AADE-05-NTCE-02

Organo-amine Surfactant Salts as Hydration Suppressants for Reactive Clay

Michael Jarrett and Billy Dye, Baker Hughes Drilling Fluids

Abstract

Organo-amine surfactant salts are gaining wide-spread use as clay inhibitors in water-based drilling fluids. Organo-amine surfactant salts are effective clay hydration suppressants (CHS) and suitable alternatives to halide-containing additives such as sodium and potassium chloride. These materials have also been used to modify clayey soil non-oilfield industries such as agriculture and highway construction.

This paper discusses the mechanisms by which organo-amine additives inhibit reactive clays. Clays have very good sorption properties, which are influenced by the clay's total charge, chemical composition and surface area. Specific surface area and cation exchange capacity are interrelated and control the adsorption rate of clay. The chemistry and charge density of the CHS influences the optimum flocculation point and molecular coverage of the clayey surface.

The paper also discusses the application of organo-amine CHS in stabilizing reactive clays and gumbo when drilling with water-based muds (WBM). The CHS have proven effective in stabilizing reactive clays, providing greater tolerance to solids contamination, reducing dilution rates and improved management of drilling fluid properties. Additionally, these additives are used with increased frequency in drill-in and completion fluid applications to stabilize reactive clays in the reservoir.

Introduction

The hydration and dispersion of reactive clays and gumbo leads to problems such as bit balling, accretion, poor solids removal efficiency, high dilution rates and problems managing rheological and filtration control properties. Clay constitutes a large proportion of shale mineralogy and clay swelling is a leading cause of shale instability. Clay inhibition is managed by limiting water absorption, which leads to swelling and dispersion. Cation exchange within the clay interlayer is an effective and proven mechanism to suppress clay hydration and dispersion. Potassium chloride (KCl) is often used as a cation (potassium) source for drilling young formations containing high concentrations of smectite. KCl reduces the swelling pressure in reactive clays, such as smectite, by reducing the hydration energy and limits the ability of clays to swell.

Clay hydration occurs from surface and ionic hydration. Surface and osmotic absorption result in two distinctly different problems: swelling, which is the expansion of the clays due to water uptake, and dispersion, which is the disintegration of the clay fabric after hydration. The CHS is an environmentally acceptable, water-soluble and high performing alternative to KCl used to stabilize reactive clays and gumbo. The CHS functions in a manner similar to potassium chloride in suppressing clay hydration. This functionality inhibits reactive clays from hydrating and becoming plastic, which provides a secondary benefit of reducing the tendency towards bit balling.

Amine Chemistry

The amino group consists of a nitrogen atom attached by single bonds to hydrogen atoms, alkyl groups, aryl groups, or a combination of these three. An organic compound that contains an amino group is called an amine. Amines are derivatives of the inorganic compound ammonia (NH_3). When one, two, or all three of the hydrogen in ammonia are replaced by an alkyl or aryl group, the resulting compound is known as a primary, secondary, or tertiary amine, respectively. Like ammonia, the amines are weak bases because the unshared electron pair of the nitrogen atom can form a coordinate bond with a proton. Amines will react with a mineral acid to form an amine salt, e.g., with hydrochloric acid to form an amine hydrochloride. A water-insoluble amine can be made to dissolve by adding acid to form its water-soluble amine salt. Amines react similarly with alkyl halides to form alkyl ammonium salts. Table 1 lists some examples of amines that have been used as clay stabilizers in WBM.

Clay Minerals

There are four major types of clay minerals, including layered silicates, oxides, amorphous/allophanes, and chained silicates. This paper will focus on the mineralogy of smectite, illite and kaolinite because they are commonly problematic when drilling with WBM. Silicate clays contain tetrahedral and octahedral layers and a 1:1 layering occurs when one octahedral sheet is bonded to one tetrahedral sheet. Surface charges give kaolinite clays their capacity to absorb cations. An octahedral sheet is bonded to two tetrahedral sheets in 2:1 clay minerals. These clays can be classified as expanding (smectite) or non-expanding clays (illite and micas) on the basis of the sheet where isomorphous substitution is predominantly taking place.[1,2,3]

Clay minerals are colloids and are characterized by a small particle size and large surface area. Clays carry charges on their surfaces, which influences their ability to attract or repulse charge ions to or from surfaces. The two main sources of charge in clay minerals are isomorphous substitution and pH-dependent charges. Charge development on silicate clay is mainly due to isomorphous substitution, where elements are exchanged without changing clay structure. Charges developed as a result of isomorphous substitution are permanent and not pH-dependent. In pH-dependent clays such as kaolinite, charges vary depending on the pH of the clay matrix.

