<u>İSTANBUL TECHNICAL UNIVERSITY ★ INSTITUTE OF SCIENCE AND TECHNOLOGY</u>

CONTROLLING RHEOLOGICAL AND FILTRATION PROPERTIES OF SEPIOLITE BASED DRILLING FLUIDS UNDER ELEVATED TEMPERATURES AND PRESSURES

M.Sc. Thesis by Ali ETTEHADI OSGOUEI

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JUNE 2010

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<u>İSTANBUL TEKNİK ÜNİVERSİTESİ 🛧 FEN BİLİMLERİ ENSTİTÜSÜ</u>

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ABBREVIATIONS

- Cake deposition indexDynamic filtration rateCarrying capacity index CDI DFR
- CCI

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CONTROLLING RHEOLOGICAL AND FILTRATION PROPERTIES OF SEPIOLITE BASE DRILLING FLUIDS UNDER EVALUATED TEMPERATURES AND PRESSURES

SUMMARY

Deep oil and gas wells, particularly geothermal well drilling is known as high temperature environment; therefore, it is difficult to formulate a drilling mud functioning adequately, particularly in temperatures above 100 °C. Another common problem associated with these drilling environments is the formations that have high salt content in liquid phase of the pores. Circulation breaks, abnormally high fluid losses and viscosities, and unacceptable high gel strengths are the main problems that are usually associated with geothermal wells. Although bentonite based mud with extremely expensive additives is commonly used in these drilling conditions, it does not meet the desired needs in higher temperatures above 150 °C. There are geothermal fields having temperatures more than 240 °C in both Turkey and the world. Therefore, sepiolite, a magnesium silicate clay mineral with fibrous texture, has been proposed as the bentonite replacement for both the high temperature and the high salinity environment. There might be temperature dependent minor changes in crystalline structure; nevertheless, sepiolite is stable at temperatures up to 260 °C. Additionally, the basic structure of sepiolite is known to be firm in saturated salinewater phase.

This study is an attempt to characterize both rheological and fluid loss behavior of water-based drilling fluid prepared with five different raw sepiolite clay samples obtained around Sivrihisar-Eskisehir district of Turkey. The samples were not treated or purified by any chemical methods before and after grinding. API standards were followed throughout the experimental study. In the first step of study, no additives other than salt have been used while formulating sepiolite muds to determine the rheological and filtration properties. Then in the second step of study, some of special additives have been used to improve the properties of muds. Four out of five samples in ambient conditions have given better rheological property than that of indicated by the API standard. Two of the four samples satisfying the requirement having the best performance were selected and used later in the study along with additives to control rheological and filtration properties in high temperature and high pressure conditions. Moreover, dynamic filtrations of drilling fluid based on these two sepiolite clays have been determined at HTHP conditions. Finally one of these clays subjected to reactive clay contamination at high salinity condition is further investigated and its properties are controlled with additives. The results have indicated that the sepiolite based drilling fluid is superior to the bentonite based and other type of drilling fluids like KCL/PAC polymer system and synthetic muds (second and third generation polymer based muds) in terms of both rheological and fluid loss properties under elevated temperature and pressure conditions, particularly at high salt concentrations. In short, under the prescribed conditions like high salinity (fully saturated with NaCl or CaCl₂) and high temperature (200 °C), sepiolite based muds formulated in this study yield better rheological and filtration loss values than those of rivals. Most importantly, the cost of sepiolite based muds compared to those of others is three to five folds less at normal conditions. Cost effectiveness of sepiolite muds increases with increasing extremities like salinity, temperature, weight and contaminants.

SEPİOLİT TEMELLİ SONDAJ ÇAMURLARININ REOLOJİK VE FİLTRASYON ÖZELLİKLERİNİN YÜKSEK SICAKLIK VE BASINÇ KOŞULLARINDA KONTROL ALTINA ALINMASI

ÖZET

Derin petrol ve gaz kuyuları, özellikle de jeotermal kuyular yüksek sıcaklıklı ortam olarak bilinmektedirler, bu yüzden, özellikle 100 °C üzerindeki sıcaklıklarda istenilen performansta çalışan bir sondaj çamuru hazırlamak oldukça zordur. Yüksek sıcaklık ortamlarındaki sondajlarda rastlanan önemli problemlerden birisi de gözeneklerindeki sıvı fazda yüksek tuzluluk içeren formasyonlardır. Jeotermal kuyularda rastlanan problemler; sirkülasyon kayıpları, yüksek su kaybı ve istenmeyen yüksek viskozite durumları, kabul edilebilir olmayan yüksek jel kuvvetleridir. Bu durumlarda yüksek maliyetli katkı maddeleri ile birlikte kullanılan bentonit camurları 150 °C'nin üzerindeki sıcaklıklarda istenilen özellikleri sağlayamamaktadır. Gerek Türkiye'de ve gerekse dünyada sıcaklığı 240 °C'nin üzerinde olan jeotermal sahalar vardır. Bundan dolayı iğne yapılı bir magnezyum silikat mineral olan sepiyolit kili yüksek sıcak ve tuzlu ortamlarda bentonit kilinin verine kullanımı önerilmektedir. Her ne kadar sıcaklık etkisi nedeniyle kristal yapısında küçük değişiklikler olsa bile, sepiyolit 260 °C sıcaklıklara kadar yapısını korumaktadır. Bununla birlikte, sepiyolitin temel yapısının doymuş tuzlu su ortamında değişime uğramadığı da bilinmektedir.

Bu çalışmada Türkiye'nin Sivrihisar Eskişehir bölgesinden alınan beş farklı sepiyolit kili kullanılarak hazırlanan su bazlı çamurların reolojik ve su kaybı özelliklerinin belirlenmesi amaçlanmıştır. Sepiyolit killeri öğütmeden önce veya sonra herhangi bir kimyasal kullanılarak işleme ve saflaştırmaya maruz bırakılmamıştır. Deneyler yapılırken Amerikan Petrol Enstitüsü (API) tarafından belirlenen standartlar takip edilmiştir. Çalışmanın birince aşamaşında, sepiyolitlerin reoloji ve su kaybı özellikleri belirlenirken, içerisindeki yabancı maddeler uzaklaştırılmamış ve içerisine tuz dışında hiçbir katkı maddesi katılmamıştır. İkinci aşamada ise ticari katkı maddeleri kullanılarak reolojik ve su kaybı özelliklerindeki değişim incelenmiş ve su kaybı kontrol altına alınmaya çalışılmıştır. Çalışma sonucunda dört sepiolit örneğinin standartlarda belirtilen reolojik değerlerden daha iyi sonuç verdiği, dolayısıyla yüksek verimli killer sınıfına girdiği belirlenmiştir. Dört sepiyolit örneklerinden en iyi performansa sahip olan ikisi seçilmiş ve yüksek sıcaklık ve yüksek basınç koşulları altında katkı malzemeleri kullanılarak reolojik ve su kaybı özelliklerinin kontrol altına alınmasına çalışılmıştır. Bununla birlikte, bu iki sepiyolit örneğinin dinamik filtrasyon özellikleri yüksek sıcaklık ve yüksek basınç koşullarında incelenmiştir. Son olarak bu killerden biri, yüksek tuzluluk ortamında çamura aktif kil girişi olması durumundaki özellikleri deneysel olarak incelenmiştir. Sonuçlar göstermiştir ki özellikle yüksek tuzlu ve yüksek sıcaklık ve basıncın hakim olduğu ortamlarda, sepiyolit bazlı çamurlar, bentonit, KCL/PAC Polymer ve sentetik camurlarından (birinci ve ikinci nesil polimer bazlı camurlar) reolojik ve su kaybı özellikleri bakımından daha iyi performans göstermiştir.

Kısaca, bu çalışma formule edilmiş sepiyolit bazlı çamurların daha önce belirtilen NaCl ve CaCl₂ tuzlarıyla tamamen doygun yüksek tuzluluk ve yüksek sıcaklık (200 °C) koşullarında diğer rakiplerine göre daha iyi reoloji ve su kaybı değerleri vermektedir. En önemlisi, sepiyolit çamurlarının maliyeti aynı koşullarda kullanılan diğer çamurlara göre beş kat kadar daha azdır. Normal şartlarda, sepiyolit bazlı çamurların maliyeti tuzluluk, sıcaklık, ağırlık ve kirleticiler gibi artan uç noktalar ile artmaktadır.

1. INTRODUCTION

Clays and clay minerals are very important industrial minerals. There are well over one hundred documented industrial applications of clay materials. Clays are utilized in the process industries, in agricultural application, in engineering and construction applications, in environmental remediation, in geology and in many other miscellaneous applications.

Clay is an abundant raw material that has an amazing variety of uses and properties that is largely depending on their mineral structure and composition. Although, clays are differently grouped in different literature sources, the clay mineral groups are kaolin, smectite and palygorskite-sepiolite which are sometimes referred to hormites (Martin-Vivaldi and Robertson, 1971), illite, chlorite, and mixed-layered clays. In drilling engineering related references such as Bourgoyne at al. (1991), they are grouped as smectite (montmorillonite), kaolin, illite, and chlorite. The properties of these clays are very different related to their structure and composition (Murray, 2000a). The structure and composition of kaolin, smectite, and palygorskite-sepiolite are very different even though the fundamental building blocks, i.e. the tetrahedral and octahedral sheets are similar. However, the arrangement and composition of the octahedral and tetrahedral sheets account for major difference in the physical and chemical properties that control the applications of a particular clay material. In addition, the type and amount of non-clay minerals (such as dolomite, calcite, mica, feldspar, quartz...) are important.

Selection and maintenance of the best drilling fluid in an oil, gas or geothermal well is one of the main interests of drilling engineers. The drilling fluid is associated either directly or indirectly with most drilling problems. If the drilling fluid did not function satisfactorily, it could become necessary to abandon the well. Therefore, extreme care must be taken into consideration when formulating and selecting drilling fluids.

In general, fresh water based bentonite mud with additives used in geothermal wells can easily be deteriorated due to flocculation phenomenon of bentonite plates when the borehole temperature is high. This phenomenon affects the drilling process unfavorably and increases the drilling cost. In addition, no logging tool along with temperature measurements could be run due to mechanical difficulties in running logging probe into a hole because of gelled mud. Another unwanted 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 dictate a complete renewal of mud system.

Common problems related to the drilling fluid in a geothermal well are circulation losses, abnormally high fluid losses and viscosities, and unacceptable high gel strengths. In addition, the salinity of water greatly reduces the hydration ability of commercial bentonite; thus, a fibrous clay mineral called attapulgite may be used when the water salinity is too high instead of bentonite. However, inadequate 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 bentonite replacement for both the high temperature and the high salinity environment. On the other hand, even though the sepiolite muds yield good rheological properties at elevated temperatures, fluid loss property of these muds is not acceptable to be used in a well.

The clay mineral sepiolite belongs to a group of magnesium silicate with a fibrous texture whose idealized formula can be written as $Si_{12}Mg_8O_{32}.nH_2O$. In nature, two types of sepiolite formation are usually exhibited in Turkey. The first type of sepiolite formation is especially found in Eskişehir and Konya regions called Lületaşı (α -sepiolite). The second type is seen mostly in Eskişehir-Sivrihisar and Mihalıççık-Yunus Emre regions. The secondary sepiolite formation is sedimentary (β -sepiolite) sepiolite which is called "industrial and bedded sepiolite".

Today, sepiolite have a wide range of application in the industry in as much as it is sorptive, catalytic and has fine rheological properties and also has high surface area. In addition, it possesses fibrous structure, porosity, crystal morphology and composition, surface activity, with high viscosity at low concentrations to create stable suspensions. The structure of sepiolite is sensitive to heat treatment. This mineral is also sensitive to operations using acid. Its crystal structure may be destroyed in case it gets in touch with acid. The operation containing heat and acid cause metamorphosis of the surface properties and porosity of the sepiolite. Thus, the sorptive, catalytic and rheological properties of this mineral may be possibly changed (Sabah, E. and M.S. Çelik, 1998).

A large amount of the world production of sedimentary sepiolite is supplied by Spain. This situation is caused by not only Spain has great reserves, but also it has more than 40 products and numerous patents as a consequence of 30 years of research and development proceedings. Also, despite of having a lack of sepiolite reserve, Japan has more than 4000 patents.

The result of various projects and studies made by Mineral Research and Exploration Institute (MTA) indicates that Turkey has the world's largest reserves after Spain and emphasizes that it exists three different qualities in sedimentary sepiolite. On the other hand, there are recent studies indicating that Turkey has the largest sepiolite deposits in the world (above 150 million ton), particularly in the amount of pure sepiolite reserves, (Balci 1999, Öztürk ve Kavak 2004).

First, second and third quality of sepiolite have mineral content respectively,>% 90, %70-89 and %50-69. The sepiolite reserves having over %50 percent of mineral is 1.5 million tons and the reserves (quality of pet litter) having less than %50 percent of mineral is a few million tons. Having high quality and short length of fibers (2-5 μ) and lack of carcinogenic effects make Turkish sepiolite more advantageous among others (Sabah, E. ve M.S. Çelik, 1998).

Sepiolite has a wide range of application areas such as pharmaceutical industry in the ceramic industry, agriculture, animal husbandry and farming sector by sector, catalytic applications of fiber reinforced cement production, rubber industry, Bioreactors, industrial waste water treatment and flue gas waste removal, etc..

In this study among of these clay minerals, the sepiolite is considered as a basic material of drilling mud. Throughout the study, controlling of rheological and filtration characteristics of sepiolite based muds are examined experimentally along with additives, when necessary, at elevated temperature and pressure.

2. LITERATURE REVIEW

There are many studies in the literature related to sepiolite, but a large part of the studies include in the application of sepiolite in other than drilling mud. These subjects contain a large area such as, sepiolite mineralogical properties, physical properties and sepiolite applications in different areas. In this section, first review of the sepiolite clay properties are given, then summary of the some studies are reviewed including application of sepiolite as a drilling fluid additive.

2.1 Properties of sepiolite

Sepiolite, deposited in the sedimentary layers, often is looking fine - grained and slick. In this type of sepiolites, the minerals of sepiolite are over 90% in composition. Generally accompanied minerals are also dolomite and smectite group of clay and magnesite, palygorskite and detritic. Besides non-clay carbonate minerals such as quartz, feldspar and phosphate may also be existed. Moreover, organic matters almost always find in the composition of this type of sepiolite clay. Stratified sepiolite that has a slick-looking, fine-grained, earthy structure, is usually white, cream, gray or pink color. Depending on the organic material content, in some species in the Neojen territory (south of Sivrihisar), may also be dark brown and blackish. β-sepiolite (stratified sepiolite) which is in the form of sedimentary formations with long fiber bundles has fiber length between 100 Å - 3 and 5 µm also its width and thickness can vary between 50-100 Å and 100-300 Å, respectively. Sepiolite has a porous structure. The average diameter of microspores is 15 Å and the radius of mesopores varies between 15 - 45 Å. The density of sepiolite is between 2-2.5 g/cm³, also the density of species that is very porous may occasionally be less than one. When it dries, it can float on water as consequence of a density drop. Sepiolite has a drying temperature of 40°C. Its melting temperature varies between 1400-1500 ° C. Table 2.1 lists some of the important physical and chemical properties of sepiolite. Sepiolite fibrous structure is seen in Figure 2.1.

Table 2.1: Properties of sepiolite (Murray 2007).

| Particle shape | Elongate |
|---------------------------------|--------------------------------------|
| Mohs' hardness | 2.0-2.5 |
| High surface area | 150-320 m²/g |
| Moderate base exchange capacity | 30-50 meq/100g |
| Charge on the lattice | Moderate |
| API yield | 100-115 bbl/ton |
| Melting point sorptivity | 1550 °C |
| Sorptivity | High |
| Water absorption | Up to 100% of the weight of the clay |
| Oil absorption | Up to 80% of the weight of the clay |



Figure 2.1: Fibrous structure of sepiolite (Web 1-2009)

Sepiolite with its own special structure has very high sorption properties, and can absorb water 200-250 times of its own weight. Taking into consideration the defined structural model of sepiolite, discontinuities in the crystal structure of sepiolite for a cross-section of the channels (3.6x10.6 Å) determine surface area which is approximately 800-900 m²/g. In comparison to other clays, the surface area and porosity of sepiolite can be changed by applying the thermal activation, acid activation, or both of them.

The internal arrangment of the layers of sepiolite is unique on which there are channels through the structure. Figure 2.2 shows the structure of the chain of sepiolite. These channels are filled with what is termed zeolitic water. When this water is driven off by heating the surface area and thus the sorptivity is increased,

chemical compounds that are of the size that will fit into these channels are readily absorbed. Absorption and adsorption are properties related to surface area. Absorption is the penetration of fluid molecules into the bulk of an absorbing clay, whereas absorption is the interaction between the fluid molecules and the clay surface.



Figure 2.2: Chain structure of sepiolite (Web 2-2009)

Sepiolite and Palygorskite are the most important two clay minerals in as much as they can create gel property. Low concentrations of these clays can form relatively high viscosity and stabilize suspensions with water or other organic solvents having high-low polarities compared to other clays. Observations made by the electron microscope show that needle-shaped particles of sepiolite have an agglomerate structure and form large cluster of fibers similar to the shrub-grass pile. These fiber piles are easily dispersed with water or solvents having high-low polarity and absorb the liquid, thereby increase the viscosity of the suspension. This type of sepiolite suspension shows Non-Newtonian behavior. This condition depends on the suspension concentration, pH, tensile stress and a lot of parameters such as the composition of the electrolyte. Rheological properties of the suspension containing sepiolite clay, 1 % sodium and potassium salts at high pressure (800-1000psi), and structural changes of sepiolite at high temperatures have been investigated by Güven et al (1988)

In their study it is observed that at 700 °F, 60% and at 800 F, 80% of sepiolite is turn into the smectite. It is also mentioned that a large part of sepiolite clay is transformed

to smectite, Tremolite and talk (water-containing magnesium silicate) as a result of structural change.

2.2 Application area

The application of sepiolite is as varied as kaolin and bentonite applications. The elongated shape of sepiolite minerals results in unique colloidal properties, especially the resistance to high concentrations of electrolytes. The shape and size of sepiolite results in high surface area and high porosity when thermally activated. This elongated needle shape is in contrast to the flake-shaped kaolin and montmorillonite which leads to some unique applications. Sepiolite, which is a natural clay mineral due to the above-mentioned features has a wide range of the application area used from animals' fields to the cleaning agent. Table 2.2 lists the many uses of sepiolite (Galan, 1996 and Murry, 2005). The first six applications consume the largest tonnages and the remaning uses are listed alphabetically. Becaues of its elongated shape, sepiolite is excellent suspension aids in system with a high electrolyte content so that its particles do not flocculate because of the hindered settling of the elongated crystals.

| Drilling fluids | Floor absorbents |
|-----------------------------|-------------------------------|
| Cat litter | Foundry sand binder |
| Agricultural carriers | Granulation binders |
| Tape joint compounds | Laundry washing powders |
| Paint | Liquid suspension fertilizers |
| Industrial floor absorbents | Medicines |
| Adhesives and caulks | Metal drawing lubricants |
| Animal feed binders | Percolation adsorbents |
| Anti – caking agents | Pharmaceuticals |
| Bleaching earths | Polishes |
| Catalyst supports | Reinforcing fillers |
| Ceramics | Wax emulsion stabilizer |
| cosmetics | |

Table 2.2: Applications of sepiolite caly, (Murray, 2007)

2.3 Sepiolite as a drilling mud clay

In direct circulation rotary drilling system, for the purpose of transportation of cutting, created during the drilling, to the surface and cooling of bit, drilling fluid is continuously circulated in drilling wells, this fluid that is called drilling mud and

pumped through the drill pipe from mud tank, return to the surface through the annulus that is the space between well's wall and drill pipe, by carrying the drilling cutting to the surface. On the surface, fluid is clean using a shale shaker or settling in the mud pool, then clean mud is pumped into the well again.

Drilling mud have several different function in drilling well. These are;

- clean the rock fragments from beneath the bit
- carry the cutting to the surface
- exert hydrostatic pressure against formations
- prevent formation fluids from flowing into the well
- keep the drilled borehole open until casing is set
- cool and lubricate the drill string and bit

In order to function properly and serve the needs explained above, it is important to control three main properties of a mud at all times during drilling operation. These three parameters are (1) rheological properties, (2) filtration properties, and (3) pH values. Drilling mud that is used, in order to carry the above-mentioned functions must have certain rheological properties such as: gel strength, thixotropy, viscosity, filtration, etc.. (A. T. Bourgoyne vd., 1991).

When pumping of mud is stopped temporarily and the mud is in static condition, drilling mud should have specific gel strength and thixotropic properties, delaying cutting settling. Besides of this, the special property of mud that is easily pumping into the well is desirable. In addition, this fluid should be as possible less affected from high pressure and variable electrolyte concentrations which occur in different environments in deep drilling.

Today, clay suspensions are usually used as the drilling mud. Due to the minimum sensitivity of sepiolite against the presence of electrolytes in saline environments it is stable in comparison to other clays (bentonite, etc...) and with this feature it is preferred as a clay mineral used for drilling fluid in petroleum industry. Sepiolite can hold its useful properties until pH=8; in cases where pH=9, a sharp decrease in peptization viscosity abserved. In such cases, rheological behavior of drilling mud is Newtonian. The property of water holding characteristic can be improved using of chemical additives such as magnesium oxide, with additives such as maleic anhydride copolymer containing ethylene. Indeed, especially after the discovery of water-binding properties of Loughlinite clay (Na-sepiolite), by using this clay as a additive in sepiolite base muds which have a poor filtration ability, have been taken

positive results in this context (serpen, 2000). Moreover, since that Loughlinite is unsusceptible in the case of salt contamination, thus this composition can be used in preparing salt mud.

Table 2.3 shows the results of the research made in the Petroleum and Natural Gas Engineering department at Istanbul Technical University to obtain sample of sepiolites belonging to Sivrihisar, taken from different regions, as a drilling mud ingredient (National Planning Organization "NPO" report, 1991). In general, when the table is evaluated, the yield of drilling mud prepared with sepiolite is very high and gives better results than commercial bentonite. However, the important disadvantage of sepiolite mud is having very high API water loss, and it is not proper for using in drilling operations with this negative property. By using CMC as a low and medium temperature filtration control agent in drilling mud, it has been seen an improvement on the API water loss, but it is still quite above general criteria determined by API. In spite of this fact that API has not specified a certain acceptable range for amount of water loss of sepiolite based drilling mud but it is observed that amount of water loss of sepiolite mud decreases below 20 ml/30 min in high temperature. Besides of these, sepiolite based mud can show better performance in terms of water loss and rheological properties in salt and hot environments in comparison to the both bentonite and attapulgite (A.T. Bourgoyne vd. 1991).

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.

In their experimental work, Serpen et al. (1992), they compared sepiolite mud with bentonite and attapulgite muds at room condition and showed that sepiolite mud gave better rheological and filtration properties for various salinities. The grain size effect on the rheological properties of sepiolite slurries at room temperature were investigated by Carney et al. (1976) and revealed the considerable differences.
| properties | Units | Brown sepiolite | Wells beige sepiolite | Beige sepiolite | Black pure sepiolite | | |
|---|------------------|--------------------|-----------------------------|--------------------|-------------------------|--|--|
| Yield | (bbl/short ton)* | 112,6 | 156,7 | 157,2 | 132,7 | | |
| Density | (g/cm^3) | 1,04 | 1,03 | 1,03 | 1,04 | | |
| Sand Concentration | (%) | < 0,25 | < 0,25 | < 0,25 | < 0,25 | | |
| Filtration | (ml) | 143 | 84 | 100 | 140 | | |
| Filtration (with %3 CMC) | (ml) | 70 | 80 | - | - | | |
| * 1bbl = 158,97 liter, 1 short ton = 2000 lbm, 1 lbm = 454 gram | | | | | | | |

Tablo 2.3: Properties of sepiolite muds as a additive materials in drilling, (NPA report, 1992).

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 bentonite based muds have trivial effect at room condition and can easily be neglected. In contrast, in an experimental study, Altun et al. (2005) it is observed that the sepiolite based muds behave in an entirely unusual manner under specified conditions mentioned above. Better viscosities and filtration properties were obtained from the sepiolite muds when the mixing speed and mixing time were increased and grain size was decreased.

Massive sepiolite deposits are found around Eskischir, Turkey. In fact, Turkey has the biggest sepiolite reserves in the world, Balci (1999) and Ozturk and Kavak (2004). These clays, having different dolomite content and organic materials, are deposited within the lacustine series in white, brown, and black colors.

Poor filtration property of sepiolite based mud is a well known fact and has already been addressed by some investigators, such as Carney and Meyer (1976), Serpen et al. (1992), and Altun et al. (2005). High water loss problem is resolved to some degree by using polymers, such as Na-polyacrylates (cypan) and synthetic resin (resinex). This study is an attempt to control both rheological and fluid loss behavior of water-based drilling fluid prepared with raw sepiolite clay named TTB obtained around Sivrihisar-Eskisehir district of Turkey.

Carney and Meyer (1976) demonstrated a new approach to high temperature drilling fields, they showed the work done on sepiolite mineral in an effort to obtain a drilling fluid capable of withstanding temperatures in the ultra high range. This included a study on the rheology of sepiolite slurries that have been subjected to

temperatures up to 800°F. There appears to be some changes in the sepiolite after being subjected to temperatures up to 800°F, however, the basic structure seems to be stable.

In studies of slurries containing sepiolite, it was observed that the rheological properties of the slurries have shown a slight increase in viscosity when measured at elevated temperatures and pressures, Due to this thermal stability, formulations containing sepiolite were prepared and tested at these extreme conditions. Parameters that are important for good drilling fluid performance were measured and recorded.

In this study sepiolite slurries were subjected to temperatures of 750°F using a specially designed ultra high temperature, high pressure thickening time tester. The viscometer data shows an increasing in viscosity after heat aging, but not unreasonable good mud rheology. The yield point has little change.

It was observed that sepiolite samples from the Amargosa Desert exhibited varying yield according to impurities and/or grind size. Grind size is an important factor in the yield of this material at ambient temperature.

