

## Investigating Improved Rheological and Fluid Loss Performance of Sepiolite Muds under Elevated Temperatures

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### ABSTRACT

The most common commercial clay used in drilling fluids is so-called Wyoming bentonite. Since the salinity of water greatly affects the hydration ability of commercial bentonite, a fibrous clay mineral called attapulgite may be used when the water salinity is too high for the use of bentonite. However, inadequate performance of attapulgite based drilling fluids in high temperature geothermal environments requires the search for substitute clays. Therefore, sepiolite, a magnesium silicate clay mineral with fibrous texture, has been proposed as the attapulgite replacement for both the high temperature and the high salinity environment. Although there could be temperature dependent minor changes in crystalline structure, sepiolite is stable at temperatures up to 260°C. Additionally, the basic structure of sepiolite is known to be firm in saturated saline-water phase.

This study is an attempt to better characterize both rheological and fluid loss behavior of water-based drilling fluid prepared with sepiolite clay. The effects of grain size, mixing time and mixing speed on elevated temperatures have been investigated. Substantial differences on rheological properties as a function of mixing time and mixing speed have been observed. No additives other than salt have been used while formulating sepiolite muds to determine the rheological and filtration properties. The results have indicated that the sepiolite based drilling fluid is superior to the bentonite and/or attapulgite based drilling fluids in terms of both rheological and fluid loss properties under elevated temperature conditions, particularly at high salt concentrations.

### 1. INTRODUCTION

Geothermal fields often exhibit highly fractured intervals, i.e., common loss circulation zones and, thus, require cementing frequently. High temperature environment coupled with cement contamination either limits or prevents the usage of any water base drilling fluid system. However, the gelation tendency of drilling fluid due to high temperature is avoided by employing sepiolite based mud systems.

A large number of clay minerals with widely different properties are present in nature. In general, high-swelling clays are desirable and added to the drilling fluid for viscosity and filtration control. Low-swelling clays, however, enter the drilling fluid in the form of cuttings and cavings, and are referred to as contaminants.

The drilling engineer is concerned with the selection and the maintenance of the best drilling fluid for the job. The drilling fluid is associated either directly or indirectly with

most drilling problems. If the drilling fluid does not perform adequately, it could become necessary to abandon the well. Therefore, extreme care must be taken into consideration when formulating drilling fluids under such restrictions.

Fresh water-bentonite mud used in geothermal wells can easily be damaged under high temperature environment; i.e. greater than 175 °C due to flocculation phenomenon of bentonite plates. This phenomenon affects the drilling process unfavorably and increases the drilling cost. In addition, no logging and temperature measurements could be run due to mechanical difficulties in lowering logging tools into a hole because of gelled mud. Another worse case situation for the same phenomenon occurs when brine intrusion is encountered as the drilling operation in progress. High temperature environment along with salt contamination result in unacceptable rheological and filtration properties for the use of fresh-water bentonite mud. Consequently, it would necessitate a complete renewal of mud system.

When the salinity of water greatly affects the hydration ability of commercial bentonite, a fibrous clay mineral called attapulgite may be used. However, insufficient performance of attapulgite based drilling fluids in high temperature environments requires the search for substitute clays. Therefore, sepiolite, a magnesium silicate clay mineral with fibrous texture, has been proposed as the attapulgite replacement for the environments having both high temperature and high saline concentration.

The clay mineral sepiolite belongs to a group of magnesium silicate with a fibrous texture whose idealized formula can be written as  $\text{Si}_{12}\text{Mg}_8\text{O}_{32}\cdot n\text{H}_2\text{O}$ , Bourgoyne Jr. et al. (1991). Structural characteristics and physical - chemical properties make sepiolite mineral unique in the clay mineral family. Although there could be temperature dependent changes in crystalline structure, sepiolite is stable at temperatures up to 425 °C (800 °F), Bourgoyne Jr. et al. (1991). Some investigators; for instance, Carney et al. (1976 and 1980), Serpen et al. (1992), and Serpen (1999) have indicated that sepiolite is temperature resistant clay with low filtration properties up to 260 °C. Additionally, the basic structure of sepiolite is known to be firm in saturated saline-water phase, Serpen et al. (1992).

Numerous investigators have studied to formulate a water-based mud system that can be used in the high temperature and the high brine environments, Carney et al. (1976, 1980, and 1982), Hillscher and Clements (1982), Moussa (1985), Guven et al. (1988), Zilch et al. (1991), Serpen et al. (1992), Serpen (1999), and Serpen (2000). Such mud can also be used in very deep oil wells that present similar conditions to the geothermal environment. One common point among the investigators is that almost all mud samples prepared with sepiolite clay contain abundant different additives to obtain suitable viscosity and filtration

properties that are necessary to accomplish safe drilling operations with minimum cost. Only one study among them compares sepiolite slurry with the other well known mud slurries, namely bentonite and attapulgite, Serpen et al. (1992). In their experimental work, they compared sepiolite mud with bentonite and attapulgite muds at room condition and obtained that sepiolite mud gave superior rheological and filtration properties for various salinities. However, mixing speed, mixing time and grain size variation when preparing sepiolite muds are not considered in this work by Serpen et al. (1992). The grain size effect on the rheological properties at room temperature of sepiolite slurries were investigated by Carney et al. (1976) and revealed the considerable differences.

It is well known fact that, the effects of mixing speed, the mixing time, and the grain size on rheological and filtration properties for the fresh water-based bentonite muds have trivial effect at room condition and can easily be neglected. On the other hand, the water-based sepiolite muds behave in an entirely unusual manner under specified conditions named above. Therefore, determination of accurate rheological and filtration properties of sepiolite based muds is essential for geothermal environment applications.

The objective of this study is to better characterize rheological and filtration properties of water-based sepiolite mud at varying mixing speeds, mixing times, and grain sizes under elevated temperatures. In other words, this study does not involve in controlling the rheological and filtration properties of sepiolite based muds by using excessive chemical additives. For this purpose, experiments are being conducted in laboratory on sepiolite samples taken from Sivrihisar-Eskisehir region in Turkey.

## 2. METHOD AND MATERIALS

API RP-13B Standard procedures are employed throughout the laboratory work to determine rheological and fluid loss properties. All the sample muds are based on the formulation of 350 ml of fluid that contains only fresh water and sepiolite. In addition, properties of sepiolite muds examined in this work are also investigated for different brine (NaCl) concentrations, 200 g/l and 400 g/l, respectively. Prepared muds are subjected to hydrothermal treatments in an aging cell that is rolled in an oven at temperatures up to 200 °C for 24 hours. First the aging cell is pressurized with nitrogen before placing it into the oven to prevent the evaporation of the sample. Then, the sample in the aging cell is cooled to room temperature. Rheological properties such as apparent viscosity, plastic viscosity, yield point and gel strength of the sepiolite slurries are measured on a Fann Model 286 variable speed viscometer before and after high temperature aging at ambient condition. Static filtration properties of the samples are measured by standard API filter press equipment.

## 3. EXPERIMENTAL WORK AND DISCUSSION

Huge sepiolite deposits are found around Eskisehir, Turkey. These clays, having different dolomite content and organic materials, are deposited within the lacustrine series in white, brown, and black colors. Composition of brown colored sepiolite used in this experimental work due to its high yield mud making value among the others is given in Table 1 below. The black sepiolite, regardless of the highest sepiolite content among others, did not provide better rheological and filtration properties due to high organic material content compared with that of the brown sepiolite.

Yields of muds prepared with sepiolite clays were also compared with that of commercial bentonite (Ca-Bentonite, so-called high yield clay) and attapulgite clays by Serpen et al (1992). Table 2 summarizes the determined yield values when the API standards were employed. Note that the all muds shown in the table were prepared for distilled water along with 200 mesh of grain size, 11,000±500 rpm of mixer speed, and 20 minutes of mixing time at room conditions. As can be seen, the mud yields increase with decreasing dolomite content and organic material that act as thinner, or dispersant, and cause the viscosity reduction.