Clay minerals have good sorption properties, which are affected by the number and density of surface charges, as well as pH and specific surface area. Previous work proposed that one could determine specific surface area and cation exchange capacity of clays through adsorption of methylene blue (MB) dye. The charges and surface area of clay minerals control their adsorption rate. The cation exchange capacity (CEC) of clay controls adsorption at low MB levels, while surface area governs adsorption rates at higher MB concentrations.[4]

Water is the most common polar compound present in the interlayer space of smectite clays. Water is intimately involved in the binding and transformation of polar organic compounds at the surface of these minerals. Sorption sites of exchangeable cations are situated between the layers within the clay structure. Clay minerals carry a permanent negative charge in their structural framework due to isomorphous substitution. This charge is balanced by exchangeable cations such as Na^+, K^+, Ca^{2+} and Mg^{2+}. The hydration of these cations imparts a hydrophilic nature to clay surfaces.

Water enters the interlayer region along with these cations. The properties of water are affected more so by the type of hydrated cation rather than the clay surface. Since uncharged polar molecules are absorbed by replacement of the interlayer water, the behavior of such molecules is likewise strongly influenced by the exchangeable cation. Cation-dipole interactions effect the adsorption of polar organic species by clay minerals.

Water uptake and retention by smectite are significantly influenced by the nature of the interlayer cation. Two distinct types of interlayer water are found in smectite crystals. The first type constitutes the inner (primary) hydration shell around the exchangeable cation, when the water is directly coordinated to the cation. The second type forms the outer (secondary) coordination sphere of the cation. This water is more labile that that of the first type because it is indirectly linked to the cation and has greater mobility. The size and valence (polarizing power) of the exchangeable cation play decisive roles in water adsorption.

CEC Measurements

Mineralogical composition and clay fraction content are the main factors governing the swelling behavior of reactive clays such as smectite. However, mineralogical composition requires relatively sophisticated test equipment and procedures, which may not be available for routine measurements. Therefore, more simplistic methods were developed to avoid these difficulties and provide testing procedures equally suitable for use in the laboratory and at the well site.

Methylene blue (MB) adsorption is a measure of the clay specific surface area. Surface area varies with clay type and affects the degree of water adsorption and swelling potential of clay. Surface area measurements are a direct reflection of clay mineralogy, but are an indirect reflection of expansivity.[5,6] Some researchers believe that the cation exchange capacity of clay can be measured by the adsorption of MB from aqueous solutions. Others found a close relationship between MB, ammonium acetate analysis and CEC.[7] Still others studied CEC and surface area using MB with smectite and found a linear relationship with these parameters.[8,9] All of these studies indicate the presence of smectite clay and provide insight into the clay's surface area and CEC. The MB dye test provides a rapid, simple and relatively inexpensive method of determining the CEC of reactive (smectite) clay.[10-13]

Clay Hydration Suppressants

A study was conducted to determine the effectiveness of commercially available amine-based CHS additives on reducing the CEC of Wyoming bentonite clay. Amine-based CHS additives are absorbed on the water-reactive sites and, to varying degrees, reduce the CEC of clay. A slurry of 20 lbs/bbl bentonite in deionized water was prepared and allowed to hydrate overnight. Synthetic filtrates samples, including a control, were treated at increments of 1 – 8 lb/bbl of the CHS.

The following MB method was used:

a. Add 2 ml of the bentonite slurry to a flask.
b. Add 5 ml deionized water.
c. Add 5 ml synthetic filtrate.
d. Stir without heat for 20 minutes.
e. Add 25 ml of 3% hydrogen peroxide.
f. Bring to slow boil and digest for 15 minutes.
g. Conduct MB measurement, using a solution of 1

ml = 0.01 meq (containing 3.20 g USP grade methylene blue per liter).

