

USE OF BORATES AS AN ACTIVATING AGENT  
FOR DRILLING MUD BENTONITES

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FOR DRILLING MUD BENTONITES**

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## **ABSTRACT**

### **USE OF BORATES AS AN ACTIVATING AGENT FOR DRILLING MUD BENTONITES**

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In drilling technologies, Na-bentonite-water mixtures are used as drilling mud. As the reserves of sodium-bentonite decline, calcium or low-quality bentonites are activated with inorganic salts and/or treated with different types of polymers as an alternative to Na-bentonite. The quality of such bentonites, commonly referred to as extended bentonite may deteriorate under severe drilling conditions, such as; bacterial activity, high temperature etc. It was considered that borates could prevent the deterioration of extended bentonites due to their well known bactericide properties. This study involves the use of Na-Borate as an activating agent for bentonites in place of the commonly used Na-carbonate.

The effects of Na-borate on the rheological and filtrate behaviours of two different bentonites, namely, Edirne Bentonite (E-Ben) and Çankırı Bentonite (C-Ben) were investigated. The results were evaluated based on API standards and in comparison to those of Na-carbonate activated bentonites.

It was found that Na-borate could be used as a bentonite activator, although somewhat higher dosages (3,78% Na<sub>2</sub>O equivalent for E-Ben and 0,76% Na<sub>2</sub>O equivalent for C-Ben) than Na-carbonate (2,90% Na<sub>2</sub>O equivalent for E-Ben and 0,58% Na<sub>2</sub>O equivalent for C-Ben) are necessary to obtain the same rheological properties. In terms of aging, it was determined that aging had no adverse effect on

the rheological and filtrate properties of bentonite treated with carboxymethyl cellulose (CMC) and polyanionic cellulose (PAC), but rheological properties of bentonites treated with xanthan gum were found to deteriorate upon aging. The presence of Na-borate in the mud appears to alleviate this adverse effect.

**Keywords:** Bentonite, Borate, Rheology, Filtrate Loss, Gel Strength.

## ÖZ

### **BORATLARIN SONDAJ ÇAMUR BENTONİTLERİ İÇİN AKTİVATÖR OLARAK KULLANIMI**

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Sondaj teknolojisinde, sodyum bentonit-su karışımları sondaj çamuru olarak kullanılmaktadır. Sodyum bentonit rezervleri azalırken sodyum bentonite alternatif olarak, kalsiyum ya da düşük kaliteli bentonitler inorganik tuzlarla aktive edilmekte ve/veya farklı tiplerdeki polimerlerle kimyasal işleme tabi tutulmaktadır. Genellikle katkılı bentonit olarak adlandırılan bu bentonitler, bakteri aktivitesi ve yüksek sıcaklık gibi zor sondaj koşulları altında bozunabilirler. Bakterilere karşı iyi olduğu bilinen boratın, katkılı bentonitlerdeki bozunmayı önleyebileceği göz önüne alınmış ve bu çalışmada bentonitlerin aktivasyonunda yaygın olarak kullanılan  $\text{Na}_2\text{CO}_3$ 'nın yerine sodyum borat kullanılmıştır.

Edirne bentoniti (E-Ben) ve Çankırı bentoniti (C-Ben) olarak isimlendirilen iki farklı bentonitin reolojik ve filtrasyon özellikleri üzerine sodyum boratın etkisi araştırılmıştır. Sonuçlar, API standardıyla ve sodyum karbonatla aktive edilen bentonitlerin sonuçlarıyla karşılaştırılarak değerlendirilmiştir.

Aynı reolojik özellikleri sağlamak için sodyum karbonata (E-Ben için 2,90%  $\text{Na}_2\text{O}$  eşdeğeri ve C-Ben için 0,58%  $\text{Na}_2\text{O}$  eşdeğeri) göre sodyum boratın (E-Ben için 3,78%  $\text{Na}_2\text{O}$  eşdeğeri ve C-Ben için 0,76%  $\text{Na}_2\text{O}$  eşdeğeri) daha yüksek miktarlarda kullanılmasına rağmen bentonitlerin aktivasyonunda kullanılabilirliği bulunmuştur. Çamurların yaşlandırılması durumunda karboksimetilselüloz (CMC) ve poliyonik

selülozlarla (PAC) kimyasal işleme tabi tutulan bentonitlerin reolojik ve filtrasyon özelliklerinde yaşlandırmanın ters bir etkisine rastlanmamıştır. Fakat zantam sakızı polimerlerle kimyasal işleme tabi tutulan bentonitlerin reolojik özelliklerinin yaşlandırma ile bozulduğu bulunmuştur. Çamur içerisinde bulunan sodyum boratın bu ters etkiyi azalttığı gözükmektedir.

**Anahtar Kelimeler:** Bentonit, Borat, Reoloji, Su Kaybı, Jel Mukavemeti.

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## NOMENCLATURE

E-Ben	= Edirne bentonite
C-Ben	= Çankırı bentonite
HV CMC	= high viscosity carboxymethylcellulose
LV CMC	= low viscosity carboxymethylcellulose
PAC R	= high viscosity polyanionic cellulose
PAC L	= low viscosity polyanionic cellulose
600 rpm	= viscosity at a rotor speed of 600 rpm
300 rpm	= viscosity at a rotor speed of 300 rpm
AV	= apparent viscosity
PV	= plastic viscosity
YP	= yield point
YP/PV	= yield point/plastic viscosity
$g_0$	= 10 second gel-strength
$g_{10}$	= 10 minute gel-strength
$\tau$	= shear stress
$\tau_0$	= yield point
$\mu_p$	= plastic viscosity
$\gamma$	= shear rate
K	= consistency index in Pa.s <sup>n</sup> or lb.s <sup>n</sup> /100 ft <sup>2</sup>
n	= the dimensionless flow behaviour index
$\phi_0$	= surface potential
$\phi_1$	= stern potential
$\zeta$	= electrokinetic potential

## CHAPTER 1

### INTRODUCTION

Since ancient times, bentonites have been used in numerous applications, e.g., in the formulation of ceramic products, cement, drilling fluids, paints and paper, due to their large adsorption capacity and swelling characteristics in aqueous suspension (Asselman and Garnier, 2000). These applications are mostly related to their specific physicochemical and rheological properties such as, gel formation, osmotic swelling, yield stress, and viscoelastic and thixotropic behaviour.

Montmorillonite is the most abundant of the smectite clay minerals. A good quality bentonite contains mainly smectites (montmorillonite) and secondary minerals such as quartz, calcite and micas. They come about in numerous forms on the market. In industry, montmorillonite are generally classified as sodium (Na), or calcium (Ca) types, depending on which exchangeable ion is dominant (Hassan and Abdel-Khalek, 1998). In Turkey the vast majority of montmorillonite deposits outside Tokat-Reşadiye are calcium type. Calcium montmorillonites are known to occur in Edirne, Çankırı, Balıkesir, Ordu, Kütahya, Çorum (Akbulut, 1996).

Bentonite has typical characteristics such as negative electric charge, fine size, and very high specific surface, high sensitivity to the hydration, which explains why this clay is employed as drilling mud (Besq et al., 2003). Its main functions in mud are the control of the flow properties and the filtration rate of drilling fluids in drilling operations which are important aspects of drilling fluid technology. Desirable rheological properties include a high shear thinning viscosity -the higher the shear rate, the lower the viscosity-, relatively high gel strength with a high ratio of yield point to plastic viscosity for cuttings transport and suspension (Ward et al., 1999). Besides, low viscosities are desirable in the interest of efficient hydraulic horsepower utilization; low filtration rates imply thin filter cakes, which are desirable in order that annular clearances be restricted to a minimal extend.

Bentonite shall be deemed to meet the physical requirements of American Petroleum Institute (API) Standard in Table 1-1.

Table 1-1 Physical Requirements of Bentonite in API (API 13A, 2004)

<b>Requirement</b>	<b>Bentonite</b>	<b>OCMA</b>	<b>Nontreated Bentonite</b>
<b>Viscosimeter Dial Reading at 600 rpm (lb/100 ft<sup>2</sup>)</b>	30 (min)	30 (min)	-
<b>Dispersed Plastic Viscosity (mPa.s)</b>	-	-	10 (min)
<b>Yield Point/Plastic Viscosity ratio (lb/100 ft<sup>2</sup>/cP)</b>	3 (max)	6 (max)	1,5 (max)
<b>Filtrate Volume, (cm<sup>3</sup>)</b>	15 (max)	16 cm <sup>3</sup> (max)	12,5 (max)

Bentonites can be used in their crude form or after various chemical treatments in order to bring out a particular quality to meet performance requirements. If the rheological properties of a bentonite for drilling application are insufficient, generally, bentonites are treated. For example, extenders (inorganic salts or polymers) may be added to make certain grades (Olphen, 1977).

A class of water soluble polymers is added to a drilling-grade clay mineral during grinding to enhance the clay's rheological performance. Among the various functions of soluble polymers, two are important: the control of the rheology of the liquid phase and the reduction of fluid loss (Simon et al 2002). Water soluble polymers, such as starch, carboxymethylcellulose (CMC), polyanionicellulose (PAC) and xanthan gum, the most suitable polymers in classical drilling operations, are frequently used as viscosifiers and fluid-loss reducers (Asselman and Garnier, 2000). There are two theoretical models for fluid loss control in drilling muds. In the first theory, long chain polymers form an impermeable filter cake by attaching themselves onto the surface of individual solids particles and achieving filtration control by a bridging mechanism. According to the second model the filtrate

reducing polymers are located in the pores of the bentonite filter cake and are plugging the pore spaces resulting in filtration control (Audibert et al., 1999; Plank and Hamberger, 1988).

There are three adsorption mechanisms between clay and polymer during the interactions of negative charge-carrying clay particles with the additives. Firstly, the ionic exchange takes place between the  $\text{OH}^-$  ions of the clay particles faces and the anionic part of the polymer. Secondly, hydrogen bonds can form between clay particles and surfactant molecules. A third possibility is the existence of polyvalent cations in bentonite, which can serve as an electrostatic bridge between the clay surfaces and polyanions (Benchabane and Bekkour, 2006; Yalçın et al., 2002; Güngör and Karaoğlan, 2001).

The long-term performance of the extended bentonite will certainly not be as good as that of the pure, high quality bentonite. Under severe drilling conditions (high temperatures, presence of bivalent cations, high bacterial activity, etc) the extended bentonite can show an unexpected deterioration in quality (Bol, 1986). Bacterial (Chatterji and Borchardt, 1981) and thermal (Thomas, 1982) degradation of the polymers is the most important problem with their use.

Most of the Ca or Na/Ca bentonites are activated with inorganic salts (such as  $\text{Na}_2\text{CO}_3$ ). Due to ion-exchange, the original Ca or Na/Ca-bentonites are converted to Na-bentonite by  $\text{Na}_2\text{CO}_3$ . But the addition of inorganic salt lead to opposed behaviour (increasing or decreasing values of yield stress and apparent viscosity) depending on the amount of salt employed (Besq et al., 2003).

Na-borate, also an inorganic salt, contains not only  $\text{Na}^+$  ion but also boron. In this study borate were thought as an alternative for  $\text{Na}_2\text{CO}_3$ . In the literature, borate was used to treat guar gum to form a complex that inhibits hydration above pH 7 (Chatterji and Borchardt, 1981). But up to now it has not been used as an extender on the activation of bentonite for drilling application.

In this experiment a calcium bentonite from Edirne (E-Ben) and a sodium bentonite from Çankırı (C-Ben) were activated with  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  mixture, separately. Besides, polymers were used in the treatment of raw and activated bentonites as viscosifiers ((HV CMC, PAC R and Xanthan Gum) and fluid-loss reducer (LV CMC, PAC L and Dextrid).



## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Clay Chemistry**

##### **2.1.1 Geologic Origin of Clay**

Clay minerals are ubiquitous on earth, both in the continents and on the ocean floors. In particular, they are concentrated in geological formations where water-rock interactions are active. Their crystal structure and chemical composition are a response to the destabilization of minerals formed in higher pressure-temperature conditions when altered in different types of environments including soils, weathered rocks, geothermal systems, burial diagenetics series, etc. (Meunier, 2006). During the weathering process, clays are degraded by the leaching of ionic potassium, magnesium, iron, aluminium, and silicon. As the weathered clays are deposited, the ions may re-enter the crystal lattice reconstituting the original minerals, sometimes with accompanied crystal growth. The same ion is not always replaced during process.

The upper limit of the particle size of clays is defined by geologists as 2  $\mu\text{m}$ , so that virtually all clay particles fall within the colloidal size range. Montmorillonite particles may be as large as 2  $\mu\text{m}$  and small as 0,1  $\mu\text{m}$  in diameter with average sizes of  $\sim 0,5 \mu\text{m}$ . The particles are of irregular shape (Lagaly and Ziesmer, 2002). As they occur in nature, clays consist of a heterorogeneous mixture of finely divided minerals, such as quartz, feldspars, calcite, pyrites, etc., (Gray and Darley, 1981).

##### **2.1.2 Clay Minerals and Their Structure**

According to the clay-mineral concept, clays generally are composed of extremely small crystalline particles of one or more members of a small group of

minerals which have come to be known as the clay minerals. The clay minerals are essentially hydrous aluminium silicates, with magnesium or iron proxying wholly or in part for the aluminium in some minerals and with alkalis or alkaline earths present as essential constituents in some of them (Grim, 1968). The term “clay” refers to any material which exhibits a plastic behaviour when mixed with water. Thus, clay minerals will be, e.g., kaolinite, illite and smectite but not, e.g., micas, talc and pyrophyllite which occur in much larger particle sizes (Giese and Oss, 2002).

The structural framework of the clay minerals is basically composed of layers comprising silica and alumina sheets joined together and stacked on top of each other. Two structural units are involved in the atomic lattices of most clay minerals. One unit is named the octahedral sheet. It consists of two sheets of closely packed oxygens and hydroxyls in which aluminium, iron or magnesium atoms are embedded in octahedral coordination, so that they are equidistant from six oxygens or hydroxyls. The second unit is built of silica tetrahedrons and is named the tetrahedral sheet. In each tetrahedron, a silicon atom is equidistant from four oxygen atoms, or hydroxyls if needed to balance the structure. The silica tetrahedral groups are arranged to form a hexagonal network, and is repeated indefinitely to form a sheet of composition  $\text{Si}_4\text{O}_6(\text{OH})_4$ .

The analogous symmetry and the almost identical dimensions in the tetrahedral and octahedral sheets allow the sharing of oxygen atoms between these sheets. In the case of the ‘three-layer minerals’ or smectite clay minerals, one alumina or magnesia sheet shares oxygen atoms with two silica sheets, one on each side. The combination of an octahedral sheet and one or two tetrahedral sheets is called a unit layer. Most clay minerals consist of unit layers that are stacked parallel to each other. A schematic representation of the atom arrangements in a unit cell for a three-layer clay, such as montmorillonite is shown in Figure 2-1. Within the unit layer the unit structure repeats itself in a lateral direction. This unit structure is also called a unit cell.

The unit layers are stacked together face-to-face to form what is known as the crystal lattice. The distance between a plane in the unit layer and the corresponding plane in the next layer is called basal spacing or c-spacing,  $d_{001}$ . This repetition distance of the layers in the stack is very important. Its determination from X-ray diffraction patterns immediately enables one to distinguish between two-layer

clays with a c-spacing of about 7.1-7.2 Å and three-layer clays with a c-spacing of at least 9.2 Å. The sheets in the unit layer are tied together by covalent bonds, so that the unit layer is stable. The layers in the lattice layer are held together only by Van der Waals forces and secondary valences between juxtaposed atoms (Luckham and Rossi, 1999).

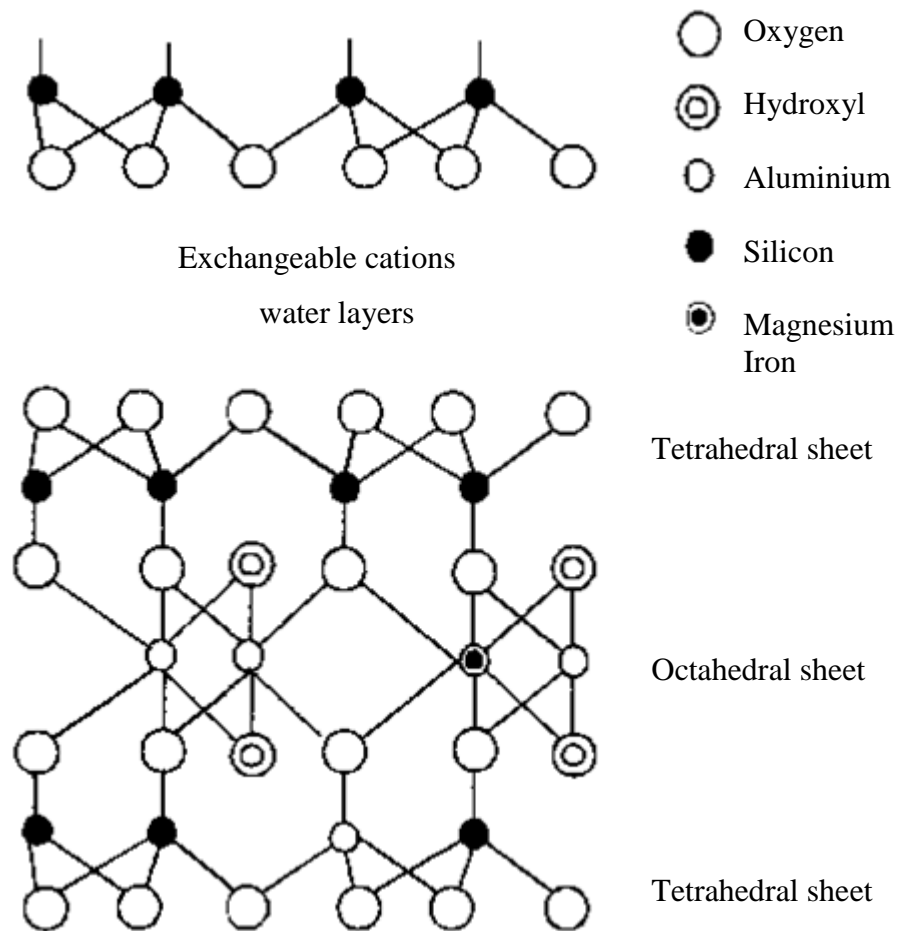


Figure 2-1 The Layer Structure of Montmorillonite (Luckham and Rossi, 1999)

Clay mineral crystals carry a charge arising from isomorphous substitutions of certain atoms in their structure for other atoms of a different valence. For example, in silicate tetrahedron sheet  $\text{Si}^{4+}$  may be replaced by trivalent cations ( $\text{Al}^{3+}$  or  $\text{Fe}^{3+}$ ) and in the octahedron alumina sheet divalent cations ( $\text{Mg}^{2+}$  or  $\text{Fe}^{2+}$ ) may replace  $\text{Al}^{3+}$ , respectively. These substitutions leave a net negative charge on the face of the sheet

which is balanced by a cloud of cations are termed exchangeable because they are easily replaced by other cations in the environment (Williams and Underdown, 1981). The total amount of cations adsorbed on the clay, expressed in milliequivalents per hundred grams of dry clay, is called the cation exchange capacity (CEC), or the base exchange capacity (BEC). The cation exchange capacity is high for sodium montmorillonite, comparing it to the other clay minerals (Luckham and Rossi, 1999).