**Table 1: Composition of Various Sepiolite Clays, Serpen et al. (1992).**

Sample	%Sepiolite	%Organic Material	%Dolomite
White	60	-	40
Brown	85-90	0.5	10-15
Black	97.5	2.5	-

**Table 2: Yield Comparison of Different Clays, Serpen et al. (1992).**

Type of Clay	Mud Yields, lbm/bbl
Sepiolite (Brown)	26.5
Sepiolite (Black)	27
Sepiolite (White)	30.5
Attapulgite	16.5
Commercial Bentonite	40

### 3.1 Apparent and Plastic Viscosity Characteristics of Fresh-Water Based Sepiolite Muds.

The effect of grain size and mixing speed on rheological properties were investigated. Variations on apparent viscosities for fresh-water based sepiolite muds for grain sizes passing 100, 140, 170 and 200 meshes are shown in Figure 1. As it is expected, apparent viscosities increase with increasing sepiolite content by weight. Three different concentrations of muds were prepared to construct Figure 1, namely, 3% (10.82 g), 6% (22.34 g), and 8% (30.44 g), respectively. On this figure, the effects of mixing speeds on apparent viscosities are also shown. It is clear that the apparent viscosity considerably increases with increasing mixing speed. For example in the case of 200 mesh sizes, a 15 cp apparent viscosity is observed at about 5.2% weight concentration for a mixing speed of 18,000 rpm whereas same apparent viscosity is observed at about 7.3% weight concentration for a mixing speed of 11,000 rpm. Apparent viscosities obtained from mixing speed of 18,000 rpm are approximately greater than twice that of 11,000 rpm as shown in Figure 1.

Yield of a clay is defined as the volume of mud that can be produced using 1 ton of clay if the mud has an apparent viscosity of 15 cp when measured in a rotational viscometer at 600 rpm. Weight percents of sepiolite that gives an apparent viscosity of 15 cp in Figure 1 were determined and corresponding yields were calculated for varying grain sizes and mixing speeds and given in Figure 2. It is found that variations in the mixing speed have important effects on mud yield whereas variations in the grain size have minor effect on mud yield. Yield of the samples with respect to increasing mesh size, in general, slightly decreases until a

grain size of 170 mesh; i.e. the finer the grains the lower the yields as shown in the figure. However, further increment in the grain size results in a noticeable increase in the yield values; for instance, a grain size of 200 mesh (74 micron). This phenomenon is postulated as the effect of grain size area that initiates different physical interaction between the sepiolite particles and fluid phase in the slurry and results in higher yields after a certain size. Similar behaviors regarding grain sizes were observed for both plastic viscosity and yield point measurements. That will be addressed later on this study. Note that the higher the yield of a clay, the lower the required clay amount to provide rheological properties and the lower the drilling fluid cost, not to mention the faster penetration rates and easier solid control. This result reveals that sepiolite based drilling muds are ought to be carefully designed to meet desired rheological properties; i.e. API Standards to prepare bentonite based muds may not be suitable to use when preparing sepiolite based muds.

### 3.2 Nonlinear Characteristics of Fresh-Water Based Sepiolite Muds.

Non-Newtonian rheological behavior of sepiolite based muds was observed. This phenomenon is shown in Figures 3 through to 5 for various grain sizes and mixing speeds. Shear stresses increase not only with increasing shear rates but also with increasing mixing speed and weight concentration of sepiolite clay. Again the effect of mixing speed is significant as in the other measured rheological properties. Note that data measured for 140, 170 and 200 mesh sized samples with weight concentration of 3% sepiolite mud mixed on 11,000 rpm given in Figure 3 are very close to each other; thus, it is not easy to distinguish their behavior curves from each other. A similar situation is encountered on the same figure for the samples with 100 and 170 mesh grain sizes for a mixing speed of 18,000 rpm. Again, enormous differences (about three fold) are shown from the Figures 3-5 at various mixing speeds. Non-linear behavior between shear stress and shear rate shown in the figures indicates that sepiolite based mud can be modeled by Non-Newtonian models, such as Bingham Plastic, Power Law, or Yield Power Law. In this study, statistical analysis is not carried out to determine a model that fits for the measured data best. However, Bingham model parameters, namely plastic viscosity and yield point, are calculated and plotted to further characterize the rheological properties of the sepiolite based muds.

### 3.3 Mixing Speed and Mixing Time Effects on Rheology of Fresh-Water Based Sepiolite Muds.

Plastic viscosity is a measure of the internal resistance to fluid flow resulting from interaction of solids and fluids in a drilling fluid. It is known that mechanical effects along with number, type, and size of solid particles in the fluid phase affect the plastic viscosity. Friction forces between the particles increases as the solid content increases, and shearing stress required to induce a unit rate of shear also increases. Plastic viscosity behavior of sepiolite mud samples with three weight concentrations were tested and results for only 6% weight concentration sample are provided with Figure 6. Mixing speed has an important role on plastic viscosity.

Yield point value is a measure of the internal resistance of a fluid to initial flow. Electrical charges holding colloidal particles together are the main causes of the resistance that is a function of the type, size, and amount of the submicron particles. Yield point value is usually known as the dominant mud property affecting circulating friction losses,

equivalent density, the transition point between laminar and turbulent flow, and cuttings transport efficiency. Experimental results obtained for sepiolite sample with 6% weight concentration at room condition is shown in Figure 7. There is more than a two fold increase in yield values obtained with a mixing speed of 18,000 rpm than that of 11,000 rpm. Similarly to the plastic viscosity case, the grain size difference has minor effect on the variation of yield point at constant mixing speeds. As stated before, increments on both plastic viscosity and yield point values at grain sizes of 200 mesh are postulated as a result of different physical interaction effect between the surface areas of clay particles and the liquid phase in the slurry after the grain sizes of sepiolite clay is reduced to certain sizes.

Mixing duration effect on apparent viscosity measurements is also conducted for 90 minutes, and measurements are carried out 15 minutes of intervals. Results obtained for the concentration of 3% sepiolite muds are shown in Figure 8, Caliskan and Misirli (2002). Four different mixing speeds 11,000, 13,000, 16,000, and 18,000 rpm were applied for the samples. Apparent viscosity increases as the mixing time increases for all the mixing speeds up to 60 minutes, and then apparent viscosity value stabilizes and remains constant until the maximum recorded time period of 90 minutes. As in the previous examinations conducted on rheological properties, maximum apparent viscosity is observed at the maximum mixing speed. It is evident that mixing time and mixing speed have enormous affect on apparent viscosity. It is also worth mentioning again that extreme care should be taken to formulate sepiolite muds when using API Standards since wide variation on rheological properties of sepiolite clays are observed at different mixing speed and mixing time.

### 3.4 Filtration Characteristics of Fresh-Water Based Sepiolite Muds at Room Condition.

The second part of this study considers the filtration characteristics for sepiolite based muds that are also used to characterize the rheological properties. Figure 9 shows the filtration loss results obtained for varying weight concentrations of sepiolite, mixing speeds, and grain sizes at room condition. It is clear that filtration characteristics of sepiolite based muds are not good to be used in any drilling operations; i.e. inadequate. Therefore, filtration control additives must be used to obtain acceptable results. Many researchers have worked on this subject and proposed many materials that perform in a good manner for different situations. However, sepiolite based muds with additives performs better than bentonite or attapulgite based muds in the environments where temperature and brine concentration are very high, Serpen et al. (1992), Carney et al. (1976, 1980), Guven et al. (1988), etc.

As expected, the filtration rate (ml/30 minutes) decreases as the sepiolite concentration increases and levels off when the concentration becomes 8% or more as shown in Figure 9. However, the filtrate property of sepiolite mud samples is still not favorable to meet the desired safe drilling process even in the range where the filtration rate seems to be leveled off. On the other hand, the mixing speed effect on filtration rate is not detrimental, but the effect gets marginal with the increasing clay concentration shown in the figure. It is clear that the higher mixing speeds yield lower filtration loss.

Filtration characteristics of sepiolite muds with 6% weight concentration and mixing speeds of 11,000 and 18,000 rpm at elevated temperatures up to 200 °C and different brine

concentrations up to 400 g/l were also investigated. After 24 hours of aging, the samples were cooled to room temperature, and the static measurements to determine API filtration loss were carried out and reported by Ergun (2004). It is revealed that the filtration rates enormously increase as the temperature increases for the fresh-water based sepiolite muds. However, it is also observed that the filtrate rates slightly decrease with increasing salt concentration. This situation is an indication of usefulness of sepiolite based muds in high brine environments that commercial bentonite based muds cannot be used. Another important result is obvious that the higher mixing speeds lower the filtration volume.

### 3.5 High Temperature and Brine Effects on Rheology of Fresh-Water Based Sepiolite Muds.

As it mentioned before, rheological properties of sepiolite muds with 6% weight concentration at elevated temperatures, brine concentrations, and mixing speeds were also studied and the results were given in Figures 10 thru 15 for apparent viscosity, plastic viscosity, and yield point variations, respectively. Grain size of 200 mesh (74 micron) sepiolite clay at 6% weight concentration was used for preparing the slurries in this part of the experiments. Direct rheological measurements were performed on using a water bath that was used for faster heating up the samples to 50 and 90 °C conditions. The samples were then placed in the viscometer's heating cup which was preheated to desired temperature, either 50 or 90 °C. Following this, the samples were tested to determine the rheological properties. Aging cell experiments were conducted in an oven for 24 hours to obtain rheological properties of the sepiolite based muds at elevated temperatures of 150 and 200 °C. After 24 hours of aging, the samples were cooled to room temperature, and the rheological properties were measured. The results shown in the figures were conducted for fresh-water based sepiolite muds along with two different sepiolite muds having brine concentrations of 200 and 400 g/l, respectively.