A standard calibration graph showing residual amine content in a filtrate versus equivalent bentonite concentration determined by MB was plotted, as illustrated in Figure 1. Note that the CHS additive had the greatest effect on lowering CEC compared to other competitive amine-based products (Products A & B).

Clay Hydration Suppression

Bentonite rapidly hydrates and swells when exposed to water and is commonly used in to build viscosity and manage filtration control in WBM. Amine-based CHS reduce the CEC of montmorillonite clay (bentonite and smectite) and it follows that this would affect rheological/filtration control properties of bentonite slurries. Bentonite should be less prone to hydration, thus affecting viscosity and filtration control when pre-treated with an amine-based CHS.

In order to test this assumption, laboratory barrel equivalents (350 ml) of deionized water were pre treated with 7 lb/bbl of CHS. While shearing, 35 lbs/bbl of a commercial bentonite was added and then sheared for 30 minutes. Figure 3 illustrates the resultant rheological and filtration control properties of the treated bentonite slurry and compares them to a control sample. The dial readings of an oilfield 6-speed viscometer were used as relative measures of viscosity.

Ideally, the CHS should block sorption sites on the clay and limit the ability of the clay to imbibe water and hydrate. Clearly the bentonite hydrates and builds viscosity and filtration control in the untreated (control) sample. As shown in Figure 2, the CHS effectively limits clay hydration as evidenced by minimal viscosity increase, whereas the competitive product was only partially effective in managing clay hydration.

Comparing CHS to KCl for Clay Inhibition

Despite its benefits in stabilizing smectite clays, there are several detractors from the use of KCl. Swelling pressures are clay-specific and, while it works well with smectite clays, KCl is only moderately effective in illite and may actually increase swelling in kaolinite clays. Additionally, monovalent cations, such as potassium, are known to have a temporary effect and can be displaced from the clay lattice by other cations. Lastly, the use of KCl is restricted in areas such as the Gulf of Mexico and onshore North America because of toxicity issues related to potassium and chloride ions, respectively.

Clay minerals have large cation exchange capacities, which enable them to be modified to enhance sorption of organic and anionic contaminants.

Referring to one past study, the influence of quaternary ammonium surfactants on sorption of five metal cations (Cs^{+}, Sr^{2+}, La^{3+}, Pb^{2+}, and Zn^{2+}) onto a clinoptilolite zeolite was investigated. Generally, the metal cation sorption capacity and affinity for the zeolite decreased, indicating that pre-sorbed cationic surfactants blocked sorption sites for metal cations, as the surfactant loading on the zeolite increased. Sorption of cationic surfactants on zeolite preloaded with different metal cations showed a strong correlation with the chain length of the surfactant tail group, while the roles of the charges and types of the metal cations were minimal. As the chain length increases, the critical micelle concentration decreases and the surfactant molecules become more hydrophobic, resulting in progressive bilayer coverage. Desorption of pre-sorbed metal cations by cationic surfactants was strongly affected by the surfactant chain length and metal type.[14]

Figure 3 presents a comparison of the CHS and KCl in minimizing the hydration of bentonite. One-bbl aliquots of deionized water were pretreated with increasing increments of salt additive. A concentration of 30 lb/bbl of bentonite were added and then sheared for 30 minutes. Rheological / filtration control properties were measured. Note that 3.5 lb/bbl of the CHS was near-equivalent to 20 lb/bbl potassium chloride in suppressing the hydration of bentonite.

Depletion Rate of CHS

It has been questioned if CHS is consumed by other drilling fluid additives such as PHPA, deflocculants and filtration control products. A study utilizing visible light spectrometry was conducted and showed that residual amine salt in a filtrate could be measuring using visible light and a dye transfer reagent extraction method (Figure 4).

However, measurements of residual amine salt in filtrates from test fluids prepared with various WBM additives showed a “consuming” effect of the amine salt. Spectrometer readings indicated apparent depletions, with results showing that one pound of each product absorbing the amine salt at the levels below:

Chrome lignosulfonate:	1.1 lb amine
Lignite:	1.2 lb amine
Quebracho:	0.7 lb amine
Polymeric defocculant:	0 lb amine
Derivatized starch:	1.9 lb amine
PAC:	4.0 lb amine
PHPA:	2.5 lb amine

Application studies were conducted to determine if there were detrimental effects upon suppression of bentonite hydration. To eliminate any viscosifying effects on data interpretation, deflocculants such lignosulfonates and lignite were evaluated, along with an amine salt. Results showed (in Figure 5) the amine salt to still be available, as evident by suppression of viscosity build-up and increasingly poor filtration control. The CHS was the most effective in suppressing clay hydration.