### **2.1.3 Types of Clay Minerals and Montmorillonite**

The most important types of clay minerals are: illites, kaolinites, attapulgites, chlorites and smectites. The first two types consist of plate-like particles that do not have an expanding lattice; therefore no water can penetrate between the layers. In illites, this is due to the strong interlayer bonding from the high layer charge, and in the case of kaolinite is due to the strong hydrogen bonding. Attapulgite particles consist of bundles of 'laths', which separate into individual laths (long shaped needles) when mixed vigorously with water. Consequently, the rheological or flow properties of attapulgite suspensions are dependent on mechanical interference between the long laths, rather than on electrostatic interparticle forces. Chlorites differ from the above in that they possess a positive charge on one layer, balanced by an additional negative charge. Thus, normally there is no interlayer water.

The smectite 2:1 structural units are three-layer clay minerals in which one tetrahedral sheet of one unit layer is adjacent to another tetrahedral sheet of another layer. They are classified as 2:1 phyllosilicates. In this case, the oxygen atoms are opposite one another and bonding between the layers is weak. Also there is a high repulsive potential on the surface of the layers resulting from isomorphous substitution. These two last factors contribute to the increase of the c-spacing between the layers due to the penetration of water. Thus smectites have an expanding lattice, where all the layer surfaces are available for hydration and cation exchange. Interlayer surface and cation hydration between smectite structural units is a unique property of smectite clays (Luckham and Rossi, 1999).

The montmorillonite structure is classified as dioctahedral, having two thirds of the octahedral sites occupied by trivalent cations (such as  $\text{Al}^{3+}$ ). Dioctahedral montmorillonite has its structural charge originating from the substitution of  $\text{Mg}^{2+}$

for  $\text{Al}^{3+}$  in the octahedral sheet. The negative charge is balanced by cations intercalated between the structural units and these cations may be alkaline earth ions ( $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ ) or the alkali metal  $\text{Na}^+$ . When  $\text{Na}^+$  cations are exclusively in exchange with the montmorillonite surface, the clay is known as  $\text{Na}^+$ -montmorillonite. The expanding lattice may provide the clay with a specific area of as high as  $800 \text{ m}^2/\text{g}$ . The chemical formula for  $\text{Na}^+$ -montmorillonite is  $\text{Na}_{0.33}[(\text{Al}_{1.67}\text{Mg}_{0.33})(\text{O}(\text{OH}))_2(\text{SiO}_2)_4]$  (Luckham and Rossi, 1999).

#### 2.1.4 Swelling of Montmorillonite

All natural clays hydrate or sorb water when in aqueous suspension and water films are formed on the particle surfaces.

In the structure of montmorillonite, only a relatively small proportion of the inorganic cations balancing the negative layer charge are located at external crystal surfaces. The majority of these cations are present in the interlayer space between the clay platelets. The thin, negatively charged sheets are held together by the electrostatic forces between alternate layers of bridging cations (typically  $\text{Na}^+$ ).

Swelling is due to the penetration of water along the silicate surfaces of the mineral, thereby increasing the cleavage spacing between adjacent sheets of clay. When dry montmorillonite is placed in liquid water, the water penetrates between the crystallites by capillary process induced by hydrogen bonding between the silicate surface and the water. When sufficient water penetrates between the crystallites the base exchange cations begin to electrolytically dissociate from the silica surface (Browning and Perricone, 1963).

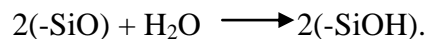
Water sorption by expandable clay minerals is generally accompanied by an increase in basal spacing. An increase in basal spacing with the hydration energy of the saturating cation:  $\text{Cs}^+ < \text{Rb}^+ < \text{K}^+ < \text{Na}^+ < \text{Li}^+ < \text{Ba}^{2+} < \text{Sr}^{2+} < \text{Ca}^{2+} < \text{Mg}^{2+}$ . The amount of adsorbed water is influenced by the exchangeable cation i.e, smaller, more hydrated, cations ( $\text{Na}^+$  and  $\text{Mg}^{2+}$ ) increases water sorption compared with larger cations ( $\text{K}^+$  and  $\text{Ca}^{2+}$ ) (Dontsova et al., 2004). For sodium bentonite, the interlayer spacing may increase abruptly up to  $30\text{-}40 \text{ \AA}$  and continues to increase to several hundred Angstroms with water content. If the exchangeable cations are calcium, however, they do not freely dissociate from the silica surface of the bentonite

crystallites; consequently, they exert little repulsive force between the bentonite particles, and interlayer hydration is limited to 2 or 3 molecular layers of water (Malfoy et al., 2003).

### 2.1.5 Interactions between Clay Platelets

The charge of a clay basal surface mainly depends on the isomorphous changes occurring in the clay crystal. For example, the  $\text{Si}^{4+}$  cation can be substituted by  $\text{Al}^{3+}$ ,  $\text{Mg}^{2+}$ , etc. As a result the crystal structure gets a negative charge that is compensated by exchangeable cations in dry conditions. In presence of water the exchangeable cations dissociate, thus the clay crystal surface gets negatively charged and the electrical double layer (EDL) is formed. Theoretically the EDL can be represented as a “condenser” where the negatively charged inner layer of the condenser is built up by the clay surface and positively charged outside layer is formed by the exchangeable cations.

When a clay crystal edge interacts with water,  $(-\text{SiO})$  –groups are hydrated and form  $(-\text{SiOH})$ -groups on the crystal edge surface:



In basic conditions the formed poly-silicon acid partially dissociates:



The produced  $\text{H}^+$  ions (or  $\text{H}_3\text{O}^+$ ) transit to the aqueous phase, but under the electrostatic attraction of  $(-\text{SiO}^-)$ -groups they remain nearby the crystal surface.

The concentration of  $\text{OH}^-$  and  $\text{H}^+$ -ions in the solution effects the degree of the  $(-\text{SiOH})$ -group dissociation and thus determines the potential of the silicon surface (i.e. clay edge charge). An increase of the  $\text{OH}^-$  ions concentration causes increasing the silicon surface negative potential and thus stabilizes clays in aqueous suspensions.

If  $\text{Si}^{4+}$  is isomorphically changed with  $\text{Al}^{3+}$ , the type of the  $(-\text{AlOH})$ -group dissociation depends on the pH. In basic conditions the  $(-\text{AlOH})$ -group dissociates by the acid type ( $\text{Al}(\text{OH})_3 \longleftrightarrow \text{Al}(\text{OH})_2\text{O}^- + \text{H}^+$ ), and in acid condition - by the base type ( $\text{Al}(\text{OH})_3 \longleftrightarrow \text{Al}(\text{OH})_2^+ + \text{OH}^-$ ). In the second case the edges of the clay particles get positively charged (Tchistiakov, 2000).

### 2.1.6 Clay Particle Electrical Double Layer Structure

In clay-water systems, a double layer is made up of the negative surface charge and the balancing cation charge. The hydration reduces the strength of bonds between a clay surface and exchangeable cations. While part of the cations are electrostatically attracted by oppositely charged surface and form the adsorbed cation layer (or so-called Stern layer), another part of the cations transits at some distance from the clay surface and forms the diffuse ionic layer (Figure 2-2, a). The distribution of the dissociated cations near a clay particle surface is determined by the balance between electrostatic attraction of the clay surface and thermal motion of the cations, tending to spread the cations away from the surface and equalize their concentration in the solution.

Consequently the concentration of the dissociated cations decreases with the distance from the particle attaining the concentration of the similar cations in the solution (Figure 2-2, b). The concentration of the anions on the contrary decreases in the direction towards the surface.

The clay surface charge is balanced by an equivalent number of oppositely charged cations in adsorption and diffusion layers. The part of the clay surface (or thermodynamic) potential, which is compensated by the adsorbed cation layer, is called consequently the adsorption potential. The remaining part of the potential, at the boundary between adsorbed and diffusion layers, is called Stern potential (Tchistiakov, 2000).

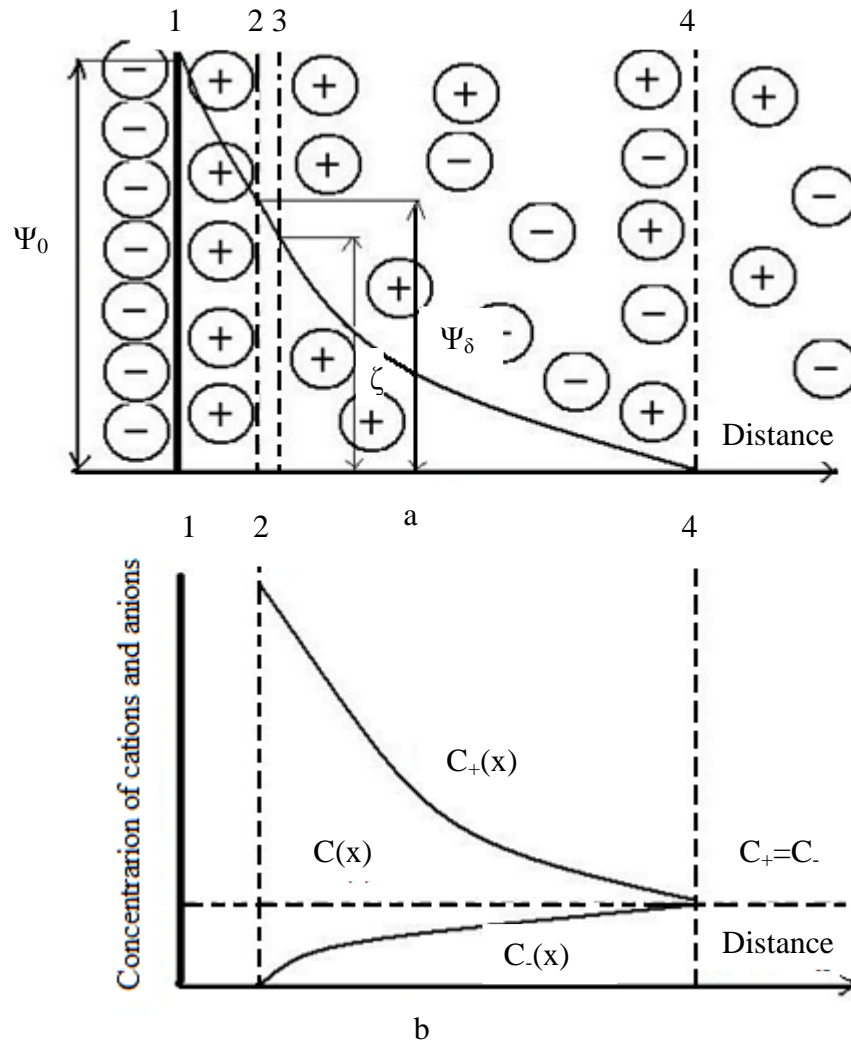


Figure 2-2 The Structure of the Diffuse Electrical Double Layer. ( $\Psi_0$  - surface potential;  $\Psi_\delta$  - Stern potential;  $\zeta$  - electrokinetic potential; 1- clay surface; 2- Stern plane; 3- plane of shear, between a particle and fluid; 4- boundary between a diffuse layer and free solution) (Tchistiakov, 2000).

Diffuse character of the counter-ion atmosphere was recognised by Gouy and Chapman who presented a theoretical treatment of the counter-ion distribution. Their theory predicts an approximate exponential decay of the electrical potential  $\Psi$  of the solution with distance  $x$  from the plane surface. This is represented by the following equation:

$$\Psi = \Psi_0 \exp(-\kappa x) \quad (2.1)$$



where  $\Psi_0$  is the surface potential and  $\kappa^{-1}$  the extension or thickness of the double layer. The above expression is valid for a certain distance from the charged surface, where the potential is relatively low and decreases exponentially

$$1/\kappa = \exp(\epsilon_0 RT / F^2 \sum_{i=1}^n c_i z_i^2) \quad (2.2)$$

where:  $F$  is the Faraday constant,  $c_i$ - the electrolyte concentration,  $z_i$ - the valence of the ion,  $\epsilon_0$ - the dielectric constant of the medium,  $R$ - the gas constant (1 atm/mol K) and  $T$ - the absolute temperature (K).

Eq. (2.2) shows that as the electrolyte concentration increases the thickness of the double layer ( $\kappa^{-1}$ ) decreases or is compressed. The surface charge of the clay particles does not change with increasing electrolyte concentration, simply the diffuse double layer is compressed. Besides, the thickness of the diffuse double layer decreases with decreasing hydrated ion radius. Since the radius of hydrated  $\text{Ca}^{2+}$  is smaller than that of  $\text{Na}^+$ , the thickness of the  $\text{Ca}^{2+}$ -montmorillonite double layer is smaller than  $\text{Na}^+$ -montmorillonite (Luckham and Rossi, 1999). As a result, the degree of compression of the double layer is governed by the concentration and valence of ions of opposite sign from that of the surface charge. The higher the concentration and the valence of the ions of opposite sign, the more the double layer is compressed.

The interaction between charged particles is governed predominantly by the overlap of their diffusion layers, so the Stern potential is often considered as the most relevant to the interaction. Nevertheless there are no direct methods for determining Stern potentials. So far instead of the Stern potential the electrokinetic potential has been often used for calculations. The electrokinetic or  $\zeta$ -potential is that at the plane of shear, between a particle and a fluid, when there is relative motion between them (e.g. due to electrophoretic clay transport or electroosmotic fluid flow). Consequently the  $\zeta$ -potential of clays can be found by means of electrophoresis or electroosmosis techniques. In spite on the fact that the shear plane does not coincide precisely with the adsorption layer boundary (the Stern plane),  $\zeta$ -potential is assumed that is equal to the Stern potential.

The edge of the clay particle is assumed to possess an electrical double layer of a different nature to that of the flat surface described above. This double layer is

due to the adsorption of potential determining ions on the broken bonds of the tetrahedral silica sheets and octahedral alumina sheets. There is a strong possibility that in a neutral clay suspension a positive double layer is created on the edge surfaces owing to the exposed alumina sheet, whereby it may become more positive with decreasing pH and its sign may be reversed with increasing pH. The value of the point of zero charge for montmorillonite is equal to pH 6,5 (Tchistiakov, 2000). Lagaly and Ziesmer (2002) stated that the position of the point of zero charge of the edges is still uncertain but there are several colloid chemical arguments that must be near pH~5 for montmorillonites.

### **2.1.7 Particle Association in Clay Suspensions**

If the concentration of clay is high enough, flocculation will cause the formation of a continuous gel structure instead of individual flocks. The gel structures build up slowly with time, as the particles orient themselves towards positions of minimum free energy under the influence of Brownian motion. The concentration of clay present in the system and the salt content are decisive factors in the length of time required for a gel to attain maximum strength.

When a suspension of plate-like clay particles flocculates, three different modes of particle association may occur: face-to-face (FF), edge-to-face (EF) and edge-to-edge (EE). The electrical interaction energy for the three types of association is governed by three different combinations of the two double layers. Also, the rate of diffusion of the particles as they approach each other in these three ways is not the same, and they may not occur simultaneously or to the same extent when the clay suspension is flocculated. The various modes of particle association are illustrated in Figure 2-3. FF association leads to thicker and larger flakes, and EF and EE association lead to three-dimensional voluminous 'house-of-cards' structures (Olphen, 1977).

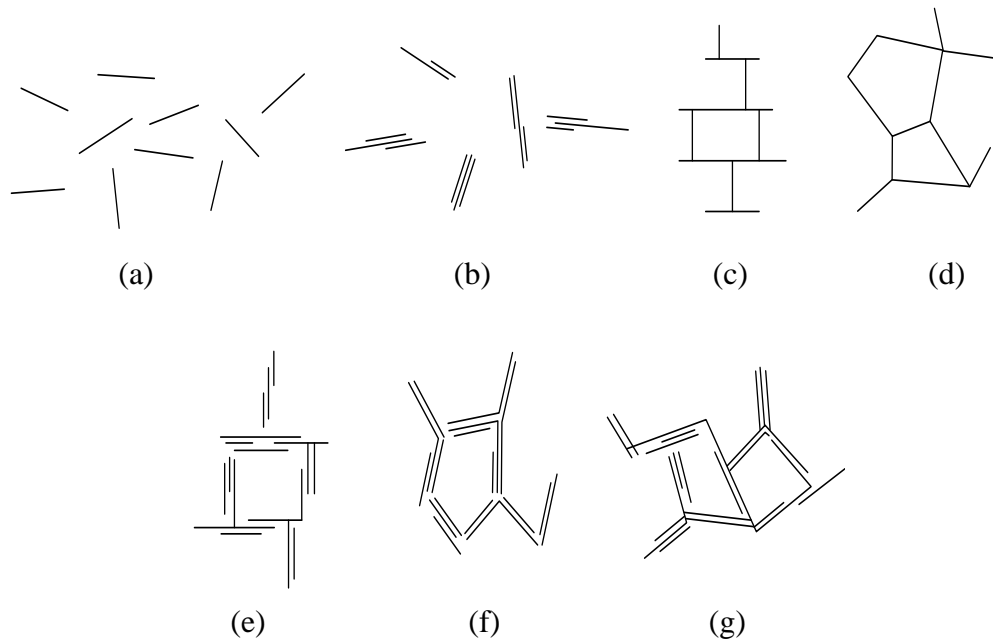


Figure 2-3 Models of Particle Association in Clay Suspensions: (a) dispersed and deflocculated; (b) aggregated but deflocculated (FF association) ; (c) EF association but dispersed; (d) EE flocculated but dispersed; (e) EF flocculated and aggregated (f) EE flocculated and aggregated; (g) EF and EE flocculated and aggregated (Olphen, 1977).