Temperature, salinity, and mixing speed effects on apparent viscosity shown in Figures 10 and 11 reveal that the apparent viscosity notably decreases with the increasing temperatures up to 150 °C, particularly for the case where the mixing speed of 18,000 rpm and the brine concentration of zero; i.e. fresh sepiolite mud. However, an improvement on apparent viscosity was observed after 150°C and more noticeable for the case of high mixing speeds. The temperature effect on the samples with a mixing speed of 11,000 rpm indicates slower reduction in the apparent viscosity. Moreover, the high mixing speeds give better apparent viscosity for the all temperature ranges. The apparent viscosity variation with respect to the brine concentrations is another point that should be further examined. As can be shown from Figure 10, the apparent viscosity gradually decreases up to temperatures of 90 - 150 °C and starts recovering back to as high as its initial values obtained at room condition as the temperature is increased to 200 °C for the all saline concentrations and the mixing speeds. This phenomenon is more evident as the brine concentration increases. It is evident that apparent viscosity of sepiolite muds as shown in Figure 11 decreases as the brine concentration increases from zero to 200 g/l concentration, but the apparent viscosity, all of a sudden, starts increasing with further increased brine concentration of 400 g/l, so-called fully saturated. In summary, it can easily be inferred that there is an inverse relationship between apparent viscosity and temperature ranges from ambient to 150 °C and linear relationship above the temperatures of 150 °C. In addition, the higher the mixing

speed and brine concentration, the higher the apparent viscosities on the temperature ranges analyzed. Note that mineralogical structure of sepiolite clay may undergo alterations due to thermal effects.

Plastic viscosity changes with temperature variations shown in Figures 12 and 13 indicate that the plastic viscosity remains fairly constant up to temperature of 90 °C and starts increasing rapidly with increasing temperatures beyond 90 °C. A faster increment on the plastic viscosity is evident particularly above the temperature of 150 °C. A minimum two fold increment on plastic viscosity was observed as shown in the figure between 90 and 200 °C temperatures. The effect of mixing speed on the plastic viscosity is more apparent as reflected in the figure. Significantly higher plastic viscosities at the mixing speed of 18,000 rpm were obtained compare to that of 11,000 rpm. The effect of brine concentrations on plastic viscosity behavior shown in Figure 13 is similar to that of mixing speeds. The plastic viscosity, in general, decreases as the brine concentration increases up to 200 g/l, and increases as the brine concentration increases further to 400 g/l. More brine concentrations such as increments of 50 g/l are of use to better characterize the effect of brine on rheological properties of sepiolite muds at elevated temperatures and mixing speeds. Note also that experiment for fresh water based sepiolite mud at 200 °C and 11,000 rpm conditions is not conducted and not shown in the figure.

Yield point properties of the sepiolite muds shown in Figures 14 and 15 indicate that high temperature has detrimental effect on preserving the yield point values. This unfavorable effect is more prominent at the mixing speed of 18,000 rpm compare to that of 11,000 rpm since better yield characteristics for sepiolite muds are attained at high speeds as explained previously. However, the yield point values level off for the all samples when the temperature reaches to 150 °C and remains fairly constant up to 200 °C. This could be the result of thermal alteration in the structure of sepiolite at temperatures higher than 150 °C. The most noticeable reduction on the yield point shown in Figure 15 was observed for the sample prepared with fresh water at the mixing speed of 18,000 rpm. Yield point values of the samples considerably decrease with increasing brine concentration, predominantly for mixing speed of 18,000 rpm.

## 5. CONCLUSIONS AND RECOMMENDATIONS

This experimental investigation is as an attempt to better characterize the rheological properties of sepiolite based drilling fluids under a variety of conditions in mixing times, mixing speeds, brine concentrations, and temperatures. Sepiolite clay behaves considerably astonishing and unstable when the conditions cited above are changed.

Elevated temperatures have detrimental effect on filtration properties of sepiolite muds. Filtration characteristics of sepiolite muds without additives are not suitable for any drilling operations.

Sepiolite based slurries perform better as the brine concentration reaches full saturation.

Yield and rheological properties of sepiolite muds are substantially affected by grain size variation of sepiolite clay. The finer the grain sizes the more the surface area of particles that controls the rheological behavior of slurry.

More grain sizes and brine concentration ranges should be examined to better characterize sepiolite suspensions.

Extensive care should be taken into consideration when using API Standards to formulate sepiolite based drilling fluids. The effects of grain size, mixing speed, and mixing time must be evaluated with proper additives.

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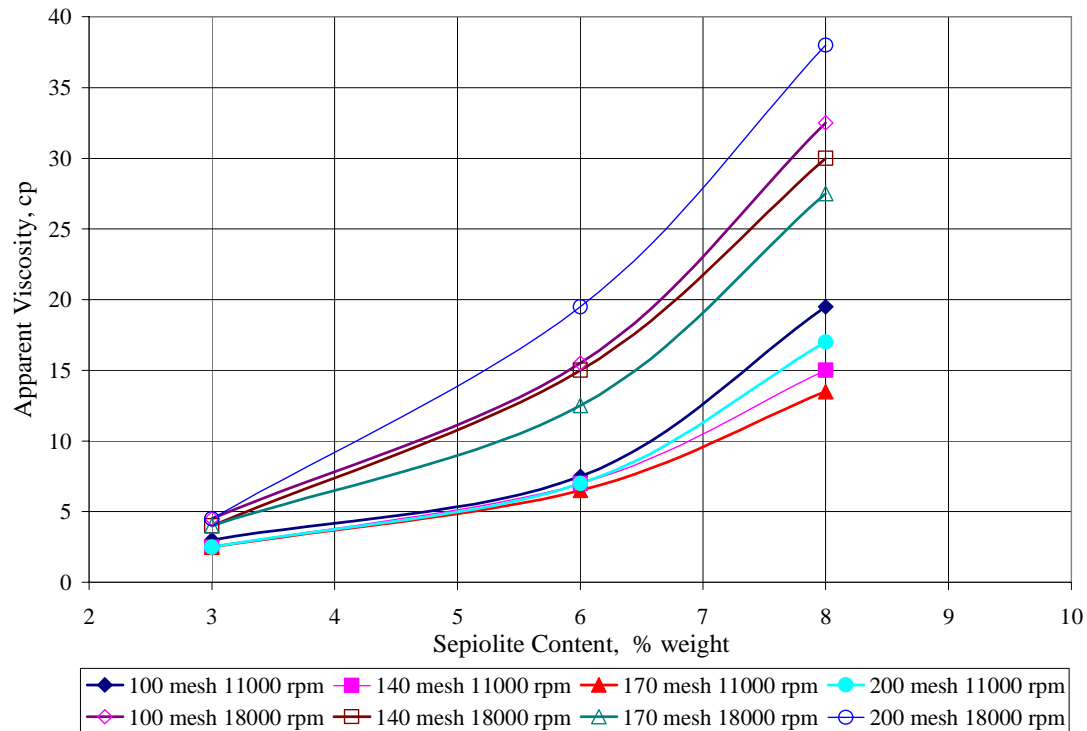


Figure 1: Grain Size and Mixing Speed Effect on Apparent Viscosity for Sepiolite Muds at Room Condition.