It is surmised that a "sequestrant effect" of lignite and other additives with the amine salt is being observed, not a "consuming effect". The reaction for amine salt to react with clay is far stronger than the bonding effects with other additives. There is competition for cationic exchange clay sites between amines and additives such as lignosulfonates and tannins. The mechanisms upon these sites though are different: lignosulfonates absorb, whereas amines function through cationic exchange. Amines have a strong hydrogen bonding capability, thus they induce clay flocculation even in the presence of lignosulfonate deflocculants.[15-22]

The Capillary Suction Test (CST) is used to measure the propensity of clay to swell once it is introduced to fresh water. A slurry of test clay or ground shale with distilled water is usually prepared to run this test. The CST measures the time of filtration, or dewatering of the clay slurry. This time is directly related to the sample's swelling potential, and the greater the time, then the higher the swelling potential. CST time can be reduced by adding halide salts such as KCl to the slurry. In samples where the clays are predicted to swell, a salt may be added to the drilling fluid in a percentage determined by the CST to inhibit swelling. This test is applicable for evaluating the depletion rate or consumption of amine salts.[23-28]

Incremental dosages of commercial bentonite were added to deionized water pretreated with 6 lb/bbl of the CHS. Solutions were sheared at 11,000 rpm for 2 minutes. CST measurements were determined, using the small funnel. Figure 6 shows the behavior of an amine salt when increasing the concentration of reactive clay (commercial bentonite). Results showed a depletion rate of 1 pound of the amine additive per 3.5 pounds of bentonite. Typical bentonite has an MBT of 80 lb/bbl equivalent, compared to Gulf of Mexico gumbo having an MBT of 20 lb/bbl. The depletion rate of the CHS in drilling gumbo with WBM is estimated to be roughly 1 pound of the CHS per 15 pounds of gumbo clay. Actual rates will vary based on the salinity of the WBM and mineralogy of the clay drilled.

Field Applications of CHS

The CHS has been used as an integral component of a high-performance water-based mud (HPWBM) on deepwater and shelf wells in the Gulf of Mexico.[29,30] Table 2 presents XRD analyses of highly reactive gumbo clays successfully drilled using the CHS in the HPWBM. CHS concentrations were monitored using a filtrate titration method at the well-site, and engineered so that an excess of the material is available for clay inhibition.

The CHS was used in the HPWBM while drilling 2,543 feet of 12 ¼" hole, with 957 feet (38 %) drilled while sliding and 1,586 feet (62 %) while rotary drilling. Rates-of-penetration ranged from 20 to 200 feet/hour with instantaneous ROP above 300 feet/hour using a rock bit. The average ROP when sliding was 85 feet/hour, while the average ROP when rotary drilling was 94 feet/hour. The solids removal efficiency ranged from 80 - 82 % and four linear motion shakers were able to process flow rates as high as 1250 gallons per minute.

The material was also used in the HWPBM to drill 5,668 feet at an average ROP of 93 feet/hour with a PDC bit on a shelf well in the Gulf of Mexico. Well bore and cuttings stability were characterized as excellent, with three wiper and two round trips being made without incidences of tight hole, fill on bottom or gumbo attacks. The interval was drilled and casing was run without problems. There were no incidents of balling or accretion on tool joints, stabilizers, drilling assembly and the PDC bit.

Conclusions

- Organo-amine surfactants are highly effective clay hydration suppressants in WBM
- The amine-based CHS effectively reduce clay the ability of the clay to imbibe water.
- The CHS is an attractive alternative to halide-containing salts such as NaCl and KCl.
- The CHS has been successfully used as a component of a new HPWBM in deepwater and shelf wells in the Gulf of Mexico.

Acknowledgments

The authors wish to thank Baker Hughes Drilling Fluids for permission to publish this paper.