Also in this study, in an effort to control fluid loss, slurries were prepared with sepiolite in conjunction with thinners and various polymers. This approach looks more promising than the previous works, but the fluid loss was still considered to be excessive. After thorough investigation, reasonable fluid loss control properties were obtained with small additions of Wyoming bentonite in conjunction with various polymers, but by considering to data obtained from this composition it is observed that the rheological properties such as yield point and gel strength have not reasonable values.

Hischer and Clements (1982) also developed a new high temperature drilling fluid. The system which uses a thermally stable deflocculant and fluid loss additive was tested in the laboratory at temperatures up to 450°F (232°C). It provides stable rheological properties and good filtration control even in the present of severe cement contamination. Lime may be used with the system to inhibit shale swelling and dispersion.

This study investigated the prospects of preparing a good high temperature, low lime mud drilling for green cement. It was found low lime mud can be stabilized at high temperatures by using a thermally stable polymeric deflocculant (TSPD). Filtration control is enhanced by adding of the lignite/polymer reaction complex (LPC). The utility and general behavior of this system at 400°F and 450°F has been

characterized. However, when we consider this study, we can find that it is used the significant amount of LPC polymer (about 15 lb/bbl). The results of this study are shown in Table 2.4.

| SAMPLE NO: | 1 | 2 | 3 | 4 | 5 | 6 |
|---------------------------------|-----|-----|-----|-----|-----|------|
| Base Mud*, bbl | 1 | 1 | 1 | 1 | 1 | 1 |
| LPC, 1b | 10 | 15 | 10 | 15 | - | 15 |
| Lignite, 1b | - | - | - | - | 15 | - |
| TSPD, 1b active | 1.5 | 1.5 | 3 | 3 | - | - |
| DMS, 1b | - | - | - | - | 4 | - |
| Cement, 1b | 3 | 3 | 3 | 3 | 3 | 3 |
| Shear Strength 1b/100 sq ft. | 180 | 130 | 210 | 120 | 610 | 1380 |
| Plastic Viscosity, cp | 22 | 26 | 26 | 20 | 42 | 34 |
| Yield Point, 1b/100 sq ft. | 14 | 22 | 22 | 22 | 84 | 54 |
| 10 sec gel, " | 3 | 7 | 6 | 7 | 35 | >300 |
| 10 min gel, " | 14 | 20 | 16 | 24 | 96 | >300 |
| HPHT filtrate (400°F), cc | 44 | 28 | 36 | 28 | 68 | - |

Table 2.4: Drilling fluid formulation with cement contamination and their propertiesafter 450°F static aging, (Hischer and Clements, 1982).

* 10 lb/bbl bentonite and 20 lb/bbl sepiolite pH adjusted to 11.0 - 11.5

Drilling fluids prepared with clay of sepiolite with no additives protect their structure at temperatures up to 300°F and performs better than bentonite and attapulgite when exposed to the same conditions (Carney and Guven, 1980). Sepiolite clay used in conjunction with saponite at higher temperatures have better rheological properties, sepiolite begins to convert to a smectite at 300°F (149°C) and this reaction is fully completed at 500°F (260°C). The new smectites in the fluid have a thin flakey morphology and they increase the viscosity and improve the filtration losses that are shown by Guven et al (1988). In conclusion, if rheological and water loss properties

of sepiolite base muds are controlled by using various additives, it can become a proper drilling fluid in high salinity and high temperature environments.

In addition, using of sepiolite in drilling industry can create important economic opportunities for Turkey as a country with the world's largest deposits of sepiolite (Balcı, 1999, Öztürk ve Kavak, 2004). Geological formation and structure sepiolites used in this study were investigated. On the other hand, API standards about sepiolite are not sufficiently defined unlike bentonite clay and attapulgite. Another aim of this study is to contribute to the development of standards and new definitions by identifying common features of sepiolite clay.

As a result of literature review, it is obvious that there are no sufficient works on sepiolite based drilling fluids. In other words, all the attempts are failed in terms of controlling rheological and filtration characteristics of sepiolite muds. Sepiolite is used in most of the work as additive to improve properties of main clay such as bentonite, saponite, attapulgite. Thus, it is obvious that the considering of sepiolite as a drilling mud main clay can be important investigation helped drilling fluid industry.

3. STATEMENT OF PURPOSE AND PROBLEM

Deep oil and gas drilling, thermal EOR wells, and geothermal well drilling are known as high temperature environment; therefore, it is difficult to formulate a drilling mud functioning adequately, particularly in temperatures above 150°C. Circulation breaks, abnormally high fluid losses and viscosities, and unacceptable high gel strengths are the main problems that are usually associated with these wells. Although bentonite based mud with extremely expensive additives is commonly used in these drilling conditions, it does not meet the desired needs in higher temperatures above 150 °C. There are geothermal fields having temperatures more than 240°C in both Turkey and the world. Therefore, sepiolite, a magnesium silicate clay mineral with fibrous texture, has been proposed as the bentonite replacement for both the high temperature and the high salinity environment.

There appears to be some changes in the sepiolite after being subjected to temperatures up to 800°F. However, the basic structure seems to be stable. (Leroy L. Carney and Robert L. Meyer, 1979). Additionally, the basic structure of sepiolite is known to be firm in saturated saline-water phase. On the other hand, sepiolite muds are known with high filtration loss values that are not acceptable and suitable in using as drilling fluid. Therefore, it is vital to control filtration characteristics in order to serve an alternative drilling fluid particularly in harsh environments instead of high cost muds such as, synthetic based drilling fluids (second or third generation polymeric muds) in some extend potassium chloride (KCl) mud, etc. It is also well known fact that harsh environment drilling fluids are highly expensive, for instance barrel cost of polymeric muds are over 100 US Dollar and increases with increased mud weight, salinity, and temperature.

In this study, the first aim is to develop sepiolite based drilling muds with acceptable rheological and filtration characteristics. Second, the field usage of such a mud will be applicable from normal to harsh drilling environment conditions in terms of wide range of salinity, temperature, pressure etc. Third, cost of sepiolite based mud must be competitive with other types of muds, particularly in harsh drilling environments.

Turkey has the largest sepiolite clay deposits in the world; on the other hand, the usage of sepiolite clay as a drilling fluid not only in Turkey and but also in the world is negligible. If such sepiolite mud with aims defined above is developed, it will also serve for Turkey as a new economical opportunity as well.

4. METHODS AND MATERIALS

API RP-13B Standard procedures are employed throughout the laboratory work to determine rheological and fluid loss properties. Also according to API 13A, drilling grade sepiolite shall be deemed to meet the requirements of the international standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 4.1.

| Test parameter | Specification |
|--|------------------------------|
| Suspension properties | |
| Viscometer dial reading at 600 r/min | Minimum 30 |
| Residue of diameter greater than 75 µm | Maximum mass fraction 8,0 % |
| Moisture, % | Maximum mass fraction 16,0 % |

Table 4.1: Sepiolite physical specifications (API-13A, 1990)

All the samples muds prepared in this work are based on the formulation of 350 ml of fresh water that contains 20 g of sepiolite clay along with different concentrations of some additives in order to provide best performance such as polymer A, polymer B, soda ash, caustic and glycol. In addition, properties of sepiolite muds examined in this work are also investigated for two 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. Moreover, in order to simulate entrance of the salt into the mud from formation fluid having high salinity or from salt zones, after preparing typical mud a certain amount of sodium chloride is added to mud. In addition, a drilling grade bentonite clay with API/ISO specifications known OCMA as a contaminant is added to mud in order to represent reactive clay/shale zones becoming unstable in contact with drilling fluid water. Rheological properties such as apparent viscosity, plastic viscosity, yield point and gel strength of the sepiolite slurries are measured on a Fann Model 35 viscometer before and after high temperature aging at ambient condition. Static filtration properties of the samples are measured by standard API filter press and HTHP filter press equipment. Experimental apparatuses used in this study are explained below extensively.

4.1 HPHT (High Pressure, High Temperature) filter press

Measurement of the filtration behavior and all-cake-building characteristics of an oil mud are fundamental to the treatment and control of a mud, as are the characteristics of the filtrate, such as the oil, water or emulsion content. Filtration characteristics of an oil mud are affected by the quantity, type, and size of solid particles and emulsified water in the mud and by properties of the liquid phase. Interactions of these various components may be influenced by temperature and pressure. Therefore, filtration tests are often performed from ambient to high-temperature conditions to provide data for comparison purposes. 175 ml volume of HPHT Filter Press units can be pressurized to 1800 psig on the cell and 750 psig on the backpressure receiver. Maximum operating temperature is 350°F. Figure 4.1 show a sample of HTHP filter press 175 ml and its specifications are given in Table 4.2.



Figure 4.1: 175 ml HTHP filter press (Web 3-2009).

| Maximum Working Pressure | 1800 PSIG |
|--------------------------|---|
| Maximum Temperature | 350 °F |
| Power Requirement | 115/230 VAC 50/60 Hz |
| Sample Cell Volume | 175 ml |
| Receiver Volume | 15ml |
| Heating Capacity | 400 watts |
| Filtering Area | 22.6 cm ² (3.5 in ²) |

Table 4.2: Specification of HTHP filter press 175 ml

4.2 Model 35 viscometer

Fann Instrument Company produces a range of true Couette coaxial cylinder rotational viscometers. The test fluid is contained in the annular space or shear gap between the cylinders. Rotation of the outer cylinder at known velocities is accomplished through precision gearing. The viscous drag exerted by the fluid creates a torque on the inner cylinder or bob. This torque is transmitted to a precision spring where its deflection is measured and then related to the test conditions and instrument constants. This system permits the true simulation of most significant flow process conditions encountered in industrial processing.

FANN Direct Indicating Viscometers combine accuracy with simplicity of design, and are recommended for evaluating materials that are Bingham plastics. These instruments are equipped with factory installed R1 Rotor Sleeve, B1 Bob, F1 Torsion Spring, and a stainless steel sample cup for testing according to American Petroleum Institute Specification RP-13B. Other rotor-bob combinations and/or torsion springs can be substituted to extend the torque measuring range or to increase the sensitivity of the torque measurement. Shear stress is read directly from a calibrated scale. Plastic viscosity and yield point of a fluid can be determined easily by making two simple subtractions from the observed data when the instrument is used with the R1-B1 combination and the standard F1 torsion spring. Model 35 of viscometer is shown in Figure 4.2.



Figure 4.2: Model 35 viscometer (Web3-2009).

4.3 Aging cells

Drilling fluid aging is the process in which a drilling fluid sample, previously subjected to a period of shear, is allowed to more fully develop its rheological and filtration properties. The time period needed to more fully develop properties varies from as little as several hours (usually 16 hours) to as much as several days. The aging can be done at either ambient or elevated temperatures.

Most drilling fluid formulations contain a base liquid and additives which must be dissolved or mechanically dispersed into the liquid to form a homogenous fluid. The resulting fluid may contain one or more of the following: water-dispersible (soluble) polymers or resins, clays or other insoluble but dispersible fine solids, and soluble salts. The fluids are mixed or sheared for times appropriate to achieve a homogenous mixture and are then set aside to "age." Aging is done under conditions which vary from static to dynamic and from ambient to highly elevated temperatures. Figure 4.3 show samples of aging cells.



Figure 4.3: Aging cells, glass liner and accessories (Web 3-2009).

4.4 Roller oven

Drilling fluid aging is the process in which a drilling fluid sample, previously subjected to a period of shear, is allowed to more fully develop its rheological and filtration properties. The time period needed to more fully develop properties varies from as little as several hours (usually 16 hours) to as much as several days. Aging is done under conditions which vary from static to dynamic and from ambient to highly elevated temperatures.

Fann Roller Ovens (Figure 4.4) provide an excellent method of aging fluid samples for further analysis. High-Temperature Aging Cells containing sample fluids are placed in the roller oven where they are subjected to moderate heat and agitation (rolling) on power driven rollers. Samples may also be heated without rolling (static aging). These Roller Ovens are constructed of polished stainless steel and other corrosion resistant materials. They are well insulated and the temperature is regulated by a digital electronic controller. An internal circulation fan assures an even temperature distribution throughout the oven.



Figure 4.4: Fann roller oven (Web 3-2009).

4.5 Multi-Mixer® model 9B

Most drilling fluid formulations contain a base liquid and additives which must be dissolved or mechanically dispersed into the liquid to form a homogenous fluid. The resulting fluid may contain one or more of the following: water-dispersible (soluble) polymers or resins, clays or other insoluble but dispersible fine solids, and soluble salts. The fluids are mixed or sheared for times appropriate to achieve a homogenous mixture and are then set aside to "age." Drilling fluid aging is the process in which a drilling fluid sample, previously subjected to a period of shear, is allowed to more fully develop its rheological and filtration properties. Aging is done under conditions which vary from static to dynamic and from ambient to highly elevated temperatures. The Five-Spindle Multi-Mixer® Model 9B mixer (Figure 4.5) is recommended for use in general purpose mixing of drilling fluids in preparation for laboratory tests of mud materials. Five Spindle Multi-Mixer mixers are supplied with No. 9B29X impeller blades. Each spindle is fitted with a single sine-wave impeller approximately 25 mm in diameter mounted flash side up. It Conforms to American Petroleum Institute (API) Specification 13A for mixing water-based and oil based drilling fluids.



Figure 4.5: Five-spindle multi-mixer (Web 3-2009).

4.6 Dynamic HTHP filtration system

The Fann DYNAMIC HPHT filtration system (Figure 4.6) is the industry's only true dynamic filtration system for conducting filter cake formation and permeability analysis for drilling fluids optimization. Utilizing a wide range of available filter mediums, the DYNAMIC HPHT filtration system can be heated and pressurized to provide the closest possible simulation of down-hole conditions.

Several safety features have been designed into the system to protect the user and help ensure reliable test results. The filter medium is a thick-walled cylinder with rock-like characteristics to simulate the build-up of filter cake on the formation. The filter medium is available in varying porosities and permeabilities to simulate down hole formations. Filtration occurs radially from the inside of the filter core to the outside. At the same time, the filter cake is formed on the inside of the filter core to simulate filter cake formation on the wall of a borehole.



Figure 4.6: Dynamic HPHT filtration system - model 90 (Web 3-2009).

Following completion of a test, the filter cake can be inspected visually. A polished stainless steel shear bob runs through the central axis of the filter core. The shear bob is rotated to produce a concentric cylinder-type shear across the filtration surface.

The LCD display allows monitoring of real-time test results, which are printed for further analysis and filing of test results. The DYNAMIC HPHT filtration system also features an interface port, which allows downloading of ASCII-formatted data to a personal computer. The system is fully automatic with a built-in computer controller. Through menu-driven software, the user can establish up to 20 sequence steps to program the following testing parameters: temperature, pressure, differential pressure, shear rate.

4.7 pH meter

pH meter is an electronic instrument used to measure the pH (acidity or alkalinity) of a liquid (though special probes are sometimes used to measure the pH of semi-solid substances). A typical pH meter consists of a special measuring probe (a glass electrode) connected to an electronic meter that measures and displays the pH reading.

The Waterproof pH Tester2 (Figure 4.7) has unique double junction electrode design and increased reference gel volume that give significantly longer electrode life, especially in harsh applications. Automatic Temperature Compensation and three point push-button calibrations provide high accuracy even under varying temperature conditions. Accuracy is ± 0.1 PH.



Figure 4.7: Waterproof PH tester 2 (Web 4-2009).

5. SEPIOLITE CLAYS USED IN THIS STUDY

Sepiolite clay samples used in this study were obtained from Sivrihisar -Eskişehir region. The experiments applied on five different Sepiolite clays, and the results were evaluated in the following section. Three of sepiolite clays were obtained from AEM Company that sells and trades sepiolite clay in Sivrihisar. The other two sepiolite samples were obtained directly from the two different sepiolite mining areas that used to be a commercially sepiolite production field. The names and places from which sepiolite samples obtained, listed in the Table 5.1. Names given to sepiolite clays obtained from AEM Company have been preserved throughout in this study and are listed in tables in this way.

| CLAY No | NAME OF SEPIOLITE | COMPANY | COLOR |
|---------|-------------------|--------------|-------------|
| 1 | Kurtşeyh | AEM | Brown |
| 2 | TTB | AEM | Beige |
| 3 | S | AEM | Black |
| 4 | YD-K | Mine of Clay | Light Brown |
| 5 | YD-S | Mine of Clay | Dark Brown |

| Table 5.1 : Set | epiolite | clays | used | in | this | study. |
|------------------------|----------|-------|------|----|------|--------|
|------------------------|----------|-------|------|----|------|--------|

The pieces of sepiolite clays were reduced to a smaller size using large crushers and then using of rotary grinding, it has been reduced to a size below 74 microns (200 mesh) in ITU, Faculty of Mines Mineral Engineering Department laboratories. During the grinding process, homogeneous mixing of clay samples is performed according to the rules of first 1/2, then 1/4, then 1/8. During this process, mineral processing methods were followed. Especially grinding of YD-S clay has resulted a major problem in screening process due to the adherence of this clay to screen. Thus the grinding process of this clay was very time consuming than that of others.

Dimensional analysis of each clay samples were also performed in Mineral Processing Engineering Department by using the laser dimension analysis equipment. In below, the results obtained from clay samples are provided as the charts in Figures from D.1 to D.6 in Appendix-D. Grain size distribution of TTB Sepiolite is given as an example in Figure 5.1. Grain size analysis also made for commercial bentonite clay, and the results are given in order to compare with sepiolite clays. If figures are evaluated, it has been observed that the grain sizes obtained for all samples are to be quite small than 74 microns. In Table 5.4 grain size values of 10 %, 50 % and 90 % are listed for the all clay samples. In each cumulative value, the grain size of bentonite is smallest as a remarkable situation. However, frequency distribution shapes of each sample (like letter S) are similar to each other. The results of all five-sepiolite samples XRF analysis are given in Table 5.2. X- Ray fluorescence (XRF) analysis provides a means of finding the chemical composition of samples (or parts of samples) without removing these. In addition, mineral compositions of the sepiolite samples are tabulated in Table 5.3. As can be seen, except Kurtşeyh clay containing 3% quartz, all of the other clays belong to sepiolite clays groups and they can be accepted as pure easily. Determination of mineral compositions and XRF analysis of these clays are conducted in TPAO research center.



Figure 5.1: Grain size distribution of TTB sepiolite.

| | Mineral composition (%3 by volume) | | | | | | | | |
|--------|------------------------------------|-----------------|---------|----------|-----------|-----------|--------|-----------|---------------------|
| Sample | | | | | | Foldenato | | Clay+Mica | |
| Ankeri | Ankerite | Hydro magnesium | Brucite | Dolomite | Magnesium | Groups | Quartz | Others | Sepiolite Groups |
| TTB | _ | _ | _ | _ | _ | Trace | Trace | _ | 100 |
| 11D | _ | - | _ | _ | _ | amount | amount | - | 100 |
| KURT | _ | _ | _ | Trace | _ | _ | 3 | _ | 97 |
| ŞEYH | _ | - | - | amount | _ | - | 5 | - | |
| S | | - | | Trace | | | Trace | | 100 |
| 5 | - | | - | amount | - | - | amount | - | 100 |
| | | | | Trace | | | Trace | | 100 |
| 10-5 - | - | - | - | amount | - | - | amount | - | 100 |
| YD-K - | | | | Trace | | | Trace | | 100 |
| | - | | | amount | - | - | amount | - | 100 |

Table 5.2: Mineral composition of sepiolite samples

Table 5.3: XRF analysis of sepiolite samples.

| Sample | CaO % by weight | MgO % by weight | SiO2 % by weight | Fe2O3 % by weight | K2O % by weight | Na2O % by weight | Al2O3 % by weight |
|----------|--------------------|--------------------|---------------------|----------------------|--------------------|---------------------|----------------------|
| TTB | 4.37 | 23.22 | 52.31 | 1.23 | 0.35 | < 0.01 | 2.69 |
| KURTŞEYH | 0.75 | 27.68 | 62.78 | 0.48 | 0.13 | < 0.01 | 1.01 |
| S | 0.28 | 23.79 | 60.12 | 1.31 | 0.4 | < 0.01 | 2.73 |
| YD-S | 0.34 | 26.38 | 62.29 | 0.81 | 0.21 | < 0.01 | 1.67 |
| YD-K | 0.18 | 26.67 | 62.17 | 0.68 | 0.18 | < 0.01 | 1.44 |

| Drobability | Grain size, micron | | | | | | | | |
|-------------|--------------------|----------|-----|-----|------|------|--|--|--|
| Probability | Bentonite | Kurtşeyh | TTB | S | YD-K | YD-S | | | |
| 10 | 1.5 | 3 | 5 | 2.5 | 4 | 4 | | | |
| 50 | 5 | 10 | 18 | 10 | 11 | 11 | | | |
| 90 | 17 | 21 | 22 | 30 | 21 | 28 | | | |

Table 5.4: Results of grain size analysis of clays.

% 97.5 by weight of clays' particle size that is used in drilling mud should be smaller than 74 microns that is the feature of clay requested by the standards of API. On the other hand, it is a fact that the smaller desired nominal value of particle sizes the more the cost of grinding. In compared with commercial bentonite (if only take into consideration the particular size) the grinding cost of sepiolite clay can be expected similar to the bentonite. However, being the particle size in very different range (for example, the difference in value of 10% and 90%) is not providing means of a homogeneous size of particles. This case can cause the difference in surface area corresponding to the unit weight of clay, so this can result in variable water sorption and rheological properties of clay. Changing of time depending high pressure high temperature (HTHP) rheological properties of sample clays prepared homogeneous in particle size as a subject of another study should be evaluated.

In this study, the experiments with sepiolite clay comprise three different groups. In the first group water loss and rheological properties of full saturated (400 g sodium chloride in 1 liter distilled water) sepiolite drilling muds were investigated. With the second semi saturated (200 g sodium chloride in 1 liter distilled water) sepiolite mud and the third group includes the experiments made with fresh water. In the first part of the study, the water loss and the rheological properties of five different sepiolite clay are examined in the room condition in order to decide about the continue of experiments with which one of used clays is effective. In the final stage of the study, water loss and rheological properties of sepiolite mud in HTHP conditions will be controlled using chemical additives.

In order to provide continuity in the study, API standards are followed. In cases does not provide standards or standards are not specified, practices of the experts in TPAO Research Center, Drilling Mud and Cement Laboratory were followed. Results of rheological and API water loss properties of muds are tabulated and presented in the appendixes. Templates of these tables are shown in Table 5.5 and 5.6. Water loss and rheological properties of mud samples are examined for not only 20 minutes that is

the time recommendation to prepare mud by API standard, but also changes of these properties are examined for mud samples that are aged for 16 hours, 24 and 48 hours. It was determined that, unlike bentonite clay, aging period in drilling mud samples prepared with sepiolite clay has remarkable effect specially on the rheological properties.

 Table 5.5: Examples of rheology measurement

Sample name :

Temperature of room: •c

Composition : 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|--------|----------|----------|----------|
| Sample mud temperature: oc | | | | |
| Density , ppg | | | | |
| θ 600 | | | | |
| θ 300 | | | | |
| θ 200 | | | | |
| θ 100 | | | | |
| θ6 | | | | |
| θ3 | | | | |
| Apparent viscosity (AV) , cp | | | | |
| Plastic viscosity (PV) , cp | | | | |
| Yeild point (YP), lb/100 ft ² | | | | |
| 10 sec gel strength, lb/100 ft ² | | | | |
| 1 min gel strength, lb/100 ft ² | | | | |
| 10 min gel strength, lb/100 ft ² | | | | |
| 30 min gel strength, lb/100 ft ² | | | | |
| Cake thickness mm | | | | |
| Cake status | | | | |
| pH (mud) | | | | |
| pH (filtration) | | | | |

Table 5.5: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ N |
|-------------------|-------------|-------------|-------------|-------------|
| | | | | 1021,8 |
| | | | | 510,9 |
| | | | | 340,6 |
| | | | | 170,3 |
| | | | | 10,218 |
| | | | | 5,109 |

 Table 5.6:
 Sample water loss table

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(ext{time}), 	ext{min}}$ |
|------------|--------|----------|----------|----------|----------------------------------|
| 1 | | | | | 1,000 |
| 2 | | | | | 1,414 |
| 2,5 | | | | | 1,581 |
| 3 | | | | | 1,732 |
| 4 | | | | | 2,000 |
| 5 | | | | | 2,236 |
| 7,5 | | | | | 2,739 |
| 10 | | | | | 3,162 |
| 20 | | | | | 4,472 |
| 30 | | | | | 5,477 |

6. RHEOLOGICAL PROPERTIES

Rheology is the study of the deformation and flow of matter. Viscosity is a measure of the resistance offered by that matter to a deforming force. Shear dominates most of the viscosity-related aspects of drilling operations. Because of that, viscosity of drilling fluids is the property that is most commonly monitored and controlled. Retention of drilling fluid on cuttings is thought to be primarily a function of the viscosity of the mud and its wetting characteristics. Drilling fluids with elevated viscosity at high shear rates tend to exhibit greater retention of mud on cuttings and reduce the efficiency of high-shear devices like shale shakers. Conversely, elevated viscosity at low shear rates reduces the efficiency of low-shear devices like centrifuges, in as much as particle settling velocity and separation efficiency are inversely proportional to viscosity. Water or thinners will reduce both of these effects. Also, during procedures that generate large quantities of drilled solids, it is important to increase circulation rate and/or reduce drilling rate. Other rheological properties can also affect how much drilling fluid is retained on cuttings and the interaction of cuttings with each other. Yield point is a good indicator of contamination by solids that affect the electrochemical environment. By contrast, plastic viscosity is a function of the base fluid viscosity and concentration of solids. Thus, plastic viscosity is a good indicator of contamination by drilled solids. Good solids control begins with good hole cleaning. One of the primary functions of the drilling fluid is to bring drilled cuttings to the surface in a state that enables the drilling-fluid processing equipment to remove them with ease. To achieve this end, quick and efficient removal of cuttings are essential.

In this section, the results of rheological properties of fully and semi salt saturated sepiolite and fresh water sepiolite muds are given separately for each clay types in ambient condition and then all of the data are given comparatively in order to select the best performing clay sample in terms of rheological properties. Experimentally obtained results are shown graphically in Appendix E. The data used in graphics

were obtained from the values of Appendix A, Appendix B and Appendix C that are given in tables.