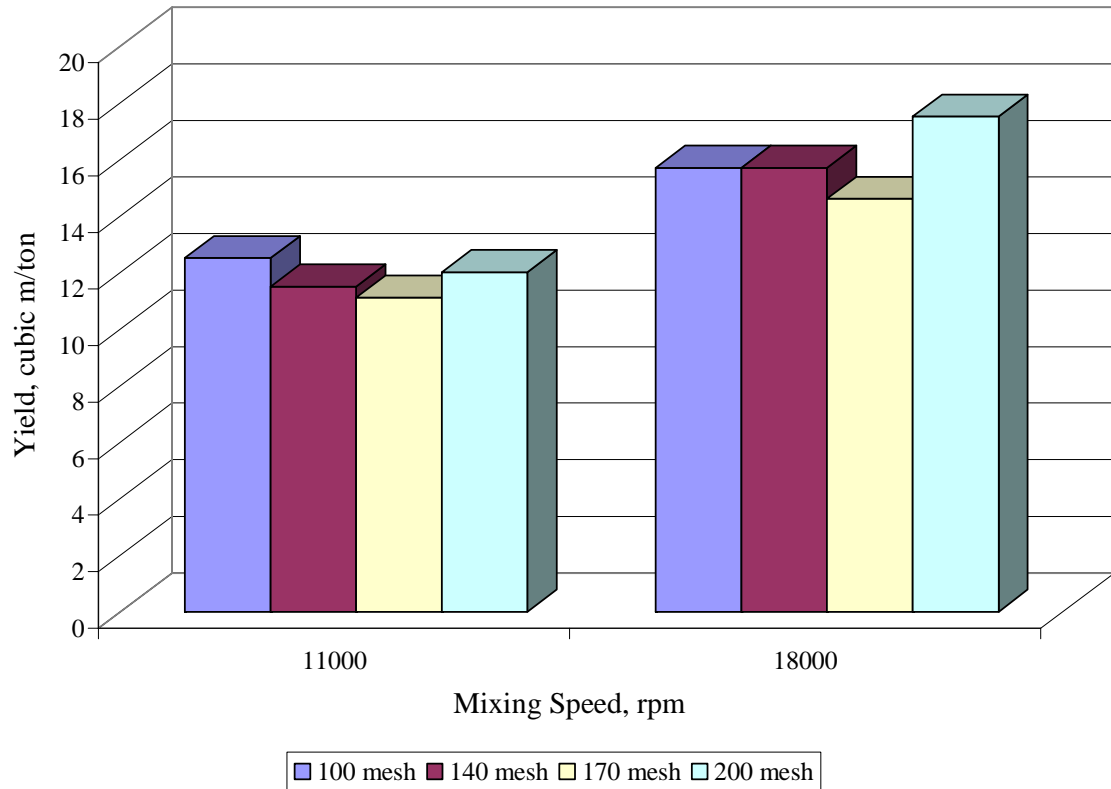


Figure 2: Grain Size and Mixing Speed Effect on Yield for Sepiolite Muds at Room Condition.

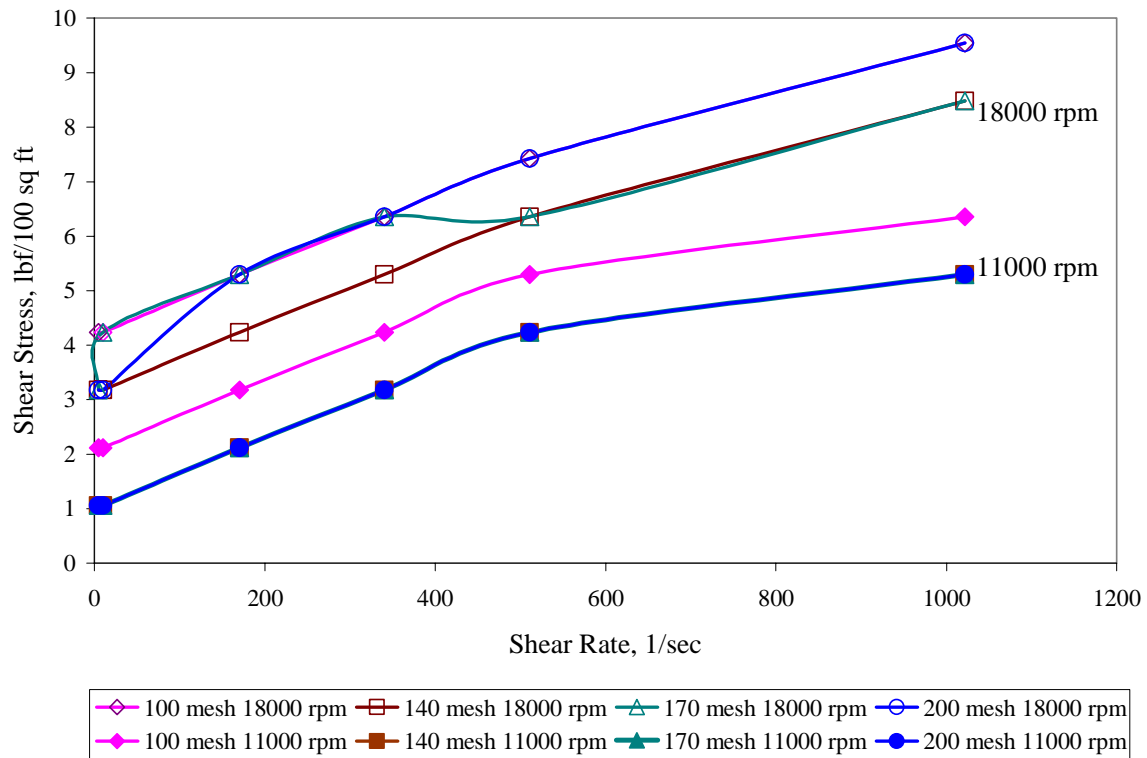


Figure 3: Shear Stress vs. Shear Rate Behavior for 3% Weight Concentration Sepiolite Mud at Room Condition.

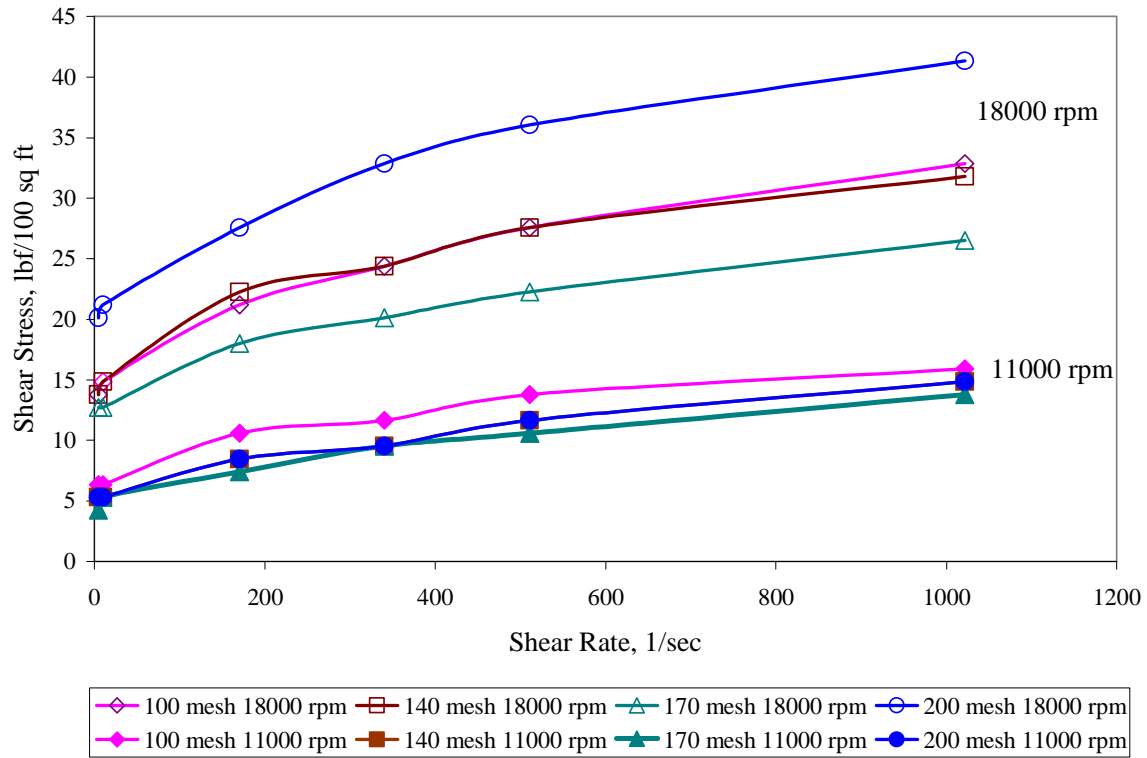


Figure 4: Shear Stress vs. Shear Rate Behavior for 6% Weight Concentration Sepiolite Mud at Room Condition.