Nomenclature

CHS = *clay hydration suppressant*
MB = *methylene blue*
lbs/bbl = *pounds per barrel*
CEC = *cation exchange capacity*

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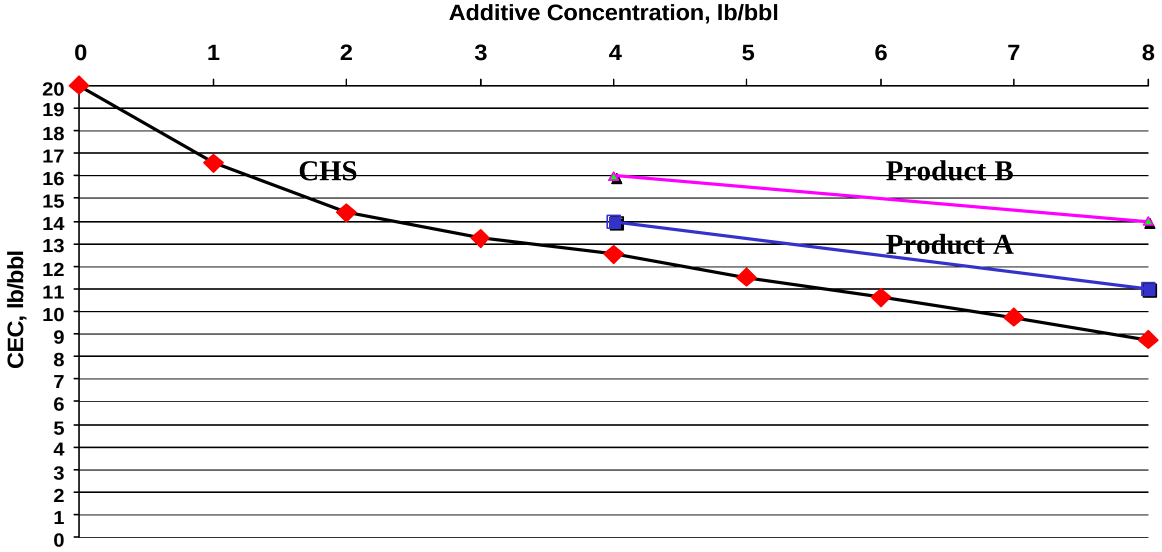


Figure 1: Comparison of commercially available amine-based additives in reducing CEC of bentonite