Face-to-face association (aggregation) refers to the collapse of the diffuse double layers and the formation of aggregates of parallel platelets spaced 20 Å or less apart. It decreases the number of particles and causes a decrease in viscosity. Dispersion, the reverse of aggregation, leads to a greater number of particles and to higher viscosities.

Edge-to-edge or edge-to-face association is a flocculation process and causes an increase in viscosity.

There are various linkages and plate orientations proposed to account for gel structure. Van Olphen (1977) suggested that cross-linking between parallel plates through positive edge to negative surface linkages, to form a house-of-cards structure. Norrish opposed Van Olphen's theory of the house of cards structure, suggesting that the gel formation in montmorillonite suspensions was due to long-range electrostatic double-layer edge-to-edge (E-E) and face-to- face (F-F) repulsions

(Laribi et al., 2006). This theory is substantiated by the fact that the area of the edge which constitutes ~5% of the total surface area is small (Rabaloli et al., 1993). Therefore the electrostatic attraction between the edge and face is small compared to the repulsion between the two faces when two particles approach each other (Luckham and Rossi, 1999). Benna et al. (1999) made yield point measurements for sodium montmorillonite at various pH. They found that in the range of pH around the isoelectric point, the yield stress decreases when  $\text{pH} > \text{pH}_{\text{iep}}$  (pH of the isoelectric point) and increases sharply when  $\text{pH} < \text{pH}_{\text{iep}}$ . This can be explained by the fact that at pH values less than the  $\text{pH}_{\text{iep}}$ , all the edges are positively charged and the mechanism by which the three-dimensional network is formed in the gel could be connection between edges and faces (card-house structure). At  $\text{pH} > \text{pH}_{\text{iep}}$  all the edges are negatively charged, which leads to the breakdown of the card-house structure and the electrical double layers are not very compressed.

## 2.2 Water Soluble Polymers

Different types of polymers are used by drilling industry to design a drilling mud to meet expected functional requirement such as appropriate mud rheology and fluid loss control property. These polymers can be classified as synthetic or natural polymers. Water-soluble polymers, such as, starch, guar gum, xanthan gum, carboxymethyl cellulose (CMC) and their derivatives are used extensively in drilling fluids as viscosifiers and fluid-loss reducers. The selection of the best polymer for a given application depends on both economic and performance consideration. Polymer solution properties that affect performance include solution viscosity in the solvent to be used, the influence of shear rate on viscosity, temperature stability, the effect of solution pH, and the tendency of the polymer to adsorb on the formation. Also, compatibility of the polymers with other solution to undergo bacterial degradation also can be very important. The presence of both cellulose-producing and hemicelluloses-producing bacteria can cause polymer degradation. Increasing the solution pH to about 12 inhibits the growth of bacteria and consequent polymer degradation (Chatterji, 1981).

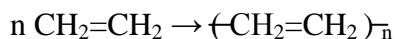
Stability of a particular polymer is a time dependent concept. The context of polymer usage must be considered before defining an upper temperature limit for the

given polymer in the desired application. The temperature limit will be higher for a short-term application than for a long-term application. The limiting temperature values quoted are for short-term applications.

### 2.2.1 Polymer Definition

Ethylene gas ( $\text{CH}_2=\text{CH}_2$ ), under proper chemical conditions, combines with hundreds or thousands of other ethylene molecules to form a solid product called polyethylene, with a molecular weight in the millions. This conversion of ethylene to polyethylene is called polymerization – the chemical combination of many small molecules to form a very large molecule.

The term polymer is derived from Greek words “polys” meaning many and “meros” meaning parts or units. The simple molecules from which polymers are formed are called monomer (“mono” meaning one) – i.e., a single unit. Monomer has at least two reactive sites at which other monomer units can be joined. The two reactive sites in ethylene are the carbon-carbon double bond. Thus, the polymerization of ethylene may be written as

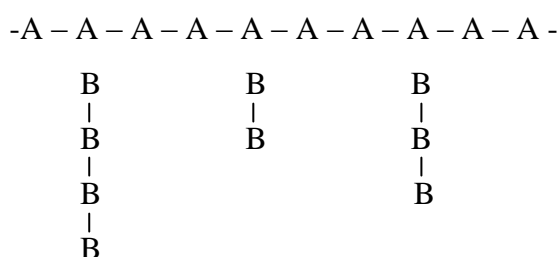


The number of monomer units,  $n$ , present in a polymer may vary from two to millions. The value of  $n$  often is referred to as the degree of polymerization.

The molecular weight of a polymer controls not only physical but also chemical properties. The molecular weight is proportional to the degree of polymerization – i.e., the number of monomer units present in a polymer. Molecular weight always implicitly refers to an average molecular weight of all the macromolecules in the polymer sample. As a general rule for anionic polymers the low molecular weights behave as deflocculants, the medium behave as filtration controllers and the high molecular weight anionics act as viscosifiers. Also, as general rule thermal stability decreases with increasing molecular weight (Clements et al., 1985).

Polymer containing repeat units (-A-A-A-A-A-A-) of only one chemical composition are termed “homopolymers” while those with two or more chemically different types of repeating units (-A-A-B-A-B-B-A-) are termed “copolymers”.

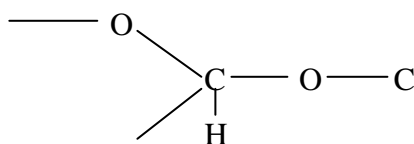
These polymers are called linear polymers because the macromolecule is a long single chain of monomer units. A graft copolymer is one in which long chains of a homopolymer are attached at various, generally random, points to the backbone of a long chain of a different homopolymer, as depicted in structure (A, B= monomer units):



This type of polymer is called a branched polymer because polymer chains branch off from each other.

Water-soluble polymers – whether naturally occurring or synthetics, whether homopolymers or copolymers, whether linear or branched – have certain common structural features. These molecules are all polar.

The typical natural polymers used in the oil field are characterized by an acetal linkage in the polymer repeat unit. In the acetal linkage, a carbon atom is bonded with a single chemical bonds to two oxygen atoms while also being bonded to at least one hydrogen atom as depicted;



In a polysaccharide, the acetal carbon atom of one monosaccharide unit is linked by way of oxygen to a nonacetal carbon atom of another monosaccharide unit to form a polymer chain (Chaterji, 1981).

In polysaccharide derivatives, two terms – DS and MS – frequently occur. MS is the total number of moles of a chemical substituted on a mole of polysaccharide, while DS refers to the number of substituted hydroxyl groups. The building block of the cellulose polymer chain is the anhydroglucose unit (a

dehydrated glucose molecule). There are three hydroxyl groups (-OH) on each anhydroglucose unit. Theoretically, all three hydroxyl groups can undergo substitution by various chemical reactions. The product of such a reaction would have a maximum degree of substitution (DS) of three. The molar substitution (MS) is defined as the total number of moles of a chemical reagent attached to all three hydroxyl groups of an anhydroglucose unit.

### **2.2.2 Starch**

Starch is the second most abundant biomass found in nature, next to cellulose. Starch, as used in drilling fluids, is a powder that disperses readily in water to give a low-viscosity fluid that can be used to seal microfractures and prevent fluid loss. This starch has been processed after separation from corn, wheat, rice, or potatoes. The approximate chemical formula is  $(C_6H_{10}O_5)_n$ , with n varying from a few hundred to a over million. When starch is heated with water the colloidal suspension that results may be separated into two major components: chemically amylose (a linear polymer) with a molecular weight in the range of 100,000-500,000 and amylopectin (a highly branched polymer) with a molecular weight in the range of 1-2 million (Figure 5-4) (Amanullah, 2004).

The thermal stability of starches does not exceed 107 °C (225 °F). Acid hydrolysis rapidly decomposes starch molecules. Long-term exposure to alkaline solutions also leads to degradation: however, alkaline solutions are far more stable than acid ones ((Thomas, 1982). Starch and starch derivatives, particularly hydroxyethyl starch and carboxymethyl starch, have been used as fluid-loss additives in drilling muds (Chatterji and Borchardt, 1981).

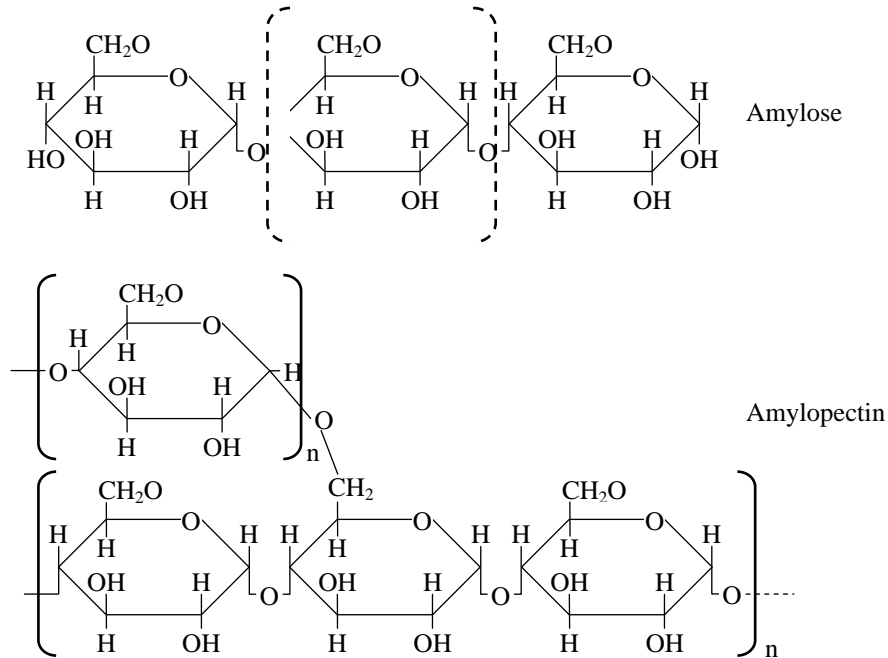


Figure 2-4 Structure of Starch-Based Polymers

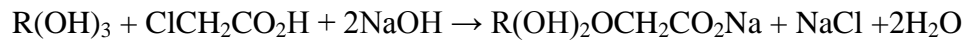
### 2.2.3 Cellulose and Cellulose Derivatives

Cellulose is the most abundant of all the natural polymers and probably comprises at least a third of all vegetable matter in the world. Only a dry basis, wood contains 40-50% cellulose.

Cellulose is a linear molecule formed by the polymerization of a simple sugar – glucose. Due to very strong intermolecular and intramolecular hydrogen bonding, cellulose is insoluble in water. Water is unable to break these hydrogen bonds and enter into association with cellulose to dissolve it. Thus, water soluble derivatives are required for drilling fluid applications.

To prepare water-soluble cellulose derivatives, cellulose is treated with an aqueous solution of a strong base, usually sodium hydroxide. This results in separation and swelling of polymer particles. The larger space between polymer chains allows facile penetration of chemical reagent. Cellulose is modified to form the sodium salt of carboxymethyl cellulose (CMC) by reaction with monochloroacetic acid in the presence of caustic soda shown in Figure 2-5 (Hughes, 1993)





Purification is accomplished by washing with an alcohol/water mixture properly proportioned to extract salt and other by products without dissolving the CMC contain only small amounts of NaCl (Thomas, 1982).

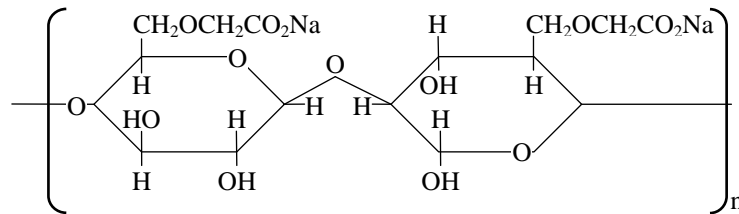


Figure 2-5 Chemical Structures of Cellulose and Sodium CMC

NaCMC is water-soluble, while cellulose is insoluble in water. The solubility changes with the degree of substitution (DS). CMC of low DS (0,2 to 0,3) is soluble in aqueous alkali. As the DS is increased, the solubility increases. Commercial CMC'S are generally in the DS range of 0,5 to 0,85 but may vary from 0,4 to 1,5. Standard CMC's used in the oil field have a DS of about 0,7 to 0,9. Maximum viscosity and best stability occur at a pH of 7 to 9 in fresh water.

Sodium carboxymethyl cellulose is preferred to starch for applications in other than high-pH and salt-saturated muds. It is an anionic polymer and is adsorbed on clays. Filtration is sharply reduced by low concentrations of CMC, and especially by the higher molecular weight (and higher viscosity) products.

The apparent viscosity decreases with rise in temperature. Thermal degradation of CMC is accelerated as temperature approaches 149 °C (300 °F).

Carboxymethyl cellulose is commonly supplied either as low-viscosity (CMC LV) or high-viscosity (CMC HV) grades, both of which have API specifications. The weight-averaged molecular weight increases systematically from the low- to the high-viscosity grade of the CMC samples. The weight-averaged molecular weight of the CMC samples varies from 57 200 to 227 700 g/mol (Hughes 1993).

Figure 2-6 shows the chemical structure of polyanionic cellulose (PAC). One characteristic of PAC is a degree of substitution for the carboxymethyl group of greater than 0,8.

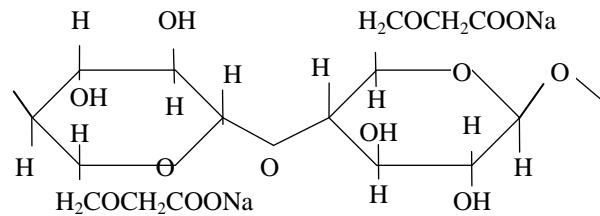


Figure 2-6 Chemical Structures of Polyanionic Cellulose

As water-soluble polymer, it dissolves immediately in cold/hot water and can be used as a good drilling mud additive. It is a good filtrate reducer with the capacity of increasing viscosity. The temperature stability of PAC is 149 °C and is not subjected to bacterial degradation (Mahto and Sharma, 2004). Its calcium tolerance, however, is limited to <2000 ppm calcium ions (Plank and Gossen, 1991).

PAC is available in two types (high or low viscosity grade), both of which impart the same degree of fluid loss control but different degrees of viscosity. The short chain products act as deflocculants and give good filtration control. The longer chain versions give viscosity as well as filtration control.

#### 2.2.4 Xanthan Gum

Xanthan gum, also known as XC polymer, is a high-molecular-weight biopolysaccharide produced by the fermentation of bacterium *xanthomonas campestris*. It can give interesting properties to the drilling fluids (Benchabane and Bekkour, 2006). The polymer is produced commercially by growing the bacteria by a fermentation process, precipitating the gum in alcohol, and then drying and milling the product to a powdered form (Figure 2-7) (Carico and Bagshaw, 1978). Xanthan gum is soluble in cold and hot water. It is useful as a viscosifier and suspending agent. The molecular weight of xanthan gum is approximately 2 million g/mol.

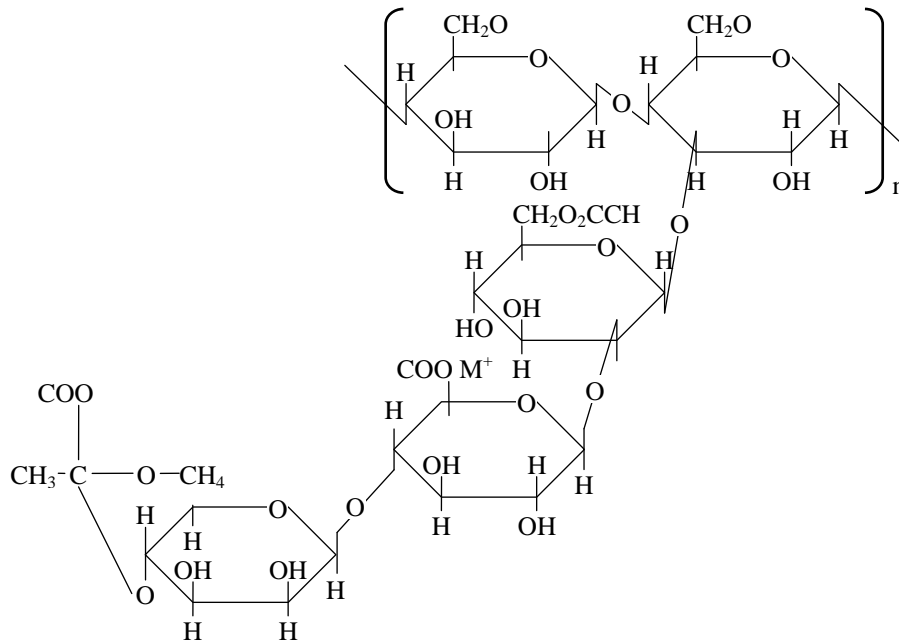


Figure 2-7 Repeating-unit Structure of Xanthan Gum

Xanthan gum solution is non-Newtonian and highly pseudoplastic. The apparent viscosity changes significantly when different shear stresses are applied. Under certain conditions the thermal stability of xanthan gum against hydrolysis is superior to many other water-soluble polysaccharides in general. Xanthan gum is stable over a board range of pH values and is fully biodegradable (Katzbauer, 1997).

Xanthan gum solutions are susceptible to bacterial degradation, and a bactericide is recommended if the solution is stored for more than 24 hours (Chatterji and Borchardt, 1981).

### 2.3 Clay Polymer Interaction

Clay-polymer interactions are important in a wide variety of applications including water treatment, stabilisation of soil structure, paper coating and drilling fluids (Breen, 1999).

Generally, for industrial applications, bentonite powders are treated by polymers to make certain grades. The interactions that take place between the bare clay particles in suspension alter the rheological behaviour of clay-polymer suspensions. A number of parameters affect the extent of interaction between clay particles and polymers. These are particle size, shape, surface charge of clay particles, solid-water ratio, polymer type, degree of hydrolysis of polymer, concentration and molecular weight of polymer, pH and temperature (Alemdar et al., 2005b). With little quantity of polymers, the flow properties can be strongly modified. For this reason, it is important to understand the mechanism by which polymer adsorption occurs. The models of interaction can be distinguished: ion-dipole interaction, hydrogen bonding, Van der Waals forces and entropy effects.

### 2.3.1 Ion-Dipole Interactions

The saturating cation on the clay surface (ex-changeable cation) serves as adsorption sites for polar non-ionic molecules by ion-dipole interactions. The greater the affinity of the exchangeable cations for electrons is, the higher the energy of interaction with polar groups of organic molecules is capable of donating electrons. Thus, the nature of the cation is of prime importance. This type of interaction may take place from one of two forms depending on the hydration of the clay and of the nature of the exchangeable cation.