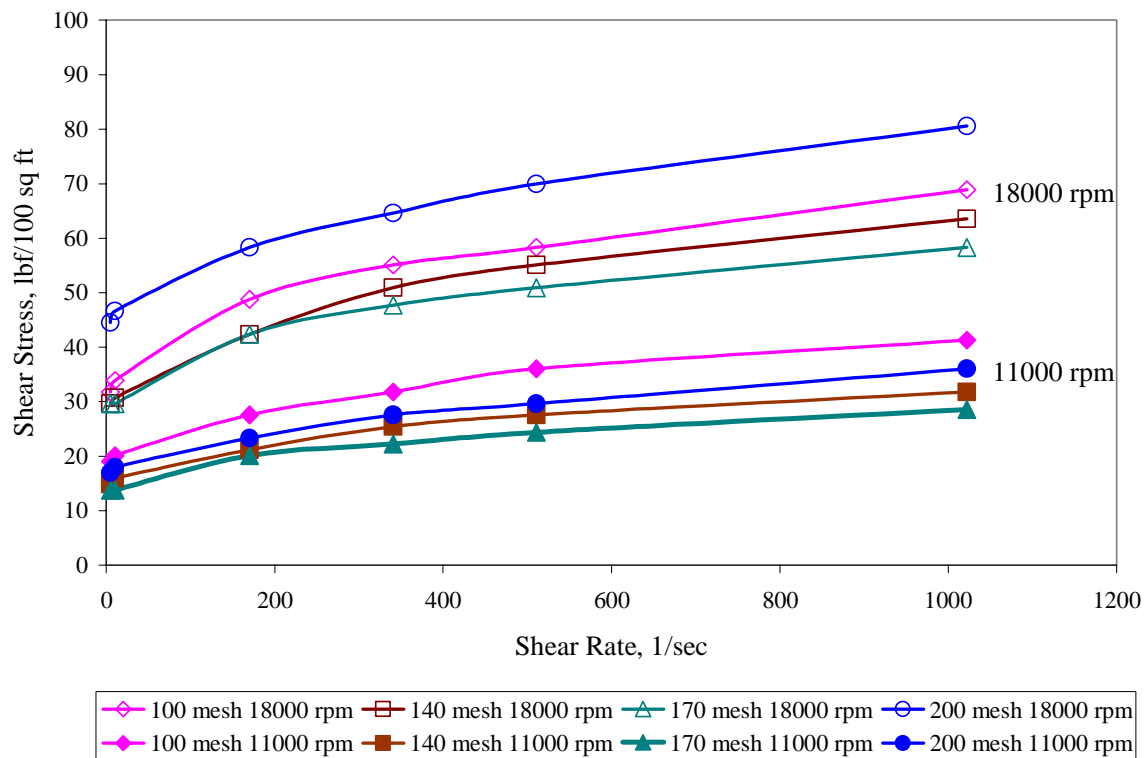
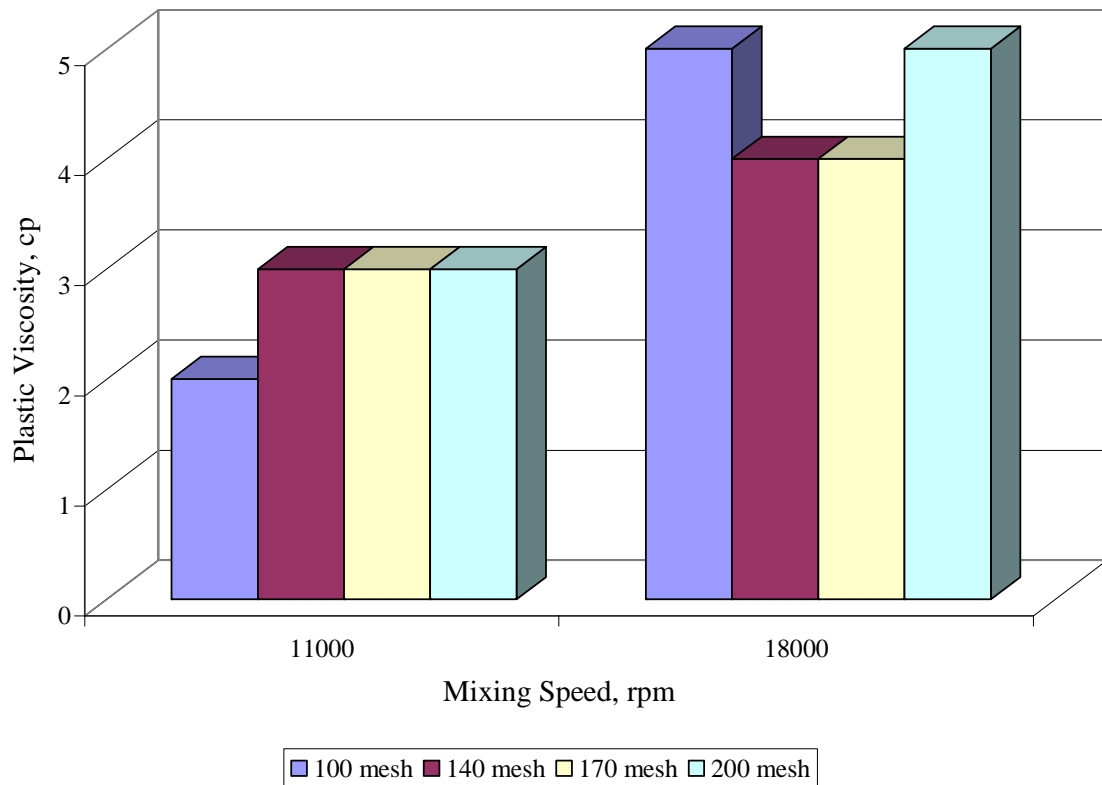
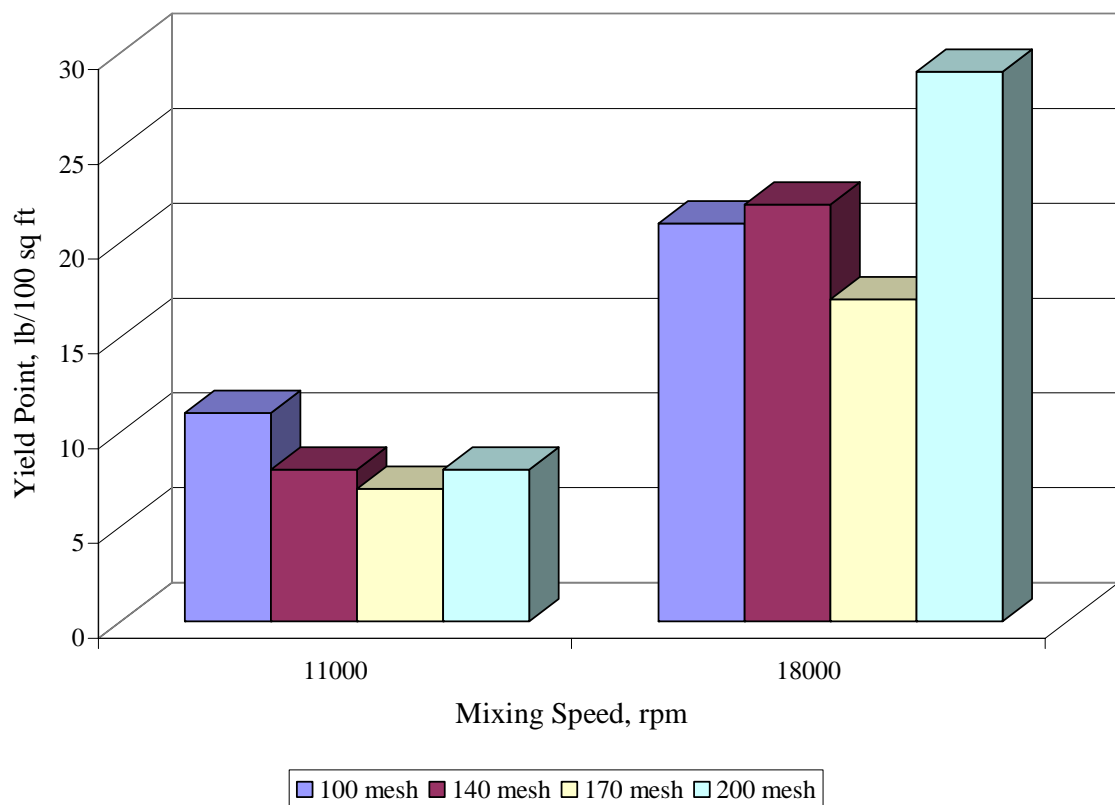


Figure 5: Shear Stress vs. Shear Rate Behavior for 8% Weight Concentration Sepiolite Mud at Room Condition.