6.1 Fully saturated sepiolite base muds

Rheological properties of full saturated (400 g sodium chloride in one litter distilled water) sepiolite muds are given at the ambient conditions. The graphics of each sepiolite clay mud are shown in Figures E.1 to E.5 presented in Appendix E respectively. These graphics indicate the variations of shear rates vs. shear stress at six different shearing speeds. As an example, the rheological properties in TTB clay is shown graphically in Figure 6.1. It has been decided to run the experiments only at 20 min, 16 hours and 24 hours of aging period, due to slight differences between pilot test results obtained from the experiments of 24 hours and 48 hours of aging. Experimental conditions and determined rheological properties are given as tables in Appendix-A. It is known that the factor between shear rate and rotary speed of viscometer is 1.703. Viscometer rotation speeds are 3, 6, 100, 200, 300 and 600 rpm. Fann model 35 viscometer is used to rheological measurements of five-types of sepiolite used in this study.



Figure 6.1: Rheological properties of fully saturated mud prepared with TTB clay.

6.2 Discussion on fully saturated mud results

Considering the rheological properties of fully saturated sepiolite mud, it has been seen that except one of sepiolite clays named S clay, in all of other clays, dial readings of 600 rpm is 30 or more. In other words, rheological properties of the S clay remain below the limit values specified by API standards. On the other hand, YD-S clay gives a dial reading of 43 at 600 rpm that is above the limit value of 30. In terms of yield point and gel strength except of S clay, all of other four clay provide acceptable results even in fully saturated system and it is noticeable in the light of cutting transportations and hole cleaning in the wells. In short, four of the five clays used in this study provides the minimum rheological condition. Evaluating the graphics of relationships between shear stress and shear rate in all of the clays, indicate the rheological model of these clays mud so that lack of linear relationship between shear rate and shear stress is the indication of non-Newtonian rheological behavior. An interesting observation is that, depending on increasing of aging time, the fluids shear stress tends to increase and it is observed that the changes of increasing is important especially in the first 24 hours. This situation is a special case of sepiolite clay, and should be taken into consideration. API standards for bentonite mud specified that the desired rheological properties are obtained after aging 16 hours, however this standard is limited to 20 minutes for sepiolite mud. On the other hand, in case of water absorption properties of clays, it is clear that sepiolite clay is water absorptive in more time due to having fibrous structure. However, the reason of being short aging period in terms of sepiolite clays has not been described in the API standards. This confirms that API standards are not appropriate for sepiolite mud formulation. Therefore, in this study, in order to compare with bentonite mud, the process of aging is done for 16 hours. Tests have been done considering to changes of rheological properties and decided to measure the values of 24-hour. Unlike bentonite clays, as a major consequence according to the graphics, the values of shear stress in sepiolite clay show great changes for different aging periods.

6.3 Semi salt saturated sepiolite base muds

Rheological properties of semi saturated (200 g sodium chloride in one litter distilled water) sepiolite mud have been investigated in the room conditions. The graphics of each sepiolite clay mud are given in Figures E.6 to E.10 presented in Appendix E.

Similar to fully saturated experiments, in semi saturated system, determination of rheological properties of five mentioned clays mud are also done at 20 min, 16 hours and 24 hours aging times apart from 48 hours tests. As mentioned before there are slightly variations in results of 48 hours tests respected to 24 hours. As an example, the rheological properties of TTB clay is shown graphically in Figure 6.2 in order to discuss about results. Experimental conditions and obtained other rheological properties shown in the table presented in Appendix-B. Behaviors and results of semi saturated sepiolite muds is similar to the results obtained from fully saturated mud. In these cases, it has been observed that the shear stresses are slightly higher than fully saturated mud samples.



Figure 6.2: Rheological properties of semi saturated mud prepared with TTB clay.

6.4 Discussion on semi saturated mud results

The procedures prepared for fully saturated sepiolite muds were also followed for semi-saturated sepiolite muds. It has been observed that the rheological properties of semi-saturated sepiolite muds are similar to fully saturated sepiolite muds results. S clay has not satisfied acceptable rheological values. On the other hand, in the other clays, dial readings of 600 rpm are 30 or more, that is, these clays can also provide the minimum rheological condition for the semi-saturated systems. For example, YD-S clay gives the reading of 600 rpm above the limit value. Non-Newtonian fluid

behavior relationship is seen between shear rate and shear stress similar in fully saturated system. Aging period has a significant effect on the increasing fluids shear stress. Yield point and gel strength properties of all clays muds are in appropriate values except S clay. There is slightly increment in rheological values of semi saturated Sepiolite muds compared to the fully saturated sepiolite clay, but this increment is not significant.

6.5 Fresh water sepiolite base muds

Rheological properties of fresh water (diluted water without salt) sepiolite mud are investigated in the room conditions. The graphics of each sepiolite clay mud are given in Figures E.11 to E.15 presented in Appendix E. Similar to fully and semi saturated experiments, in fresh water system, determination of rheological properties of five mentioned clays mud are also done at 20 min, 16 hours and 24 hours aging times apart from 48 hours tests. As an example, the rheological properties of TTB clay are shown graphically in Figure 6.3 in order to discuss about results. Experimental conditions and obtained other rheological properties shown in the table presented in Appendix-C. The highest shear stress values are observed in the muds prepared with fresh water compare to semi and fully saturated systems.



Figure 6.3: Rheological properties of fresh water mud prepared with TTB clay.

6.6 Discussion on fresh water mud results

The procedures prepared for full and semi salt saturated sepiolite muds were followed for fresh water sepiolite muds. It has been observed that the rheological behaviors of sepiolite muds prepared with fresh water are similar to semi and fully saturated sepiolite muds. Even in fresh water system, S clay could not provide acceptable value for dial reading of 600 rpm that other four clays could satisfy the limitations of API standards. Therefore, it will be logical that S clay are not used in the next experiments. But as it seen, dial reading in different speed of viscometer for S clay mud prepared with fresh water is a little higher than S clay mud prepared with full and semi salt saturated water. This situation is also valid for other clays. On the other hand, especially YD-S clay gives the reading of 600 rpm above the limit value. In short, four of the five clays used in this study, meets the minimum rheological condition. Non-Newtonian fluid behavior is also seen in this system due to existence of non-linear relationship in the graphs of shear stress versus shear rate. Depending on increase in aging time, the fluids shear stress tends to increase. These muds have high yield point and gel strength with respect to salt saturated system. Generally, it is observed that fresh water sepiolite muds have higher rheological values than full and semi salt saturated sepiolite muds. This confirms that shear stress values of sepiolite clay is inversely proportional with increasing salinity. However, not only the rheological properties of clay are distinctive features in the selection of proper clay, but also water loss properties are important factors.

6.7 The effect of salinity and aging on shear stress

The results of rheological properties of fully and semi salt saturated sepiolite and fresh water Sepiolite muds are given comparatively at the room condition between Figures E.16 to E.35 shown graphically in the Appendix E. The data used in graphics were obtained from the values of Appendix A, Appendix B and Appendix C that are given in tables. In addition, effects of aging period and salt content on rheological behavior of sepiolite clays at room conditions are tabulated and given in Appendix E in Tables between 6.1 to 6.5. The rheological properties of TTB clay are reported in fully and semi saturated and fresh water systems comparatively for 20 min aging, as an example in order to discuss about results, (Figure 6.4). In this section, changes in rheological properties are shown for the results of 20 minutes, 16 hours, 24 hours and 48 hours aging of muds.

As mentioned before in all of three systems, lack of linear relationship between shear rate and shear stress is the indication of non-Newtonian rheological behavior. It is also noticeable that shear stress increases with increasing aging period. It can be also said that 24 hours of aging is required for four clays that can provide API standards to hydrate adequately.

Another important observation is the effect of salt concentration on rheological behavior. Simply, higher the salt content in the mud, lower the shear stress. Salt content and aging period have incredible effects on rheological behavior of sepiolite muds. These effects are listed in Tables 6.1 to 6.5 for all of the clays. As shown in the table, increased salt content reduces the shear stress or apparent viscosity; in other words, higher the salt content, lower the yield of sepiolite clay. Shear stress recorded at 600 rpm of viscometer speed and 48 hours of aging decreases from 42 for fresh water mud to 32 for saturated mud. Total change referenced to fresh water mud has a value of 24 % and it is very important. The change in shear stress is more dominant at lower aging periods. Another important parameter effecting shear stress is aging period of sepiolite muds. As indicated in Table 6.1, shear stress increases with increasing aging period; for instance, there is a 11 % variation for fresh water mud case when the period of aging varies from 20 min to 48 hours. The aging effect gets higher when the salt content increases. This condition is seen in other four clays. As a result, unlike bentonite based muds, the rheological properties of sepiolite based muds are more dependent on aging period and salt concentration.

Additionally, because of having close results in semi-salt saturated and salt saturated mud, the change of shear stress values is stabilized after certain salinity and changing in shear stress values (decreasing) are unimportant with increasing of salinity. As explained in sections of 3, 4 and 5 in this chapter, YD-S clay gives the best results in terms of rheological and it is followed respectively with Kurtşeyh, TTB, and YD-K and S clays mud. It is interesting that the mud prepared with S clay does not show requested shear stress in spite of 48 hours of aging. This case may be is due to other materials exited in S clay. This clay will not be used in the next phases of the study.

| | _ | Shear str | Aging effect | | | |
|--------|-----------------|-----------|--------------|--------|--------|-----|
| _ | TTB clay | 20 min | 16 hrs | 24 hrs | 48 hrs | (%) |
| d Type | Fresh water | 38 | 38 | 42 | 42 | 11 |
| | Semi -saturated | 26 | 33 | 35 | 36,5 | 40 |
| Mu | Saturated | 21,5 | 26 | 29 | 32 | 49 |
| | Salt effect (%) | 43 | 32 | 31 | 24 | |

Table 6.1. Effects of aging and salt content on rheological behavior of TTB sepiolite at room conditions.

Table 6.2. Effects of aging and salt content on rheological behavior of Kurtşeyh sepiolite at room conditions.

| Shear stress @ 600 p | | | | rpm, lbf/1 | 100 sq ft | Aging effect |
|----------------------|-----------------|--------|--------|------------|-----------|--------------|
| | kurtşeyh clay | 20 min | 16 hrs | 24 hrs | 48 hrs | (%) |
| d Type | Fresh water | 37 | 40 | 42 | 43 | 16 |
| | Semi -saturated | 27 | 31 | 33,5 | 35 | 30 |
| Mu | Saturated | 25 | 30 | 33 | 36 | 44 |
| | Salt effect (%) | 32 | 25 | 21 | 16 | |

Table 6.3. Effects of aging and salt content on rheological behavior of S sepiolite at room conditions.

| | Shear stress @ 600 rpm, lbf/100 sq ft | | | | | Aging effect |
|--------|---------------------------------------|--------|--------|--------|--------|--------------|
| | S clay | 20 min | 16 hrs | 24 hrs | 48 hrs | (%) |
| d Type | Fresh water | 18,5 | 21,5 | 24 | | 30 |
| | Semi -saturated | 15 | 18 | 20 | | 33 |
| Mu | Saturated | 14 | 17 | 19 | | 36 |
| | Salt effect (%) | 24 | 21 | 21 | | |

Table 6.4. Effects of aging and salt content on rheological behavior of YD-K sepiolite at room conditions.

| | _ | Shear str | Aging effect | | | |
|--------|-----------------|-----------|--------------|--------|--------|-----|
| _ | YD-K clay | 20 min | 16 hrs | 24 hrs | 48 hrs | (%) |
| d Type | Fresh water | 31 | 31 | 33 | 34 | 10 |
| | Semi -saturated | 23 | 26 | 28 | 30 | 30 |
| Mu | Saturated | 22 | 26 | 28 | 31 | 41 |
| | Salt effect (%) | 29 | 16 | 15 | 9 | |

| | Shear stress @ 600 rpm, lbf/100 sq ft | | | | | Aging effect |
|--------|---------------------------------------|--------|--------|--------|--------|--------------|
| _ | YD-S clay | 20 min | 16 hrs | 24 hrs | 48 hrs | (%) |
| d Type | Fresh water | 45 | 49 | 53 | 55 | 22 |
| | Semi -saturated | 29 | 35 | 38 | 42 | 45 |
| Mu | Saturated | 29 | 36 | 41,5 | 45 | 55 |
| | Salt effect (%) | 36 | 27 | 22 | 18 | |

Table 6.5. Effects of aging and salt content on rheological behavior of YD-S sepiolite at room conditions.



Figure 6.4: Changes in Rheological properties of mud prepared with TTB clay (20 min).

7. FILTRATION PROPERTIES

When the hydrostatic pressure of the drilling fluid is greater than the pore pressure, drilling fluid invades the formation. Suspended solids attempt to flow in with the liquid fraction, but very quickly particles of the appropriate size (generally one-sixth to one-third the size of pore throats at the well bore) bridge the pores and begin to build a filter cake. In time, finer and finer particles fill the interstices left by the bridging particles and ultimately form such a tight web that only liquid (filtrate) is able to penetrate. Once this filter cake is established, the flow rate of fluid into the formation is dictated by the permeability of the cake. When mud is not being circulated, filter cake grows undisturbed (static fluid loss) and the rate of filtration after the cake is established is proportional to the square root of time. When the mud is being circulated, the filter cake grows to the point at which the shear stress exerted by the mud balances the shear strength of the filter cake (dynamic fluid loss). Under this condition, the cake has a limiting thickness and the rate of filtration after the cake is established is proportional to time. Often, spurt loss is greater under dynamic conditions. Whether static or dynamic, the particles that invaded the formation during the spurt-loss phase may or may not ultimately help to form an internal filter cake, too. The API Fluid Loss Test (30 min, 100 psi through No. 50 Whatman filter paper, ambient temperature) is the standard static filtration test used in the industry; however, because it uses very fine mesh paper as the filter medium, all of the bridging particles are stopped at the surface of the paper and the spurt-loss phase is not simulated properly. Usually this leads to gross underestimates of the spurt loss. A better static filtration test is the PPT, or permeability-plugging test, which uses a 1/4inch-thick ceramic disk of known permeability [API 13B1/API 13B2]. Dynamic filtration, such as in the Fann 90 test, uses a core made of the same ceramic material and simulates shearing of the filter cake by the fluid in the annulus. Dynamic filtration properties of sepiolite drilling fluids are examined in Chapter 9 of this study. Flocculation causes particles to join together to form a loose, open network. When a drilling fluid is flocculated (e.g., through the addition of salts), the filter cake that it generates at the well bore contains some of that flocculated character, and the

rate of filtration increases. High temperature causes clay particles to flocculate that result in unacceptable high viscosities and water losses. Conversely, thinners (deflocculants) disperse clay flocculated, thereby decreasing cake permeability.

In this section, the results of API water loss properties of fully and semi salt saturated sepiolite and fresh water sepiolite muds are given separately for each clay in the ambient condition and then in order to discussion about best performance and compare these clay together in terms of API water loss properties, all of the data are shown comparatively.

7.1 Fully saturated sepiolite base muds

In addition to rheological experiments, drilling fluids prepared by all of five clays examined in this study subjected to filtration tests in the fully saturated system at room conditions. The graphics of API water loss versus filtration time are given in Appendix F between Figures F.1 to F.10 for each of the sepiolite base mud. Experimental conditions and obtained other filtration properties shown in the table in Appendix-A. Variations of water loss with time and square time are shown in different graphics. Unacceptable high water loss is a remarkable fact in all of the clays. As an example variations of API water loss versus square root of time in TTB clay is given below in the Figure 7.1 in order to discussion.



Figure 7.1: API water loss versus square root of time for TTB clay mud-fully saturated.

7.2 Discussion on fully saturated sepiolite mud results

As can be seen from the graphics, API water loss of S and Kurtseyh clay yield water losses higher than 100 ml/30 min and the observed amount of water loss is between 80-90 ml/min for the other clays. Moreover, with increasing aging time, it is clear a decrease in water loss even if be less. This decrement in TTB and YD-S clays is higher than that of other clays. A zero or little spurt loss is a desirable objective in drilling muds. As shown in Figure 7.1 for TTB clay, it has been observed a spurt loss even it is a little. This condition is valid for other type of clays. It is also noticeable that amount of spurt loss decreases with increasing aging time. Water loss graphics of samples are comparatively given in Appendix-F between Figures F.11 to F.16. These graphics shows the superiority of clays against each other for 20 min, 16 hours and 24 hours. Due to the structure of sepiolite clays, water loss of sepiolite mud are known to be high and this condition are clearly visible in the graphics. Changes of API water loss with time for all of clays are given graphically in the Figure 7.2 for 16 hours aging in full salt saturated system. As shown in this figure, YD-S and TTB clays provide the best performance in terms of API water loss; they are followed by YD-K, Kurtşeyh and S clays respectively.



Figure 7.2: Comparison of API water loss changes –fully saturated (16 hours).

7.3 Semi saturated sepiolite base muds

Similar to fully saturated system experiments, drilling fluids prepared by all of five clays, examined in this study subjected to filtration tests in the fully saturated system at room conditions. The graphics of API water loss versus time are given in Appendix F between Figures F.17 to F.26 for each of sepiolite base mud. Experimental conditions and obtained other filtration properties shown in the table in Appendix-B. Variations of water loss with time and square time are shown in different graphics. Unacceptable high water loss is a remarkable fact in all of the clays. As an example variations of API water loss versus square root of time in TTB clay is given below in the Figure 7.3 in order to discuss. In this case, water losses were obtained slightly higher than the full saturated.



Figure 7.3: API water loss versus square root of time for TTB clay mud-semi salt saturated.

7.4 Discussion on semi saturated sepiolite mud results

Similar to the fully saturated systems, water loss properties of semi saturated drilling fluids prepared by the same method. As can be seen from the graphics, API water loss of S and Kurtşeyh clay is higher than 120 ml/30 min and it is observed amount of water loss is between 100-110 ml/min for the other clays. Moreover, with increasing aging time, it is clear a decrease in water loss even if be less. This
decrement in YD-S, S and Kurtşeyh clays is higher than other clays. A zero or little spurt loss is a desirable objectives in drilling muds that as shown in figure 7.1 for TTB clay, it has been observed a spurt loss even be a little (extrapolating last three point of API water loss results to zero) This condition is valid for other type of clays. It is also noticeable that amount of spurt loss decreases with increasing aging time. Similar to full salt saturated results, water loss graphics of samples are comparatively given in Appendix-F between Figures F.27 to F.32. These graphics shows the superiority of clays against each other for 20 min, 16 hours and 24 hours. Due to the structure sepiolite clays, water loss of sepiolite mud are known to be high and this condition are clearly visible in the graphics. Changes of API water loss with time for all of clays are given graphically in the figure of 7.4 for 16 hours aging in semi salt saturated system. As shown in this figure, YD-S and TTB and YD-K clays indicate the very close results in terms of API water loss; they are following with Kurtşeyh clay that can be seen it shows better properties than S clay. Semi saturated sepiolite muds have a little more water loss than fully saturated sepiolite muds, but the amounts of water loss are still very high in terms of field usage and must be controlled.



Figure 7.4: Comparison of API water loss changes –semi salt saturated (16 hours).

7.5 Fresh water sepiolite base muds

Drilling fluids prepared by all of five clays, examined in this study subjected to filtration tests in the fresh water system at room conditions. The graphics of API water loss versus time are given in Appendix F between Figures F.33 to F.42 for each of sepiolite base mud. Experimental conditions and obtained other filtration properties shown in the table in Appendix-C. Variations of water loss with time and square time are shown in different graphics. Unacceptable high water loss is a remarkable fact in all of the clays. As an example, variations of API water loss versus square root of time in TTB clay is given below in the Figure 7.5 in order to discussion. It can be seen that, the most high water loss was obtained in Sepiolite mud prepared with fresh water.



Figure 7.5: API water loss vs. square root of time for TTB clay mud-fresh water.

7.6 Discussion on fresh water sepiolite mud results

Water loss properties of fresh water drilling muds prepared by the same methods, similar to the fully and semi saturated systems. As can be seen from the graphics, API water loss of S and Kurtşeyh clay is higher than 120 ml/30 min and it is observed amount of water loss is between 105-110 ml/min for the other clays. Unlike to fully and semi saturated systems, effect of increasing aging time, in amount of

water loss is not noticeable. A in terms of spurt loss that as shown in Figure 7.5 for TTB clay, it has been observed a spurt loss even be a little (extrapolating last three point of API water loss results to zero) This condition is valid for other type of clays. Similar to full and semi salt saturated results mentioned above, water loss graphics of samples are comparatively given in Appendix-F between Figures F.45 to F.50. These graphics shows the superiority of clays against each other for 20 min, 16 hours and 24 hours. Due to the structure sepiolite clays, water loss of sepiolite mud are known to be high and this condition are clearly visible in the graphics. Changes of API water loss with time for all of clays are given graphically in the Figure 7.6 for 16 hours aging in fresh water system. As shown in this figure, YD-S and TTB clays indicate the very close results in terms of API water loss; they are following with YD-K clay that can be seen it shows better properties than Kurtşeyh and S clays. Fresh water sepiolite muds give higher loss of water than full and semi salt saturated sepiolite muds that are not acceptable values.



Figure 7.6: comparison of API water loss changes –fresh water (16 hours).

7.7 The effects of aging and salinity on filtration changes

The results of filtration properties of fully and semi salt saturated sepiolite and fresh water sepiolite muds are given comparatively in the room condition between Figures F.51 to F.60 shown graphically in the Appendix F. The data used in graphics were

obtained from the values of Appendix A, Appendix B and Appendix C that are shown in tables. In addition, effects of aging and salt content on rheological behavior of sepiolite clays at room conditions are tabulated and given between Tables 7.1 to 7.5. The filtration properties of TTB clay are represented in fully and semi saturated and fresh water system comparatively as an example in order to discuss about results, (Figure 7.7). In this section, changes in filtration properties are examined for the results of 20 minutes, 16 hours, 24 hours and 48 hours aging of muds.

A good filtration characteristic is expected from any mud used in a drilling well, particularly in high temperature environments. High temperature causes clay particles to flocculate that result in unacceptable high viscosities and water losses. The problem gets worsen with increasing salt intrusion into the mud. The results of experiments related to high temperature are given and discussed in Chapter 8. As a result, both high salt content and high temperature make sepiolite muds unique among others in terms of rheological and filtration properties. Even though the sepiolite muds perform better in harsh environments (high salinity and high temperature), their natural water loss values are not suitable for the most well drilling, and must be controlled to achieve secure drilling conditions. Mud cakes obtained from sepiolite muds are too thick due to high filtration and unacceptable, ranging from 6 to 10 mm.

In spite of this fact that the API dose not determine a certain value for amount of water loss in the case of sepiolite base muds, however, as shown in Figures, API water losses of the muds are much above the industry standards that is maximum 15 ml/30 min in ambient conditions for bentonite based fresh water systems. If the drilling environment is exposed to sensitive formations, almost zero water loss is needed and should be achieved by means of additives. The results of filtration experiments with additives are given in Chapter 8 also. As seen, filtration characteristic of all sepiolite clays examined in this study gets better with increasing salt content. Actually, there is negligible difference between fresh water mud and semi-saturated mud cases. It can be said that unlike rheological properties, the sepiolite-based muds performs better with increasing salt concentration. As indicated in Table 7.5 that shows the effect of salinity and aging time on filtration properties of YD-S, amount of water loss recorded 24 hours of aging decreases from 106 ml for fresh water mud to 80 ml for saturated mud. Total change referenced to fresh water mud has a value of 25 % and it is very important. It can be said that if the water

phase salinity is concerned, sepiolite muds are performing much better compared to the bentonite mud (actually, bentonite cannot be used in high salinity).

The aging effect shown in Table 7.3 indicates that there is negligible effect of aging periods on sepiolite muds prepared by using fresh water. The same behavior is observed when the sepiolite muds are prepared with different salt content. When rheological and water loss properties were evaluated together, YD-S and TTB clays provide the best performance, they are followed by Kurtşeyh, YD-K and S clays respectively. However, considering to this fact that filtration control is more critical in the case of sepiolite base muds, therefore it was decided to use of TTB and YD-S clays in the next phase of the study. When pH measurements all of the experiments are compared, as expected, pH values of muds are lower than filtrations pH measurements. This case is due to exist of some alkaline materials in structure of clay and salt in the water phase of the drilling muds, which go into solution.



Figure 7.7: Changes in API water loss in mud prepared with TTB clay (20 min).

| | _ | API | water loss | Aging effect | |
|-----|-----------------|--------|------------|--------------|-----|
| | TTB clay | 20 min | 16 hrs | 24 hrs | (%) |
| ype | Fresh water | 108 | 108 | 108 | 0 |
| d T | Semi -saturated | 110 | 110 | 108 | 2 |
| Mu | Saturated | 98 | 92 | 86 | 14 |
| | Salt effect (%) | 9 | 15 | 20 | |

Table 7.1: Effect of salt content and aging on API water loss in TTB clay.

Table 7.2: Effect of salt content and aging on API water loss in kurtşeyh clay.

| | _ | API | water loss | Aging effect | |
|-----|-----------------|--------|------------|--------------|-----|
| _ | kurtşeyh clay | 20 min | 16 hrs | 24 hrs | (%) |
| ype | Fresh water | 124 | 128 | 126 | -2 |
| d T | Semi -saturated | 124 | 122 | 120 | 3 |
| Mu | Saturated | 108 | 106 | 100 | 8 |
| | Salt effect (%) | 13 | 17 | 21 | |

Table 7.3: Effect of salt content and aging on API water loss in S clay.

| | | API water loss, ml | | | Aging effect | |
|-----|-----------------|--------------------|--------|--------|--------------|--|
| | S clay | 20 min | 16 hrs | 24 hrs | (%) | |
| ype | Fresh water | 135 | 136 | 134 | 1 | |
| d T | Semi -saturated | 140 | 135 | 135 | 4 | |
| Mu | Saturated | 120 | 112 | 112 | 7 | |
| | Salt effect (%) | 11 | 18 | 16 | | |

Table 7.4: Effect of salt content and aging on API water loss in YD-K clay.

| | | API | water loss | Aging effect | |
|-----|-----------------|--------|------------|--------------|-----|
| | YD-K clay | 20 min | 16 hrs | 24 hrs | (%) |
| ype | Fresh water | 108 | 112 | 112 | -4 |
| d T | Semi -saturated | 106 | 106 | 104 | 2 |
| Mu | Saturated | 90 | 92 | 89 | 1 |
| | Salt effect (%) | 17 | 18 | 21 | |

| | | API water loss, ml | | | Aging effect | |
|--------|-----------------|--------------------|--------|--------|--------------|--|
| | YD-S clay | 20 min | 16 hrs | 24 hrs | (%) | |
| ype | Fresh water | 108 | 108 | 106 | 2 | |
| Mud Ty | Semi -saturated | 104 | 108 | 92 | 13 | |
| | Saturated | 90 | 80 | 80 | 13 | |
| | Salt effect (%) | 17 | 26 | 25 | | |

Table 7.5: Effect of salt content and aging on API water loss in YD-S clay.