Exchangeable cation directly influences the bonding strength between the polymer and the smectite. Figure 2-8 shows interactions or coordination between the exchangeable cations and the amide groups. The amide group ( $\text{CONH}_2$ ) is dipolar. The ion-dipole interaction mainly occurred between the carbonyl oxygen and the exchangeable cation through  $\text{CO} \cdots \text{M}^{n+}$  bonding, where  $\text{M}^{n+}$  is the exchangeable cation. The amino group ( $\text{NH}_2$ ) has also contributed to ion-dipole interaction or coordination through the  $\text{H}_2\text{N} \cdots \text{M}^{n+}$  bonding, but this interaction was less important than the  $\text{CO} \cdots \text{M}^{n+}$  bonding. The alkaline earth cations  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  have higher charge densities than alkali cations  $\text{Na}^+$  and  $\text{K}^+$ , therefore,  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  had

stronger ion-dipole interactions with the amide group than  $\text{Na}^+$  and  $\text{K}^+$ . Stronger ion-dipole interaction weakened the bond strengths of the N- H and the C = O bonds (Deng et al., 2006).

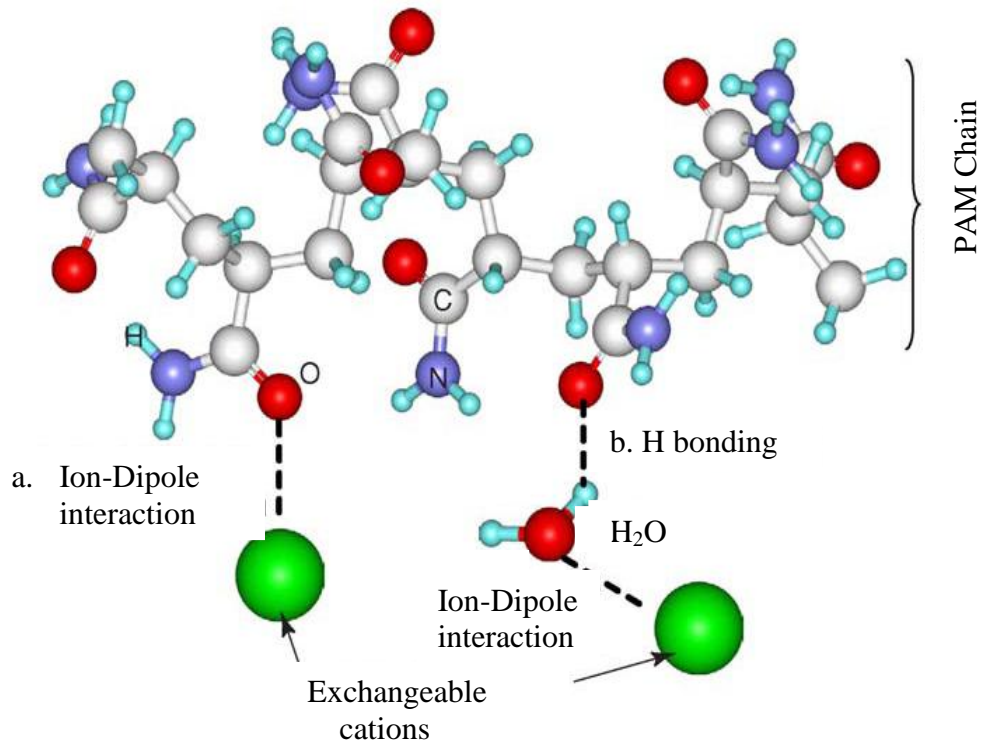


Figure 2-8 Conceptual Model of Major Bonding between a PAM Chain and Exchangeable Cations in the Interlayer of Smectite, (a) ion-dipole interaction or coordination and (b) H-bonding of amide groups with water molecules in the hydration shells of exchangeable cations (Deng et al., 2006).

### 2.3.2 Hydrogen Bonding

This type of bonding occurs by the direct interaction between the adsorbed molecule and the oxygens or hydroxyls of the clay mineral surface. Infrared analysis of clay-organic complexes indicated that basal oxygens of clay surfaces are very weak electron donors and they are incapable of forming this type of hydrogen bond. When water molecules are present in the interlayer, the exchangeable cations are

hydrated. Therefore, the polymers might react with the exchangeable cations through water bridging. The amide groups of the polymer form H-bonds with water molecules in the hydration shells of the exchangeable cations (Figure 2-8b) (Deng et al., 2006).

### **2.3.4 Van der Waals Forces**

Van der Waals or physical forces operate between all atoms, ions or molecules, but are relatively weak. They result from the attraction between oscillating dipoles in adjacent atoms and decrease very rapidly with distance between the interacting species. In clay organic compounds containing polymers of large molecular weight, these attractive forces become quite significant.

### **2.3.5 Entropy Effects**

The adsorption of an uncharged, flexible linear polymer onto the clay surface generally leads to the desorption of numerous solvent (here water) molecules from the surface Figure 2-9. The entropy so gained provides the driving force for adsorption because the enthalpy change of the process is commonly very small and may even be positive (Theng, 1982). This results in an overall increase in translational entropy contributing to the free energy of adsorption. Thus, the adsorption of polymers, especially high molecular weight species, is largely an ‘entropy-driven’ process.

Adsorption also leads to a change in conformation in that the polymer, which in solution tends to exist as a random coil, uncoils and spreads out at the solid/solution interface. Thus, the surface conformation is one in which contiguous sequences of adsorbed segments or “trains” alternate with three-dimensional “loops” extending from the surface, and the polymer chain terminates in two free-dangling “tails” (Breen, 1999).

(a)

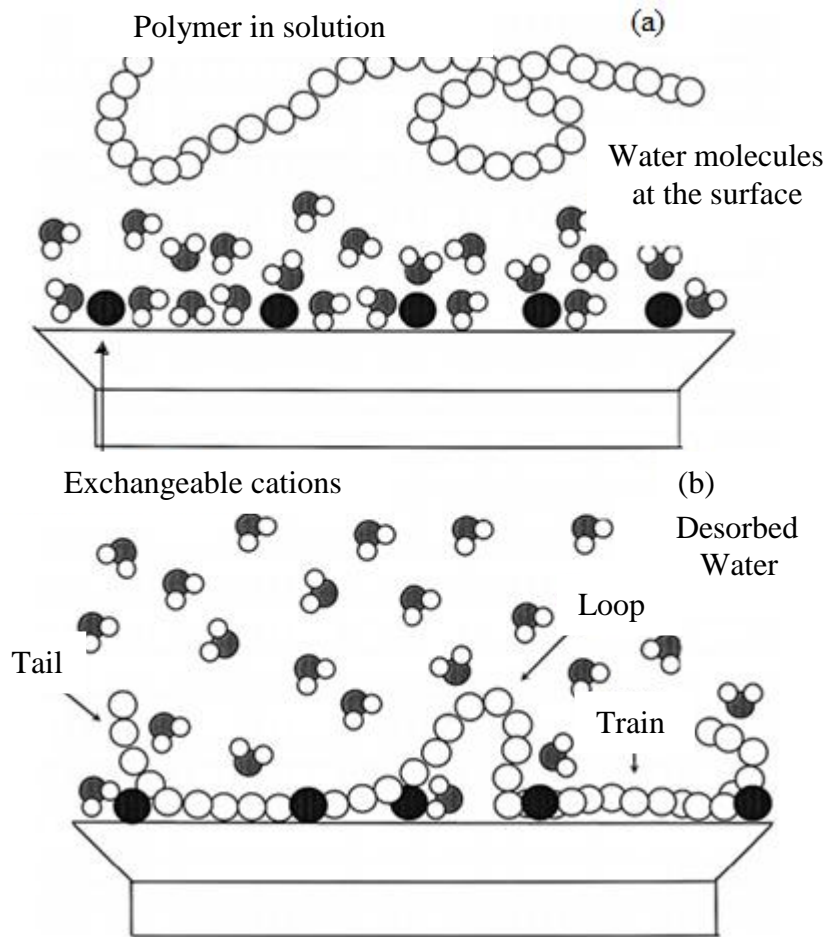


Figure 2-9 Adsorption of Polymers on a Clay Surface as an “Entropy-driven” Process (Breen, 1999)

## 2.4 The Rheological Behaviour of Bentonite Suspensions

Rheology is the science of deformation of materials (if they are solid), or of their flow (if they are liquid) under applied stress (Monicard, 1982).

Rheological data can be evaluated and used to control fluid flow. The major applications of rheological properties for evaluating drilling fluid behaviour are important in the following determinations: (1) hole cleaning and hole erosion, (2) suspension of cuttings, (3) hydraulic calculations, and (4) mud treatment (Chilingarian and Vorabutr, 1981).

The flow behaviour of any system is described in terms of the relationship between the shear stress ( $\tau$ ) and the shear rate ( $\dot{\gamma}$ ). The action of a force (i.e., a stress,

e.g., pump pressure) causes a fluid to flow (i.e., the resulting strain, pump output). The amount of force applied to a fluid determines the shear rate is determined by the flow rate of the fluid through a particular geometrical configuration. Resistance of the fluid to the applied rate of shear or force is called the shear stress, which is analogous to the pump pressure. Physically, the share rate relates to particles or planes of the fluid moving or sliding past one another as illustrated in Figure 2-10 which depicts a fluid flowing in a pipe. In this case, the fluid velocity is 1,0 cm/sec at a distance of 1,0 cm from the wall of the pipe. In as much as, theoritetically, at the pipe wall the velocity will be zero, the fluid velocities will vary between 0 to 1 cm/sec. The ratio of velocity to distance from the pipe wall (velocity gradient) is called the shear rate. If velocity is expressed in cm/sec and the distance in cm, then the units of shear rate is in 1/sec ( $\text{sec}^{-1}$ ). The magnitude of the rate depends on the geometrical configuration of the pipe (or annulus, i.e., area between two concentric pipes), the overall velocity of the fluid, and the viscous properties of the fluid.

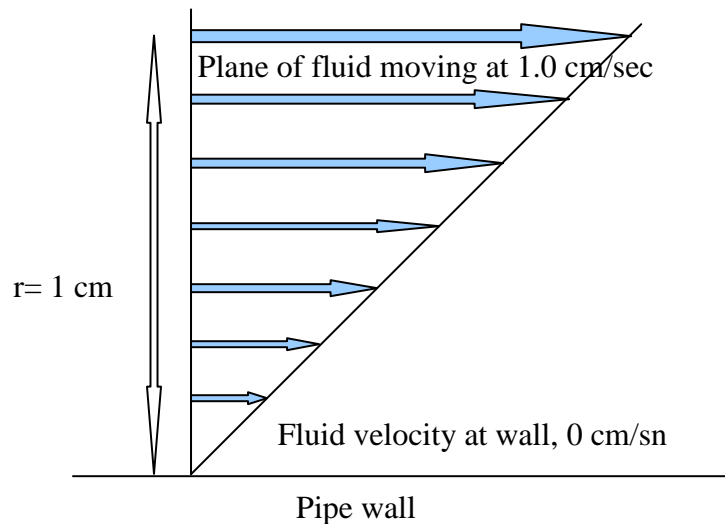


Figure 2-10 A Schematic Diagram of the Velocity Gradient in a Flowing Fluid Assuming a Straight-line Relationship, i.e.,  $\gamma = v/r = \text{constant}$ , which in case is equal to  $1.0 \text{ sec}^{-1}$ .



The shear rate,  $\gamma$ , is defined as follows:

$$\gamma = \frac{dv}{dr} = \frac{\text{Velocity difference between two adjacent layers}}{\text{Distance between the two layers}} \quad (2.3)$$

The shear stress,  $\tau$ , is defined as follows

$$\tau = \frac{F}{A} = \frac{\text{applied force}}{\text{surface area}} \quad (2.4)$$

The plot of the shear stress vs. the shear rate is called a consistency curve. Four different types of flow may be distinguished: Newtonian, pseudoplastic, Bingham plastic, and dilatant, as illustrated in Figure 2-11. Only the first three are of interest in drilling fluid technology.

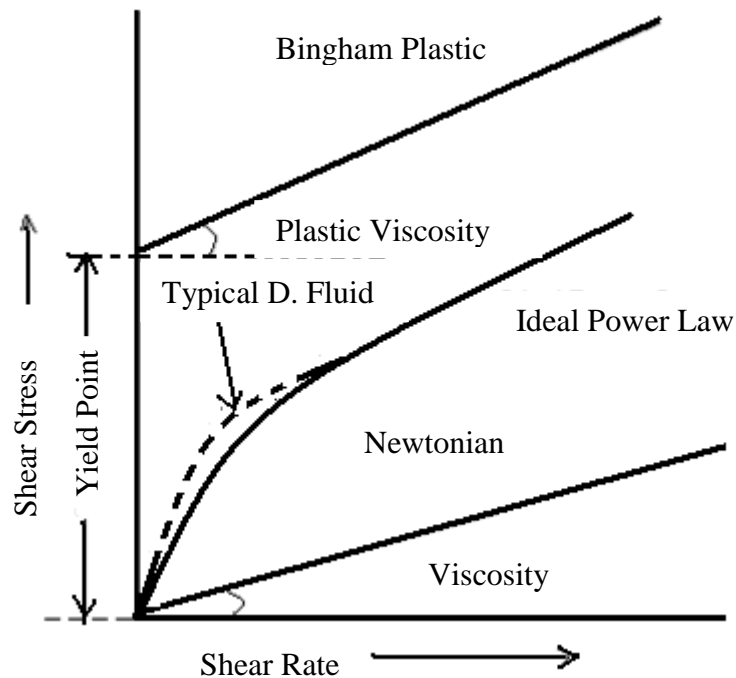


Figure 2-11 Ideal Consistency Curves for Common Flow Models.

In general, the flow behaviour of clay suspensions depends on clay concentration, particle size, shape, and the strength of the interactions among clay particles. When there is little or no interaction between the clay particles in an aqueous suspension is Newtonian in behaviour. The shear stress of Newtonian fluids is directly proportional to the shear rate: if one variable is doubled, the other one is doubled also. Water, glycerine, and light oil are examples. The rheological equation is,

$$\tau = \mu \dot{\gamma} \quad (2.5)$$

The graph is a straight line passing through the origin; the fluid begins to move as soon as a nonzero force is applied.

For a Newtonian fluid, the ratio  $\mu = \tau / \dot{\gamma}$  is constant at constant temperature and pressure, and is the viscosity.

Most drilling fluids are not Newtonian; the shear stress is not directly proportional to shear rate. When the clay particles interact, the flow of the suspension is non-Newtonian in behaviour. Drilling fluids are shear thinning when they have less viscosity at higher shear rates than at lower shear rates. This non-Newtonian behaviour is due to the in-resistance to flow either from electrical forces of attraction and repulsion of the charged surfaces of the solids, or from purely mechanical resistance to flow of the particles and the fluids (Annis, 1967). Two types of non-Newtonian fluids are most often encountered in drilling muds; Bingham Fluids and Pseudo-plastic or power-law fluids. Rheological modelling of drilling fluids in the field is usually described by Bingham plastic or the power law model. Although these models are fairly easy to solve for specific descriptive parameters, they do not simulate fluid behaviour across the entire rheological spectrum very well, particularly in the low shear rate range (Hemphill et al., 1993).

In Bingham plastic fluids the shear stress also varies linearly with shear rate but, unlike Newtonian fluids, a minimum force must be applied to impart motion to them. This force is known as the yield-point or yield-stress. The yield stress and the flow behaviour of clay suspensions are both sensitive indicators of clay particle interactions. They are related to the degree of association between clay platelets, and

can be used as an index of particle-particle association strength (Heller and Keren, 2002).

Such fluids are characterized by two constants:

- i- Yield-point,  $\tau_o$ , which corresponds to the smallest force required to set the fluid in motion.
- ii- Plastic viscosity,  $\mu_p$ , which is the ratio between the increment in the shear stress and corresponding increment in the shear rate, i.e., the slope of the curve obtained by plotting  $\tau$  as a function of  $\gamma$ .

The equation for the Bingham model is:

$$\tau = \tau_o + \mu_p \gamma \quad (2.6)$$

In Cartesian coordinates, this is a straight line.

Determination of plastic viscosity and yield point are carried out in a Fann viscometer. The values to be determined are the shear stress  $\tau_{1020}$  at the shear rate  $\gamma_2 = 1020 \text{ s}^{-1}$  (at 600 rpm), and the shear stress  $\tau_{510}$  at the shear rate  $\gamma_1 = 510 \text{ s}^{-1}$  (at 300 rpm). These values are illustrated by the Figure 2-12.

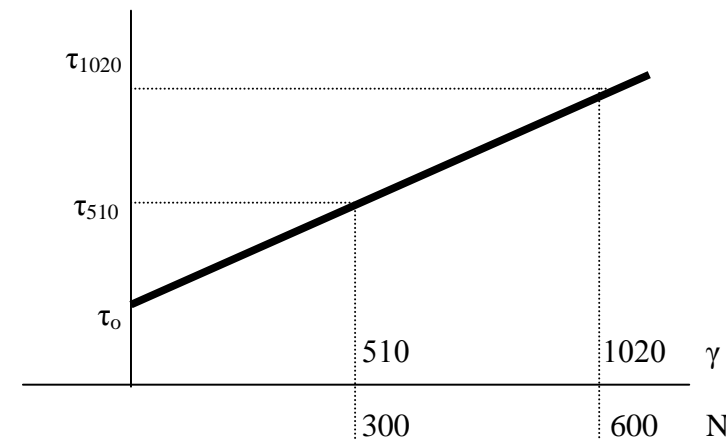


Figure 2-12 Determination of Plastic Viscosity and Yield Point

- Determination of the apparent viscosity  $\mu_a$ .

$$\mu_a = \text{Fann reading at 600 rpm } (\theta_{600})/2 \quad (2.7)$$

$\mu_a$  is expressed in centipoises.

- Determination of the plastic viscosity  $\mu_p$ .

$$\mu_p = \text{Fann reading at 600 rpm } (\theta_{600}) - \text{Fann reading at 300 rpm } (\theta_{300}) \quad (2.8)$$

$\mu_p$  is expressed in centipoises.

- Determination of yield point  $\tau_o$ .

$$\tau_o = \text{Fann reading at 600 rpm } (\theta_{600}) - 2\mu_p \quad (2.9)$$

$$\tau_o = \theta_{600} - 2(\theta_{600} - \theta_{300}) \quad (2.10)$$

$\tau_o$  is expressed in lb/100 ft<sup>2</sup>.