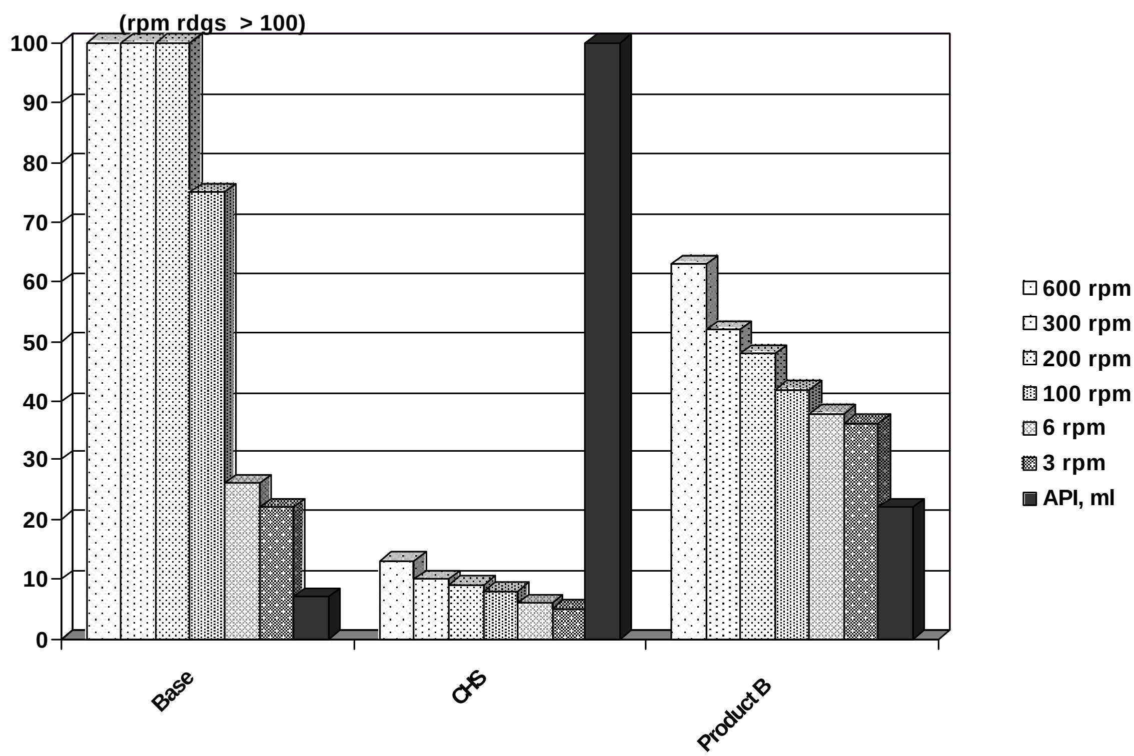


Figure 2: Suppression of Bentonite Hydration

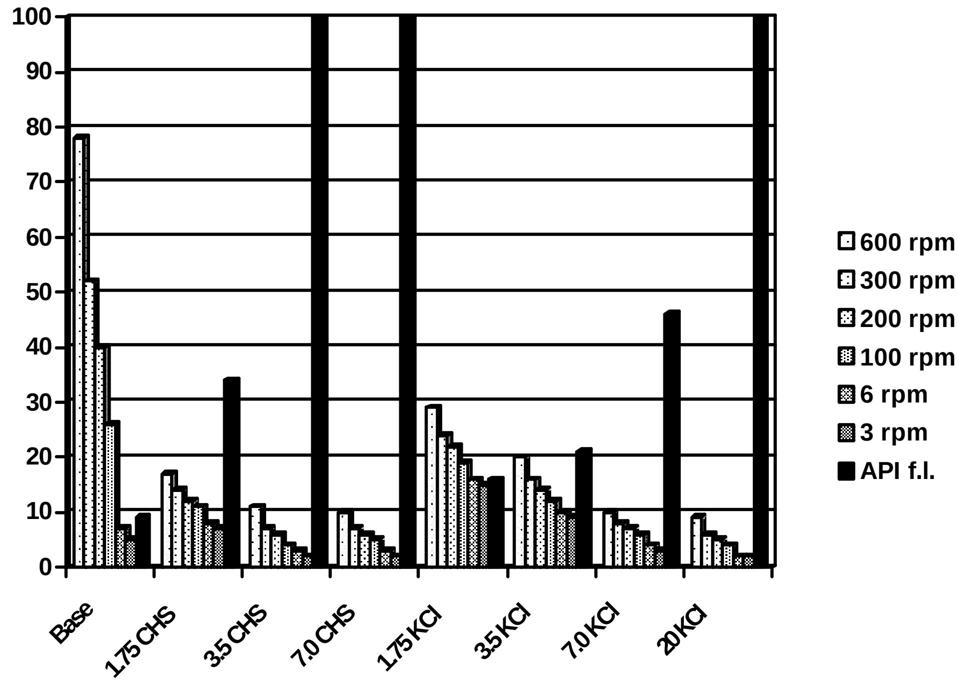


Figure 3: Suppression of Bentonite Hydration – CHS vs. Potassium Chloride (concentration in lb/bbl)