8. RHEOLOGICAL AND FILTRATION PROPERTIES OF SEPIOLITE MUDS CONTAINING ADDITIVES

As stated in earlier chapters, sepiolite clays used in the study satisfy the limit value of rheological properties given in the API standards, except one (S clay). On the other hand, because of its rod-like shape, sepiolite provides no filtration control. In terms of API water loss, all of the clays are given the unacceptable values. Therefore, it is necessary to control the mud filtration loss. For this purpose, changes in rheological and water loss properties were examined using different additives.

8.1 Additives

Experiments, in this section, have been done using of YD-S and TTB clays that yield the best performance among the others. Some special materials are used as additives in order to control of sepiolite base mud properties specially for filtration control that is the major problems of drilling fluids prepared with sepiolite clays. Polymer A is a filtration control agent provides filtration control and secondary viscosity in water-based drilling fluids at temperature beyond 400°F (204°C). It has good salt stability and will tolerate moderate levels of calcium contamination. Polymer 2 is a liquid polymer used to reduce the viscosity of the mud and functions extremely well in reducing high-temperature gelation. Polymer B is also effective in the presence of chlorides and aids in filtration control. It is used to pretreat the mud system prior to drilling cement and stable at temperature above 400 °F (204 °C) that may be used as a deflocculant to maintain low rheological properties in high temperature wells. Caustic or soda ash are used to control of mud pH and water hardness. Experiments were carried out using fresh water, semi-salt saturated and full salt saturated systems then the results were discussed.

8.2 Problems and experimental methods

High temperature in deep oil and gas wells or geothermal wells is a harsh environment problem that can affect the properties of drilling fluid. From the drilling fluid point of view, high temperatures can be considered as those above which conventional drilling fluid additives begin to thermally degrade at an appreciable rate. The degradation leads to loss of product function, and system maintenance becomes difficult and expensive. The majority of drilling fluid treatment chemicals derived from natural products begin to degrade at temperatures between 250 and 275°F. However, many systems designed for hot wells are based on clay containing lignosulphonates and lignites that can exhibit temperature stability up to approximately 350°F. However, management of these drilling fluids above 300°F can be difficult and expensive.

Thermal degradation can be simplistically thought of as the result of putting so much energy into a chemical substance that some portion of its structure can break off or change form. Similar results can be affected at lower temperatures by the presence of certain chemicals. Oxygen (from air) can promote oxidation; water (present in the drilling fluid) can promote hydrolysis. Whatever the cause, or particular chemical reaction involved, the end result is that at higher temperatures formerly stable drilling fluids become difficult to control. Unfortunately, elevated temperatures are usually not the only stresses experienced by drilling fluids in high bottom hole temperature wells. In this section, it is tried to prepare a economical typical sepiolite base mud, containing additive that would satisfy acceptable properties especially in terms of filtration control at high temperature and high-pressure conditions. Due to lack of HPHT viscometer in laboratory, the rheological experiments in high temperature condition are done using aging cells that the results of these are given in the next chapter along with the effect of shale (reactive clay) contamination.

Fresh water and salt-water sepiolite based mud have been subject to water loss experiments in different pressure and temperature. Experiments were made under pressure differentials at 100 psi and 500 psi and 350 °F temperatures. In addition, tests are repeated in room condition. Different concentration of additive materials added to samples in fresh water and salt water systems. Typical compositions of sepiolite base mud used in this section to prepare fresh water and salt-water mud system are shown in Tables 8.1 to 8.3. In addition, results of water loss experiments of sepiolite base muds containing additives in room condition and 350 °F under pressures differentials of 100 psi and 500 psi for fresh water and salt water drilling condition are given between Tables 8.4 to 8-6. Moreover, changes in API water loss as a function of time are shown between Figures G.1 to G.6 presented in Appendix

G. In order to discuss about the results, only two figure of API water loss properties are shown in Figures 8.1-8.2 in this section.

| Mud composition | | | | | | | | |
|-----------------|-------------------------|-------------|---------------|--|--|--|--|--|
| No | Additive | Function | Concentration | | | | | |
| 1 | Sepiolite (TTB or YD-S) | Viscosifier | 20 ppb | | | | | |
| 2 | Polymer 1 | Fluid loss | 3 ppb | | | | | |

Table 8.1: Typical composition of fresh water sepiolite base mud containing additives at room condition (80°F).

Table 8.2: Typical composition of salt saturated sepiolite base mud at HTHP condition (350 °F).

| | Mud Composition | | | | | | | | |
|----|------------------------|---------------------------------|-----------------------|--|--|--|--|--|--|
| No | additive | Function | concentration | | | | | | |
| 1 | NAOH | PH control | 0.75 lb/bbl | | | | | | |
| 2 | Sepiolite (TTB – YD-S) | Viscosifier | 20 lb/bbl | | | | | | |
| 3 | Polymer 1 | Deflocculant | 5 lb/bbl | | | | | | |
| 4 | Polymer 2 | Fluid loss | 7 lb/bbl | | | | | | |
| 5 | Glycol | Fluid loss and shale stabilizer | % 3 percent of volume | | | | | | |

| Table 8.3: | Typical composition | of fresh water | sepiolite base | mud at HTHP |
|-------------------|---------------------|----------------|----------------|-------------|
| | condition (350 °F). | | | |

| | Mud Composition | | | | | | | | |
|----|--|---------------------------------|-----------------------|--|--|--|--|--|--|
| No | additive | Function | concentration | | | | | | |
| 1 | Sepiolite clay (TTB – YD-S) Viscosifier | | 15 lb/bbl | | | | | | |
| 2 | Bentonite | Fluid loss | 5 lb/bbl | | | | | | |
| 3 | NAOH | PH control | 0.75 lb/bbl | | | | | | |
| 4 | Polymer 1 | Fluid loss | 4 lb/bbl | | | | | | |
| 5 | Polymer 2 | Deflocculant | 3 lb/bbl | | | | | | |
| 6 | Glycol | Fluid loss and shale stabilizer | % 3 percent of volume | | | | | | |

| Water loss | API water loss, ml | | | | | | | |
|------------|--------------------|----------|----------|--------|----------|----------|--|--|
| test | | TTB | | YD-S | | | | |
| Time, min | 20 min | 16 hours | 24 hours | 20 min | 16 hours | 24 hours | | |
| 1 | 1 | 0,4 | 0,2 | 2,4 | 2,2 | 1,8 | | |
| 2 | 2,4 | 1,4 | 1 | 3,8 | 3,6 | 3 | | |
| 2,5 | 2,8 | 2 | 1,4 | 4,4 | 4,2 | 3,4 | | |
| 3 | 3,4 | 2,4 | 1,8 | 5 | 4,8 | 4 | | |
| 4 | 4,2 | 3,2 | 2,4 | 5,8 | 5,6 | 4,8 | | |
| 5 | 5 | 4 | 3 | 6,5 | 6,3 | 5,6 | | |
| 7,5 | 6,6 | 5,4 | 4,5 | 8 | 7,8 | 7 | | |
| 10 | 7,8 | 6,6 | 5,6 | 9,4 | 9,2 | 8,4 | | |
| 20 | 12 | 10,6 | 9,4 | 13,3 | 13,1 | 12,2 | | |
| 30 | 14,8 | 13,4 | 12,2 | 16,6 | 16,4 | 15 | | |

Table 8.4: API water loss of fresh water sepiolite base mud containing additives at room condition (80 °F).

Table 8.5: API HTHP water loss of fresh water sepiolite base mud at HTHP
condition $(350 \text{ }^\circ\text{F}) - 20 \text{ min.}$

| Water | API HTHP Water Loss, ml | | | | | | |
|-----------|-------------------------|----------|------------------|------|--|--|--|
| Loss Test | 100 psi | @ 350 °F | 500 psi @ 350 °F | | | | |
| Time, min | TTB | YD-S | TTB | YD-S | | | |
| 4 | 5,2 | 6,2 | 6,8 | 9 | | | |
| 7,5 | 7,4 | 8,4 | 10,2 | 12,2 | | | |
| 20 | 13,4 | 14,6 | 17,4 | 19 | | | |
| 30 | 17,6 | 18 | 21,6 | 23,6 | | | |

Table 8.6: API HTHP water loss of salt saturated sepiolite base mud at HTHP condition (350 °F) – 20 min.

| Water | API HTHP Water Loss, ml | | | | | | | | |
|-----------|-------------------------|----------|---------|----------------|----------------|----------------|------|----------------|--|
| Loss Test | 100 psi @350 °F | | | | 500psi @350 °F | | | | |
| | Semi s | aturated | Full sa | Full saturated | | Semi saturated | | Full saturated | |
| Time, min | TTB | YD-S | TTB | YD-S | TTB | YD-S | TTB | YD-S | |
| 4 | 3,6 | 4,2 | 2,8 | 4 | 4,6 | 5,2 | 4 | 4 | |
| 7,5 | 5,6 | 6,2 | 4,4 | 5,6 | 7 | 7,4 | 6 | 6,2 | |
| 20 | 10,4 | 11,2 | 7,8 | 9,2 | 13 | 13,8 | 10,2 | 10,8 | |
| 30 | 13,8 | 14,6 | 9,6 | 11 | 17 | 17,8 | 13 | 13,8 | |

8.3 Discussion

As can be seen from the figures and the tables, it is observed that in both of the sepiolite sample muds, amount of water loss has been decreased significantly compare to sepiolite sample mud without additives. With due attention to results obtained for typical sepiolite mud given in Table 8.4, it is obvious that only by adding 3 lb/bbl of special copolymer into mud system amount of filtration decrease to reasonable value in both TTB and YD-S clays under room conditions. In both clays, it is observed that amount of water loss decrease slightly with increasing aging time under room conditions; this can be one of characteristic properties of sepiolite clays. In addition, it is observed that filtration properties of TTB clay is better than YD-S clay at room conditions. Despite of obtained data we can say that amount of water loss decrease with increasing aging time and this is interesting and important event.

At high pressure high temperature conditions a typical composition of sepiolite (both TTB and YD-S clays) base mud is examined and obtained positively proper amount of filtration that is suitable for field applications. The results of these test are given in Table 8.5. Sepiolite base mud prepared with TTB and YD-S clays in fresh water system have amount of reasonable filtration at 350 °F temperature in both low pressure (100 psi) and high pressure (500 psi). In addition, similar to results obtained in room condition, TTB clay has lower filtration rate compare to YD-S clay. On the other hand, in the case of salt saturated system in sepiolite base mud, it is observed that controlling of filtration is done successfully. Salinity environment has fatal effect in properties of conventional drilling fluids especially on filtration control of muds, but from obtained results that are given in Table 8.6, it is clear that sepiolite base mud prepared with both clays have low amount of water loss even in fully saturated system. Similar to fresh water in semi and fully saturated system, TTB clay has proper filtration properties compare to YD-S clay. There is an interesting result in filtration salt saturated sepiolite base mud. When the results of water loss in semi saturated and fully saturated systems are compared together, it is very obvious that when amount of environment salinity increase, in both sepiolite clays amount of water loss decrease that is due to characteristic properties of sepiolite clays. In spite of this fact that API standards do not determine a specified values for water loss of

sepiolite base muds, results obtained in this study evaluated according to field data of some companies such as TPAO.



Figure 8.1: API water loss versus square root of time in sepiolite (TTB) fresh water base mud containing additive at room condition (80°F).



Figure 8.2: API HTHP water loss versus square root of time in sepiolite (YD-S and TTB) salt saturated base mud containing additive at 500 psi @ 350°F.

9. DYNAMIC FILTRATION PROPERTIES OF SEPIOLITE MUDS

There are two kinds of filtrations during drilling and completion operations. One is the static filtration after stopping the circulation of drilling fluids. The mud cake thickness increase gradually. On the other hand, the filtration rate decreases with time. Another is the dynamic filtration when the mud cake formed is eroded by circulating of drilling fluids. The erosion rate depends on the shear rate of the fluid at the face of the cake. If the shear rate remains constant, cake thickness and filtration rate reach steady state, usually in a matter of hours. When the conditions change, a new steady state will be established. There is no a proportional relationship between static filtration and dynamic filtration of differential drilling fluids, therefore the measurement of dynamic filter loss is particularly important.

9.1 Experimental equipment

The FANN Model 90 is a dynamic radial filtration apparatus, manufactured and sold by the Fann Instrument Company, Houston, Tex. The device evaluates the filtration properties of a circulation fluid through a ceramic core. Dynamic filtration simulates the effect of fluid movement (shear rate) on the filtration rate and filter cake deposition in an actual well.

The Model 90 is a device used in the industry for conducting filter cake formation and permeability analysis for drilling fluid optimization. The Model 90 can be heated and pressurized to provide the closest possible simulation of downhole conditions. The filter medium is a thick walled cylinder with rock like characteristics to simulate the formation. The filter medium is available in varying porosities and permeabilities. A simulated view is depicted at Figure 9.1.

Filtration occurs radially from the inside of the filter core to the outside. At the same time, the filter cake is formed on the inside of the filter core to simulate filter cake formation on the wall of a borehole. A polished stainless steel shear bob runs through the central axis of the filter core. The shear bob is rotated to produce a concentric cylinder type shear across the filtration surface.



Figure9.1: A schematic view of ceramic filter core (web 3-2009)

9.2 Interpretation of dynamic filtration test:

The test determines if the fluid is properly conditioned to drill through permeable formations. Currently there are no standard methods for the interpretation of Dynamic Filtration Data. Some parameters that can be determined from the data obtained using the model 90 are:

- Spurt loss volume Usually considered to the volume of filtrate obtained initially when filtration begins. This could be considered as the difference in the volume from the start of filtration volume at the end of the first 10 seconds. It is desirable to have a low spurt loss volume.
- Particle plugging of pores It is the primary factor controlling spurt loss volume. If the particle size distribution of the sample solids is optimized for a specified pore size, plugging will occur and the spurt loss volume will be minimized.
- 3. **Dynamic filtration rate** It can be evaluated over any interval during the filtration process. This is accomplished by calculating the rate of change in the filter volume versus time (ml/min). It is desirable to have a low filtration rate. The rate should be less than 0.2 ml/min for most oil well drilling fluid systems.
- 4. Cake Deposition Index (CDI) May be determined by calculating the rate of change in the filtration rate versus time [(ml/hr)/hr]. A low CDI indicates that the formation of the filter cake has almost reached steady state. New cake is being deposited at almost the same rate that cake it is being washed away or

the additional cake has no effect on filtration. For most oil well drilling fluid systems a CDI of 10 ml/hr² or less is desired.

Moreover, the values are mentioned above for CDI and filtration rate have been revised for a field mud and for the most oil well drilling fluid systems the desired maximum recommendation values of CDI and filtration rate are give in Table 9.1.

In addition, the typical results of this test shown graphically in Figure 9.2. It must be mentioned that these values are given for oil well drilling fluids and for other systems there are no standard method for dynamic filtration system.

| Table 9.1: FANN | 90 maximum | recommended | values, | (Web 3-2009). |
|-----------------|------------|-------------|---------|---|
| | | | | ())))))))))))))))))) |

| FANN 90 maximum recommended values | | | | |
|--|------|----|--|--|
| Mud weight, Ib/gal (sg) Rate, mL/min CDI | | | | |
| 9–12 (1.08-1.44) | 0.22 | 25 | | |
| 12–15 (1.44-1.80) | 0.18 | 20 | | |
| 15 or greater (1.80 or greater) | 0.14 | 16 | | |



Figure 9.2: Typical FANN 90 test results. (Web 3-2009)

9.3 Composition of sepiolite muds to be tested

Seven fresh water based muds were chosen for detailed study. All muds are prepared with 350 ml of fresh water and their compositions are same except one observed at 200 ⁰F. Compositions of two different muds are given in Table 9.2.

| Substance | Quantity (lbm/bbl) | | | | | |
|------------|--------------------|-------------|------------------------|--|--|--|
| Substance | Muds @ high | temperature | Muds @ low temperature | | | |
| Mud type | Unweighted | Weighted | Weighted | | | |
| Sepiolite | 20 | 20 | 20 | | | |
| (TTB-YD-S) | | | | | | |
| Soda ash | 0,1 | 0,1 | 0,1 | | | |
| Polymer B | 5 | 5 | 4 | | | |
| Polymer A | 5,5 | 5,5 | - | | | |
| Polymer C | - | - | 3 | | | |
| Barite | - | 200 | 200 | | | |

Table 9.2: Compositions of sepiolite muds used in dynamic filtration test.

Two different weighted type of muds are chosen because the use of polymer types varies depending on the temperature that the experiments are made at. In addition, dynamic filtration of unweighted sepiolite mud (density = 8,70 lb/gal) is conducted in order to compare the results. Polymer B is a chemical substance which is used to control the rheology of the muds at high temperatures. Polymer A and C are chemicals used to control the water loss. Polymer A is effective at high temperatures over 300 ⁰F. Polymer C has efficiency at temperatures below 300 ⁰F. All muds are prepared by following the American Petroleum Institute (API) standards. Their densities, pH and rheological properties are presented at Table 9.3.

In terms of rheological properties, there is high plastic viscosity and moderate yield point due to the existence of considerable amount of solids (barite). The effect of polymer A providing secondary viscosity along with its filtration control feature should not be forgotten. These properties may be affecting on rheological properties of muds at different temperature conditions ($300\ {}^{0}\text{F} - 350\ {}^{0}\text{F} - 400\ {}^{0}\text{F}$). Viscosity and yield point of the mud at 200 ${}^{0}\text{F}$ is lower than the mud at different temperature conditions. This situation is due to type of polymer used at 200 ${}^{0}\text{F}$ to control fluid

loss (polymer C). This polymer helps minimize viscosity impact due to being low molecular weight polymer along with its fluid loss decreasing property. It is also observed that in both muds, amounts of gel strength values are not significantly progressive and it is seen that gel strength is broken right after mixing at mixer.

| Property | | Muds @ high | Muds @ low |
|----------|---|-------------|-------------|
| | | temperature | temperature |
| Densi | ity, ppg | 11,5 | 11,6 |
| | 600 | 120 | 64 |
| gu | 300 | 80 | 39 |
| eadi | 200 | 65 | 30 |
| al Ro | 100 | 44 | 18 |
| Di | 6 | 12 | 4 |
| | 3 | 9 | 3 |
| Appa | rent Viscosity (AV), cp | 60 | 32 |
| Plasti | c Viscosity (PV), cp | 40 | 25 |
| Yield | Point (YP), lb/100 ft ² | 40 | 14 |
| 10 see | c gel strength , lb/100 ft ² | 13 | 3 |
| 1 min | gel strength, lb/100 ft ² | 18 | 4 |
| 10 mi | n gel strength, lb/100 ft ² | 35 | 10 |
| pH (n | nud) | 9 | 9 |

Table 9.3: Properties of two different weighted muds

9.4 Discussion and results

Experiments in this section have been done using of YD-S and TTB clays. Experiments were carried out using fresh water, and then the results were discussed. Typical fresh water Sepiolite base mud have been subject to dynamic filtration test in different high temperatures at 100 psi differential pressure using dynamic filtration system model FANN 90. The entire tests are done by using filter core with 35 micron pore size. In order to obtain best performance in terms of particle plugging pores, and to compare dynamic fluid loss between clay having different grain size, we select three type of TTB clay that have different grain size. These three different particle sizes are observed such as below 20 microns, above 75 microns and particles having

grain size between 20 and 75 microns. After first experiments, the study attended with the particle size between 20 and 75 microns. Effective bridging of a reservoir depends upon both the particle size distribution of materials comprising the drill-in fluid and the pore throat diameters of the reservoir rock. A bridging material is chosen by matching its size to the diameter of formation pore throats. The industry-accepted rules for selecting size and concentration of bridging materials are based upon work carried out by A. Abrams 1977, and include the following:

The medium particle size of the bridging additive should be equal to or slightly greater than one-third the medium pore size of the formation. Once the mean pore diameter is known, the particle size distribution of the bridging solids must be measured and adjusted to meet the required specifications and included in the drill-in fluid formulation. An alternative method of determining optimal particle size distribution is to use the ideal packing theory. According to Dick et al (2000). that stated Ideal Packing Theory, ideal packing theory can be defined as the full range of particle size distribution required to effectively seal all voids, including those created by bridging agents' results in a tighter and less invading cake (Dick et al, 2000). In spite of this theory and considering to the results obtained from dynamic filtration tests, it is clear that drilling mud prepared with sepiolite having grain size between 20 and 75 microns provides better sealing and proper results in terms of filtration rate and cake deposition index while using a ceramic filter core having 35 micron (5,5 darcy) pore size. The results of experiments are shown graphically between Figures H.1-H.9 in Appendix-H and presented as a summary in Table 9.4.

During the forming an external filter cake, fine solids are especially forced into the formation, building an internal filter cake. An internal filter cake plugs the near surface pore and reduces the formation permeability. Fine particles penetrate deeply into the pores and are not easily removed by back flushing. Invasion of larger particles is usually localized to near surface. Studies conducted by Bailey et al. (1999) show a strong correlation between invasion and damage. Because of that, minimizing of internal filter cake and quickly forming of external cake is very important for both fluid loss and formation damage control. A semi permeable slicker external filter cake can significantly reduce the invasion of the solids and the filtrate. In all the tests, the internal filter cake is formed during the first 5 seconds, as can be seen in the results, once this internal filter cake is formed; the incremental filtration volume is minimal. These results also show no considerable increase in

spurt loss. Amount of spurt losses of samples are given in Table 9.4. It is observed that TTB clay base mud have dynamic filtration rate lower than YD-S clay in same conditions. Data obtained from tests shows the proper filtration rate (0.22 - 0.27)ml/min) for sepiolite base mud's at high temperature and 100 psi pressure (according to confirmation by FANN Company). Also in temperature of 300°F and 500 psi pressure, it is observed that amount of 0.33 ml/min in terms of filtration rate that is a reasonable value in high pressure condition for sepiolite base muds. Moreover it is observe that in the entire tests amount of low CDI (below 10 ml/hr/hr) that is a desirable value. Also, as can be seen from graphics, a decrease in the CDI value is observed with increasing pressure, which indicates a compressible filter cake. However, in temperature of 400 °F at 100 psi pressure, we can see a little high filtration rate (0.34 ml/min) for our typical composition of mud. It can be improved with mud treatment, using of other concentration of additives. In the case of unweighted sepiolite mud presented in Figure H.9 in Appendix H, amount of filtration rate (0.32 ml/min) is higher than weighted mud at same condition, also unweighted sepiolite mud have amount of very low CDI (0.75 ml/hr/hr) that indicate, external cake is not formed, as seen in the test process. Unweighted system have a high amount of spurt loss (13,60 ml), as can be seen from results.

| Sample Code | Temperature, ⁰ F | ∆p, psia | DFR, ml/min | CDI, ml/hr/hr | Spurt loss, ml | Particle size, d micron |
|----------------|--------------------------------|-------------|----------------|------------------|-------------------|-------------------------------|
| TTB | 300 | 100 | 0,22 | 6,3 | 3,50 | 20< d <75 |
| YD-S | 300 | 100 | 0,25 | 10,53 | 3,20 | d=28 |
| TTB* | 300 | 100 | 0,32 | 0,75 | 13,60 | 20< d <75 |
| TTB | 300 | 100 | 0,25 | 12,6 | 2,60 | 75 <d< td=""></d<> |
| TTB | 300 | 100 | 0,26 | 6,95 | 3,00 | 20>d |
| TTB | 350 | 100 | 0,27 | 6,98 | 3,70 | 20< d <75 |
| TTB | 400 | 100 | 0,34 | 6,75 | 3,25 | 20< d <75 |
| TTB | 300 | 500 | 0,33 | 3 | 4,40 | 20< d <75 |
| TTB | 200 | 100 | 0,12 | 5,47 | 2,90 | 20< d <75 |
| | TTB*: Un | weight | ed TTB cla | ay base mud | system | |

Table 9.4: Summary of the experiments' results and conditions.



Figure 9.3: Dynamic filtration results of weighted TTB clay (20 < d < 75) mud at 300° F and 100 psi.

10. PROPERTIES OF SEPIOLITE MUDS CONTAMINATED WITH REACTIVE CLAYS

The composition and treatment of drilling fluids depends on the formation encountered or material added during drilling operations. Some of these materials under certain circumstances, along with formation cuttings, can be considered contaminants. During drilling and/or completion, it is not uncommon to encounter strata comprising reactive shales. Up to 80% of the rocks we drill are shales (clayrich rocks). As referred to herein, the term "shale" will be understood to mean materials such as certain types of clays (for example, bentonite) and related subterranean materials that may "swell," or increase in volume, when exposed to water. In other words, zones that comprise shales and/or reactive clays can become unstable, when they are in contact with water in a drilling fluid. These zones contain clays that have been dehydrated over geologic time by overburden pressure. When these zones are exposed to a water containing material such as a drilling fluid, the clays osmotically imbibe water from the drilling fluid and swell. Reactive shales may be problematic during drilling operations because of their tendency to degrade when exposed to aqueous media such as aqueous-based drilling fluids. This degradation, of which swelling is one example, can result in undesirable drilling conditions and undesirable interference with the drilling fluid. For instance, the degradation of the shale may interfere with attempts to maintain the integrity of drilled cuttings traveling up the well bore until such time as the cuttings can be removed by solids control equipment located at the surface. Degradation of drilled cuttings prior to their removal at the surface greatly prolongs drilling time, because shale particles traveling up the well bore break up into smaller and smaller particles, which increasingly exposes new surface area of the shale to the drilling fluid, which leads to still further absorption of water and further degradation. The swelling of the shale induces stresses, loss of mechanical strength, and shale failure. Shale crumbling into the borehole ("sloughing") can ultimately place a burden on the drill bit which makes it impossible to retrieve. For this reason, it is important that the rheological and water

loss properties of drilling mud are examined with respected to entrance active clay into mud.