In practice, the most common use of plastic viscosity (PV) and yield point (YP) is for the wellsite evaluation of drilling mud performance, particularly as a guide to maintenance treatments. Thus the PV is sensitive to concentration of solids, and therefore indicates dilution requirements; the YP is sensitive to the electrochemical environment, and hence indicates the need for the chemical treatment (Gray and Darley, 1981).

Pseudo plastic fluids have a nonlinear relationship between shear stress and shear rate without a yield stress. Very dilute clay suspensions or drilling fluids that contain polymers behave as pseudoplastic fluids, which may be described by the power-law equation.

$$\tau = K\dot{\gamma}^n \quad (2.11)$$

where K is the consistency index in Pa.s<sup>n</sup> or lb.s<sup>n</sup>/100 ft<sup>2</sup>, and n is the dimensionless flow behaviour index, which is unity or smaller than unity.

If n=1, the equation becomes identical with the equation of flow of a Newtonian fluid having the viscosity.

Other models have been considered in describing the rheological behaviour of clay suspensions, such as the Casson equation:

$$\tau^{1/2} = k_o + k_I \gamma^{1/2} \quad (2.12)$$

and the yield-power law model or the Herschel-Bulkley equation

$$\tau = \tau_0 + K\gamma^n \quad (2.13)$$

In both cases, the suspension has an initial yield stress at low shear rates, and afterwards presents pseudoplastic or ‘shear-thinning’ type behaviour at higher shear rates. In the last case, the viscosity decreases with shear rate.

The yield-power law, or Herschel-Bulkley, rheological model better describes the behaviour of most water-based and oil-based drilling fluids across the shear rate spectrum than the Bingham plastic or power law models (Hemphill et al., 1993).

Clay suspensions frequently show a time dependent flow behaviour, known as thixotropy. After mixing the suspension, the yield stress and plastic viscosity decrease but will recover with time if left standing.

Various factors affect the flow behaviour of clay suspensions. Naturally, the clay concentration will bring about an increase of all rheological properties. Due to the negative particle charge and double-layer structure, the yield stress and viscosity change with variations of the pH of the suspension and electrolyte concentration. Also, differences in the rheological properties are observed depending on the type of electrolyte in solution and on the nature of the exchangeable ions. A rise of the temperature increases the interparticle attractive forces, which in turn, leads to enhanced particle-particle interactions affecting the yield stress and suspension viscosity. The suspension viscosity also depends to a great extent on the viscosity of the medium, which is also a function of temperature. Pressure effects may also alter the flow properties on clay suspensions, due to the exponential increase of liquid viscosity with pressure and the differential compression of clays and liquids.

Na-montmorillonite suspensions behave according to the Bingham model. The electrical double layer forces are responsible for the gel formation in Na-montmorillonite suspensions, for both  $Mg^{2+}$  and  $Ca^{2+}$  montmorillonite suspensions, the flow behaviour changes from Newtonian to pseudoplastic upon the addition of  $Na^+$  as the exchangeable cation, exhibiting a yield stress and an increased viscosity. This was attributed to the presence of  $Na^+$  ions which break down the clay

aggregates into smaller units. According to the Stern model, the thickness of the diffuse double layer decreases with decreasing hydrated ion radius. Since the radius of hydrated  $K^+$  and  $Ca^{2+}$  is smaller than that of  $Na^+$ , the thickness of the  $K^+$  or  $Ca^{2+}$ -montmorillonite double layer is smaller than Na-montmorillonite. This means that montmorillonite possessing  $Ca^{2+}$  and  $K^+$  as exchangeable cations are less sensitive to osmotic phenomena than the clay containing  $Na^+$  (Luckham and Rossi, 1999).

Compared to clay muds, treatments of bentonite mud by CMC may eliminate the true yield-point value in most cases and will significantly reduce the cuttings carrying capacity at the low shear rates present in the annulus flow. Under these conditions, CMC acts as a thinner or deflocculant; it is anionic material that adsorbs on the clays and acts like any other thinner. So the drilling fluid has no true yield point (calculated). A simple way to detect this error is to compare the calculated yield point to that of initial gel. If the initial-gel value and the calculated yield point are close, then the true yield-point value is not far from the value calculated. The difference between this initial gel and the calculated yield point is a measure of the error of the calculated value. For Fann Viscometers capable of indicating 3-rpm values, these values may be taken as approximations of the true yield point. Of course, complete shear-stress/shear-rate plots are the best way to obtain the true value of yield point (Kelly, 1983). Benchabane and Bekkour (2006) showed that in contrast to CMC, the xanthan biopolymer (because it is produced by bacterial action) solutions exhibit a yield stress that increases with concentration.

## CHAPTER 3

### STATEMENT OF THE PROBLEM

The control of flow properties and the filtration rate of drilling fluids in deep drilling operations are two critical aspects of drilling fluid technology. Over the years various chemicals have been used to control the rheological properties and filtration characteristics of clay base drilling fluids. The extended bentonite, activated with inorganic salt and/or treated with the polymer, can show an unexpected deterioration in quality under severe drilling conditions. Bacterial and thermal degradation of the polymers is the most important problem in drilling application.

In this experiment a Ca-bentonite from Edirne (E-Ben) and a Na/Ca-bentonite from Çankırı (C-Ben) were activated with  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ , and  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture, separately and treated with polymers (Xanthan Gum, HV CMC, and PAC R as a viscosifier, Dextrid, LV CMC, and PAC L as a fluid-loss reducer).  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  was used first time in the activation of bentonites. It was considered that borates can overcome the bacterial degradation problem as they are well known for their bactericide properties.

This study compares the rheological and filtration properties of the raw bentonite mud treated with polymers,  $\text{Na}_2\text{CO}_3$  activated bentonite mud treated with polymers,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  activated bentonite mud treated with polymers, and  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated bentonite mud treated with polymers. The susceptibility of polymers to bacterial and thermal degradation are inferred from the decrease or increase in viscosity and increase in the filter loss following exposure of the treated muds to 36 °C for a 21-day period and high temperature (121 °C for xanthan gum and 149 °C for CMC and PAC) for 16 hours, respectively.

## CHAPTER 4

### MATERIALS AND METHODS

#### 4.1 Materials

A commercially available calcium bentonite from Edirne and sodium/calcium bentonite from Çankırı used all of the experiments were supplied by Bensen and Çanbensen Company. These clays are denoted “E-Ben” and C-Ben”, respectively.

The API standard bentonite and Reşadiye Na-bentonite, which has been studied in some works, have been used for comparison with the two clays. These clays are denoted “API” and “Reşadiye”, respectively.

Mineralogical analysis of the bentonites was done by using X-ray diffraction analysis (XRD). The XRD patterns of the samples were obtained Rigaku-Geigerflex X-Ray diffractometer using Ni-filter Cu-K $\alpha$  radiation. XRD patterns of bentonites showed the presence of montmorillonite in all samples (Figures 4-1 through 4-4). When the mineralogical structure of the samples were investigated, it was observed that E-Ben sample was mainly composed of montmorillonite and minor amounts of quartz and calcite and C-Ben was composed of montmorillonite, quartz, opal CT, feldspar and gypsum. The XRD analyses of the API samples showed that the dominant clay mineral is montmorillonite with minor amounts of mica, gypsum, and calcite. Quartz is also present in the clay fractions. The Reşadiye bentonite contains mostly montmorillonite, an aluminous smectite. The rest of the sample is composed of impurities such as quartz, calcite, mica, zeolite, and feldspar.



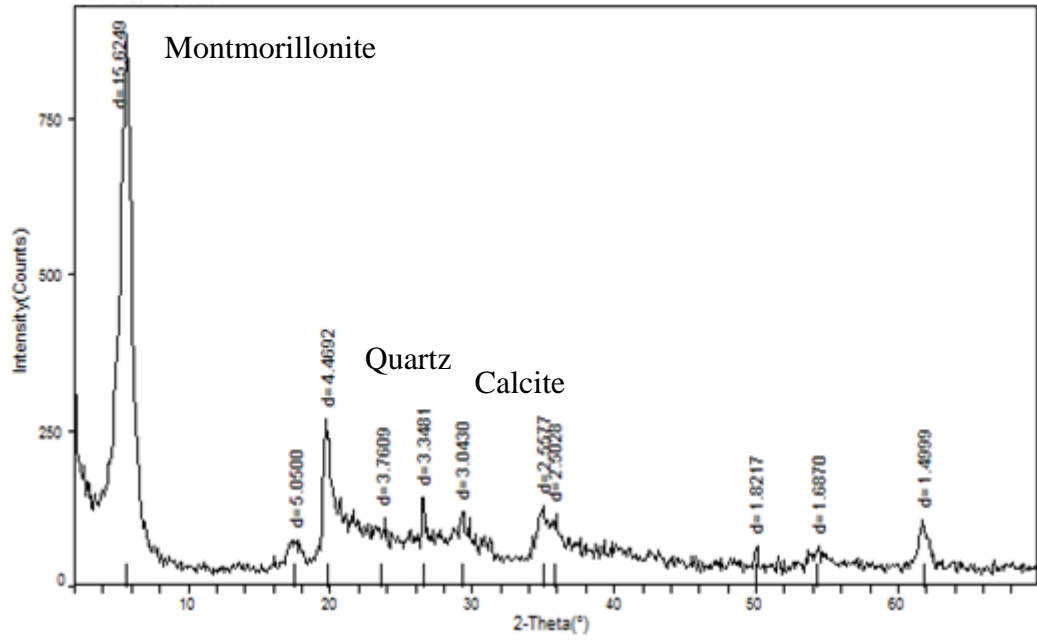


Figure 4-1 X-Ray Diffraction of E-Ben

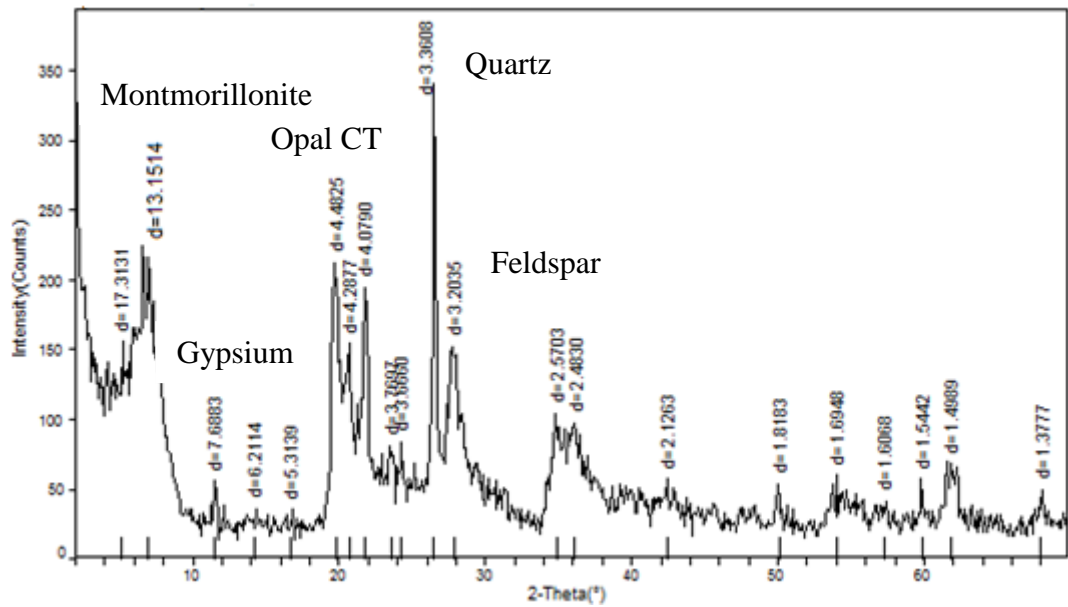


Figure 4-2 X-Ray Diffraction of C-Ben

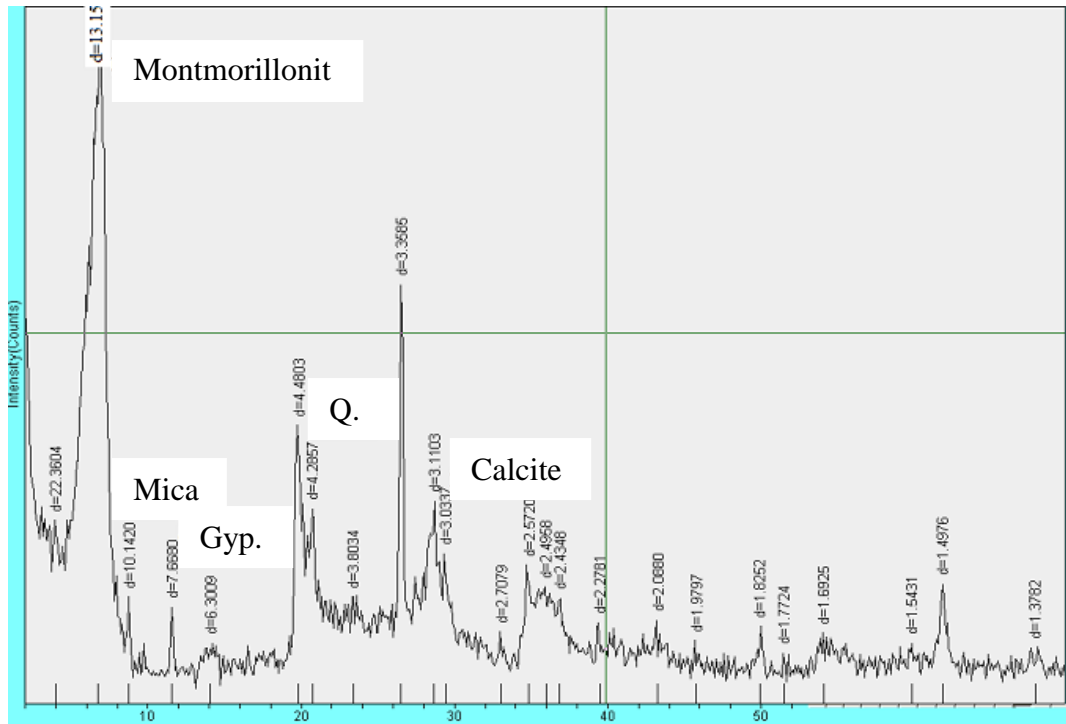


Figure 4-3 X-Ray Diffraction of API

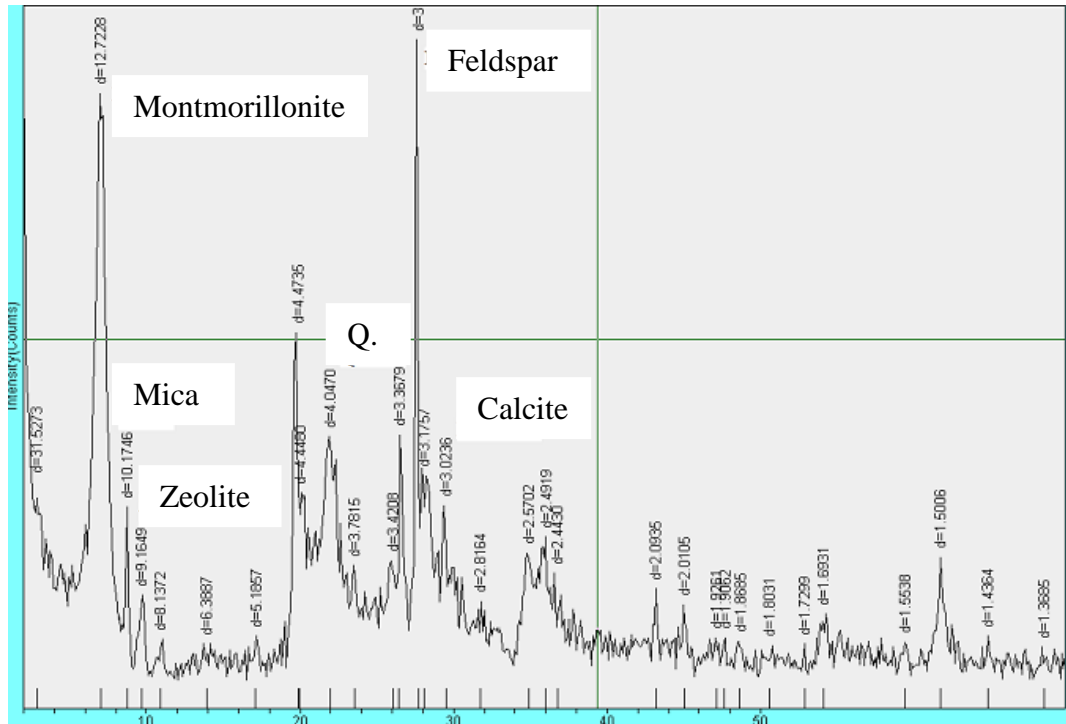


Figure 4-4 X-Ray Diffraction of Reşadiye

Chemical composition of bentonites was determined by X-ray spectrometer method. Chemical analysis results are showed in Table 4-1 that the  $\text{Al}_2\text{O}_3/\text{SiO}_2$  ratios are about 1/3 to 1/4 as expected for the montmorillonite which is the main component of bentonites under study (Kuru et al., 1995). The amount of  $\text{Na}_2\text{O}$  is varied in the range (0,92-2,92%), Reşadiye has the highest amount, and E-Ben has the lowest. The largest amounts of  $\text{CaO}$  were found in the E-Ben, API, and Reşadiye samples which are related to calcite observed in XRD pattern (Figures 4-1, 4-3 and 4-4). In E-Ben the amount of  $\text{MgO}$  was found to be greater compared to the other three samples, C-Ben, API, and Reşadiye.  $\text{Fe}_2\text{O}_3$  shows significant variations between the samples, with API sample having the highest (5,69%) and E-Ben having the lowest (1,96%). The presence of  $\text{K}_2\text{O}$  in C-Ben and Reşadiye bentonite can be attributed to the indication of K-feldspar, a non-clay mineral.

