed from larger particles. Greater surface areas enable heat conduction and successive dispersion. In addition, with the reduction of particle sizes, a greater percentage of the particles are found at the surface of the material. This enables additional electrons to be available for heat transfer. The small size of the nanoparticles also permits free movement and hence micro-convection, which helps heat transfer (Das *et al.*, 2006).

WBMs preserve their rheological properties if their shear stresses change minimally with temperature. Stability can be achieved by taking away the impact of heat on the fluid, maintaining the shear stress and eventually the fluid's rheology. The impact of temperature is opposed by the presence of nanoparticles, and this achieved by the conduction and dispersion of heat by the metal elements and the large surface areas of the nanoparticles.

Table 2 Calculated Shear Stress Standard Deviations for WBM Samples

	Standard Deviation of Shear Stresses (120 °F – 360 °F), cP					
Shear Rate (s <sup>-1</sup> )	Control	E-Z WBM Samples				
	0 g	0.5 g	1 g	1.5 g		
10.2204	15.059	12.333	4.218	3.312		
170.34	16.291	13.061	3.616	2.722		
511.02	18.644	16.150	3.235	2.438		
1022.04	26.774	20.147	2.353	3.800		
Average	19.192	15.422	3.356	3.068		



## **4** Conclusions

The following key findings were made at end the study:

- (i) E-Z significantly decreased the fluids' PVs, YPs and gel strengths, whereas increasing the filtrate volume during filtration control tests
- (ii) E-Z succeeded in thermally stabilising the mud samples over the study temperatures range, with the 1 g sample providing the optimum outcomes. Per the shear stress standard deviation calculations made, 1.5 g is the optimum concentration.
- (iii) E-Z indicates from the study, a potential to act as a mud thinner. This is obvious from the continued decrease of the yield, plastic viscosities points and gel strengths of the fluids.
- (iv) E-Z generally caused the mud samples to become more acidic.
- (v) The fluid model behaviours of all the mud samples portrayed yield power law model (combination of Herschel Buckley and Bingham Plastic models).
- (vi) Since the E-Z nanoparticles offered some degree of thermal stability, in their right concentrations, they can be used in WBMs as alternatives to OBMs.

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