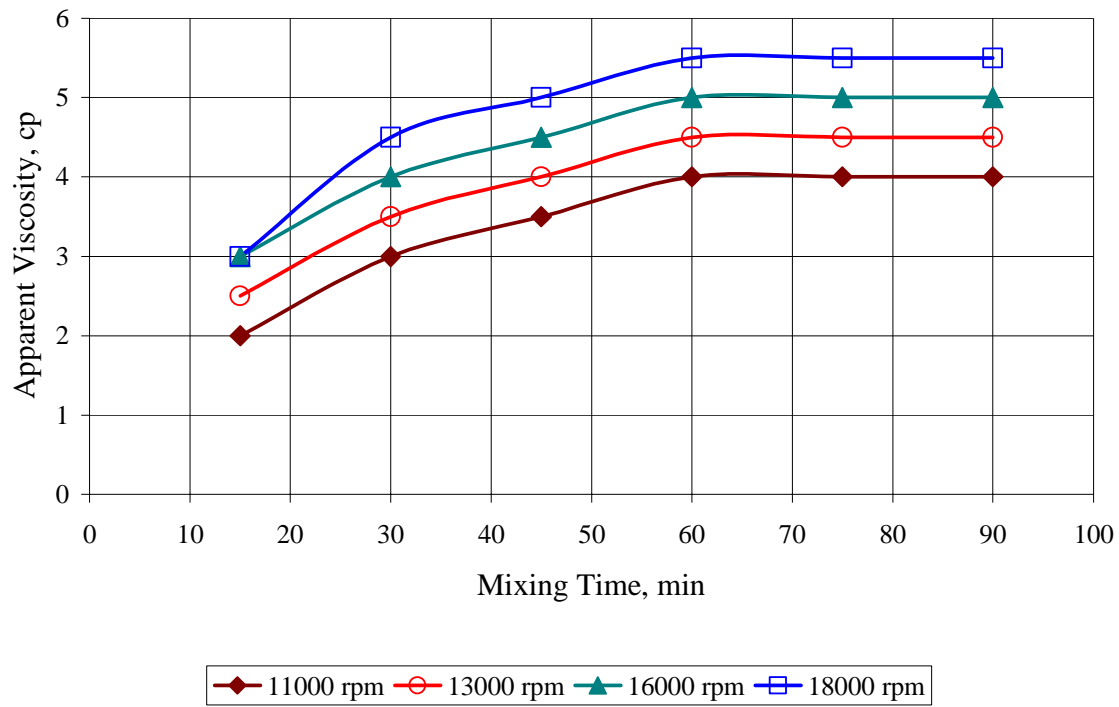


**Figure 6: Plastic Viscosity Behavior for 6% Weight Concentration Sepiolite Mud at Room Condition.**

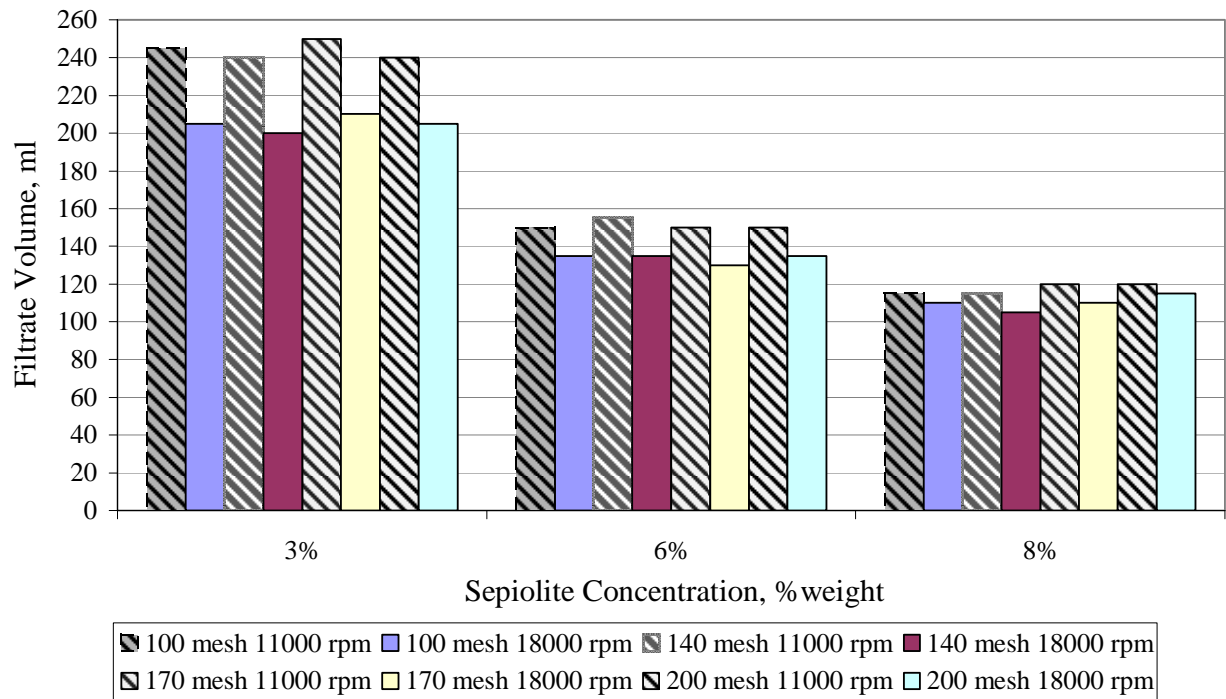


**Figure 7: Yield Point Behavior for 6% Weight Concentration Sepiolite Mud at Room Condition.**





**Figure 8: Apparent Viscosity Variation for 3% Weight Concentration Sepiolite Mud at Room Condition.**



**Figure 9: Filtrate Volume Variation Collected in 30 Minutes for Sepiolite Muds at Room Condition.**

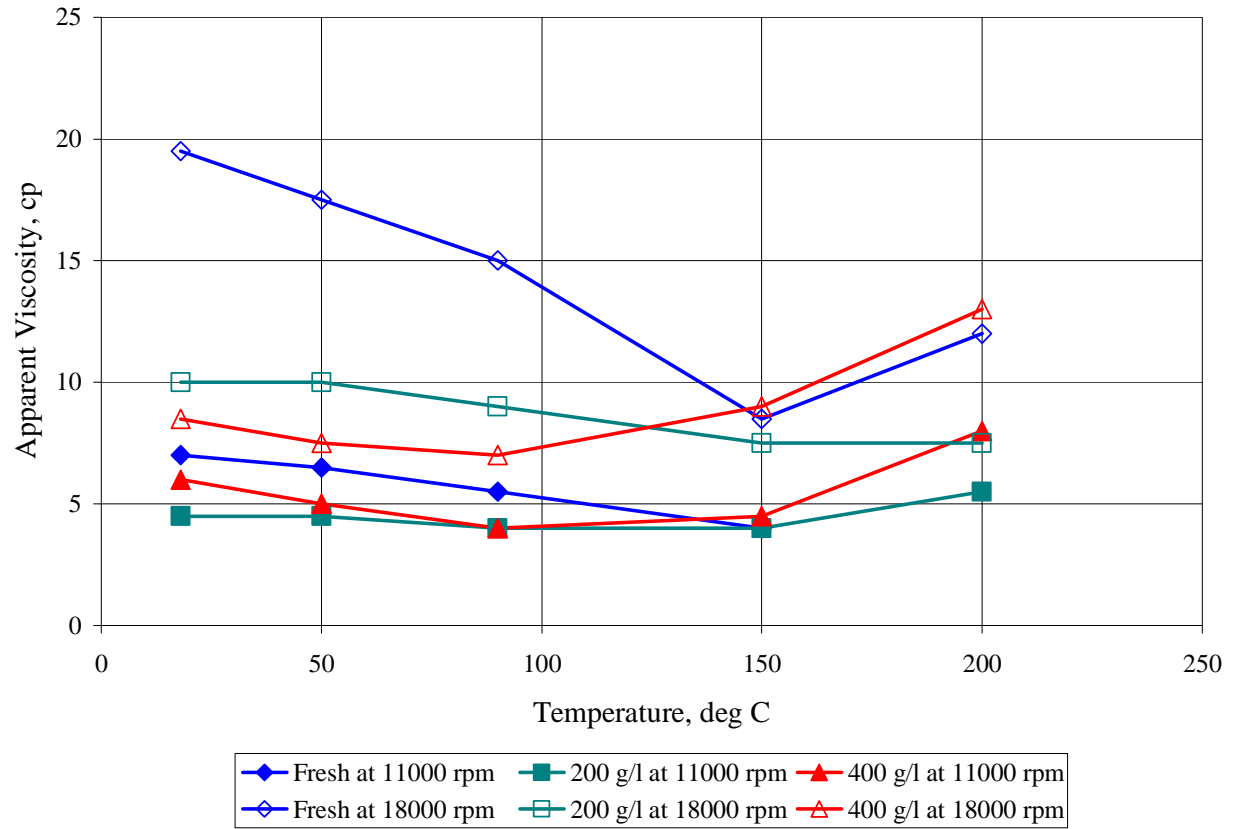


Figure 10: Salinity and Temperature Effect on Apparent Viscosity for Sepiolite Muds with 6% Weight Concentration.