Table 4-1 Chemical Analyses of the Samples

<b>Analysis (%)</b>	<b>E-Ben</b>	<b>C-Ben</b>	<b>API</b>	<b>Reşadiye</b>
<b><math>\text{Na}_2\text{O}</math></b>	0,23	2,26	1,89	2,92
<b><math>\text{MgO}</math></b>	5,21	2,66	2,49	1,95
<b><math>\text{Al}_2\text{O}_3</math></b>	16,91	16,88	19,18	17,12
<b><math>\text{SiO}_2</math></b>	60,25	62,59	59,22	61,48
<b><math>\text{P}_2\text{O}_5</math></b>	0,03	<0,01	<0,01	0,15
<b><math>\text{K}_2\text{O}</math></b>	0,42	0,88	0,62	0,98
<b><math>\text{CaO}</math></b>	3,04	1,54	2,34	3,12
<b><math>\text{TiO}_2</math></b>	0,18	0,72	0,13	0,30
<b><math>\text{MnO}</math></b>	<0,01	<0,01	0,13	0,10
<b><math>\text{Fe}_2\text{O}_3</math></b>	1,96	5,56	5,69	3,87
<b>Ignition Loss</b>	11,66	6,23	7,55	7,75

$\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  was obtained from Eti Mine Works General Management.  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  is the most common form of sodium borate used in industry. Generally, boron minerals form buffer solutions at about pH 9,1-9,4 at which exhibit minimum solubility (Özbayoğlu, 2004). The solubility of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  in water as a function of temperature was presented in Figure 4-5 (these values were obtained from web page <http://www.borax.com> on 20.01.2008).

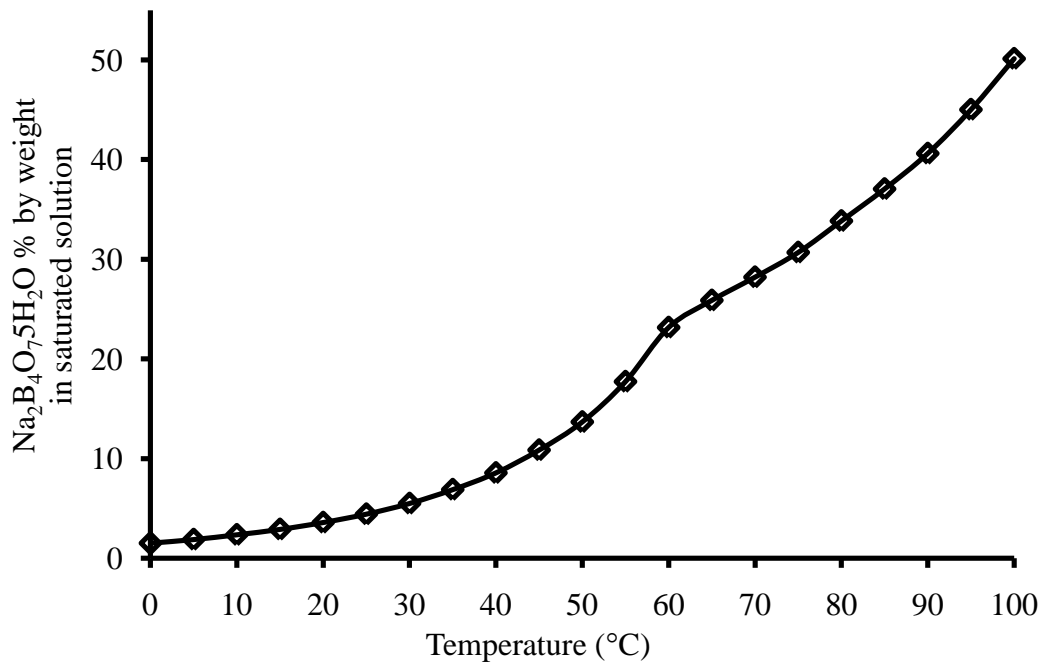


Figure 4-5 Solubility of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  in Water as a Function of Temperature

HV CMC, LV CMC, PAC R, and PAC L were produced by Karkim Company. HV CMC is high viscosity sodium carboxymethyl cellulose and is utilized to increase viscosity and control fluid loss in most water-based drilling fluids. LV CMC is low viscosity sodium carboxymethyl cellulose and is utilized to control fluid loss in water-based drilling fluids. HV CMC and LV CMC are resistant to bacterial attack and calcium contamination. PAC R is a polyanionic cellulose polymer utilized to control filtration and increase viscosity of water-based drilling fluids. PAC LV is purified grade low molecular weight polyanionic cellulose polymer utilized to be

used in filtration control in most water-based drilling fluid systems. All of these polymers are a thermally stable material up to 149 °C (300 °F)

The Barazan-D (xanthan gum) and Dextrid-LT (dextrid) were produced by Baroid Company. Barazan-D dispersion-enhanced xanthan biopolymer helps provide viscosity and suspension in fresh water. Barazan-D is stable to 121 °C (250 °F). Dextrid-LT, modified potato starch, provides filtration control with minimum viscosity buildup in-water based drilling fluids. Dextrid-LT is stable against bacterial degradation.

Deionised water was used for activation of bentonites and the preparation of polymer and bentonite suspension.

## 4.2 Methods of Activation

In the production of Na-activated bentonite, firstly  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  were dissolved in water and samples were mixed with different concentration of  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ .

Two bentonite samples (E-Ben and C-Ben) were activated with  $\text{Na}_2\text{CO}_3$  at various ratios ( $\text{Na}_2\text{CO}_3$  addition level; %  $\text{Na}_2\text{O}$  equivalent: 0,58, 1,16, 1,74, 2,32, 2,90, 3,48, 4,06, and 4,64 for E-Ben, 0,29, 0,44, 0,58, 0,73, 0,87, 1,16, and 1,74 for C-Ben).

Also the two bentonite samples were activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  saturated solution at various ratios ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; %  $\text{Na}_2\text{O}$  equivalent: 0,76, 1,52, 2,29, 3,05, 3,82, 4,58, and 5,35 for E-Ben, 0,38, 0,57, 0,76, 0,95, 1,14, and 1,52 for C-Ben).

As it can be seen from Appendix Table A-8, 5000 ml  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  saturated solution ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 3,82%  $\text{Na}_2\text{O}$  equivalent) was required to maximum activation for one kg E-Ben. In reality it is impossible to activate bentonite with this high water content solution. So  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  was dissolved at 55 °C. The bentonite was activated with this saturated solution at various ratios ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; %  $\text{Na}_2\text{O}$  equivalent: 3,02, 3,40, 3,78, 4,15, 4,53, 4,90 and 5,66).

To see the effect of  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture two bentonite samples were activated at different ratios ( $\text{Na}_2\text{CO}_3/\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; %  $\text{Na}_2\text{O}$

equivalent: 0,73/2,86, 1,45/1,91, and 2,18/0,95 for E-Ben, 0,14/0,57, 0,29/0,38, and 0,44/0,19 for C-Ben).

All samples after activation were dried at 55 °C and then crushed using a jaw crusher to 100% below 5 mm in size. Each sample was ground to 100% below 200 meshes using a porcelain mill. These samples were used for characterization and physico-chemical tests. All experiments were performed at atmospheric conditions and room temperature.

### **4.3 Equipment and Procedure for Measurement of Rheological and Filtrate Volume Properties**

#### **4.3.1 Equipment for Measurement of Rheological Properties**

In this thesis, the Model Fann 35 viscometer was used to measure the rheological properties of the test fluids by measuring the shear stress at specific shear rates. Fann 35 viscometer has six rotation speeds (600, 300, 200, 100, 6 and 3 rpm). It is a concentric cylinder apparatus which simulates the fluid interface at the wall of the pipe or annulus.

The Model 35 Fann viscometer is a rotational viscometer with a rotating cylinder and a stationary bob (Figure 4-6). Fluid is contained in the annular space between the co-axial cylinders. The outer cylinder (rotor) is driven at a predetermined rotational speed and the torque arising from the fluid's viscous drag is exerted on the inner cylinder or the bob. The torque is balanced by a helically wound spring and the dial reading which indicates the spring deflection is used to calculate the torque.

For the Newtonian fluids, the shear stress and shear rate data can be plotted to represent the rheogram of the test fluid. The slope of the data is the viscosity in centipoises (cP).

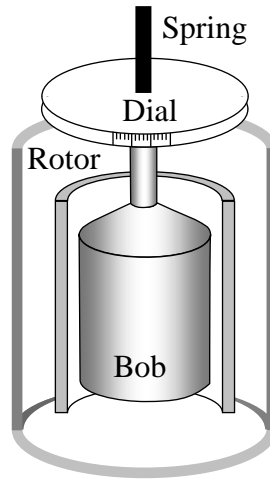


Figure 4-6 Concentric Cylinder Viscometer

#### 4.3.2 Determination of Rheological Properties of Drilling Muds

Rheological properties of drilling muds were determined according to API Specification 13A-ISO 13500;

- i- A suspension of bentonite was prepared by adding an amount of clay (22,5 g $\pm$ 0,01 for E-Ben and C-Ben and 25 g $\pm$ 0,01 for nontreated bentonite) to 350 cm<sup>3</sup> $\pm$ 5 cm<sup>3</sup> of deionised water while stirring on the mixer.
- ii- After stirring 5 min $\pm$ 0,5 min, container was removed from mixer and scraped its sides with the spatula to dislodge any bentonite adhering to container walls.
- iii- The container was replaced on the mixer and continued to stir. Total stirring time shall equal 20 min $\pm$ 1 min.
- iv- The bentonite suspension was aged up to 16 h in a sealed or covered container at room temperature.
- v- After ageing bentonite suspension, sealed or covered container was shaken well and then poured the suspension into the mixer container. The suspension was stirred on the mixer for 5 min $\pm$ 0,5 min.

- vi- The suspension was poured into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer were recorded when a constant value for each rpm is reached.

#### 4.3.3 Determination of Plastic Viscosity and Yield-Point

The plastic viscosity, yield-point, yield-point/plastic viscosity ratio and apparent viscosity were calculated from 300 and 600 rpm readings using following formulas from API Specification for Drilling Fluid Materials (API Specification 13A-ISO 13500:1998).

$$\text{Plastic Viscosity} = [600 \text{ r/min reading}] - [300 \text{ r/min reading}] \quad (4.1)$$

$$\text{Yield Point} = [300 \text{ r/min reading}] - [\text{Plastic viscosity}] \quad (4.2)$$

$$\text{Yield Point/Plastic Viscosity ratio} = [\text{yield point}] / [\text{plastic viscosity}] \quad (4.3)$$

$$\text{Apparent Viscosity} = 600 \text{ rpm}/2 \quad (4.4)$$

Plastic viscosity (PV) is a measure of the viscosity of the dispersed phase (base liquid) and friction between the insoluble solid particles in the mud.

Yield point is a measure of the resistance to initial flow, or represents the stress required to start fluid movement. This resistance is due to electrical charges located on or near the surfaces of the particles. The charges bring about inter particle forces.

#### 4.3.4 Determination of Gel Strength

Gel strength is a measure of the ability of a colloid to form gels and is related to inter particle forces of the mud. It is measured under static conditions. Gel strength measurements are normally taken as initial gel strength (zero quiescent time) and final gel strength (ten-minute quiescent time).

The following procedure was employed to determine two values – the initial gel-strength ( $g_0$ ) and the 10 minute gel-strength ( $g_{10}$ ).



- i- The mud sample was stirred at high speed (600 rpm) for ten second and then the mud was allowed to stand undisturbed for ten second.
- ii- The instrument was rotated at 3 rpm and the maximum reading attained after the start of the rotation was recorded. This maximum reading was the initial gel strength in lb/100 ft<sup>2</sup>.
- iii- The mud was re-stirred at high speed for ten seconds and then allowed to stand undisturbed for ten minutes.
- iv- The maximum reading after the start of rotation at 3 rpm was recorded. This maximum reading was termed the ten-minute gel in lb/100 ft<sup>2</sup>.

#### **4.3.5 Determination of Filtrate Volume**

A filter press was used to determine the filtration of drilling fluid. Filtrate volumes of drilling muds were determined according to API Specification 13A-ISO 13500:1998.

- i- The samples for rheological test were used for filtrate volume determination and the sample was stirred in container for 1 min±0,5 min on mixer.
- ii- The suspension was poured into filter press cell within about 13 mm of the top of the cell. Assembly of the filter press cell was completed. Filter cell was placed in frame and closed the relief valve. A container was placed under the drain tube.
- iii- One timer was set for 7,5±0,1 minutes and the second timer for 30±0,1 minutes. Both timers were started and adjusted pressure on cell to 690 kPa.
- iv- The container was removed at 7,5 min on the first timer. The 10 cm<sup>3</sup> graduated cylinder was placed under the drain tube and continued collecting filtrate to the end of the second timer set at 30 min. The graduated cylinder was removed and recorded the volume of filtrate collected.
- v- The dispersed filtrate volume of the clay suspension was calculated as:

$$\text{Filtrate Volume, cm}^3 = 2 \times V_c \quad (4.5)$$

Where  $V_c$  is the filtrate volume collected between 7,5 min and 30 min.

#### 4.4 Aging of Bentonite Samples

In this study the the rheology and filtrate properties of various bentonites treated with polymers were examined when subjected to bacterial and thermal degradation.

All the mud compositions were aged in an orbital incubator at 36 °C for a 21-day period for to examine whether the mud compositions were degrading when subjected to bacteria. The prepared suspensions were opened to atmospheric conditions for one hour a day and the rheological and filtrate properties of them were examined on the first, fourth, eighth, fifteenth and twenty-first days.

In addition the mud compositions were aged at high temperature (149 °C for CMC and PAC, and 121 °C for xanthan gum) for 16 hours in a roller oven. The aged samples were cooled to room temperature, removed from the aging cell, and stirred on a mixer for 5 minutes to break down any gels formed in the aging process. Then, the fluid properties such as apparent and plastic viscosities, gel strengths (initial/10 minutes), yield points and filtrate volumes were determined.

#### 4.5 Determination of Swelling Test

The sheets in clay structures bound to each other with covalent bonds and therefore, their crystal structure is stable. In contrast, the layers bound with Van der Waals bonds and water or organic molecules expands the layers when it introduces the interlayers. In this way, the distance between basal spacings increases. An increase in basal spacing is considered as a criteria for the degree of swelling of clay minerals due to introducing of organic molecules (İşçi et al., 2004).

The procedure was as following:

- i- 1,40 g clay sample, 2,60 g  $\text{Al}_2\text{O}_3$  and 0,20 g MgO were weighted.
- ii- They were added in Erlenmeyer flask with 100 ml distilled water.

- iii- They were stirred in magnetic mixer for 1 hour.
- iv- After stirring, the flask was removed from mixer and the sample was poured into 100 ml cylinders very slowly.
- v- After 24 hours the level of the swollen bentonite in graduated cylinders was recorded as relative swelling index value.

Table 4-2 shows the swelling volume of bentonite samples. One way of classifying bentonite is based on its swelling capacities (Elzea and Murray, 1994) when added to water. Reşadiye and API, containing Na<sup>+</sup>, have very high swelling capacities (73 and 70 mL). E-Ben in which exchangeable calcium (Ca<sup>++</sup>) is more abundant than other ions has much lower swelling capacities (8,5 mL) than sodium bentonites. Intermediate bentonites (C-Ben), called the mixed bentonites (Ca/ Na), tend to swell moderately (29 mL) and form gels of lesser volumes than equal masses of sodium type bentonite.

Table 4-2 Swelling of Bentonite Samples Studied

<b>Type of Bentonite</b>	<b>Swelling Volume (mL)</b>
<b>E-Ben</b>	8,5
<b>C-Ben</b>	29
<b>API</b>	70
<b>Reşadiye</b>	73

#### **4.6 Determination of Cation Exchange Capacity**

The cation exchange capacity (CEC) of fine grained materials, and especially clay minerals, is a fundamental property of these materials. Methods of measurement are based on a determination of the quantity of a particular exchangeable cation, by a variety of means, expressed per 100 g of dry clay (Borden and Giese, 2001). Table 4-3 lists the average CEC of various clays (Bol, 1986). A 100% pure bentonite should have a CEC value about the same as that of the montmorillonite. The presence of

other clays or nonclay materials decreases the CEC, and hence this can give an indication of bentonite purity. The CEC of studied samples was measured by methylene blue adsorption method.

Table 4-3 CEC of some Common Clay Minerals

<b>Clay Type</b>	<b>CEC (meq/100 g)</b>
<b>Montmorillonite</b>	70 to 130
<b>Illite</b>	10 to 40
<b>Kaolinite</b>	3 to 15
<b>Chlorite</b>	10 to 40

The procedure of CEC determination by methylene blue adsorption method was as follows:

- i- 0,01 N methylene blue dye solution was prepared by dissolving 3,74 g basic blue methylene blue dry powder per liter of distilled water.
- ii- Bentonite sample was dried at 105 °C for 2 hours.
- iii- A weighed quantity (0,57 g) of clay was dispersed into 10 cm<sup>3</sup> water in the Erlenmeyer flask followed by addition of 0,5 ml of 5 N sulphuric acids.
- iv- This mixture was boiled gently for 10 minute and then diluted to about 50 cm<sup>3</sup> with distilled water.
- v- Methylene blue solution was added in 0,5 ml increments from burette to the flask. After each addition, the contents of the flask were swirled for about 30 second. While the solids were still suspended in the liquid, one drop of the suspension was removed with a stirring rod and placed the drop on a piece of filter paper.
- vi- The end point of the titration was reached when dye appears as a blue ring surrounding the dyed solids.

- vii- When the blue tint spreading from the spot was detected, the flask was shaken an additional 2 minutes and placed another drop on the filter paper. If the blue ring was again evident, the end point had been reached.
- viii- The cation exchange of the clay was reported as the methylene blue capacity calculated as follow (ASTM C 837-81):

$$MBI = \frac{E \times V}{W} \times 100 \quad (4.6)$$

where:

MBI = methylene blue index for the clay in meq/100 g clay

E = miliequivalents of methylene blue per millilitre

V = millilitres of methylene blue solution required for the titration, and

W = grams of dry material.

Cation exchange capacity values of bentonites were found to be in range 52-90 meq/100 g. From Table 4-4 it can be seen that API and Reşadiye bentonite have higher CEC than the others, E-Ben, and C-Ben. Higher CEC may indicate higher amount of montmorillonite. Typical commercial grade bentonites have CEC values between 40 and 120 meq/100 g (Hassan and Abdel-Khalek, 1988). This means that the CEC values of investigated bentonites are within the desirable range. The CEC values also can be converted to surface areas as m<sup>2</sup>/g by multiplying the CEC in meq/100 g clay with 7.826. This relationship is based on the assumption that the methylene blue molecules are adsorbed as a monolayer lying flat on the clay surface (Güven et al., 1988).