Salts such as potassium chloride have been widely used in drilling treatments to convert the formation material from the sodium form by ion exchange to the potassium form, which is less vulnerable to swelling. In addition, the use of high concentrations of potassium salts affects the osmotic balance and tends to inhibit the flow of water away from the high potassium salt concentration fluids into the shale. However, it is difficult to maintain the required high concentrations of potassium salts and the physical introduction of such salts causes difficulties with the use of the viscosifier materials typically used for drilling.

10.1 Remarkable knowledge

Carrying Capacity of drilling mud is a very significant criterion in order to evaluate drilling fluids. Only three drilling-fluid parameters are controllable to enhance moving drilled solids from the well bore: AV (annular velocity), density (mud weight [MW]), and viscosity. By examining cuttings discarded from shale shakers in vertical and near-vertical wells during a 10-year period, it was learned that sharp edges on the cuttings resulted when the product of those three parameters was about 400,000 or higher [Robinson]. AV was measured in ft/min, MW in lb/gal, and viscosity (the consistency, K, in the Power Law model) in cp. When the product of these three parameters was around 200,000, the cuttings were well rounded, indicating grinding during the transport up the well bore. When the product of these parameters was 100,000 or less, the cuttings were small, almost grain sized. Thus, the term carrying capacity index (CCI) was created by dividing the product of these three parameters by 400,000:

CCI = (AV). (MW). (K)/400,000

To ensure good hole cleaning, CCI should be 1 or greater. This equation applies to wellbores up to an angle of 35° , just below the 45° angle of repose of cuttings. The AV chosen for the calculation should be the lowest value encountered (e.g., for offshore operations, probably in the riser). If the calculation shows that the CCI is too low for adequate cleaning, the equation can be rearranged (assuming CCI=1) to predict the change in consistency, K, required to bring most of the cuttings to the surface:

K = 400,000/ (MW). (AV)

Since mud reports still describe the rheology of the drilling fluid in terms of the Bingham Plastic model, a method is needed to readily convert K into PV and YP. The chart given in Figure 10.1 (Taken from Drilling Fluid Processing Handbook, Gulf professional publishing, 2005) serves well for this purpose. Generally, YP may be adjusted with appropriate additives without changing PV significantly.



Figure 10.1: Conversion of Bingham plastic yield point to power law K (Bourgoyne at al).

10.2 Experimental procedure

In this section, the rheological and water loss properties of sepiolite base drilling fluids are examined when amount of clay enters to mud system. In order to prepare this condition, clay called OCMA is used as enterer clay that simulates the clay enters from shale formation into mud. After mixing procedure and preparing mud, OCMA clay is added to the system and mixed for 30 minute. In addition, tests are repeated in high salinity environments. In order to simulate entrance of the salt into the mud from formation fluid having high salinity or from salt zones, after preparing typical mud a certain amount of sodium chloride is added to mud and along with OCMA, the experiments are conducted. After determining rheological properties, prepared mud put into aging cell and rolled in roller oven for 16 hours at high

temperature that in this study aging temperature is 300 °F and 350 °F. After 16 hours aging the rheological and water loss properties of mud samples are determined and obtained results are discussed in terms of best performance. First fresh water system is evaluated then typical sepiolite drilling mud subjected to salt and clay entered into the system.

10.3 Mud properties in fresh water system along with entrance of reactive clays

A typical composition of unweighted and weighted sepiolite (TTB clay) base mud for fresh water system is given in Table 10.1. In the first stage, after preparing of mud in a special order and mixing time, rheological properties of drilling mud are measured and then put into aging cells and pressurized in order to age for 16 hours at different temperature in roller oven to simulate well conditions. It is important that aging cells should be pressurized above pressure that prevent vaporization. Appropriate pressures are available in steam tables (web). Rheological properties and water loss of sepiolite base mud after 16 hours aging are determined. In this section TTB clay is selected as mud clay due to its best properties in previous chapters. Rheological properties are determined in 80 °F and 120 °F. Moreover, filtration operation is done in 300 °F temperature for all of the tests using HTHP filtration press. The results of rheological properties of muds aged in high temperature for 16 hours are given graphically in Figures 10.2 to 10.3. In addition, results of these properties along with amount of water loss results are tabulated in Table I.1 to I.2 in the appendix I.

In the second stage of this section, bentonite clay with API specifications (OCMA) is added into mud after preparing base drilling sepiolite mud to simulate actual conditions in the well when the unstable shale formations are drilled and reactive clays enter into the mud. It should be mentioned that a polyalkylene glycol is added to base mud before adding OCMA clay in order to stabilize reactive clays, increase lubricity and help HTHP filtration. Thus in this manner, it would be comprised a new composition of mud formulated in Table 10.2 for weighted and unweighted system in fresh water experiments. The results of rheological properties with respect to entering of reactive clay into mud system are given graphically in Figures 10.4 to 10.5. These results along with filtration results are also given in Table I.3 to I.4 in the appendix I. In this section methylene blue test is also done. When using water based drilling fluid, the MBT is one of the most meaningful tests available to indicate the general condition of the drilling fluid. The results of this test give an indication of the amount and size of active clays in the drilling fluid. In normal wells, a non-dispersed polymer drilling fluid should have an MBT no greater than 20 lb/bbl. In high density drilling fluids, 15 lb/bbl should be considered the upper limit. High temperatures can rapidly increase the yield of commercial bentonite and reactive solids; this in turn will produce a rapid increase in values obtained from MBT tests. Drilling fluids with high MBTs are susceptible to contaminants that would not normally cause problems in low solid drilling fluids (e.g. calcium, carbonates, etc.).

| Substance | Quantity (lbm/bbl) | | | | |
|-------------|--------------------|----------|--|--|--|
| Bubstance | Unweighted | Weighted | | | |
| Sepiolite | 20 | 20 | | | |
| Soda Ash | 0,1 | 0,1 | | | |
| Polymer - 1 | 4 | 4 | | | |
| Polymer - 2 | 5 | 5 | | | |
| Barite | - | 150 | | | |

Table 10.1: Compositions of unweighted and weighted typical sepiolite mud.

| Table 10.2: | Compositions of unweighted and weighted typical sepiolite mud i | n |
|-------------|---|---|
| | erms of reactive clay enter into mud system. | |

| Substance | Quantity (lbm/bbl) | | | | |
|-------------|--------------------|---------------|--|--|--|
| Substance | Unweighted | Weighted | | | |
| Sepiolite | 20 | 10-20 | | | |
| Soda Ash | 0,1 | 0,1 | | | |
| Polymer - 1 | 4 | 4 | | | |
| Polymer - 2 | 5 | 5 | | | |
| Barite | - | 150 | | | |
| Glycol | % 3 by volume | % 3 by volume | | | |

10.4 Discussion

With respect to the results of rheological properties of fresh water typical weighted and unweighted sepiolite base mud listed in Table I.1 to I.2 for first stage, it is observed that with increasing of temperature apparent viscosity values of mud are first decreased until 300 °F, and then began to increase. For example in weighted mud the reading of 600 rpm decreases from 95 cp in ambient condition to 43 cp in 300 °F temperature and then increase to 48 cp in 400 °F (Rheological properties is measured in 120 °F using heating jacket after aging in different temperatures). This situation is also valid in the case of yield point and gel strength. Increasing in yield point and gel strength is considerable while variation of plastic viscosity above 300 °F is not remarkable. This behavior is one of characteristic properties of sepiolite clays. As mentioned in literature chapter, sepiolite begins to convert to a smectite at 300 °F (149 °C) and this reaction is fully completed at 500 °F (260°C). The new smectites in the fluid have a thin flakey morphology and they increase the viscosity and improve the filtration losses that are shown by N. Guven et al (1988). Therefore, it will be logical that in the case of salt or reactive clays entrance into mud system during drilling operation, a lower amount of sepiolite clays is used. It is obvious that during drilling of salt zones or unstable shale formations, salt and reactive clays enter into mud system and this situation along with high temperatures cause to flocculation of drilling fluids. However, the rheological properties obtained under this condition for this typical sepiolite mud are in appropriated values both in weighted and unweighted mud in fresh water systems without entering of reactive clays into the mud. Amount of filtrations, it can be said that, they have the proper results even in high temperature environments, especially in terms of weighted mud amount of high temperature API water loss is in proper interval between 13 to 18 ml, measured at 300 °F. High temperature API water loss is increased with increasing aging temperature. Amount of filtration at 400 °F has higher values comparing to low temperatures, this will be due to decreasing of polymers efficiency up to 400 °F. However, as seen amount of water loss still has an acceptable value at such high temperature.

The results of second stage experiments in terms of reactive clays entering to the mud system indicate considerable values given in Tables I.3 to I.4. Viscosity of unweighted base mud decreases with increasing temperature so that the reading of 600 rpm decreases from 61 cp to 38 cp at 350 °F temperature. By adding amount of 80 lb/bbl into the mud system, viscosity increases with increasing temperature after 16 hours aging at 300 °F and 350 °F. In terms of hole cleaning adequately, suppose a vertical well is being drilled with this typical sepiolite mud, contaminated with 80 lb/bbl reactive clay circulating at an AV, mud weight = 8.70 lb/gal ,with PV = 19 cp and YP = 23 lb/100 ft² and temperature is 300 °F, as can be seen in Table I.3. From

Figure 10.1, K= 630 cp, and from the CCI equation, Solving the equation for the K value needed to give CCI=1 generates AV= 73 ft/min, that is, with an 73 ft/min annular velocity, amount of 23 lb/100 ft² yield point is a proper value for hole cleaning adequately. Similarly in order to evaluate results of mud aged in 350 °F, as can be seen in Table I.3, mud weight = 8.70 lb/gal, PV = 24 cp and YP = 29 lb/100 ft². From Figure 10.1, K= 950 cp, and from the CCI equation, Solving the equation for the K value needed to give CCI=1 generates AV= 48 ft/min. The AV chosen for the calculation should be the lowest value encountered, for cleaning hole, amount of 29 lb/100-ft² yield point is a proper value with 48 ft/min annular velocity in such drilling mud aged at 350 °F. In addition, amount of water loss is in proper interval at high temperature. With increasing pressure to 500 psi amount of filtration is increased slightly indicated compressibility of mud cake. It can be seen in the results, mud system subjected to entrance of reactive clays has 10 ml loss of water at 300 °F and 13 ml at 350 °F in the fresh water system. Water loss is decreased, when amount of unstable clay formations enter into fresh water mud system and play the solid roles in the mud.

Results of experiments accomplished by amount of 10 and 20 lb/bbl sepiolite (TTB), given in Table I.4 in the appendix I, indicate that filtration and rheological properties in drilling fluid containing 10 lb/bbl sepiolite are more logical than fluid containing 20 lb/bbl amount of sepiolite at high temperature conditions considering to contamination of drilling fluid with reactive clays.

Results of investigating weighted typical sepiolite drilling mud, when encountered to unstable formations, given in Table I.4, indicate that, by adding amount of 80 lb/bbl into the mud system, viscosity increase with increasing temperature. It can be seen reading of 600 rpm increase from 68 in 300 °F to 73 in 350 °F aging temperature. Similar to unweighted system, in order to evaluate this mud with respect to hole cleaning and cutting transportation, for weighted mud sample aged in 350 °F, as seen from Table I.4, mud weight = 10.90 lb/gal , PV = 24 cp and YP = 25 lb/100 ft². From Figure 10.1, K= 650 cp, and from the CCI equation, Solving the equation for the K value needed to give CCI=1 generates AV= 56 ft/min. Amount of 25 lb/100 ft² yield point is a proper value with 57 ft/min annular velocity in such weighted drilling mud aged at 350 °F. This evaluation can be done for weighted mud aged in 300 °F for 16 hours. Obtained results of MBT test confirm that amount of reactive clays in drilling

mud system is not progressive and it is a good indicator to denote that, mud system is in proper condition after entrance of 80 lb/bbl clay into the system.



Figure 10.2: Rheological properties of unweighted typical sepiolite (TTB) base mud aged at high temperature for 16 hours.



Figur 10.3: Rheological properties of weighted typical sepiolite (TTB) base mud aged at high temperature for 16 hours.



Figure 10.4: Effect of reactive clay entering into unweighted TTB clay base mud aged at high temperature for 16 hours in fresh water system.



Figure 10.5: Effect of reactive clay entering into weighted TTB clay base mud aged at high temperature for 16 hours in fresh water system.

Consequently sepiolite base drilling mud represented in this section is stable in fresh water system at high temperature; In addition shows appropriate properties when subjected to entrance of reactive clays during drilling of unstable formations.

10.5 Mud properties in saline environments along with entrance of reactive clay

In previous section, it was shown that in terms of entrance of reactive clays into mud during drilling unstable shale formations, the lower amount of sepiolite used in mud preparing, the better rheological and filtration properties. In this section, this situation will be more obvious than previous section. For this purpose experiments are conducted with three amount of sepiolite (TTB clay). Typical compositions of weighted sepiolite (TTB clay) base mud in salinity systems are given in Table 10.3.

| Table 10.3: Compositions of weighted typical sepiolite mud in terms of reactive clay |
|--|
| enter into mud system in high salinity condition. |

| Substance | Quantity (lbm/bbl) |
|-------------|--------------------|
| Substance | Weighted |
| Sepiolite | 10-15-20 |
| Soda Ash | 0,1 |
| Polymer - 1 | 5 |
| Polymer - 2 | 7 |
| Barite | 150 |
| Glycol | % 3 by volume |
| NaCl | 120 |

As be mentioned before after preparing typical mud a certain amount of sodium chloride is added to mud along with OCMA to simulate entrance of the salt into the mud from formation fluid having high salinity or from salt zones. Amount of 120 lb/bbl is added to the mud system in order to have a fully saturated system. The purity of sodium chloride used in experiment is lower than 100% purity. Adding salt into composition of mud can be considered as one of drilling mud components in order to weighting mud or as mentioned, simulate entrance of the salt to the system. Rheological and filtration properties are determined in high temperature and high salinity conditions and the results are given between Table I.5 to I.7 in the appendix I

and shown graphically between Figure 10.6 to 10.7. Table 10.4 and 10.5 are represented in this chapter to discus about results of filtration and rheological properties of weighted sepiolite base mud containing three amount of TTB clay formulated in Table 10.3. Moreover effect of reactive clay entering into weighted mud system containing three concentration of TTB clay shown in Figure 10.8.

| Composition | | Weighted M | ud (10 lb/bbl s | sepiolite con | ncentration) |
|--------------------------|---|------------|-----------------------|---------------|--------------|
| NaCl, lb/bbl | | | 120 | 1 | 20 |
| OCMA, ppb | | | | 8 | 30 |
| Aging temperature, | °F | 80 | 80 | 3 | 50 |
| Aging time, hrs | | 20 min | 20 min |] | 16 |
| MBT, lb/bbl | | | | 11,00 | 11,00 |
| Mud density, lb/gal | | | | 12,70 | 12,70 |
| Mud PH(with pH m | eter) | 8,00 | 7,20 | 7,20 | 7,20 |
| Filtration PH (with | pH meter) | | | 8,00 | 8,00 |
| Temperature of PH | measurement, °F | 80 | 80 | 80 80 | |
| | 600 | 95 | 48 | 55 | 62 |
| ing | 300 | 61 | 29 | 34 | 41 |
| ead | 200 | 46 | 21 | 26 | 33 |
| al R | 100 | 29 | 13 | 17 | 24 |
| Di | 6 | 4 | 3 | 4 | 10 |
| | 3 | 3 | 2 | 3 | 9 |
| Temperature of rhe | ology measurement, °F | 80 | 80 | 80 | 120 |
| PV | | 34 | 19 | 21 21 | |
| YP | | 27 | 10 | 13 20 | |
| Gel strength, 10s./ 1 | min./ 10min. | 3/4/5 | /5 2/2/5 3/4/9 5/7/15 | | 5/7/15 |
| Temperature of filtr | Temperature of filtration measurement, °F | | | 300 | |
| API water loss, cc (7 | .5/30min)@100 psi | | | 8/ | /17 |
| cake thickness, mm | | | | | 5 |

Table 10.4: Effect of reactive clays entering into weighted mud containing 10 ppb sepiolite along with high salinity.

10.6 Discussion

As seen from results obtained for three concentration of sepiolite in high salinity environments and high temperature condition, the typical sepiolite mud with concentration of 10 lb/bbl sepiolite clay indicate the best performance compare to other muds having concentration of 15 lb/bbl and 20 lb/bbl sepiolite, on account of entrance of reactive clay into the mud system along with high salinity conditions. On the other words, the concentration of sepiolite as a mud clay is fundamental factor when this fluid system encountered with unstable formations and entrance of clays at high salinity condition. This can be due to stability of sepiolite at high temperature and high salt concentration. When a large amount of reactive clay enter into mud system (especially in clay base muds), affects on the rheological properties of drilling mud, yield point and gel strength are increased. This condition along with high temperature and high salt concentration cause to flocculation of mud, so initially concentration of sepiolite must be lowered to prevent from increasing of yield point and gel strength values. On the other hand, as be mentioned before, sepiolite begins to convert a smectite above 300 °F and result in higher yield point and gel strength values. This condition in conjunction with salinity and entrance of large amount of clays lead to increasing in rheological properties expeditiously. Therefore using of low amount of sepiolite during drilling of shale and unstable formation is a reasonable decision.

Table 10.5: Effect of reactive clays entering into weighted mud containing 20 ppb sepiolite along with high salinity.

| Composition | | Weighted Mud (20 lb/bbl sepiolite concentration) | | | | |
|---|-----------------------|---|--------|--------|----------|--|
| NaCl, lb/bbl | aCl, lb/bbl 120 120 | | 120 | | | |
| OCMA, ppb | | | | 50 | 50 | |
| Aging temperature, | ° F | 80 | 80 | 350 | 350 | |
| Aging time, hrs | | 20 min | 20 min | 16 | 16 | |
| MBT, lb/bbl | | | | 11,50 | 11,50 | |
| Mud density, lb/gal | | 10,90 | | 12,60 | 12,60 | |
| Mud PH(with pH m | eter) | 8,00 | 7,20 | 7,20 | 7,20 | |
| Filtration PH (with pH meter) 8,0 | | 8,00 | 8,00 | | | |
| Temperature of PH | measurement, °F | 80 | 80 | 80 | 80 | |
| | 600 | 126 | 65 | 73 | 83 | |
| ng | 300 | 84 | 43 | 50 | 58 | |
| Dial Readi | 200 | 66 | 34 | 38 | 48 | |
| | 100 | 44 | 24 | 22 | 36 | |
| | 6 | 10 | 11 | 9 | 19 | |
| | 3 | 8 | 10 | 8 | 18 | |
| Temperature of rhe | ology measurement, °F | 80 | 80 | 80 | 120 | |
| PV | | 42 | 22 | 23 | 25 | |
| үр | | 42 | 21 | 27 | 33 | |
| Gel strength, 10s./ 1min./ 10min. | | 11/15/25 | 6/8/15 | 6/8/18 | 20/25/38 | |
| Temperature of filtration measurement, °F | | | | 300 | | |
| API water loss, cc (7.5/30min)@100 psi | | | | 16/33 | | |
| cake thickness, mm | | | | | 7 | |

It is obvious that by addition of salt into the mud system rheological properties of drilling fluid are affected; however, it can be seen rheological properties in each three system with different concentration of sepiolite result in a proper viscosity, yield point and gel strength even at high concentration of salt (fully saturated). For example the reading of 600 rpm in the mud system having 10 lb/bbl of sepiolite decrease from 95 to 48 after entrance large amount of salt, the value of yield point and gel strength is still in acceptable values. It will be shown that keeping amount of drilling mud yield point and gel strength in acceptable minimum values is critical factor especially in clay base drilling muds. Entrance of reactive clay into the mud system along with high temperature and high salinity condition increase the rheological properties of muds (as seen from results), however, increasing of rheological values are not in progressive rate in this system. Increasing of sepiolite concentration has an improper effect on rheological properties especially on yield point and gel strength. For example, amount of yield point in the mud system containing 20 ppb of sepiolite at 120 °F is 33 lb/100 ft² while in the mud system containing 10 lb/bbl of sepiolite is 20 lb/100 ft² at 120 °F. On the other hand amount of gel strength is in fragile values in drilling fluid containing 10 lb/bbl of sepiolite but it can be seen a progressive value in other two systems at 120 °F (temperature of rheology measurement). Two of the most important rheology parameters are the 3 rpm reading which defines the lifting capacity at very low flow rates, and the 10 sec gel strength which defines the suspension capability of the system. It is important that these two parameters are within the specifications to ensure that the hole cleaning is optimum and that build-up of cuttings is reduced to a minimum. This condition is satisfied in the mud systems containing 10 lb/bbl rather than other two systems containing 15 lb/bbl and 20 lb/bbl respectively. If it is not possible to maintain the 3 RPM reading and 10 sec gel strengths without producing high 10 min gel strengths, the drilling fluid system has incorporated too much ultrafine drill solids and a part of the system (20 - 40 %) will have to be replaced with fresh drilling fluid. To avoid any new drilling fluid being dumped or ending up in the reserve system, dilution should be performed over one circulation. In order to evaluate this mud system with respect to hole cleaning and cutting transportation, for weighted mud sample aged in 350 °F and consider to the rheological properties in 120 °F, as seen from Table I.4, mud weight = 12.70 lb/gal, PV = 21 cp and YP = $20 \text{ lb}/100 \text{ ft}^2$. From Figure 10.1, K = 550 cp, and from the CCI equation, Solving the equation for the K

value needed to give CCI=1 generates AV= 57 ft/min. Amount of 20 lb/100 ft² yield point is a proper value with 57 ft/min annular velocity in such weighted drilling mud in high salinity environment that aged at 350 °F.

Furthermore, the results obtained from mud systems on account of filtration indicate that, the lower sepiolite concentration the lower amount of water loss so that amount of filtration is 17 ml in the system having 10 ppb sepiolite that is lower than filtration in fluid containing in 15 ppb sepiolite (19 ml) and fluid system having 20 ppb sepiolite (33 ml) at 300 °F. MBT reading is in favorable value in spite of large amount of reactive clay enter into the mud system that is indicate the mud system is in appropriate condition.



Figure 10.6: Effect of high salinity on rheological properties of weighted mud containing three concentration of sepiolite in ambient conditions.


Figure 10.7: Effect of reactive clays entering into sepiolite base weighted mud aged in 350 °F along with high salinity.



Figure 10.8: changes in gel strength in terms of reactive clay entering into sepiolite base weighted mud aged in 350 °F along with high salinity.

11. COST ANALYSIS OF RECOMMENDED SEPIOLITE BASE MUDS

One of our propose in this study is preparing a type of drilling mud not only that can provide the API standards and proper rheological and water loss properties but also it is very important that the cost of recommended mud be economic in comparison to the other muds that is used under same conditions.

In this section total cost of recommended sepiolite base mud are analyzed and given below. Total cost of fresh water sepiolite base mud is given in Table 11.1. Moreover, the cost of typical sepiolite drilling mud used in high salinity environments is given in Table 11.2. Use of glycol increased the cost of mud. But use of proper glycol improves the performance of drilling mud encountering to shale formations and entering active clays into mud. However, as seen in tables, it is clear that total cost of typical sepiolite base mud that is used in high temperature and high pressure conditions is lower than some drilling fluids used in the same applications, such as synthetic drilling mud and polymer base drilling fluids that have a expensive cost about 100\$ that increases with harsh drilling conditions. Moreover, our typical recommended sepiolite mud has almost the same cost with KCl/PAC polymer base mud that is an inhibitive drilling mud used only until 250 ° F.

| Additive material | Unit | Unit cost * (\$) | Concentration (ppb) | Total Cost (\$) | Total Cost (\$) | | |
|--|---------------|------------------------|------------------------|-----------------------|-----------------------|--|--|
| Soda ash, 50 kg/sack | 110 lb/sack | 10,00 | 0,1 | 0,0090 | 0,0090 | | |
| Sepiolite, 50 kg/sack | 110 lb/sack | 11,78 | 20 | 2,1418 | 2,1418 | | |
| Polymer A, 5 gal/pail | 51,77 lb/pail | 73,50 | 4 | 5,6789 | 5,6789 | | |
| Polymer B, 25 lb/sack | 25 lb/sack | 75,00 | 5 | 15 | 15 | | |
| Glycol, 55 gal/drum | | 596,70 | 3% by Vol | | 13,67 | | |
| Total cost per barrel of sep | 22,8 | 36,5 | | | | | |
| Unit cost*: Extraced from TPAO research center | | | | | | | |

Table 11.1: Total cost of fresh water sepiolite base mud at 350 °F

| Additive material | Unit | Unit cost* (\$) | Concentration ppb | Total Cost (\$) | Total Cost (\$) |
|----------------------------|-----------------|-----------------------|----------------------|--------------------|--------------------|
| soda ash, 50 kg/sack | 110 lb/sack | 10,00 | 0,1 | 0,0090 | 0,0090 |
| sepiolite, 50 kg/sack | 110 lb/sack | 11,78 | 20 | 2,1418 | 2,1418 |
| Polymer A, 5 gal/pail | 51,77 lb/pail | 73,50 | 5 | 7,0987 | 7,0987 |
| Polymer B, 25 lb/sack | 25 lb/sack | 75,00 | 7 | 21 | 21 |
| Glycol, 55 gal/drum | | 596,70 | 3% | | 13,67 |
| Total cost per barrel of s | 30,2 | 43,9 | | | |
| Unit | cost*: Extraced | from TPA | O research center | | |

Table 11.2: Cost of typical sepiolite drilling mud used in full saturated and 350 °F conditions.