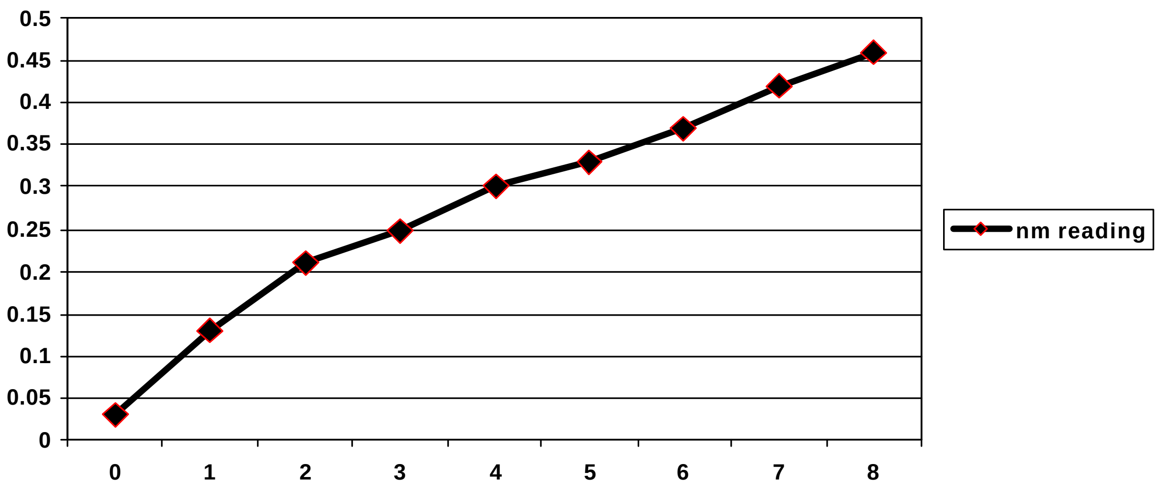


Figure 4: Visible Light Spectrophotometric Results, with increasing CHS

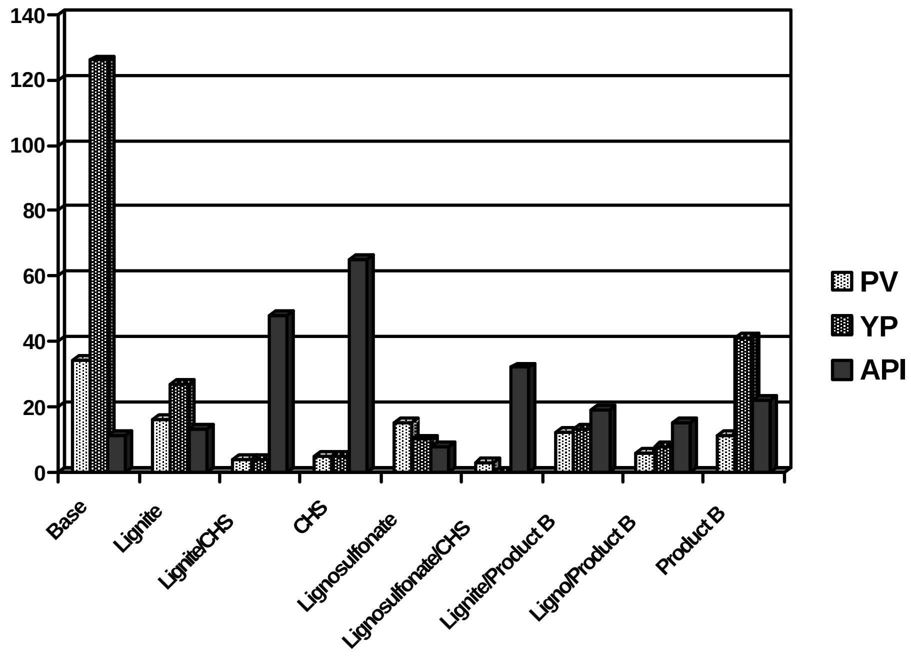


Figure 5: Suppression of Bentonite Hydration, evaluating Lignite, Lignosulfonate and Amines