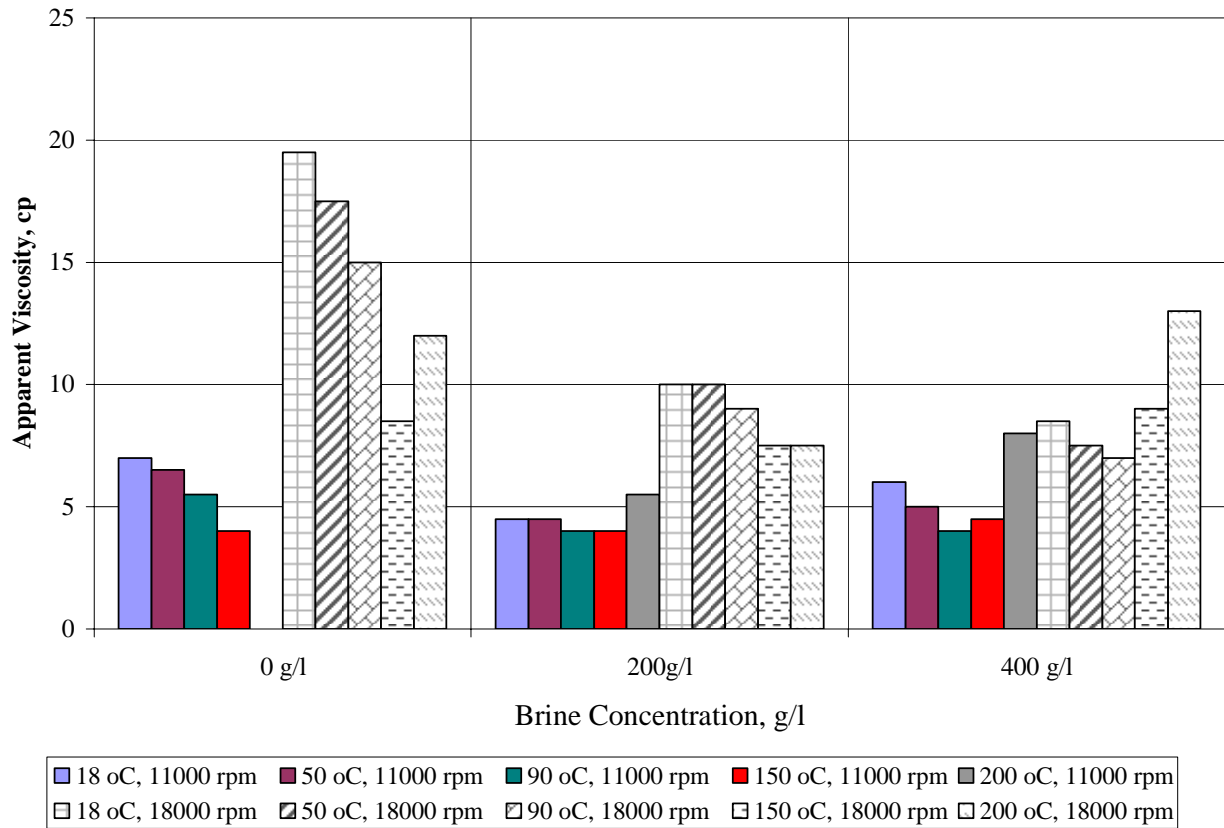


Figure 11: Brine Concentration Effect on Apparent Viscosity for Sepiolite Muds with 6% Weight Concentration.

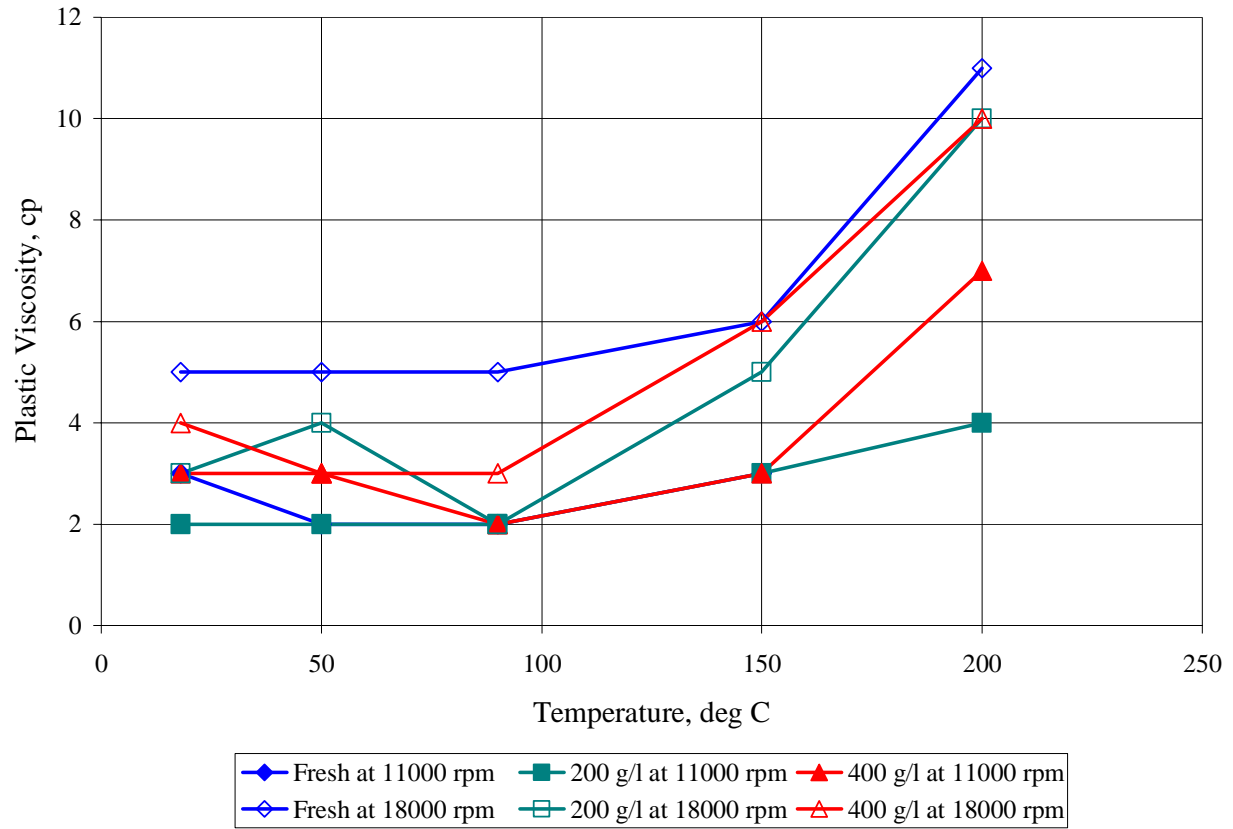


Figure 12: Salinity and Temperature Effect on Plastic Viscosity for Sepiolite Muds with 6% Weight Concentration.