12. CONCLUSIONS AND RECOMMENDATIONS

In this study, rheological and filtration properties of drilling fluids prepared with sepiolite clays are experimentally investigated. Experiments are conducted with and without some special additives for different temperatures and pressures. Moreover, the rheological and filtration properties of sepiolite base muds are evaluated and controlled in terms of reactive clays entering into mud system during drilling of unstable formations at high temperature in fresh water and high saline systems. In consequence of this study the following results are experimentally obtained:

- Five different clays used in this study were obtained from Sivrihisar-Eskişehir region. The names of these clays are coded as Kurtşeyh, TTB, S, and YD-S and YD-K, respectively.
- Except of YD-S clay, the grinding of other clays can be easily carried out. The process of crushing and sieving of YD-S clay was very difficult because the particles of this clay tend to stick to sieves, for this reason the process of grinding of this clay is long and laborious. All of clays were sieved from 74 micron screen. According to the results of particle size analysis of clay the average value of grain size is 24 microns (with probability of 90%).
- Since the relationship between shear stress-shear rates is not linear, the rheological properties of muds show a non- Newtonian behavior. All of fluids have pseudo plastic structure because apparent viscosity of these fluids decreases with increasing shear rates.
- In the case of rheological properties, four clays (YD-S, TTB, Kurtşeyh and YD-K respectively) can meet the criteria specified in API standards. Only S clay 600 rpm reading has lower value.
- As specified in API standards, rheological values measured after mixing mud for 20 min, has significantly changed depending on aging period, for this reason the rheological values are controlled after 16 hours, 24 hours and 48 hours aging. In all of the tests shear stress values increase with increasing

aging time. This increment declines with increasing time and effect of aging period after 24 hours is negligible.

- S Clay did not comply with criteria of the API standards even though after 48 hours aging.
- In terms of rheological properties, the highest shear stress values were obtained from the mud prepared with fresh water. Shear stress values obtained from full-saturated and semi-salt saturated mud's are slightly lower, however difference between these two muds is insignificant. Changes in shear stress above certain salinity are becoming irrelevant. This value of salinity can be investigated in another study.
- Examined sepiolite muds give relatively high gel strength values. The ratio of yield point to plastic viscosity of the mud (YP / PV) is typically about 3.
- API water loss of mud's prepared with five sepiolite samples for all salinities is very high and are not suitable for drilling applications. It was observed that thickness of mud cakes are in range of 4-9 mm and very thick due to high water losses. Mud cakes obtained from samples are generally hard and fragile. This is an unfavorable condition for mud. YD-K and YD-S clays have the best results in terms of water loss in all of the salinities. The lowest value of water loss is 80 ml/30 minutes. As the same rheological properties, mud's prepared with S clay, have the worst performance. Water loss values show a decline in consequence of increasing aging period, however, this decrement is not important as much as rheological changes.
- It is observed that API water loss changes are in inversely proportional with salinity, that is, API water loss decrease with increasing salinity. For each of mud sample, the lowest water loss was obtained in full-saturated mud. Water loss values of fresh water muds and semi-salt saturated muds are close to each other and the difference between these is insignificant. After a certain salinity value changes in water loss are becoming irrelevant. This value of salinity can be examined in another study. Examined all sepiolite muds have amount of spurt water loss due to high water loss values.
- It is observed that the rheological results of semi-salt saturated sepiolite base muds are close to full-salt saturated sepiolite muds. Moreover, fresh water muds have close results to semi-salt saturated sepiolite muds in terms of

filtration properties. These are interesting observations and the reason of these can be investigated.

- When the rheological properties were evaluated as well as water loss properties, it is observed that the best performance was obtained from the YD-S and TTB clays. Therefore, these two clays are used in the next phase of experiments in terms of dynamic filtration test and stability of mud system when a large amount of reactive clays enter into system.
- Amount of water loss in sepiolite base drilling fluid prepared with TTB and YD-S clays can be controlled by using different concentration of additives at high temperature and high pressure conditions. It is indicated that sepiolite base drilling mud prepared for this study have an acceptable water loss at 350 °F and 500 psi pressure.
- It can be mentioned that filtration properties of commercial drilling fluids used at high temperature (above 300 °F) and especially with high salinity conditions are not presented in both literature and drilling fluid service companies documents. This can be important point of this study's results.
- The large part of clays used in this study comply with API standards in terms of rheological properties. This confirms that sepiolite reserves of country could be better evaluated and in this case, other studies are encouraging. At the same time, the results of this study can provide significant economic opportunities for Turkey as a country having the world's largest reserves of sepiolite.
- Dynamic filtration properties of chosen fresh water based sepiolite muds are observed under different conditions by the use of a computerized dynamic filtration system. Since there is no proportional relationship between static and dynamic filtration, the examinations of dynamic filtration properties are particularly important. Drilling muds prepared with sepiolite having average grain size 25<d<75 provides better sealing and proper results in terms of dynamic filtration rate and cake deposition index. Dynamic filtration rates are affected from the temperature variations. DFR increases with increasing temperature. Cake deposition index are similarly affected by the temperature variations. However, the temperature augmentations slightly increase and CDI. As the pressure differential increases, DFR proportionally increases and CDI</p>

decreases, that indicate a compressible filter cake. On the other hand, CDI decreases at high pressure differential that indicates a compressible filter cake.

- Due to lack of filter core used in dynamic filtration test, experiments associated with sepiolite muds in saline conditions were not conducted; this can be a good topic for another study. In addition, these experiments can be done with other filter core having different pore sizes in order to measure filtration properties of drilling fluids in different formations encountered during drilling operation.
- Sepiolite base drilling mud represented in this study is stable in fresh water and high salinity systems at high temperature. In addition, it shows appropriate properties when subjected to entrance of large reactive clays during drilling of unstable formations at high temperature and high salinity condition. It has been indicated that using of lower amount of sepiolite in drilling mud system is recommended on account of entering reactive clays into the mud system at high temperature and high salinity conditions.
- Total cost of typical sepiolite base mud (recommended in this study) that is used in high temperature and high pressure conditions is lower than some drilling fluids used in the same applications, such as synthetic drilling mud and polymer base drilling fluids that have a high cost about 100\$ that increases with harsh drilling conditions. Moreover, our typical recommended sepiolite mud has almost the same cost with KCl/Pac polymer base mud that is an inhibitive drilling mud used only until 250°F.

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APPENDICES

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APPENDIX – A: Results of rheological and filtration properties of sepiolite base mud at ambeint condition-fully saturated

In this section, relationships between shear rate shear stress and filtration properties of sepiolite base drilling fluid prepared in full saturated system are tabluated. The salt content of the mud is 400 g of NaCl per one liter of dilluted water. All of the tests are conducted in room conditoin.

Table A.1: Rheological properties of kurtşeyh clay – full saturated

Sample name : kurtşeyh clay – full saturated **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------------|
| Sample mud temperature: °c | 31°c | 27∘c | 29°c | 29°c |
| Density , ppg | 10,2 | 10,2 | 10,2 | 10,2 |
| θ 600 | 25 | 30 | 33 | 36 |
| θ 300 | 20 | 24 | 26,5 | 29 |
| θ 200 | 17,5 | 20,5 | 23 | 26 |
| θ 100 | 15 | 17 | 19 | 22 |
| θ 6 | 11 | 11,5 | 13 | 14,5 |
| 03 | 11 | 11,5 | 13 | 14,5 |
| Apparent viscosity (AV) , cp | 12,5 | 15 | 16,5 | 18 |
| Plastic viscosity (PV) , cp | 5 | 6 | 6,5 | 7 |
| Yeild point (YP), lb/100 ft ² | 15 | 18 | 20 | 22 |
| 10 sec gel strength, lb/100 ft ² | 12 | 10 | 11 | 11 |
| 1 min gel strength,lb/100 ft ² | 17 | 14 | 15 | 15 |
| 10 min gel strength, lb/100 ft ² | 29 | 22 | 24 | 27 |
| 30 min gel strength, lb/100 ft ² | 34 | 29 | 32 | 31 |
| Cake thickness mm | 4,9 | 5,05 | 5,05 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| Tuzluluk, mg/l | Full saturated | Full saturated | Full saturated | Full saturated |
| PH (mud) | 7,4 | 7,4 | 7,4 | |
| PH (filtration) | 7,9 | 8,1 | 8,2 | |

Table A.1: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 27 | 32 | 35 | 38 | 1021,8 |
| 21 | 26 | 28 | 31 | 510,9 |
| 19 | 22 | 25 | 28 | 340,6 |
| 16 | 18 | 20 | 23 | 170,3 |
| 12 | 12 | 14 | 15 | 10,218 |
| 12 | 12 | 14 | 15 | 5,109 |

 Table A.2: Water loss of kurtşeyh clay mud - full saturated

| | | API wa | | | |
|------------|--------|----------|----------|----------|-----------------|
| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
| 1 | 22 | 22 | 22 | | 1,000 |
| 2 | 30 | 30 | 28 | | 1,414 |
| 2,5 | 32 | 32 | 31 | | 1,581 |
| 3 | 36 | 36 | 34 | | 1,732 |
| 4 | 42 | 41 | 39 | | 2,000 |
| 5 | 46 | 46 | 44 | | 2,236 |
| 7,5 | 56 | 56 | 52 | | 2,739 |
| 10 | 64 | 63 | 60 | | 3,162 |
| 20 | 88 | 86 | 83 | | 4,472 |
| 30 | 108 | 106 | 100 | | 5,477 |

Table A.3: Rheological properties of TTB clay - full saturated

Sample name : TTB clay – full saturated **Temperature of room:** 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------|
| Sample mud temperature: oc | 29 °c | 27 ∘c | 26 °c | 25 ∘c |
| Density , ppg | 10,2 | 10,2 | 10,2 | 10,2 |
| θ 600 | 21,5 | 26 | 29 | 32 |
| θ 300 | 16 | 21 | 23 | 26 |
| θ 200 | 14 | 18,5 | 20 | 22,5 |
| θ 100 | 12 | 15 | 17 | 19 |
| θ 6 | 9,5 | 11 | 12 | 13 |
| 03 | 9,5 | 11 | 12 | 13 |
| Apparent viscosity (AV) , cp | 10,75 | 13 | 14,5 | 16 |
| Plastic viscosity (PV) , cp | 5,5 | 5 | 6 | 6 |
| Yeild point (YP), lb/100 ft ² | 10,5 | 16 | 17 | 20 |
| 10 sec gel strength, lb/100 ft ² | 12 | 10 | 11,5 | 12 |
| 1 min gel strength, lb/100 ft ² | 14 | 14 | 13 | 15 |
| 10 min gel strength, lb/100 ft ² | 22 | 22 | 22 | 26 |
| 30 min gel strength,lb/100 ft ² | 24 | 28 | 29 | 33 |
| Cake thickness mm | 5,9 | 5,4 | 5 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| PH (mud) | 7,4 | 7,2 | 7,4 | |
| PH (filtration) | 7,8 | 7,6 | 7,6 | |

Table A.3: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 23 | 28 | 31 | 34 | 1021,8 |
| 17 | 22 | 25 | 28 | 510,9 |
| 15 | 20 | 21 | 24 | 340,6 |
| 13 | 16 | 18 | 20 | 170,3 |
| 10 | 12 | 13 | 14 | 10,218 |
| 10 | 12 | 13 | 14 | 5,109 |

 Table A.4: Water loss of TTB clay - full saturated

| | | API wa | | | |
|------------|--------|----------|----------|----------|-----------------|
| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
| 1 | 18 | 18 | 17 | | 1,000 |
| 2 | 26 | 26 | 24 | | 1,414 |
| 2,5 | 29 | 29 | 27 | | 1,581 |
| 3 | 31 | 31 | 29 | | 1,732 |
| 4 | 36 | 34 | 32 | | 2,000 |
| 5 | 38 | 39 | 36 | | 2,236 |
| 7,5 | 48 | 47 | 44 | | 2,739 |
| 10 | 56 | 55 | 51 | | 3,162 |
| 20 | 78 | 78 | 71 | | 4,472 |
| 30 | 98 | 92 | 86 | | 5,477 |

Table A.5: Rheological properties of S clay - full saturated

Sample name : S clay – full saturated **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------|
| Sample mud temperature: •c | 28 °c | 29 °c | 29 ∘c | |
| Density , ppg | 10,2 | 10,2 | 10,2 | |
| θ 600 | 14 | 17 | 19 | |
| θ 300 | 10 | 13 | 14 | |
| θ 200 | 8,5 | 11 | 12 | |
| θ 100 | 7 | 9 | 10 | |
| θ 6 | 4,5 | 5 | 6 | |
| θ3 | 4,5 | 5 | 6 | |
| Apparent viscosity (AV) , cp | 7 | 8,5 | 9,5 | |
| Plastic viscosity (PV) , cp | 4 | 4 | 5 | |
| Yeild point (YP), lb/100 ft ² | 6 | 9 | 9 | |
| 10 sec gel strength, lb/100 ft ² | 6 | 6 | 7 | |
| 1 min gel strength, lb/100 ft ² | 7 | 7 | 8 | |
| 10 min gel strength, lb/100 ft ² | 12 | 11 | 13 | |
| 30 min gel strength,lb/100 ft ² | 15 | 14 | 16 | |
| Cake thickness mm | 4,3 | 4,2 | 4,2 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| Tuzluluk, mg/L | Full saturated | Full saturated | Full saturated | |
| PH (mud) | 7,6 | 7,4 | 7,3 | |
| PH (filtration) | 8,2 | 8,2 | 8,1 | |

Table A.5: Continue

| | Shear stress | | | | | | |
|-------------------|--------------|-------------|-------------|-----------|--|--|--|
| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ | | | |
| 15 | 18 | 20 | | 1021,8 | | | |
| 11 | 14 | 15 | | 510,9 | | | |
| 9 | 12 | 13 | | 340,6 | | | |
| 7 | 10 | 11 | | 170,3 | | | |
| 5 | 5 | 6 | | 10,218 | | | |
| 5 | 5 | 6 | | 5,109 | | | |

Table A.6: Water loss of S clay mud - full saturated

| | | API wa | | | |
|------------|--------|----------|----------|----------|----------|
| time (min) | 20 min | 16 hours | 24 hours | 48 hours | √ (time) |
| 1 | 20 | 20 | 24 | | 1,000 |
| 2 | 30 | 28 | 30 | | 1,414 |
| 2,5 | 34 | 31 | 35 | | 1,581 |
| 3 | 38 | 34 | 38 | | 1,732 |
| 4 | 44 | 40 | 43 | | 2,000 |
| 5 | 50 | 44 | 48 | | 2,236 |
| 7,5 | 60 | 56 | 58 | | 2,739 |
| 10 | 69 | 64 | 66 | | 3,162 |
| 20 | 99 | 90 | 92 | | 4,472 |
| 30 | 120 | 112 | 112 | | 5,477 |

Table A.7: Rheological properties of YD-K clay - full saturated

Sample name : YD-K clay – full saturated **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------|
| Sample mud temperature: •c | 28°c | 27 °c | 26 °c | 27 °c |
| Density , ppg | 10,15 | 10,15 | 10,15 | 10,15 |
| θ 600 | 22 | 26 | 28 | 31 |
| θ 300 | 17 | 20 | 22 | 24 |
| θ 200 | 14,5 | 17 | 19 | 21 |
| θ 100 | 12 | 14 | 15 | 17 |
| θ 6 | 9 | 10 | 10 | 12 |
| θ 3 | 9 | 10 | 10 | 12 |
| Apparent viscosity (AV) , cp | 11 | 13 | 14 | 15,5 |
| Plastic viscosity (PV) , cp | 5 | 6 | 6 | 7 |
| Yeild point (YP), lb/100 ft ² | 12 | 14 | 16 | 17 |
| 10 sec gel strength, lb/100 ft ² | 9 | 10 | 10 | 10 |
| 1 min gel strength, lb/100 ft ² | 14 | 13 | 12 | 13 |
| 10 min gel strength, lb/100 ft ² | 19 | 19 | 19 | 20 |
| 30 min gel strength,lb/100 ft ² | 20 | 21 | 22 | 23 |
| Cake thickness mm | 4,3 | 4,8 | 4,6 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| Tuzluluk, mg/L | Full saturated | Full saturated | Full saturated | |
| PH (mud) | 7,1 | 7,1 | 7,1 | |
| PH (filtration) | 8,1 | 8,1 | 8 | |

Table A.7: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 23 | 28 | 30 | 33 | 1021,8 |
| 18 | 21 | 23 | 26 | 510,9 |
| 15 | 18 | 20 | 22 | 340,6 |
| 13 | 15 | 16 | 18 | 170,3 |
| 10 | 11 | 11 | 13 | 10,218 |
| 10 | 11 | 11 | 13 | 5,109 |

Table A.8: Water loss of YD-K clay mud - full saturated

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
|------------|--------|----------|----------|----------|-----------------|
| 1 | 16 | 20 | 18 | | 1,000 |
| 2 | 24 | 27 | 25 | | 1,414 |
| 2,5 | 26 | 30 | 28 | | 1,581 |
| 3 | 28 | 32 | 30 | | 1,732 |
| 4 | 34 | 37 | 34 | | 2,000 |
| 5 | 38 | 40 | 38 | | 2,236 |
| 7,5 | 46 | 48 | 46 | | 2,739 |
| 10 | 52 | 55 | 53 | | 3,162 |
| 20 | 72 | 76 | 73 | | 4,472 |
| 30 | 90 | 92 | 89 | | 5,477 |

Table A.9: Rheological properties of YD-S clay - full saturated

Sample name : YD-S clay – full saturated Temperature of room: 23.3 °c Composition : 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------------|
| Sample mud temperature: ∘c | 29∘c | 29∘c | 29°c | 29°c |
| Density , ppg | 10,1 | 10,1 | 10,1 | 10,1 |
| θ 600 | 29 | 36 | 41,5 | 45 |
| θ 300 | 23 | 29 | 34 | 36 |
| θ 200 | 20 | 25 | 30 | 32 |
| θ 100 | 17 | 21 | 25 | 27 |
| θ 6 | 14 | 16 | 18 | 19 |
| θ 3 | 14 | 16 | 18 | 19 |
| Apparent viscosity (AV) , cp | 14,5 | 18 | 20,75 | 22,5 |
| Plastic viscosity (PV) , cp | 6 | 7 | 7,5 | 9 |
| Yeild point (YP), lb/100 ft ² | 17 | 22 | 26,5 | 27 |
| 10 sec gel strength, lb/100 ft ² | 17 | 15 | 16 | 15 |
| 1 min gel strength,lb/100 ft ² | 22 | 19 | 19 | 22 |
| 10 min gel strength, lb/100 ft ² | 33 | 33 | 32 | 31 |
| 30 min gel strength, lb/100 ft ² | 35 | 34 | 34 | 42 |
| Cake thickness mm | 4,9 | 4,65 | | 5,1 |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | Hard and rigid |
| Tuzluluk | Full saturated | Full saturated | Full saturated | Full saturated |
| PH (mud) | 7,5 | 7,4 | | 7,4 |
| PH (filtration) | 8,1 | 7,6 | | 7,4 |

Table A.9: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 31 | 38 | 44 | 48 | 1021,8 |
| 25 | 31 | 36 | 38 | 510,9 |
| 21 | 27 | 32 | 34 | 340,6 |
| 18 | 22 | 27 | 29 | 170,3 |
| 15 | 17 | 19 | 20 | 10,218 |
| 15 | 17 | 19 | 20 | 5,109 |

Table A.10: Water loss of YD-S clay mud - full saturated

| API water loss | | | | | |
|----------------|--------|----------|----------|----------|------------------------|
| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{\text{(time)}}$ |
| 1 | 20 | 15 | | 16 | 1,000 |
| 2 | 26 | 20 | | 22 | 1,414 |
| 2,5 | 28 | 22 | | 24 | 1,581 |
| 3 | 30 | 24 | | 26 | 1,732 |
| 4 | 34 | 28 | | 30 | 2,000 |
| 5 | 38 | 30 | | 32 | 2,236 |
| 7,5 | 46 | 38 | | 41 | 2,739 |
| 10 | 54 | 45 | | 46 | 3,162 |
| 20 | 74 | 64 | | 66 | 4,472 |
| 30 | 90 | 80 | | 80 | 5,477 |

APPENDIX – B: Results of rheological and filtration properties sepiolite base mud at ambeint conditions-semi salt saturated

In this section, relationships between shear rate shear stress and filtration properties of sepiolite base drilling fluid prepared in semi salt saturated system are shown. Ratio of salinity is 200 g/l dilluted water. All of the tests are conducted in room conditoin.

Table B.1: Rheological properties of kurtşeyh clay – semi saturated

Sample name : kurtşeyh clay – semi salt saturated **Temperature of room:** 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------------|
| Sample mud temperature: °c | 29∘c | 25∘c | 27∘c | |
| Density , ppg | 9,5 | 9,55 | 9,55 | 9,55 |
| θ 600 | 27 | 31 | 33,5 | 35 |
| θ 300 | 21,5 | 25 | 27 | 29 |
| θ 200 | 19 | 22 | 24 | 25,5 |
| θ 100 | 16 | 19 | 20 | 21 |
| θ 6 | 12 | 13 | 14 | 14 |
| 03 | 12 | 13 | 14 | 14 |
| Apparent viscosity (AV) , cp | 13,5 | 15,5 | 16,75 | 17,5 |
| Plastic viscosity (PV) , cp | 5,5 | 6 | 6,5 | 6 |
| Yeild point (YP), lb/100 ft ² | 16 | 19 | 20,5 | 23 |
| 10 sec gel strength, lb/100 ft ² | 12 | 12 | 12 | 12 |
| 1 min gel strength, lb/100 ft ² | 18 | 16 | 16 | 16 |
| 10 min gel strength, lb/100 ft ² | 27 | 22 | 24 | 24 |
| 30 min gel strength, lb/100 ft ² | 32 | 25 | 28 | 27 |
| Cake thickness mm | 6,8 | 7,2 | 6,6 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| Tuzluluk, mg/l | semi saturated | semi saturated | semi saturated | semi saturated |
| PH (mud) | 8,1 | 8,1 | 8,2 | |
| PH (filtration) | 8,6 | 8,2 | 8,2 | |

Table B.1: Continue

| |] | | | |
|-------------------|-------------|-------------|-------------|-----------|
| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
| 29 | 33 | 36 | 37 | 1021,8 |
| 23 | 27 | 29 | 31 | 510,9 |
| 20 | 23 | 26 | 27 | 340,6 |
| 17 | 20 | 21 | 22 | 170,3 |
| 13 | 14 | 15 | 15 | 10,218 |
| 13 | 14 | 15 | 15 | 5,109 |

Table B.2: Water loss of kurtşeyh clay mud - semi saturated

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
|------------|--------|----------|----------|----------|-----------------|
| 1 | 24 | 24 | 22 | | 1,000 |
| 2 | 34 | 34 | 33 | | 1,414 |
| 2,5 | 38 | 38 | 37 | | 1,581 |
| 3 | 40 | 40 | 40 | | 1,732 |
| 4 | 46 | 46 | 46 | | 2,000 |
| 5 | 52 | 51 | 51 | | 2,236 |
| 7,5 | 64 | 62 | 62 | | 2,739 |
| 10 | 74 | 72 | 72 | | 3,162 |
| 20 | 102 | 100 | 98 | | 4,472 |
| 30 | 124 | 122 | 120 | | 5,477 |

Table B.3: Rheological properties of TTB clay - semi saturated

Sample name : TTB clay – semi salt saturated **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------|
| Sample mud temperature: •c | 29 °c | 28 °c | 26 ∘c | 25 ∘c |
| Density , ppg | 9,6 | 9,6 | 9,6 | 9,6 |
| θ 600 | 26 | 33 | 35 | 36,5 |
| 0 300 | 21 | 27 | 29 | 30 |
| θ 200 | 19 | 24 | 25,5 | 26,5 |
| θ 100 | 16,5 | 20 | 21,5 | 22,5 |
| θ 6 | 13,5 | 14 | 15,5 | 16 |
| 03 | 13,5 | 14 | 15,5 | 16 |
| Apparent viscosity (AV) , cp | 13 | 16,5 | 17,5 | 18,25 |
| Plastic viscosity (PV) , cp | 5 | 6 | 6 | 6,5 |
| Yeild point (YP), lb/100 ft ² | 16 | 21 | 23 | 23,5 |
| 10 sec gel strength, lb/100 ft ² | 17 | 15 | 15 | 15 |
| 1 min gel strength, lb/100 ft ² | 20 | 19 | 19 | 19 |
| 10 min gel strength, lb/100 ft ² | 26 | 28 | 25 | 29 |
| 30 min gel strength, lb/100 ft ² | 32 | 33 | 38 | 35 |
| Cake thickness mm | 5,6 | 6 | 6,3 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| PH (mud) | 8,2 | 8,1 | 8 | |
| PH (filtration) | 8,7 | 8,2 | 8,2 | |

Table B.3: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 28 | 35 | 37 | 39 | 1021,8 |
| 22 | 29 | 31 | 32 | 510,9 |
| 20 | 26 | 27 | 28 | 340,6 |
| 18 | 21 | 23 | 24 | 170,3 |
| 14 | 15 | 17 | 17 | 10,218 |
| 14 | 15 | 17 | 17 | 5,109 |

Table B.4: Water loss of TTB clay - semi saturated

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | √ (time) |
|------------|--------|----------|----------|----------|----------|
| 1 | 22 | 22 | 22 | | 1,000 |
| 2 | 28 | 28 | 28 | | 1,414 |
| 2,5 | 32 | 32 | 30 | | 1,581 |
| 3 | 36 | 36 | 34 | | 1,732 |
| 4 | 40 | 40 | 38 | | 2,000 |
| 5 | 46 | 44 | 44 | | 2,236 |
| 7,5 | 56 | 54 | 52 | | 2,739 |
| 10 | 64 | 64 | 62 | | 3,162 |
| 20 | 90 | 88 | 86 | | 4,472 |
| 30 | 110 | 110 | 108 | | 5,477 |

Table B.5: Rheological properties of S clay - semi saturated

Sample name : S clay – semi salt saturated Temperature of room: 23.3 °c Composition : 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------|
| Sample mud temperature: oc | 28 ∘c | 26 ∘c | 27 ∘c | |
| Density , ppg | 9,6 | 9,6 | 9,6 | |
| θ 600 | 15 | 18 | 20 | |
| θ 300 | 12 | 14 | 16 | |
| θ 200 | 10,5 | 13 | 14 | |
| θ 100 | 9 | 10,5 | 12 | |
| θ 6 | 6 | 7 | 8 | |
| θ3 | 6 | 7 | 8 | |
| Apparent viscosity (AV) , cp | 7,5 | 9 | 10 | |
| Plastic viscosity (PV) , cp | 3 | 4 | 4 | |
| Yeild point (YP), lb/100 ft ² | 9 | 10 | 12 | |
| 10 sec gel strength, lb/100 ft ² | 8 | 8 | 8,5 | |
| 1 min gel strength, lb/100 ft ² | 11 | 10 | 10,5 | |
| 10 min gel strength, lb/100 ft ² | 16 | 16 | 17,5 | |
| 30 min gel strength, lb/100 ft ² | 18 | 20 | 21 | |
| Cake thickness mm | 4,8 | 5,2 | 5 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| Tuzluluk, mg/L | semi saturated | semi saturated | semi saturated | |
| PH (mud) | 7,6 | 7,6 | 7,6 | |
| PH (filtration) | 8,2 | 8,2 | 8,2 | |