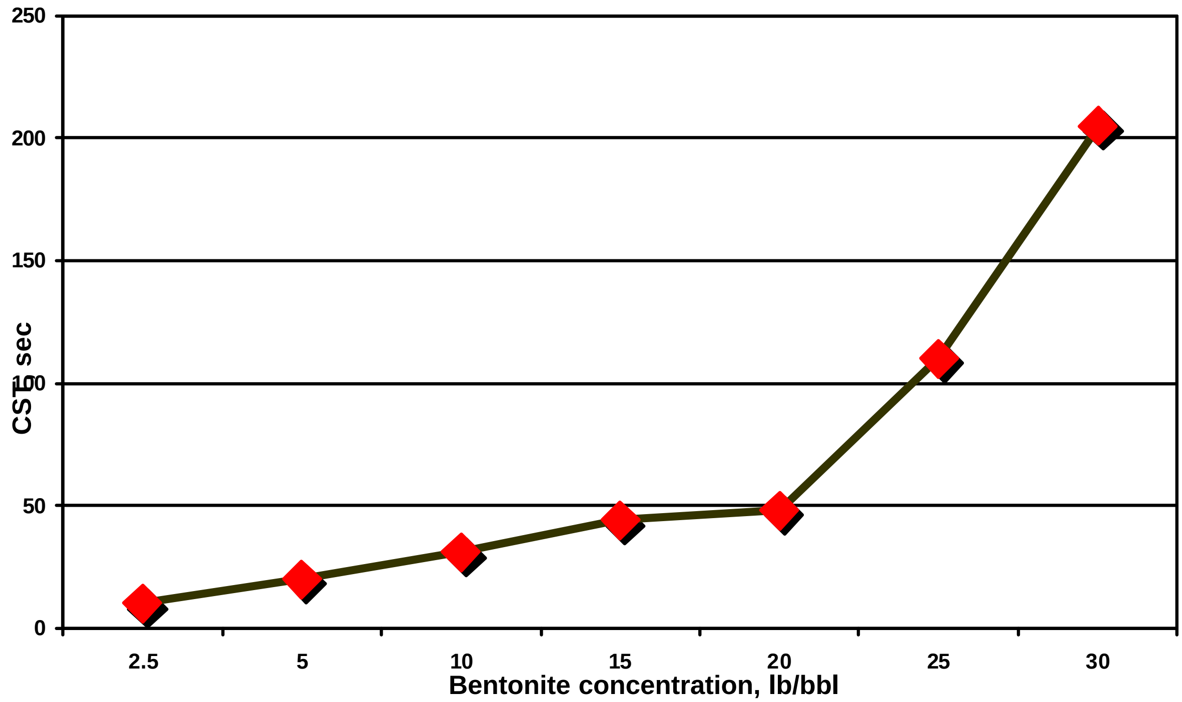
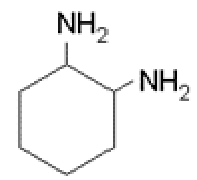


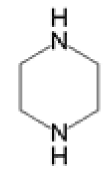
Figure 6: CST Study of Amine Depletion on Reactive Clay

Table 1
Amine-based clay hydration suppressants commonly used in WBM

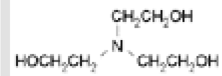
Synonyms	1,2-Cyclohexanediamine
Molecular Formula	$C_6H_{14}N_2$
Molecular Weight	114.19



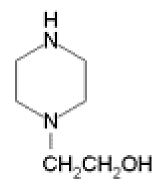
Synonyms	Diethylenediamine
	1,4-Diazacyclohexane
Molecular Formula	$C_4H_{10}N_2$
Molecular Weight	86.14



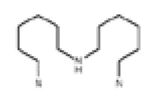
Synonyms	2,2',2''-Nitrilotriethanol
	Tris(2-hydroxyethyl)amine
Molecular Formula	$C_6H_{15}NO_3$
Molecular Weight	149.19



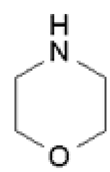
Synonyms	2-Piperazinoethanol
	Piperazine-1-ethanol
Molecular Formula	$C_6H_{14}N_2O$
Molecular Weight	130.19



Synonyms	6,6'-Iminodihexylamine
	Bis(6-aminohexyl)amine
Molecular Formula	$C_{12}H_{29}N_3$
Molecular Weight	215.38



Synonyms	Tetrahydro-1,4-oxazine, morpholine
Molecular Formula	C_4H_9NO
Molecular Weight	87.12



Synonyms	(2-Hydroxyethyl)trimethylammonium chloride, choline chloride
Molecular Formula	$C_5H_{14}ClNO$
Molecular Weight	139.62

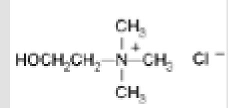


Table 2
X-Ray Diffraction Analysis of GoM Gumbo Drilled with CHS

Quartz	Illite	Mixed Layer Clays	Kaolinite
10 – 15 %	15 – 20 %	45 – 50 % > 95 % expandable	10 – 15 %
20 – 25 %	20 – 25 %	35 – 40 % 100 % expandable	10 – 15 %