Table 4-4 Cation Exchange Capacity of Bentonite Samples Studied

Property	E-Ben	C-Ben	API	Reşadiye
C.E.C (meq/100 g)	68	52	90	78

## CHAPTER 5

### RESULTS AND DISCUSSION

#### 5.1 The Effect of Inorganic Salts on the Swelling of E-Ben and C-Ben

In order to understand whether added  $\text{Na}^+$  ions replaced by  $\text{Ca}^{+2}$  ions in the interlayer of bentonites or not, swelling tests were employed for E-Ben and C-Ben activated with  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ .

##### 5.1.1 The Effect of $\text{Na}_2\text{CO}_3$ on the Swelling of E-Ben and C-Ben

In this test, the swelling volumes of E-Ben and C-Ben activated with  $\text{Na}_2\text{CO}_3$  were determined. The values of the swelling volume shown in Figure 5-1 and 5-2 (see also appendix Tables A-1 and A-2) revealed an increase by mixing with different amounts of  $\text{Na}_2\text{CO}_3$  addition level; 0,58–2,90% of  $\text{Na}_2\text{O}$  equivalent for E-Ben and  $\text{Na}_2\text{CO}_3$  addition level; 0,29–0,58%  $\text{Na}_2\text{O}$  equivalent for C-Ben. For example, the swelling volumes of activated samples reached to 40 mL for E-Ben (Figure 5-1) and 41 mL for C-Ben (Figure 5-2) when 2,90% and 0,58%  $\text{Na}_2\text{O}$  were added, respectively. Improvement in swelling indicates that the calcium ions on the E-Ben and C-Ben were replaced by sodium ions.

The reason for the increase in swelling can be explained as follows. In smectite containing  $\text{Ca}^{++}$  as the exchangeable cation, interlayer hydration is limited to 2 or 3 molecular layers of water because the unit layers are closely spaced due to strong electrostatic attraction between the cations and the negatively charged clay surfaces. In Na-montmorillonites the exchangeable  $\text{Na}^+$  ions have large hydration radii and form a wide, diffuse double layer. Thus the electrostatic attractions between clay layers are weak, forming a loose and dispersed structure of particles. As a result the interlayer spaces are very expansive and accommodate a large portion of the total water contained in the system (Malfoy et al., 2003; Alemdar et al., 2005a).

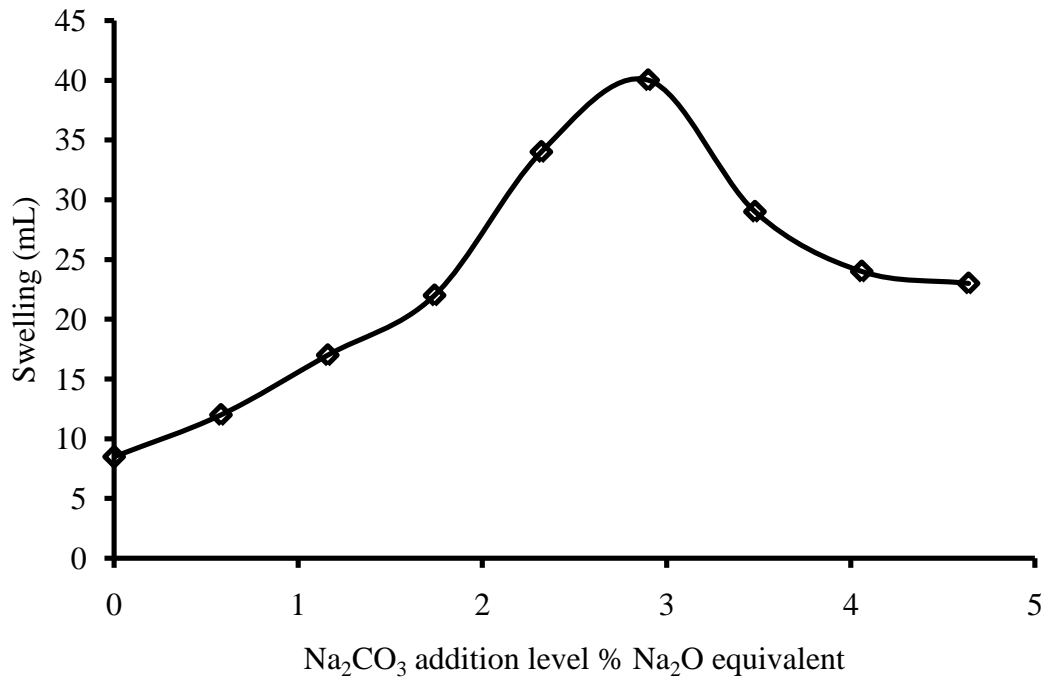


Figure 5-1 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Swelling of E-Ben

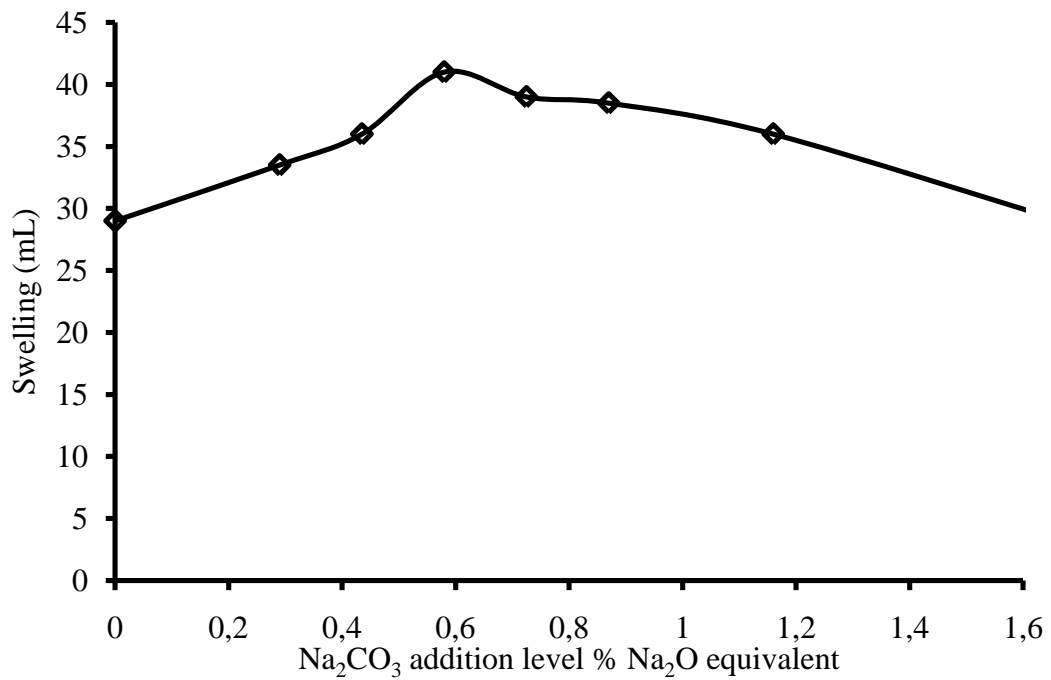


Figure 5-2 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Swelling of C-Ben

### 5.1.2 The Effect of $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ on the Swelling of E-Ben and C-Ben

The swelling volume of E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  dissolved at room temperature ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level between 0,76-5,35%  $\text{Na}_2\text{O}$  equivalent) and 55 °C ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level between 3,02-5,66%  $\text{Na}_2\text{O}$  equivalent) were determined separately. Figure 5-3 and 5-4 (see also appendix Tables A-3 and A-4) indicate that the swelling volume of activated samples reached to 36 mL and 34 mL for E-Ben when 3,82% and 3,78%  $\text{Na}_2\text{O}$  were added, respectively. The swelling volumes of C-Ben activated with different concentration of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; between 0,38-1,52%  $\text{Na}_2\text{O}$  equivalent) are given in Figure 5-5 (see also appendix Table A-5). The swelling volume of activated samples reached to 37,5 mL for C-Ben when 0,76%  $\text{Na}_2\text{O}$  was added.

$\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  treatment of bentonite like  $\text{Na}_2\text{CO}_3$  activated bentonite depends essentially on an ion exchange reaction;  $\text{Ca}^{+2}$  ions in the bentonite are replaced by  $\text{Na}^+$  ions in borate.

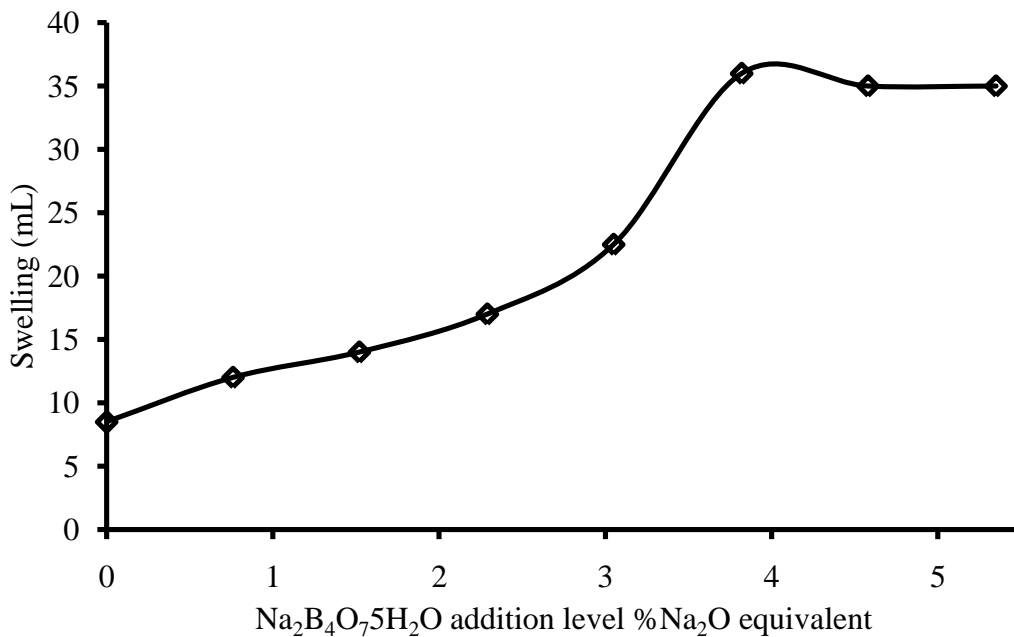


Figure 5-3 Effect of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Dissolved at Room Temperature on the Swelling of E-Ben



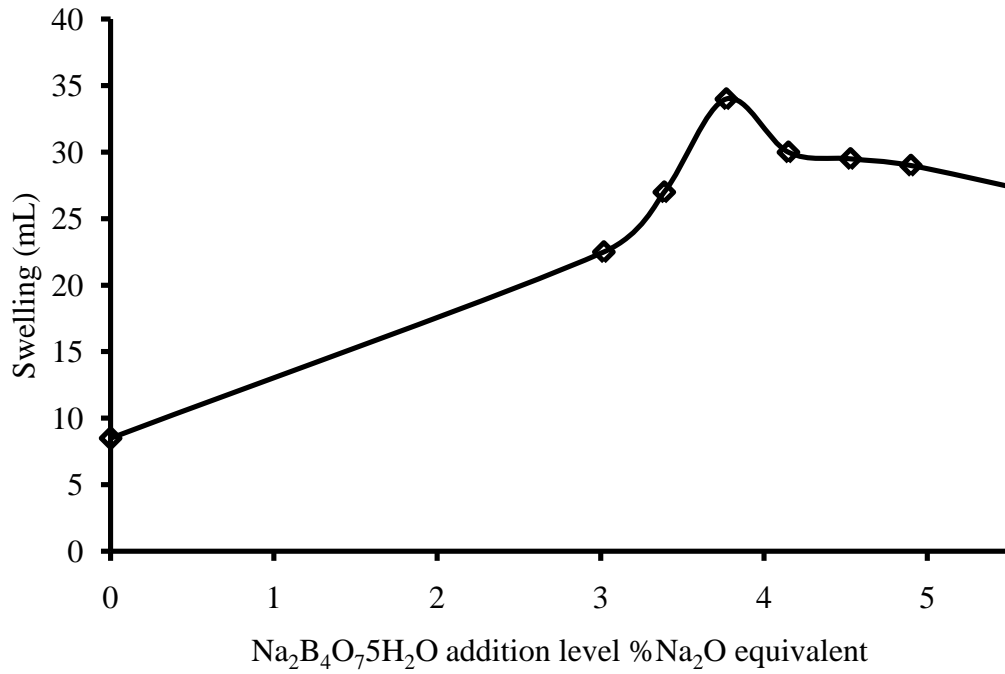


Figure 5-4 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at 55 °C on the Swelling of E-Ben

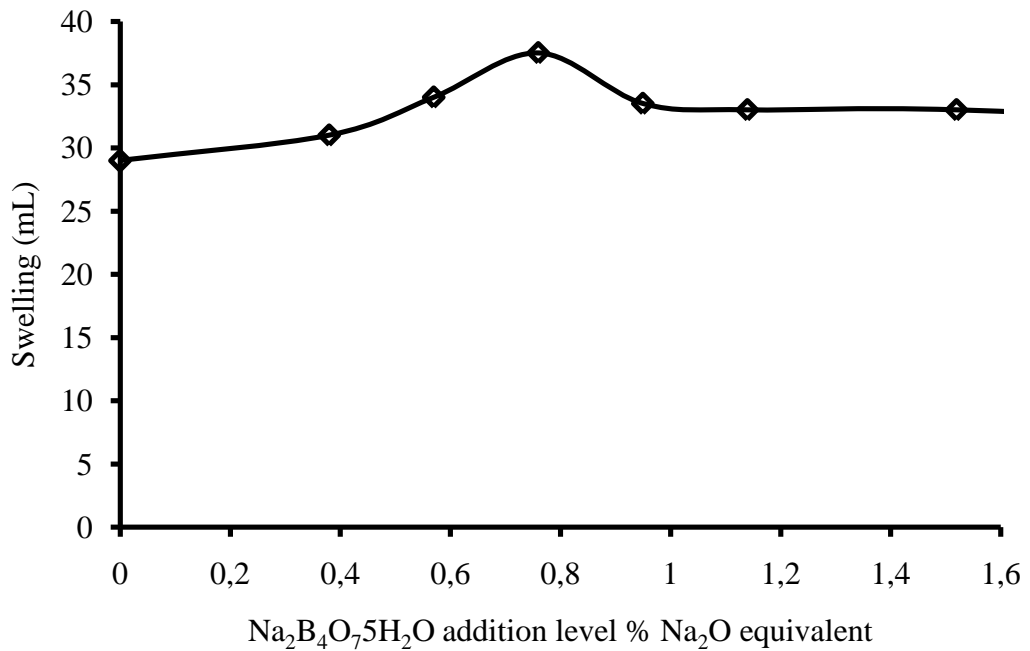


Figure 5-5 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Swelling of C-Ben

### 5.1.3 Comparison of the Effect of $\text{Na}_2\text{CO}_3$ and $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ on the Swelling of E-Ben and C-Ben

The effect of inorganic salts on the swelling of E-Ben and C-Ben was compared according to  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; % $\text{Na}_2\text{O}$  equivalent.

The swelling volumes of E-Ben activated with  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  at various ratios are shown in Figure 5-6. The swelling of bentonite were improved by  $\text{Na}_2\text{CO}_3$  addition level; 2,85%  $\text{Na}_2\text{O}$  equivalent and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 3,82% (dissolved at room temperature) and 3,78%  $\text{Na}_2\text{O}$  (dissolved at 55 °C) equivalent, respectively.

It was found in Figure 5-7 that the swelling properties of C-Ben activated with  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  were improved by  $\text{Na}_2\text{CO}_3$  addition level; 0,57%  $\text{Na}_2\text{O}$  equivalent and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,76%  $\text{Na}_2\text{O}$  equivalent, respectively.

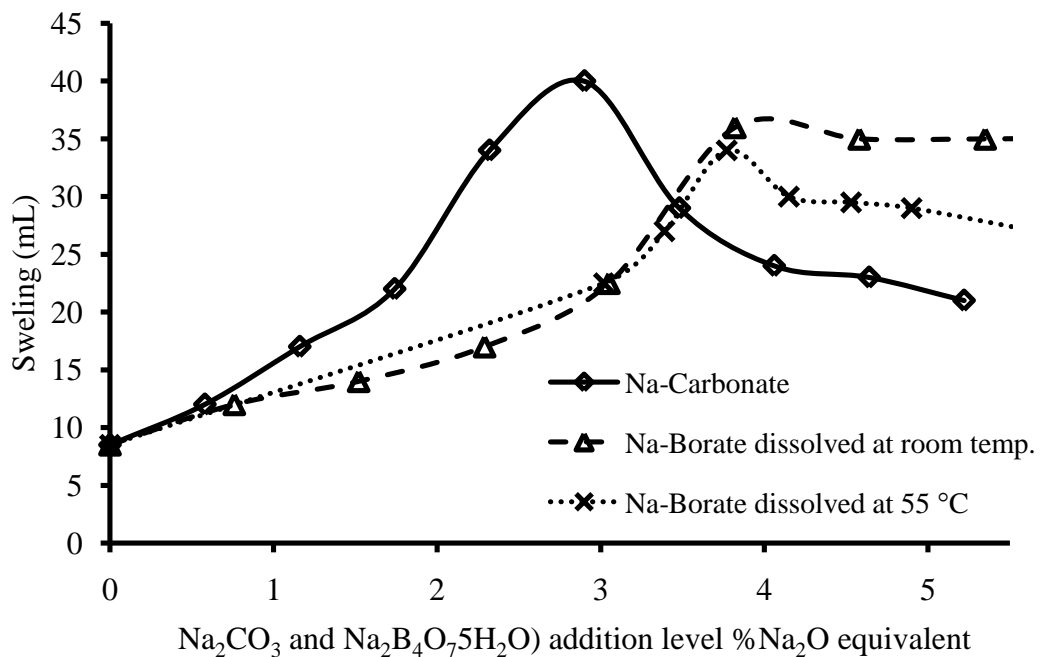


Figure 5-6 Comparison of the Effect of  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  on the Swelling of E-Ben

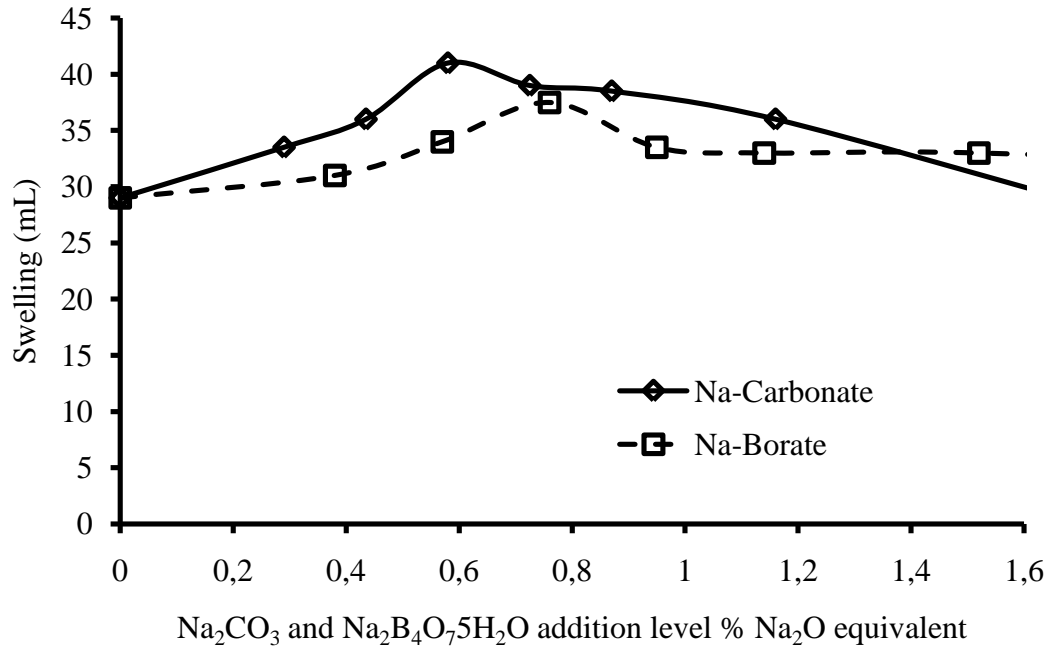


Figure 5-7 Comparison of the Effect of Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O on the Swelling of C-Ben

These results showed that the increase in the swelling volume ratio of E-Ben activated with inorganic salts were higher than that of C-Ben. Besides, the CEC of E-Ben (66 meq/100g clay) was higher than C-Ben (52 meq/100g clay). So the inorganic salt treatment of bentonite depends essentially on cation exchange capacity; the higher the CEC, the higher the swelling ratio. However, the maximum swelling volumes of high grade smectite (API and Reşadiye) used in drilling industry shown in Table 4-2 were still much greater than activated E-Ben and C-Ben.