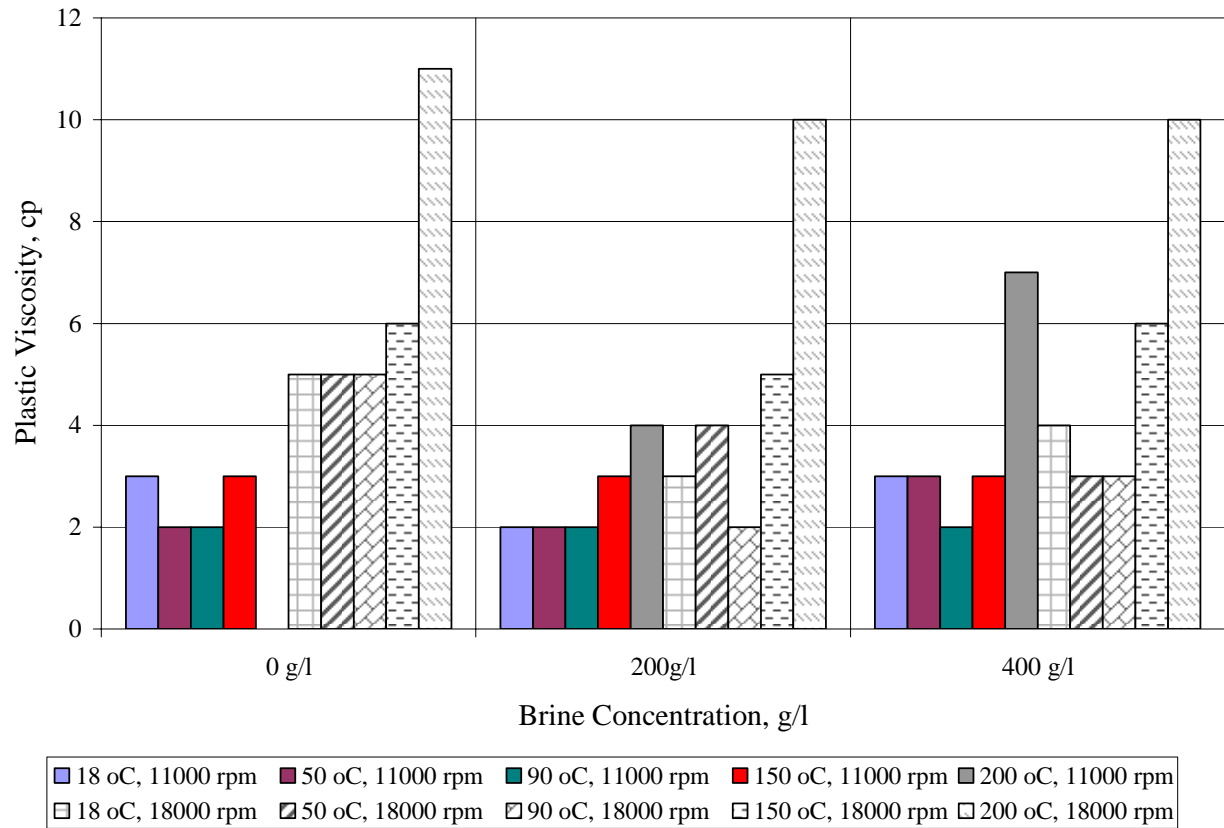


Figure 13: Brine Concentration Effect on Plastic Viscosity for Sepiolite Muds with 6% Weight Concentration.

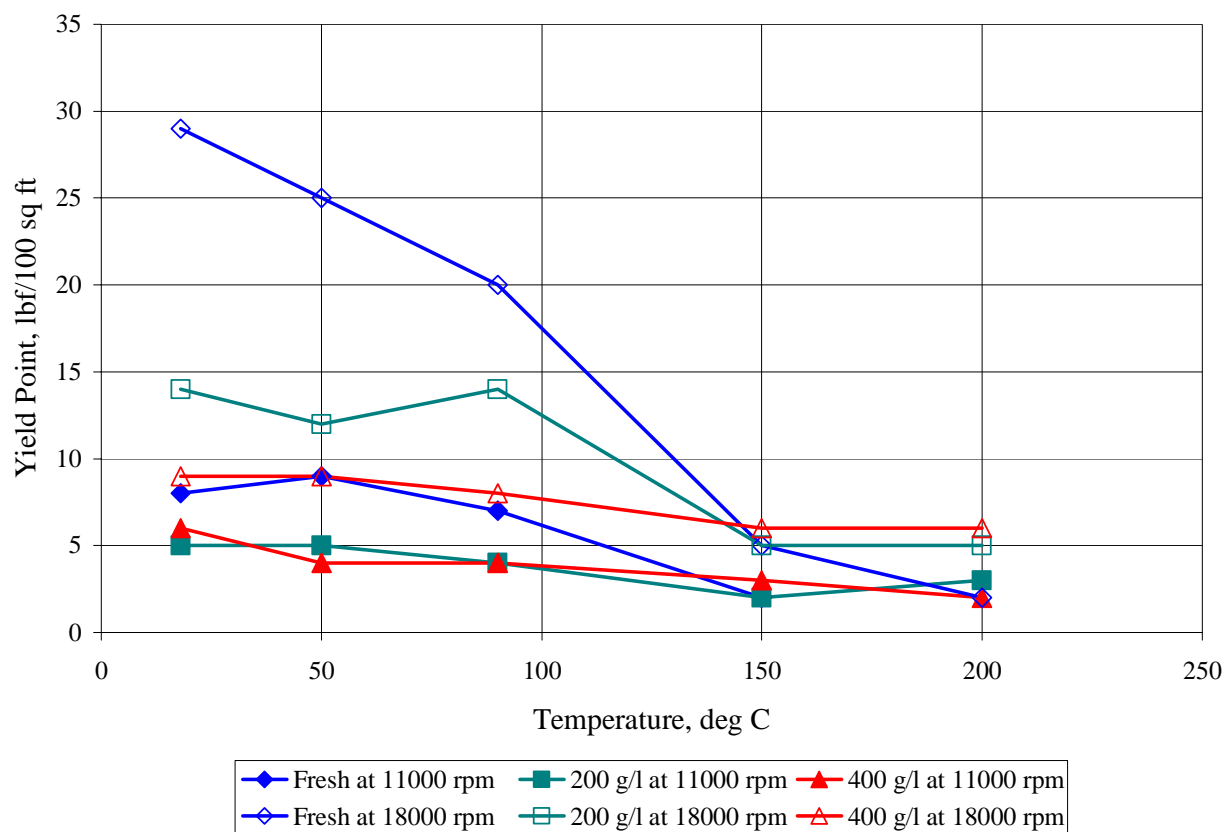


Figure 14: Salinity and Temperature Effect on Yield Point for Sepiolite Muds with 6% Weight Concentration.

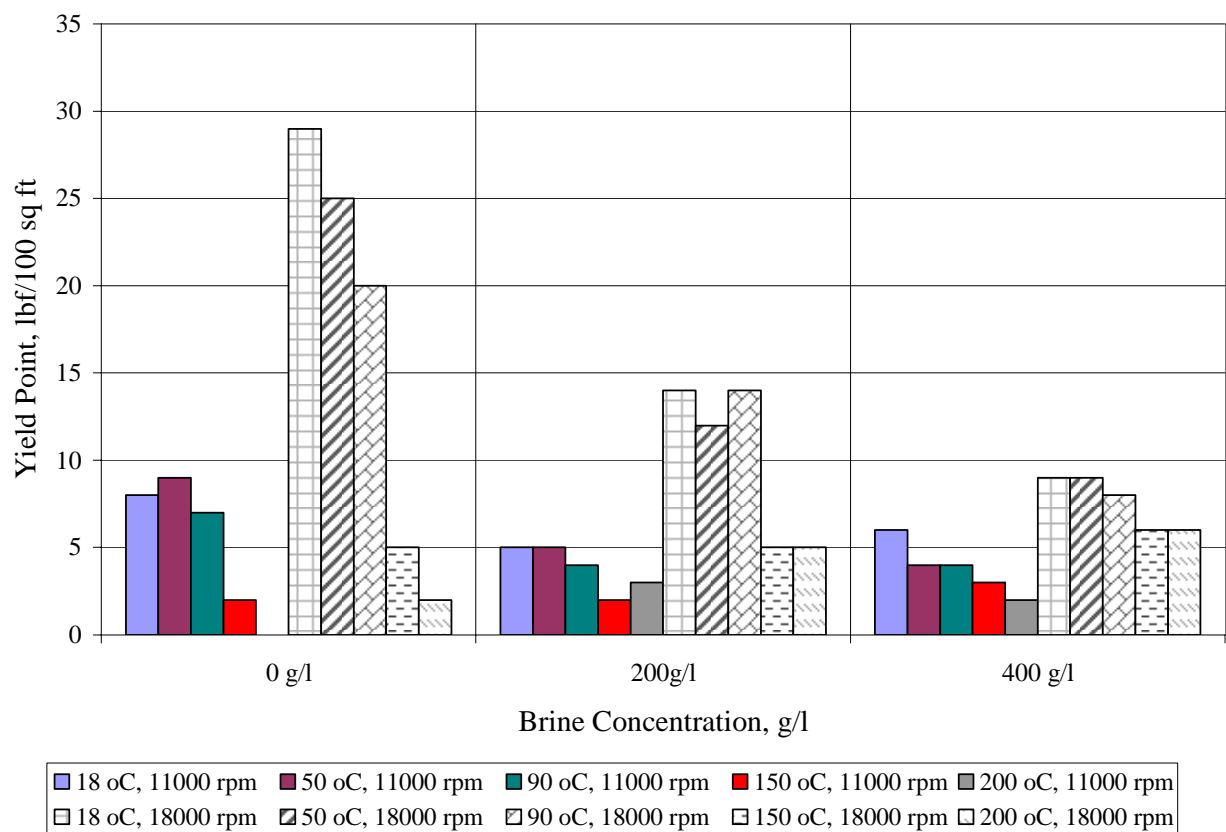


Figure 15: Brine Concentration Effect on Yield Point for Sepiolite Muds with 6% Weight Concentration.