Table B.5: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 16 | 19 | 21 | | 1021,8 |
| 13 | 15 | 17 | | 510,9 |
| 11 | 14 | 15 | | 340,6 |
| 10 | 11 | 13 | | 170,3 |
| 6 | 7 | 9 | | 10,218 |
| 6 | 7 | 9 | | 5,109 |

Table B.6: Water loss of S clay mud - semi saturated

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(extsf{time})}$ |
|------------|--------|----------|----------|----------|-------------------------|
| 1 | 27 | 24 | 28 | | 1,000 |
| 2 | 38 | 34 | 38 | | 1,414 |
| 2,5 | 42 | 39 | 42 | | 1,581 |
| 3 | 46 | 43 | 46 | | 1,732 |
| 4 | 52 | 50 | 52 | | 2,000 |
| 5 | 60 | 55 | 58 | | 2,236 |
| 7,5 | 72 | 68 | 70 | | 2,739 |
| 10 | 82 | 78 | 80 | | 3,162 |
| 20 | 116 | 110 | 112 | | 4,472 |
| 30 | 140 | 135 | 135 | | 5,477 |

Table B.7: Rheological properties of YD-K clay - semi saturated

Sample name : YD-K clay – semi salt saturated Temperature of room: 23.3 °c Composition : 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------|
| Sample mud temperature: oc | 28°c | 24.5 C | 26 C | 24 C |
| Density , ppg | 9,52 | 9,52 | 9,52 | 9,52 |
| θ 600 | 23 | 26 | 28 | 30 |
| θ 300 | 18 | 20,5 | 22 | 23 |
| θ 200 | 16 | 17,5 | 19 | 19,5 |
| θ 100 | 13 | 14 | 16 | 16 |
| θ 6 | 10 | 10 | 11 | 11 |
| 03 | 10 | 10 | 11 | 11 |
| Apparent viscosity (AV) , cp | 11,5 | 12,5 | 14 | 15 |
| Plastic viscosity (PV) , cp | 5 | 5,5 | 6 | 7 |
| Yeild point (YP), lb/100 ft ² | 13 | 15 | 16 | 16 |
| 10 sec gel strength, lb/100 ft ² | 12 | 12 | 12 | 10 |
| 1 min gel strength, lb/100 ft ² | 14 | 13 | 12,5 | 14 |
| 10 min gel strength, lb/100 ft ² | 22 | 19 | 19 | 20 |
| 30 min gel strength, lb/100 ft ² | 23 | 23 | 23 | 25 |
| Cake thickness mm | 6 | 6 | 5,5 | |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | |
| Tuzluluk, mg/L | semi saturated | semi saturated | semi saturated | |
| PH (mud) | 7,6 | 7,6 | 7,6 | |
| PH (filtration) | 8,9 | 8,2 | 8,9 | |

Table B.7: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 25 | 28 | 30 | 32 | 1021,8 |
| 19 | 22 | 23 | 25 | 510,9 |
| 17 | 19 | 20 | 21 | 340,6 |
| 14 | 15 | 17 | 17 | 170,3 |
| 11 | 11 | 12 | 12 | 10,218 |
| 11 | 11 | 12 | 12 | 5,109 |

Table B.8: Water loss of YD-K clay mud - semi saturated

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
|------------|--------|----------|----------|----------|-----------------|
| 1 | 22 | 20 | 22 | | 1,000 |
| 2 | 30 | 28 | 30 | | 1,414 |
| 2,5 | 32 | 30 | 32 | | 1,581 |
| 3 | 36 | 34 | 36 | | 1,732 |
| 4 | 40 | 38 | 40 | | 2,000 |
| 5 | 44 | 44 | 44 | | 2,236 |
| 7,5 | 54 | 52 | 52 | | 2,739 |
| 10 | 64 | 62 | 64 | | 3,162 |
| 20 | 88 | 86 | 88 | | 4,472 |
| 30 | 106 | 106 | 104 | | 5,477 |

Table B.9: Rheological properties of YD-S clay - semi saturated

Sample name : YD-S clay – semi salt saturated

Temperature of room: 23.3 °c

Composition : 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|----------------|----------------|----------------|----------------|
| Sample mud temperature: ∘c | 29 ∘c | | | |
| Density , ppg | 9,4 | 9,45 | 9,45 | 9,45 |
| θ 600 | 29 | 35 | 38 | 42 |
| θ 300 | 23 | 28,5 | 30,5 | 34 |
| θ 200 | 21 | 25 | 27 | 30 |
| θ 100 | 18 | 21 | 23 | 26 |
| θ 6 | 15 | 15 | 17 | 18 |
| θ 3 | 15 | 15 | 17 | 18 |
| Apparent viscosity (AV) , cp | 14,5 | 17,5 | 19 | 21 |
| Plastic viscosity (PV) , cp | 6 | 6,5 | 7,5 | 8 |
| Yeild point (YP), lb/100 ft ² | 17 | 22 | 23 | 26 |
| 10 sec gel strength, lb/100 ft ² | 14 | 17 | 17 | 17,5 |
| 1 min gel strength,lb/100 ft ² | 20 | 21 | 21 | 22 |
| 10 min gel strength, lb/100 ft ² | 27 | 27 | 28 | 32 |
| 30 min gel strength, lb/100 ft ² | 39 | 35 | 35 | 35 |
| Cake thickness mm | 6 | 6,4 | 6,1 | 6,7 |
| Cake status | Hard and rigid | Hard and rigid | Hard and rigid | Hard and rigid |
| Tuzluluk | semi saturated | semi saturated | semi saturated | semi saturated |
| PH (mud) | 8 | 8 | 8,1 | 8,2 |
| PH (filtration) | 8,4 | 8,2 | 8,1 | 8,2 |

Table B.9: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 31 | 37 | 41 | 45 | 1021,8 |
| 25 | 30 | 33 | 36 | 510,9 |
| 22 | 27 | 29 | 32 | 340,6 |
| 19 | 22 | 25 | 28 | 170,3 |
| 16 | 16 | 18 | 19 | 10,218 |
| 16 | 16 | 18 | 19 | 5,109 |

Table B.10: Water loss of YD-S clay mud - semi saturated

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{\text{(time)}}$ |
|------------|--------|----------|----------|----------|------------------------|
| 1 | 20 | 24 | 12 | 17 | 1,000 |
| 2 | 28 | 32 | 18 | 23 | 1,414 |
| 2,5 | 32 | 36 | 22 | 26 | 1,581 |
| 3 | 36 | 40 | 24 | 29 | 1,732 |
| 4 | 40 | 42 | 30 | 33 | 2,000 |
| 5 | 44 | 46 | 34 | 39 | 2,236 |
| 7,5 | 54 | 56 | 42 | 47 | 2,739 |
| 10 | 62 | 66 | 50 | 53 | 3,162 |
| 20 | 84 | 88 | 74 | 76 | 4,472 |
| 30 | 104 | 108 | 92 | 93 | 5,477 |
APPENDIX – C: Results of rheological and filtration properties sepiolite base mud at ambeint conditions-fresh water

In this section, relationships between shear rate shear stress and filtration properties of sepiolite base drilling fluid prepared in fresh water system are shown. Salinity is zero. All of the tests are conducted in room conditoin.

Table C.1: Rheological properties of kurtşeyh clay – fresh water

Sample name : kurtşeyh clay – fersh water **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|--------------|--------------|--------------|-------------|
| Sample mud temperature: •c | 29∘c | 24∘c | 24∘c | |
| Density , ppg | 8,6 | 8,6 | 8,6 | 8,9 |
| θ 600 | 37 | 40 | 42 | 43 |
| θ 300 | 31 | 33,5 | 35 | 35 |
| θ 200 | 28 | 29 | 32 | 31 |
| 0 100 | 24 | 25 | 27 | 26 |
| θ 6 | 16 | 17 | 18 | 17,5 |
| 03 | 16 | 17 | 18 | 17,5 |
| Apparent viscosity (AV) , cp | 18,5 | 20 | 21 | 21,5 |
| Plastic viscosity (PV) , cp | 6 | 6,5 | 7 | 8 |
| Yeild point (YP), lb/100 ft ² | 25 | 27 | 28 | 27 |
| 10 sec gel strength, lb/100 ft ² | 15 | 15 | 15,5 | 17 |
| 1 min gel strength,lb/100 ft ² | 18 | 18 | 18,5 | 19 |
| 10 min gel strength, lb/100 ft ² | 22 | 21 | 25 | 25 |
| 30 min gel strength, lb/100 ft ² | 23 | 23 | 28 | 30 |
| Cake thickness mm | 8 | 7,8 | 7,7 | |
| Cake status | Hard - rigid | Hard - rigid | Hard - rigid | |
| Tuzluluk, mg/l | Fresh water | Fresh water | Fresh water | Fresh water |
| PH (mud) | 8,9 | 9,1 | 8,8 | |
| PH (filtration) | 9,4 | 9,3 | 9,3 | |

Table C.1: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 39 | 43 | 45 | 46 | 1021,8 |
| 33 | 36 | 37 | 37 | 510,9 |
| 30 | 31 | 34 | 33 | 340,6 |
| 26 | 27 | 29 | 28 | 170,3 |
| 17 | 18 | 19 | 19 | 10,218 |
| 17 | 18 | 19 | 19 | 5,109 |

 Table C.2: Water loss of kurtşeyh clay mud – fresh water

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
|------------|--------|----------|----------|----------|-----------------|
| 1 | 24 | 24 | 24 | | 1,000 |
| 2 | 33 | 34 | 34 | | 1,414 |
| 2,5 | 37 | 38 | 38 | | 1,581 |
| 3 | 40 | 42 | 42 | | 1,732 |
| 4 | 46 | 48 | 48 | | 2,000 |
| 5 | 53 | 54 | 54 | | 2,236 |
| 7,5 | 63 | 66 | 64 | | 2,739 |
| 10 | 73 | 76 | 76 | | 3,162 |
| 20 | 102 | 106 | 104 | | 4,472 |
| 30 | 124 | 128 | 126 | | 5,477 |

Table C.3: Rheological properties of TTB clay – fresh water

Sample name : TTB clay – fresh water **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|--------------|--------------|--------------|-------------|
| Sample mud temperature: °c | 29 °c | 24 °c | | |
| Density , ppg | 8,6 | 8,6 | 8,6 | 8,6 |
| θ 600 | 38 | 38 | 42 | 42 |
| θ 300 | 32 | 32 | 35 | 35 |
| θ 200 | 29 | 28 | 31 | 30 |
| θ 100 | 25 | 24,5 | 27 | 26 |
| θ 6 | 19 | 17 | 18,5 | 18 |
| θ 3 | 19 | 17 | 18,5 | 18 |
| Apparent viscosity (AV) , cp | 19 | 19 | 21 | 21 |
| Plastic viscosity (PV) , cp | 6 | 6 | 7 | 7 |
| Yeild point (YP), lb/100 ft ² | 26 | 26 | 28 | 28 |
| 10 sec gel strength, lb/100 ft ² | 21 | 19 | 21 | 21 |
| 1 min gel strength,lb/100 ft ² | 25 | 22 | 24 | 25 |
| 10 min gel strength, lb/100 ft ² | 34 | 30 | 34 | 32 |
| 30 min gel strength, lb/100 ft ² | 40 | 36 | 41 | 40 |
| Cake thickness mm | 7,8 | 5,9 | 6,5 | |
| Cake status | Hard - rigid | Hard - rigid | Hard - rigid | |
| Tuzluluk, mg/L | Fresh water | Fresh water | Fresh water | Fresh water |
| PH (mud) | 8,7 | 8,9 | 8,9 | |
| PH (filtration) | 8,9 | 9,1 | 9,1 | |

Table C.3: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 41 | 41 | 45 | 45 | 1021,8 |
| 34 | 34 | 37 | 37 | 510,9 |
| 31 | 30 | 33 | 32 | 340,6 |
| 27 | 26 | 29 | 28 | 170,3 |
| 20 | 18 | 20 | 19 | 10,218 |
| 20 | 18 | 20 | 19 | 5,109 |

 Table C.4: Water loss of TTB clay – fresh water

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
|------------|--------|----------|----------|----------|-----------------|
| 1 | 24 | 22 | 24 | | 1,000 |
| 2 | 30 | 30 | 30 | | 1,414 |
| 2,5 | 32 | 32 | 32 | | 1,581 |
| 3 | 36 | 34 | 36 | | 1,732 |
| 4 | 40 | 38 | 40 | | 2,000 |
| 5 | 44 | 44 | 44 | | 2,236 |
| 7,5 | 54 | 54 | 54 | | 2,739 |
| 10 | 64 | 62 | 64 | | 3,162 |
| 20 | 88 | 86 | 88 | | 4,472 |
| 30 | 108 | 108 | 108 | | 5,477 |

Table C.5: Rheological properties of S clay – fresh water

Sample name : S clay – fresh water **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|--------------|--------------|--------------|--------------|
| Sample mud temperature: •c | 25∘C | | | |
| Density , ppg | 8,6 | 8,6 | 8,6 | 8,6 |
| θ 600 | 18,5 | 21,5 | 24 | 25 |
| 0 300 | 15,5 | 18 | 20 | 21 |
| θ 200 | 14 | 16,5 | 18 | 19 |
| θ 100 | 12 | 14 | 16 | 16,5 |
| θ 6 | 8 | 9 | 10 | 10,5 |
| θ 3 | 8 | 9 | 10 | 10,5 |
| Apparent viscosity (AV) , cp | 9,25 | 10,25 | 12 | 12,5 |
| Plastic viscosity (PV) , cp | 3 | 3,5 | 4 | 4 |
| Yeild point (YP), lb/100 ft ² | 12,5 | 14,5 | 16 | 17 |
| 10 sec gel strength, lb/100 ft ² | 9 | 10 | 10 | 11 |
| 1 min gel strength, lb/100 ft ² | 11 | 13 | 13 | 15 |
| 10 min gel strength, lb/100 ft ² | 14 | 16 | 18 | 20 |
| 30 min gel strength, lb/100 ft ² | 17 | 19 | 21 | 27 |
| Cake thickness mm | 7,7 | | | 6,2 |
| Cake status | Hard - rigid | Hard - rigid | Hard - rigid | Hard - rigid |
| Tuzluluk, mg/L | Fresh water | Fresh water | Fresh water | Fresh water |
| PH (mud) | | | 8,8 | 8,9 |
| PH (filtration) | | | 9,2 | 9,3 |

Table C.5: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 20 | 23 | 26 | 27 | 1021,8 |
| 17 | 19 | 21 | 22 | 510,9 |
| 15 | 18 | 19 | 20 | 340,6 |
| 13 | 15 | 17 | 18 | 170,3 |
| 9 | 10 | 11 | 11 | 10,218 |
| 9 | 10 | 11 | 11 | 5,109 |

Table C.6: Water loss of S clay mud – fresh water

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | √ (time) |
|------------|--------|----------|----------|----------|----------|
| 1 | 26 | 26 | 26 | 26 | 1,000 |
| 2 | 34 | 36 | 36 | 34 | 1,414 |
| 2,5 | 38 | 40 | 40 | 38 | 1,581 |
| 3 | 42 | 44 | 44 | 42 | 1,732 |
| 4 | 48 | 50 | 50 | 48 | 2,000 |
| 5 | 54 | 56 | 56 | 54 | 2,236 |
| 7,5 | 67 | 70 | 68 | 67 | 2,739 |
| 10 | 79 | 80 | 80 | 79 | 3,162 |
| 20 | 110 | 112 | 112 | 110 | 4,472 |
| 30 | 135 | 136 | 134 | 135 | 5,477 |

Table C.7: Rheological properties of YD-K clay – fresh water

Sample name : YD-K clay – fresh water **Temperature of room**: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|--------------|--------------|--------------|-------------|
| Sample mud temperature: °c | 29∘c | 24°c | 24°c | |
| Density , ppg | 8,55 | 8,55 | 8,55 | 8,55 |
| θ 600 | 31 | 31 | 33 | 34 |
| 0 300 | 26,5 | 26,5 | 28 | 28 |
| θ 200 | 22 | 22,5 | 24,5 | 24,5 |
| θ 100 | 19 | 19 | 21 | 20 |
| θ 6 | 14 | 14 | 14 | 14 |
| θ 3 | 14 | 14 | 14 | 14 |
| Apparent viscosity (AV) , cp | 15,5 | 15,5 | 16,5 | 17 |
| Plastic viscosity (PV) , cp | 4,5 | 4,5 | 5 | 6 |
| Yeild point (YP), lb/100 ft ² | 22 | 22 | 23 | 22 |
| 10 sec gel strength, lb/100 ft ² | 15 | 15 | 14 | 15 |
| 1 min gel strength,lb/100 ft ² | 18 | 18 | 17 | 17 |
| 10 min gel strength, lb/100 ft ² | 21 | 23 | 23 | 21 |
| 30 min gel strength, lb/100 ft ² | 26 | 27 | 27 | 27 |
| Cake thickness mm | 6 | 6,4 | 6,2 | |
| Cake status | Hard - rigid | Hard - rigid | Hard - rigid | |
| Tuzluluk, mg/L | Fresh water | Fresh water | Fresh water | Fresh water |
| PH (mud) | 8,9 | 8,8 | 8,4 | |
| PH (filtration) | 9,5 | 9,4 | 9,1 | |

Table C.7: Continue

| |] | | | |
|-------------------|-------------|-------------|-------------|-----------|
| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
| 33 | 33 | 35 | 36 | 1021,8 |
| 28 | 28 | 30 | 30 | 510,9 |
| 23 | 24 | 26 | 26 | 340,6 |
| 20 | 20 | 22 | 21 | 170,3 |
| 15 | 15 | 15 | 15 | 10,218 |
| 15 | 15 | 15 | 15 | 5,109 |

Table C.8: Water loss of YD-K clay mud – fresh water

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{(time)}$ |
|------------|--------|----------|----------|----------|-----------------|
| 1 | 22 | 22 | 22 | | 1,000 |
| 2 | 29 | 30 | 30 | | 1,414 |
| 2,5 | 32 | 34 | 33 | | 1,581 |
| 3 | 36 | 36 | 36 | | 1,732 |
| 4 | 41 | 42 | 42 | | 2,000 |
| 5 | 46 | 48 | 46 | | 2,236 |
| 7,5 | 55 | 58 | 56 | | 2,739 |
| 10 | 63 | 66 | 65 | | 3,162 |
| 20 | 89 | 90 | 91 | | 4,472 |
| 30 | 108 | 112 | 112 | | 5,477 |

Table C.9: Rheological properties of YD-S clay – fresh water

Sample name : YD-S clay – fresh water

Temperature of room: 23.3 °c **Composition :** 20 gr clay +350 ml water

| property | 20 min | 16 hours | 24 hours | 48 hours |
|---|--------------|--------------|--------------|-------------|
| Sample mud temperature: ∘c | 29°c | | | |
| Density , ppg | 8,6 | 8,6 | 8,6 | 8,6 |
| θ 600 | 44,5 | 49 | 53 | 55 |
| 0 300 | 38 | 41 | 45 | 45,5 |
| θ 200 | 34 | 37 | 39 | 40 |
| θ 100 | 30 | 32 | 34 | 35 |
| θ 6 | 23 | 23 | 24 | 25 |
| θ3 | 23 | 23 | 24 | 25 |
| Apparent viscosity (AV) , cp | 22,25 | 24,5 | 26,5 | 27,5 |
| Plastic viscosity (PV) , cp | 6,5 | 8 | 8 | 9,5 |
| Yeild point (YP), lb/100 ft ² | 31,5 | 33 | 37 | 36 |
| 10 sec gel strength, lb/100 ft ² | 24 | 22 | 22 | 23 |
| 1 min gel strength, lb/100 ft ² | 27 | 25 | 26 | 25 |
| 10 min gel strength,lb/100 ft ² | 39 | 35 | 35 | 36 |
| 30 min gel strength,lb/100 ft ² | 43 | 37 | 43 | 38 |
| Cake thickness mm | 9 | 6,3 | 7,4 | |
| Cake status | Hard - rigid | Hard - rigid | Hard - rigid | |
| Tuzluluk | Fresh water | Fresh water | Fresh water | Fresh water |
| PH (mud) | 8,6 | 8,8 | 8,5 | |
| PH (filtration) | 9,1 | 9,1 | 9,1 | |

Table C.9: Continue

| T (20 min) | T(16 hours) | T(24 hours) | T(48 hours) | γ=1.703xθ |
|-------------------|-------------|-------------|-------------|-----------|
| 47 | 52 | 56 | 59 | 1021,8 |
| 41 | 44 | 48 | 49 | 510,9 |
| 36 | 39 | 42 | 43 | 340,6 |
| 32 | 34 | 36 | 37 | 170,3 |
| 25 | 25 | 26 | 27 | 10,218 |
| 25 | 25 | 26 | 27 | 5,109 |

Table C.10: Water loss of YD-S clay mud – fresh water

| time (min) | 20 min | 16 hours | 24 hours | 48 hours | $\sqrt{\text{(time)}}$ |
|------------|--------|----------|----------|----------|------------------------|
| 1 | 22 | 24 | 22 | | 1,000 |
| 2 | 30 | 30 | 30 | | 1,414 |
| 2,5 | 34 | 34 | 32 | | 1,581 |
| 3 | 36 | 36 | 36 | | 1,732 |
| 4 | 42 | 40 | 40 | | 2,000 |
| 5 | 46 | 46 | 44 | | 2,236 |
| 7,5 | 54 | 56 | 54 | | 2,739 |
| 10 | 64 | 64 | 62 | | 3,162 |
| 20 | 88 | 88 | 86 | | 4,472 |
| 30 | 108 | 108 | 106 | | 5,477 |

APPENDIX – D: Results of grain size analysis of sepiolite samples

In this section, results of grain size analysis are given graphically for each type of sepiolite examined in this study. These experiments are done in the libratory of mineral processing engineering department.



Figure D.1: Grain size distribution of commercial bentonite.



Figure D.2: Grain size distribution of Kurtşehl sepiolite.



Figure D.3: Grain size distribution of TTB sepiolite.



Figure D.4: Grain size distribution of (S) sepiolite.



Figure D.5: Grain size distribution of YD-K sepiolite.



Figure D.6: Grain size distribution of YD-S sepiolite.

APPENDIX – E: Figures of rheological properties of sepiolite muds at ambient conditions

In this section, relationships between shear rate shear stress of sepiolite base drilling fluid are shown. Results involve the fully, semi saturated systems and fresh water system experiments. All of the tests are conducted in room condition.



Figure E.1: Rheological properties of fully saturated mud prepared with kurtşeyh clay.



Figure E.2: Rheological properties of fully saturated mud prepared with TTB clay.



Figure E.3: Rheological properties of fully saturated mud prepared with S clay.



Figure E.4: Rheological properties of fully saturated mud prepared with YD-K clay.



Figure E.5: Rheological properties of fully saturated mud prepared with YD-S clay.



Figure E.6: Rheological properties of semi salt saturated mud prepared with kurtşeyh clay.



Figure E.7: Rheological properties of semi salt saturated mud prepared with TTB clay.



Figure E.8: Rheological properties of semi salt saturated mud prepared with S clay.



Figure E.9: Rheological properties of semi salt saturated mud prepared with YD-K clay.



Figure E.10: Rheological properties of semi salt saturated mud prepared with YD-S clay.



Figure E.11: Rheological properties of fresh water mud prepared with Kurtşeyh clay.



Figure E.12: Rheological properties of fresh water mud prepared with TTB clay.



Figure E.13: Rheological properties of fresh water mud prepared with S clay.



Figure E.14: Rheological properties of fresh water mud prepared with YD-K clay.



Figure E.15: Rheological properties of fresh water mud prepared with YD-S clay.







Figure E.17: Changes in rheological properties of mud prepared with Kurtşeyh clay (16 hours).



Figure E.18: Changes in rheological properties of mud prepared with Kurtşeyh clay (24 hours).



Figure E.19: Changes in rheological properties of mud prepared with Kurtşeyh clay (48 hours).



Figure E.20: Changes in rheological properties of mud prepared with TTB clay (20 min).



Figure E.21: Changes in rheological properties of mud prepared with TTB clay (16 hours).



Figure E.22: Changes in rheological properties of mud prepared with TTB clay (24 hours).



Figure E.23: Changes in rheological properties of mud prepared with TTB clay (48 hours).



Figure E.24: Changes in rheological properties of mud prepared with S clay (20 min).



Figure E.25: Changes in rheological properties of mud prepared with S clay (16 hours).



Figure E.26: Changes in rheological properties of mud prepared with S clay (24 hours).



Figure E.27: Changes in rheological properties of mud prepared with S clay (48 hours).



Figure E.28: Changes in rheological properties of mud prepared with YD-K clay (20 min).



Figure E.29: Changes in rheological properties of mud prepared with YD-K clay (16 hours).



Figure E.30: Changes in rheological properties of mud prepared with YD-K clay (24 hours).



Figure E.31: Changes in rheological properties of mud prepared with YD-K clay (48 hours).



Figure E.32: Changes in rheological properties of mud prepared with YD-S clay (20 min).



Figure E.33: Changes in rheological properties of mud prepared with YD-S clay (16 hours).



Figure E.34: Changes in rheological properties of mud prepared with YD-S clay (24 hours).



Figure E.35: Changes in rheological properties of mud prepared with YD-S clay (48 hours).
APPENDIX – F: Figures of API water loss properties of sepiolite muds at ambient conditions

In this section, filtration properties of sepiolite base drilling fluid are shown. Results involve the full and semi saturated systems and fresh water system experiments. All of the tests are conducted in room condition.



Figure F.1: API water loss of Kurtşeyh clay-fully saturated.



Figure F.2: API water loss versus square root of time for Kurtşeyh clay mud-fully saturated.



Figure F.3: API water loss of TTB clay-fully saturated.



Figure F.4: API water loss versus square root of time for TTB clay mud-fully saturated.



Figure F.5: API water loss of S clay-fully saturated.



Figure F.6: API water loss versus square root of time for S clay mud-fully saturated.



Figure F.7: API water loss of YD-K clay-fully saturated.



Figure F.8: API water loss versus square root of time for YD-K clay mud-fully saturated.



Figure F.9: API water loss of YD-S clay-fully saturated.



Figure F.10: API water loss versus square root of time for YD-S clay mud-fully saturated.



Figure F.11: Comparison of API water loss changes – fully saturated (20 min).



Figure F.12: Comparison of API water loss versus square root of time –fully saturated (20 min).



Figure F.13: Comparison of API water loss changes –fully saturated (16 hours).



Figure F.14: Comparison of API water loss versus square root of time –fully saturated (16 hours).



Figure F.15: Comparison of API water loss changes –fully saturated (24 hours)



Figure F.16: Comparison of API water loss versus square root of time –fully saturated (24 hours).



Figure F.17: API water loss of Kurtşeyh clay-semi salt saturated.



Figure F.18: API water loss versus square root of time for Kurtşeyh clay mud-semi salt saturated.



Figure F.19: API water loss of TTB clay-semi salt saturated.



Figure F.20: API water loss versus square root of time for TTB clay mud-semi salt saturated.



Figure F.21: API water loss of S clay-semi salt saturated.



Figure F.22: API water loss versus square root of time for S clay mud-semi salt saturated.



Figure F.23: API water loss of YD-K clay-semi salt saturated.



Figure F.24: API water loss versus square root of time for YD-K clay mud-semi salt saturated.



Figure F.25: API water loss of YD-S clay-semi salt saturated.



Figure F.26: API water loss versus square root of time for YD-S clay mud-semi salt saturated.



Figure F.27: Comparison of API water loss changes -semi salt saturated (20 min).



Figure F.28: Comparison of API water loss vs. square root of time –semi salt saturated (20 min).



Figure F.29: Comparison of API water loss changes –semi salt saturated (16 hours).



Figure F.30: Comparison of API water loss versus square root of time –semi salt saturated (16 hours).



Figure F.31: Comparison of API water loss changes -semi salt saturated (24 hours).



Figure F.32: Comparison of API water loss vs. square root of time –semi salt saturated (24 hours).



Figure F.33: API water loss of Kurtşeyh clay-fresh water.



Figure F.34: API water loss versus square root of time for Kurtşeyh clay mud-fresh water.



Figure F.35: API water loss of TTB clay-fresh water.



Figure F.36: API water loss versus square root of time for TTB clay mud-fresh water.



Figure F.37: API water loss of S clay-fresh water.



Figure F.38: API water loss versus square root of time for S clay mud-fresh water.



Figure F.39: API water loss of YD-K clay-fresh water.



Figure F.40: API water loss versus square root of time for YD-K clay mud-fresh water.



Figure F.41: API water loss of YD-S clay-fresh water.



Figure F.42: API water loss versus square root of time for YD-S clay mud-fresh water.



Figure F.43: Comparison of API water loss changes –fresh water (20 min).



Figure F.44: Comparison of API water loss versus square root of time –fresh water (20 min).



Figure F.45: Comparison of API water loss changes –fresh water (16 hours).



Figure F.46: Comparison of API water loss versus square root of time –fresh water (16 hours).



Figure F.47: Comparison of API water loss changes –fresh water (24 hours).



Figure F.48: Comparison of API water loss versus square root of time –fresh water (24 hours).



Figure F.49: Change in API water loss in mud prepared with Kurtşeyh clay (20 min).



Figure F.50: API water loss versus square root of time in mud prepared with Kurtşeyh clay (20 min).



Figure F.51: Change in API water loss in mud prepared with TTB clay (20 min).



Figure F.52: API water loss versus square root of time in mud prepared with TTB clay (20 min).



Figure F.53: Change in API water loss in mud prepared with S clay (20 min).



Figure F.54: API water loss versus square root of time in mud prepared with S clay (20 min).



Figure F.55: Change in API water loss in mud prepared with YD-K clay (20 min).



Figure F.56: API water loss versus square root of time in mud prepared with YD-K clay (20 min).



Figure F.57: Change in API water loss in mud prepared with YD-S clay (20 min).



Figure F.58: API water loss versus square root of time in mud prepared with YD-S clay (20 min).

APPENDIX – G: Figures of sepiolite base muds containing additives at HTHP conditions

In this section, filtration properties of sepiolite base drilling fluids containing additives at high pressure and high temperature in fully, semi saturated systems, and fresh water systems are given. Moreover, results of tests containing additives are shown graphically in ambient conditions.



Figure G.1: Changes of API water loss in sepiolite (TTB) fresh water base mud containing additive at room condition (80°F).



Figure G.2: API water loss versus square root of time in sepiolite (TTB) fresh water base mud containing additive at room condition (80°F).



Figure G.3: Changes of API water loss in sepiolite (YD-S) fresh water base mud containing additive at room condition (80°F).



Figure G.4: API water loss versus square root of time in sepiolite (YD-S) fresh water base mud containing additive at room condition (80°F).



Figure G.5: API HTHP water loss versus square root of time in sepiolite (YD-S and TTB) fresh water base mud containing additive at 100 psi@ 350°F.



Figure G.6: API HTHP water loss versus square root of time in sepiolite (YD-S and TTB) salt saturated base mud containing additive at 100 psi @ 350°F.



Figure G.7: API HTHP water loss versus square root of time in sepiolite (YD-S and TTB) salt saturated base mud containing additive at 500 psi @ 350°F.
APPENDIX – H: Figures of dynamic filtration sepiolite base muds containing additives at HTHP conditions

In this section, dynamic filtration properties of typical sepiolite base drilling fluids containing additives at high pressure and high temperature in fresh water systems through the filter core with pore size of $35 \,\mu m$ are shown.



Figure H.1: Dynamic filtration results of weighted TTB clay ($20\mu < d < 75\mu$) mud at 300°F and 100 psi.



Figure H.2: Dynamic filtration results of weighted YD-S clay (d = 28μ) mud at 300° F and 100 psi.



Figure H.3: Dynamic filtration results of weighted TTB clay $(75\mu < d)$ mud at 300°F and 100 psi.



Figure H.4: Dynamic filtration results of weighted TTB clay (20μ >d) mud at 300°F and 100 psi.



Figure H.5: Dynamic filtration results of weighted TTB clay ($20\mu < d < 75\mu$) mud at 350°F and 100 psi.



Figure H.6: Dynamic filtration results of weighted TTB clay $(20\mu < d < 75\mu)$ mud at 400°Fand 100 psi.



Figure H.7: Dynamic filtration results of weighted TTB clay ($20\mu < d < 75\mu$) mud at 300°F and 500 psi.



Figure H.8: Dynamic filtration results of weighted TTB clay $(20\mu < d < 75\mu)$ mud at 200°F and 100 psi.



Figure H.9: Dynamic filtration results of unweighted TTB clay $(20\mu < d < 75\mu)$ mud at 300°Fand 100 psi.

APPENDIX – I: Effect of reactive clays entering into mud systems

In this section, rheological and filtration properties of weighted and unweighted sepiolite (TTB) mud aged at high temperature condition are tabulated in the light of reactive clays entered into mud system during drilling of unstable formations in fresh water and high salinity system.

| Composition | | Unweighted Mud | | Unweighted Mud | | Unweighted Mud | | Unweighted Mud | |
|---|--------------------------------------|----------------|--------|----------------|-------|----------------|--------|----------------|--------|
| Aging temperature, °F | | 80 | | 300 | | 350 | | 400 | |
| Agin | g time, hrs | - | | 16 | | 16 | | 16 | |
| Mud density, lb/gal | | 8,70 | 8,70 | 8,70 | 8,70 | 8,70 | 8,70 | 8,70 | 8,70 |
| Mud | PH(with pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 |
| Filtra | ation PH (with pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 |
| Tem | perature of PH measurement, °F | 80 | 80 | 80 | 80 | 80 | 80 | 80 | 80 |
| | 600 | 71 | 59 | 43 | 59 | 31 | 40 | 36 | 46 |
| | 300 | 46 | 38 | 28 | 38 | 20 | 26 | 25 | 32 |
| ng | 200 | 35 | 30 | 22 | 29 | 16 | 20 | 20 | 26 |
| adi | 100 | 22 | 19 | 14 | 19 | 11 | 13 | 12 | 18 |
| l Re | 6 | 5 | 4 | 3 | 4 | 3 | 3 | 4 | 6 |
| Dia | 3 | 4 | 3 | 2 | 3 | 2 | 2 | 3 | 5 |
| Tem | perature of rheology measurement, °F | 80 | 120 | 120 | 80 | 120 | 80 | 120 | 80 |
| PV | | 25 | 21 | 15 | 21 | 11 | 14 | 11 | 14 |
| YP | ҮР | | 17 | 13 | 17 | 9 | 12 | 14 | 18 |
| gel strength, 10s./ 1min./ 10min. | | 4/6/13 | 4/6/13 | 3/4/6 | 4/5/7 | 3/4/13 | 4/5/15 | 4/5/9 | 6/7/10 |
| Temperature of filtration measurement, °F | | 300 | | 300 | | 300 | | 300 | |
| API water loss, cc (7.5/30min)@100 psi | | 12,8/29,2 | | 12,8/29 | | 14,4/31,4 | | 23,6/49 | |
| cake | thickness, mm | 2 | | 2 | | 2,5 | | 3 | |

Table I.1: Rheological and filtration properties of unweighted sepiolite mud aged at high temperature condition in fresh water system.

| Composition | | Weighted Mud | | Weighted Mud | | Weighted Mud | | Weighted Mud | |
|---|--------------------------------------|--------------|---------|--------------|-------|--------------|--------|--------------|--------|
| Aging temperature, °F | | 80 | | 300 | | 350 | | 400 | |
| Aging time, hrs | | - | | 16 | | 16 | | 16 | |
| Mud density, lb/gal | | 11,10 | 11,10 | 11,10 | 11,10 | 11,10 | 11,10 | 11,10 | 11,10 |
| Mud | PH(with pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 |
| Filtra | ation PH (with pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 |
| Temp | perature of PH measurement, °F | 80 | 80 | 80 | 80 | 80 | 80 | 80 | 80 |
| | 600 | 107 | 95 | 43 | 56 | 46 | 61 | 52 | 64 |
| | 300 | 70 | 62 | 27 | 35 | 30 | 39 | 38 | 48 |
| ng | 200 | 55 | 51 | 20 | 26 | 23 | 30 | 30 | 34 |
| adi | 100 | 36 | 35 | 12 | 16 | 15 | 19 | 19 | 25 |
| l Re | 6 | 8 | 8 | 3 | 3 | 4 | 4 | 5 | 6 |
| Dia | 3 | 7 | 6 | 2 | 2 | 3 | 3 | 4 | 4 |
| Temp | perature of rheology measurement, °F | 80 | 120 | 120 | 80 | 120 | 80 | 120 | 80 |
| PV | | 37 | 33 | 16 | 21 | 16 | 22 | 14 | 16 |
| УР | | 33 | 29 | 11 | 14 | 14 | 17 | 24 | 32 |
| gel strength, 10s./ 1min./ 10min. | | 7/13/23 | 7/13/24 | 3/3/4 | 3/3/5 | 4/5/13 | 4/5/14 | 3/5/16 | 4/7/18 |
| Temperature of filtration measurement, °F | | 300 | | 300 | | 300 | | 300 | |
| API water loss, cc (7.5/30min)@100 psi | | 5,8/13 | | 6/15,2 | | 8/18 | | 14,4/31 | |
| cake | thickness, mm | 3 | | 3,5 | | 3,5 | | 4 | |

Table I.2: Rheological and filtration properties of weighted sepiolite mud aged at high temperature condition in fresh water system.

| Composition | | Unweighted Mud | | Unweighted Mud Unwei | | ghted Mud | |
|---|--------------------------------------|----------------|-------|----------------------|---------|-----------|--|
| OCM | IA, ppb | | | 80 | 80 | | |
| Aging temperature, °F | | 80 | 350 | 300 | 350 | | |
| Aging time, hrs | | - | 16 | 16 | | 16 | |
| MBT, lb/bbl | | 5,00 | 5,00 | 12,00 | 12 | 2,00 | |
| Mud | density, lb/gal | 8,7 | 8,7 | 8,90 | 8,90 | 8,90 | |
| Mud | PH(with pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | |
| Filtration PH (with pH meter) | | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | |
| Temperature of PH measurement, °F | | 80 | 80 | 75 | 80 | 80 | |
| | 600 | 61 38 | | 61 | 77 | 94 | |
| ing | 300 | 38 | 24 | 42 | 53 | 64 | |
| eadi | 200 | 28 | 18 | 34 | 43 | 52 | |
| ıl R | 100 | 17 | 11 | 25 | 30 | 30 | |
| Dia | 6 | 3 | 2 | 9 | 9 | 9 | |
| | 3 | 3 | 2 | 7 | 7 | 7 | |
| Tem | perature of rheology measurement, °F | 80 | 80 | 120 | 120 | 80 | |
| PV | | 23 | 14 | 19 | 24 | 30 | |
| YP | | 15 | 10 | 23 | 29 | 34 | |
| gel strength, 10s./ 1min./ 10min. | | 3/3/5 | 2/3/5 | 8/10/13 | 8/11/20 | 8/10/16 | |
| Temperature of filtration measurement, °F | | 300 | 300 | 300 | 300 | 300 | |
| API water loss, cc (7.5/30min)@100 psi | | 11/26 | 12/28 | 4,4/10 | 6 | /13 | |
| cake | thickness, mm | 2 | 2 | 2,5 | 2 | 2,5 | |
| API | water loss, cc (7.5/30min)@500 psi | | | 6/15 | 8 | /18 | |

Table I.3: Rheological and filtration properties of unweighted sepiolite mud aged at high temperature condition subjected to entrance of reactive clays in fresh water system.

| Composition | | Weighted mud (20 lb TTB) | | Weighted mud (10 lb TTB) | | Weighted mud (10 lb TTB) | |
|---|--------------------------------|-----------------------------|--------|-----------------------------|-------|-----------------------------|---------|
| OCMA, pp | b | | | | | 80 | |
| Aging temp | perature, °F | 80 | 350 | 80 | 350 | 300 | 350 |
| Aging time, hrs | | - | 16 | - | 16 | 16 | 16 |
| MBT, lb/bl | ol | | | 5,00 | 5,00 | 12,50 | 13,00 |
| Mud densit | ty, lb/gal | 11,00 | 11,00 | 10,50 | 10,50 | 10,90 | 10,90 |
| Mud PH(w | ith pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 |
| Filtration | PH (with pH meter) | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 | 8,00 |
| Temperature of PH measurement, °F | | 80 | 80 | 80 | 80 | 80 | 80 |
| | 600 | 87 | 64 | 69 | 41 | 68 | 73 |
| ing | 300 | 55 | 40 | 42 | 25 | 44 | 49 |
| ead | 200 | 42 | 30 | 31 | 16 | 37 | 39 |
| ıl R | 100 | 26 | 18 | 19 | 9 | 27 | 27 |
| Di | 6 | 4 | 4 | 3 | 2 | 8 | 7 |
| | 3 | 3 | 3 | 2 | 2 | 6 | 6 |
| Temperatu | re of rheology measurement, °F | 80 | 80 | 80 | 80 | 120 | 120 |
| PV | | 32 | 24 | 27 | 16 | 24 | 24 |
| ҮР | | 23 | 16 | 15 | 9 | 20 | 25 |
| gel strength, 10s./ 1min./ 10min. | | 3/4/7 | 3/3/7 | 3/3/4 | 2/2/3 | 8/9/20 | 8/10/25 |
| Temperature of filtration measurement, °F | | | 300 | | 300 | 300 | 300 |
| API water loss, cc (7.5/30min)@100 psi | | | 6,2/16 | | 5/13 | 5/12 | 6,2/15 |
| cake thickr | ness, mm | | 3 | | 3 | 6 | 6 |

Table I.4: Rheological and filtration properties of weighted sepiolite mud aged at high temperature condition subjected to entrance of reactive clays in fresh water system.

| Composition Weighted Mud (10 lb/bbl sepiolite com | | | | | |
|--|---|-------------------------|---|---|--|
| NaCl, lb/bbl | | 120 | 12 | 20 | |
| OCMA, ppb | | | 80 | | |
| Aging temperature, °F | | 80 | 350 | | |
| Aging time, hrs | | 20 min | 1 | 6 | |
| , lb/bbl | | | 11,00 | 11,00 | |
| density, lb/gal | | | 12,70 | 12,70 | |
| Mud PH(with pH meter) | | 7,20 | 7,20 | 7,20 | |
| Filtration PH (with pH meter) | | | 8,00 | 8,00 | |
| Temperature of PH meassurment, °F | | 80 | 80 | 80 | |
| 600 | 95 | 48 | 55 | 62 | |
| 300 | 61 | 29 | 34 | 41 | |
| 200 | 46 | 21 | 26 | 33 | |
| 100 | 29 | 13 | 17 | 24 | |
| 6 | 4 | 3 | 4 | 10 | |
| 3 | 3 | 2 | 3 | 9 | |
| perature of rheology meassurment, °F | 80 | 80 | 80 | 120 | |
| | 34 | 19 | 21 | 21 | |
| ҮР | | 10 | 13 | 20 | |
| gel strength, 10s./ 1min./ 10min. | | 2/2/5 | 3/4/9 | 5/7/15 | |
| Temperature of filtration meassurment, °F | | | 300 | | |
| API water loss, cc (7.5/30min)@100 psi | | | 8/17 | | |
| | | | | 5 | |
| | bosition lb/bbl A, ppb g temperature, °F g time, hrs bb/bbl density, lb/gal PH(with pH meter) tion PH (with pH meter) tion PH (with pH meter) rerature of PH meassurment, °F 600 300 200 100 6 3 rerature of rheology meassurment, °F rength, 10s./ 1min./ 10min. rerature of filtration meassurment, °F vater loss, cc (7.5/30min)@100 psi chickness,mm | oositionWeighted.lb/bbl | weighted Mud (10 lb/bb lb/bbl 120 A, ppb 120 g temperature, °F 80 80 g time, hrs 20 min 20 min lb/bbl 20 min 20 min density, lb/gal PH(with pH meter) 8,00 7,20 tion PH (with pH meter) 8,00 7,20 tion PH (with pH meter) 80 80 erature of PH meassurment, °F 80 80 600 95 48 300 61 29 13 3 2 erature of PH meassurment, °F 80 80 300 61 29 13 6 4 3 3 2 erature of rheology meassurment, °F 80 80 34 19 27 10 3/4/5 2/2/5 2/2/5 erature of filtration meassurment, °F Substruct of filtration meassurment, °F Substruct of filtration meassurment, °F Substruct of filtration meassurment, °F Substruct of filtration meassurment, °F | weighted Mud (10 lb/bbl sepiolite com lb/bbl 120 12 A, ppb 80 80 33 g temperature, °F 80 80 33 g time, hrs 20 min 20 min 1 Jb/bbl 20 min 20 min 1 Jb/bbl 20 min 20 min 1 jb/bbl 11,00 20 min 1 density, lb/gal 7,20 7,20 7,20 PH(with pH meter) 8,00 7,20 7,20 tion PH (with pH meter) 80 80 80 erature of PH meassurment, °F 80 80 80 G00 95 48 55 300 61 29 34 200 46 21 26 100 29 13 17 6 4 3 4 3 3 2 3 erature of rheology meassurment, °F 80 80 80 <t< th=""></t<> | |

Table I.5: Effect of reactive clays entering into weighted mud containing 10 ppb sepiolite along with high salinity.

| Com | position | Weighted Mud (15 lb/bbl sepiolite concentration) | | | | | | | |
|---|--------------------------------------|--|--------|--------|----------|--------|----------|--|--|
| NaCl | , lb/bbl | | 120 | 120 | | 1 | 20 | | |
| OCMA, ppb | | | | 50 | | 80 | | | |
| Aging temperature, °F | | 80 | 80 | 3 | 350 | 350 | | | |
| Aging time, hrs | | 20 min | 20 min | | 16 | 16 | | | |
| MBT | , lb/bbl | | | 1 | 1,50 | 12,00 | | | |
| Mud | density, lb/gal | | | 12 | 2,50 | 12,85 | | | |
| Mud | PH(with pH meter) | 8,00 | 7,20 | 7 | ,20 | 7,20 | | | |
| Filtra | ntion PH (with pH meter) | | | 8 | ,00 | 8,00 | | | |
| Temperature of PH measurement, °F | | 80 | 80 | 80 | | 80 | | | |
| | 600 | 114 | 56 | 52 | 62 | 79 | 86 | | |
| ng | 300 | 73 | 36 | 33 | 42 | 52 | 58 | | |
| eadi | 200 | 60 | 28 | 26 | 36 | 41 | 47 | | |
| al R | 100 | 40 | 19 | 17 | 25 | 29 | 35 | | |
| Dia | 6 | 9 | 7 | 5 | 12 | 10 | 17 | | |
| | 3 | 8 | 6 | 4 | 11 | 8 | 16 | | |
| Temp | perature of rheology measurement, °F | 80 | 80 | 80 | 120 | 80 | 120 | | |
| PV | | 41 | 20 | 19 | 20 | 27 | 28 | | |
| УР | | 32 | 16 | 14 | 22 | 25 | 30 | | |
| gel strength, 10s./ 1min./ 10min. | | 8/12/19 | 4/5/12 | 3/5/12 | 10/12/23 | 5/7/17 | 13/18/32 | | |
| Temperature of filtration measurement, °F | | | | 300 | | 300 | | | |
| API water loss, cc (7.5/30min)@100 psi | | | | 12/26 | | 9,2/19 | | | |
| cake | thickness, mm | | | 6 | | 7 | | | |

Table I.6: Effect of reactive clays entering into weighted mud containing 15 ppb sepiolite along with high salinity.

| oosition | Weighted Mud (20 lb/bbl sepiolite concentration) | | | | | |
|---|---|--------------------------|--|--|--|--|
| NaCl, lb/bbl | | 120 | 1 | 20 | | |
| OCMA, ppb | | | 50 | 50 | | |
| Aging temperature, °F | | 80 | 350 | 350 | | |
| Aging time, hrs | | 20 min | 16 | 16 | | |
| , lb/bbl | | | 11,50 | 11,50 | | |
| density, lb/gal | 10,90 | | 12,60 | 12,60 | | |
| PH(with pH meter) | 8,00 | 7,20 | 7,20 | 7,20 | | |
| Filtration PH (with pH meter) | | | 8,00 | 8,00 | | |
| Temperature of PH measurement, °F | | 80 | 80 | 80 | | |
| 600 | 126 | 65 | 73 | 83 | | |
| 300 | 84 | 43 | 50 | 58 | | |
| 200 | 66 | 34 | 38 | 48 | | |
| 100 | 44 | 24 | 22 | 36 | | |
| 6 | 10 | 11 | 9 | 19 | | |
| 3 | 8 | 10 | 8 | 18 | | |
| erature of rheology measurement, °F | 80 | 80 | 80 | 120 | | |
| | 42 | 22 | 23 | 25 | | |
| ҮР | | 21 | 27 | 33 | | |
| gel strength, 10s./ 1min./ 10min. | | 6/8/15 | 6/8/18 | 20/25/38 | | |
| Temperature of filtration measurement, °F | | | 300 | | | |
| API water loss, cc (7.5/30min)@100 psi | | | 16/33 | | | |
| hickness, mm | | | | 7 | | |
| | oosition lb/bbl A, ppb g temperature, °F g time, hrs lb/bbl density, lb/gal PH(with pH meter) tion PH (with pH meter) erature of PH measurement, °F 600 300 200 100 6 3 erature of rheology measurement, °F rength, 10s./ 1min./ 10min. erature of filtration measurement, °F yater loss, cc (7.5/30min)@100 psi hickness, mm | ossitionWeighted Ilb/bbl | Weighted Mud (20 lb/bl lb/bbl 120 A, ppb 120 it temperature, °F 80 80 g temperature, °F 80 80 g temperature, °F 80 80 g temperature, °F 80 80 g temperature, °F 80 80 g temperature, °F 80 80 g temperature, °F 80 80 density, lb/gal 10,90 Ptemperature PH(with pH meter) 8,00 7,20 tion PH (with pH meter) 80 80 erature of PH measurement, °F 80 80 G 100 144 24 6 10 11 3 8 10 erature of rheology measurement, °F 80 80 42 22 42 21 rength, 10s./ 1min./ 10min. 11/15/25 6/8/15 erature of filtration measurement, °F 9 6/8/15 water loss, cc (7.5/30min)@100 psi 5 < | Weighted Mud (20 lb/bbl sepiolite cord Ib/bbl 120 1 A, ppb 50 50 g temperature, °F 80 80 350 g time, hrs 20 min 20 min 16 lb/bbl 11,50 20 min 16 lb/bbl 10,90 12,60 12,60 PH(with pH meter) 8,00 7,20 7,20 tion PH (with pH meter) 8,00 7,20 7,20 tion PH (with pH meter) 80 80 80 erature of PH measurement, °F 80 80 80 200 66 34 38 10 200 666 34 38 10 3 8 10 8 80 42 22 23 23 42 22 23 422 21 27 27 27 27 rength, 10s./ 1min./ 10min. 11/15/25 6/8/15 6/8/18 erature of filtration measurement, °F | | |

Table I.7: Effect of reactive clays entering into weighted mud containing 20 ppb sepiolite along with high salinity.

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Author was born in Osku a city near the Tabriz in Iran on May 31, 1979. He started to his education life in Iran. He has received his BSc. degree from Civil Engineering Department of Tabriz University, in Iran as a civil engineer in 2004. He graduated as an honor student from the undergraduate program; however, because of his strong passion in Petroleum Engineering, he moved to Turkey and successfully completed a one-year formal pre-Master's preparation program in order to qualify to enter a Master's program in Petroleum & Natural Gas Engineering, which he currently is attending at Istanbul Technical University since 2007.