## 5.2 The Effect of Inorganic Salts on the Rheology and Filtrate Properties of E-Ben and C-Ben

In this experiment bentonite water suspensions were prepared with each of the activated products according to API requirements and rheological (apparent viscosity, plastic viscosity, yield point, initial and 10 minute gel strengths) and filtrate properties were measured.

### 5.2.1 The Effect of $\text{Na}_2\text{CO}_3$ on the Rheology and Filtrate Properties of E-Ben

The results of the rheological and filtrate volume of E-Ben activated with  $\text{Na}_2\text{CO}_3$  (addition level 0,58, 1,16, 1,74, 2,32, 2,90, 3,48, 4,06 and 4,64%  $\text{Na}_2\text{O}$  equivalent) are given in Figures 5-8 through 5-10 (see also appendix Table A-6).

Rheological data of raw bentonite sample recorded apparent viscosity (AV) which was well below the minimum required value of 15 cP. Due to ion exchange, the original E-Ben (Ca-bentonite) was converted to Na-bentonite by  $\text{Na}_2\text{CO}_3$  addition level; 2,90%  $\text{Na}_2\text{O}$  equivalent. The apparent viscosity values of raw bentonite and activated bentonite were found at 1,5 cP and 6 cP, respectively (Figure 5-8). This difference was caused by dispersion behaviours of the bentonite after the activation process. In Ca-bentonite dispersions,  $\text{Ca}^{++}$  ions, which are adsorbed onto two adjacent surfaces, do not freely dissociate from the silica surface of the bentonite crystallites. In Na-bentonite dispersions,  $\text{Na}^+$  ions, which are adsorbed onto one clay surface pass through into the solution when they interact with water and easily leave out the clay minerals. In contrast, water molecules easily moved into the layers of clay minerals which caused swelling that create an increase in viscosity values. Since the water associated with the clay is loosely bonded to it, it cannot flow as easily as free unbounded water. Thus the more the clay hydrates, the more viscous a fluid is formed. As a result of this process, the decrease in water content of the solution enhanced much bigger resistance against flowing (İşçi et al., 2004). Besides the yield points which indicate attractive forces between particles increased with  $\text{Na}_2\text{CO}_3$  addition level; 2,90%  $\text{Na}_2\text{O}$  equivalent. These forces exist due to the electrically negative planer surfaces and to positive sites at the edges of the plates where the lattice is disrupted and a broken bond surface is exposed (Annis, 1967).

Figure 5-18 shows that the 10 gel-strength of bentonite increased the maximum level with  $\text{Na}_2\text{CO}_3$  addition level; 2,90%  $\text{Na}_2\text{O}$  equivalent, i.e., the gel strength of activated bentonite is better than the unactivated bentonite.

The decrease in the rheological properties (Figures 5-8 and 5-9) and the increase in the filtrate volumes (Figure 5-10) after 2,90% of  $\text{Na}_2\text{O}$  were due to increasing of the  $\text{Na}^+$  concentration. This means that the increase of sodium ( $\text{Na}^+$ ) ion concentration causes the compression of the electrical double layer and a decrease of the electrokinetic potential ( $\zeta$ ). This is explained by the fact that if the salt

concentration in the solution increases, part of cations moves from the diffuse layer to the adsorption layer and consequently the  $\zeta$ -potential decreases (Tchistiakov, 2000). At high concentrations of sodium ions where silicate layers approach to close distances ( $\leq 10 \text{ \AA}$ ), the structure of the diffuse layer changes (Luckham and Rossi, 1999).

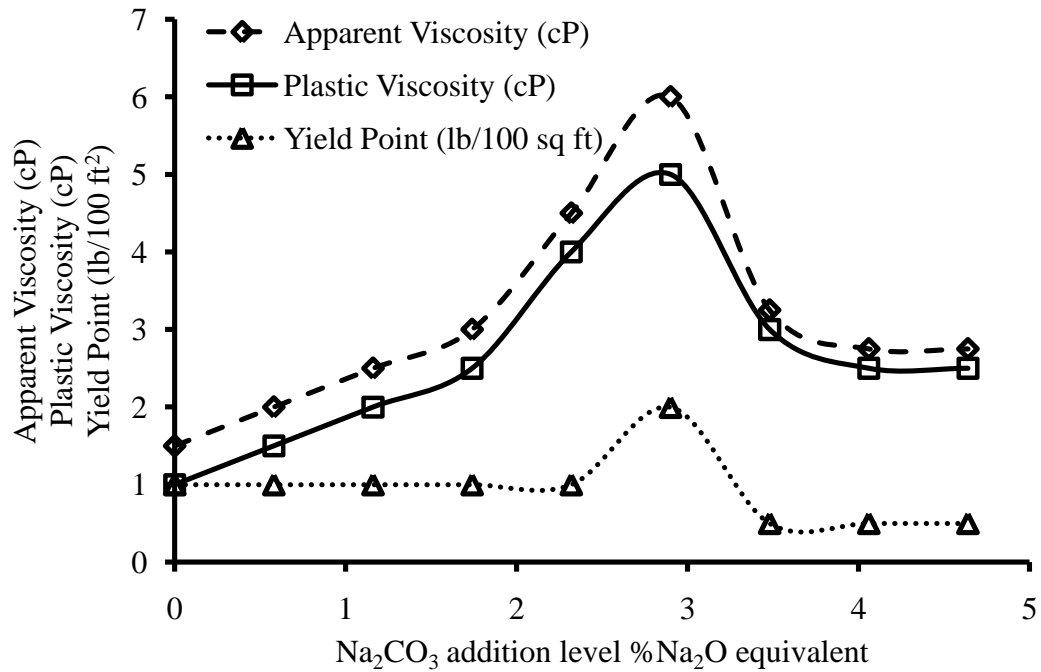


Figure 5-8 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Rheology of E-Ben

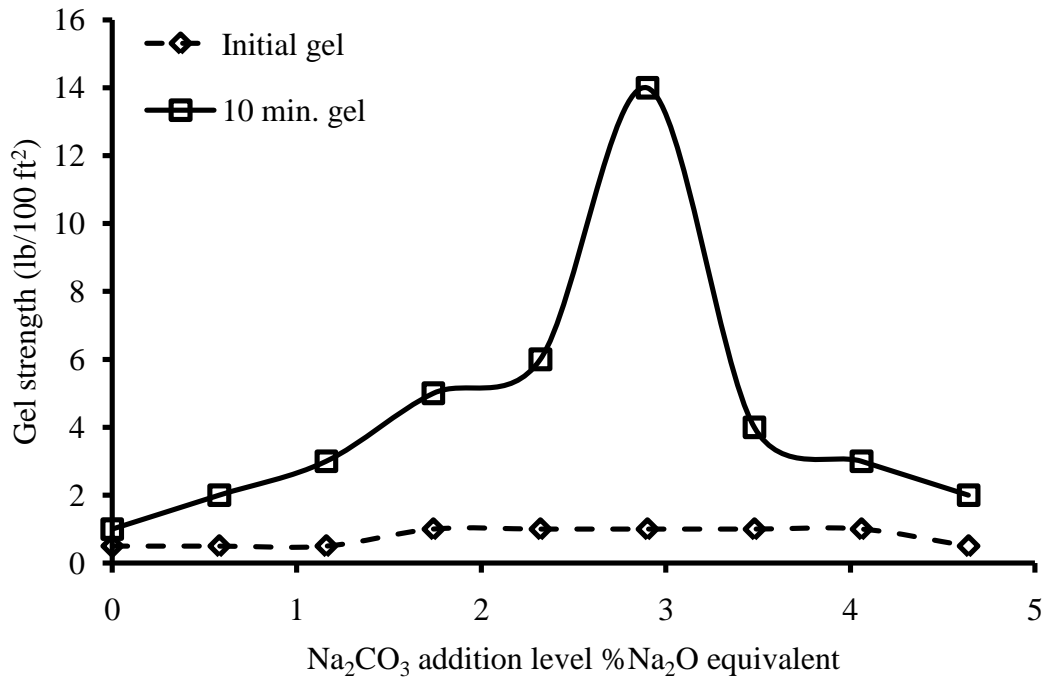


Figure 5-9 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Gel Strength of E-Ben

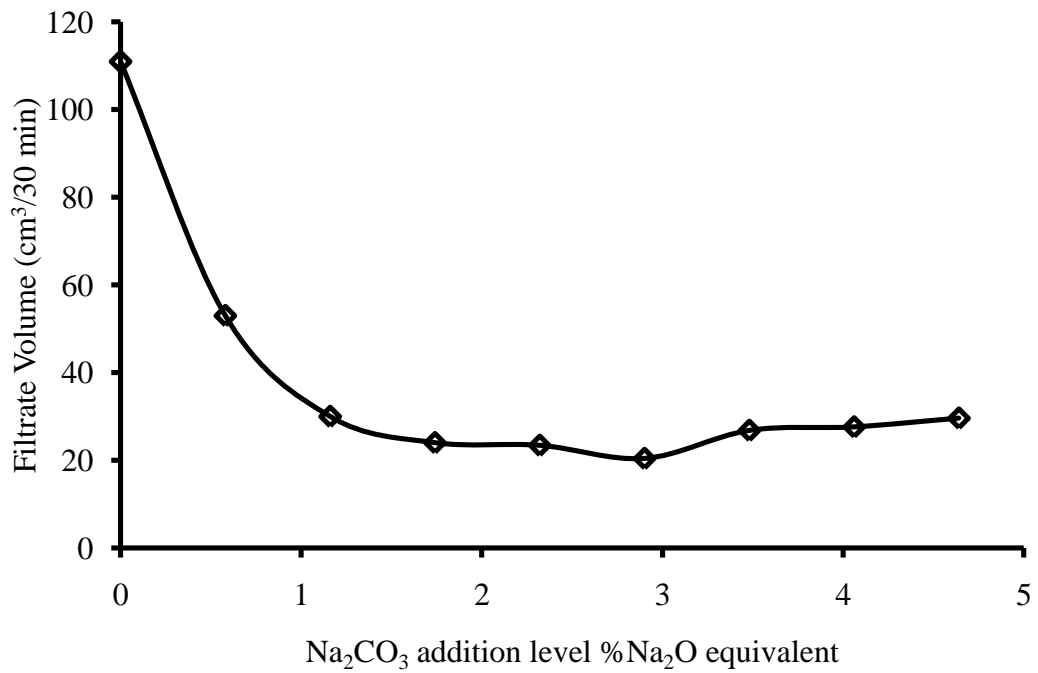


Figure 5-10 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Filtrate Volume of E-Ben

### 5.2.2 The Effect of Na<sub>2</sub>CO<sub>3</sub> on the Rheology and Filtrate Properties of C-Ben

The results of the effect of Na<sub>2</sub>CO<sub>3</sub> on the rheology and filtrate properties of C-Ben are given in Figures 5-11 through 5-13 (see also appendix Table A-7).

With the Na<sub>2</sub>CO<sub>3</sub> addition to the bentonite, the increase in the rheological parameters was determined up to 0,58% of Na<sub>2</sub>O and the decrease after 0,58% of Na<sub>2</sub>O. The reason for increasing and decreasing in rheological parameters was the same as discussed in page 55 and 56. The filtrate volume of activated bentonite decreased from 30 to 22,8 cm<sup>3</sup> by Na<sub>2</sub>CO<sub>3</sub> addition level; 0,58% Na<sub>2</sub>O equivalent. In conclusion, due to ion exchange, the original C-Ben was converted to Na-Bentonite by Na<sub>2</sub>CO<sub>3</sub> addition level; 0,58% Na<sub>2</sub>O equivalent.

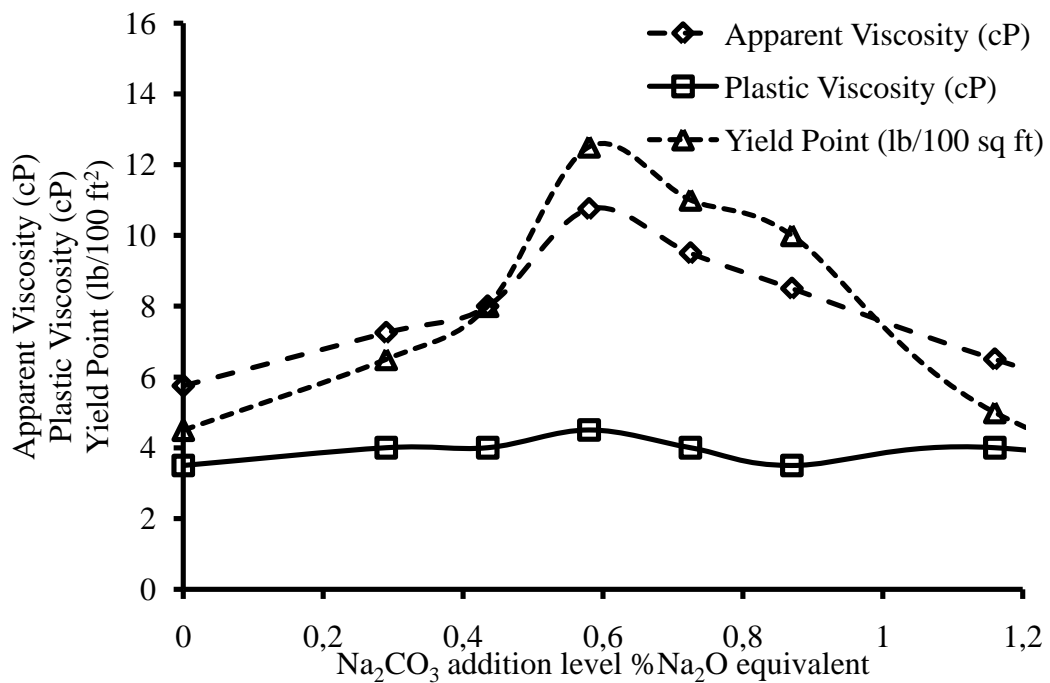


Figure 5-11 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Rheology of C-Ben

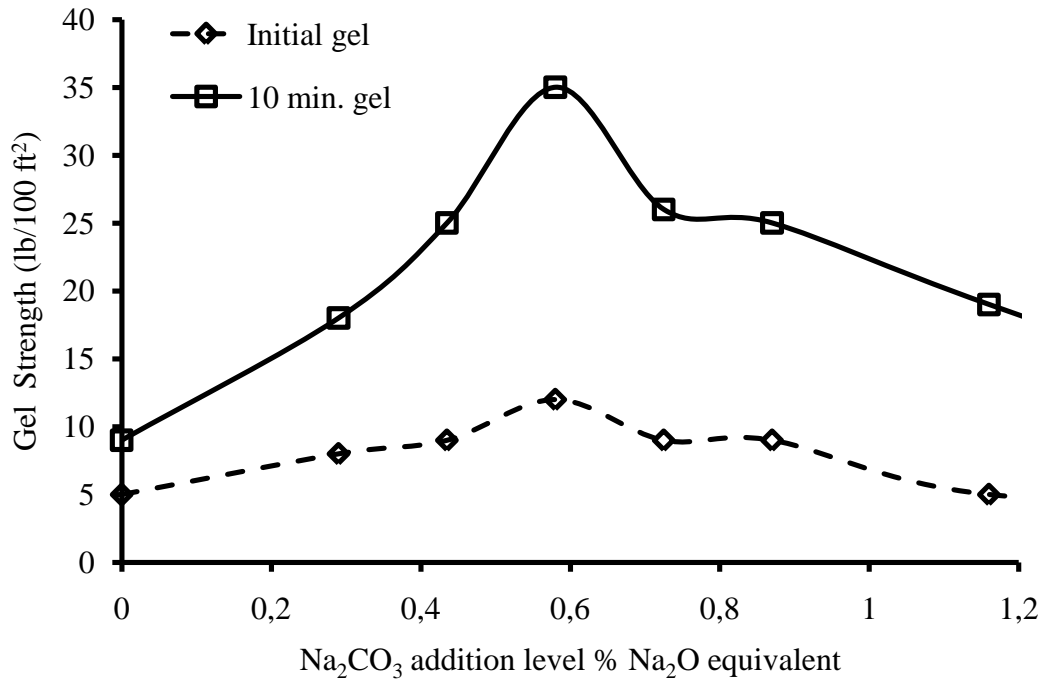


Figure 5-12 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Gel Strength of C-Ben

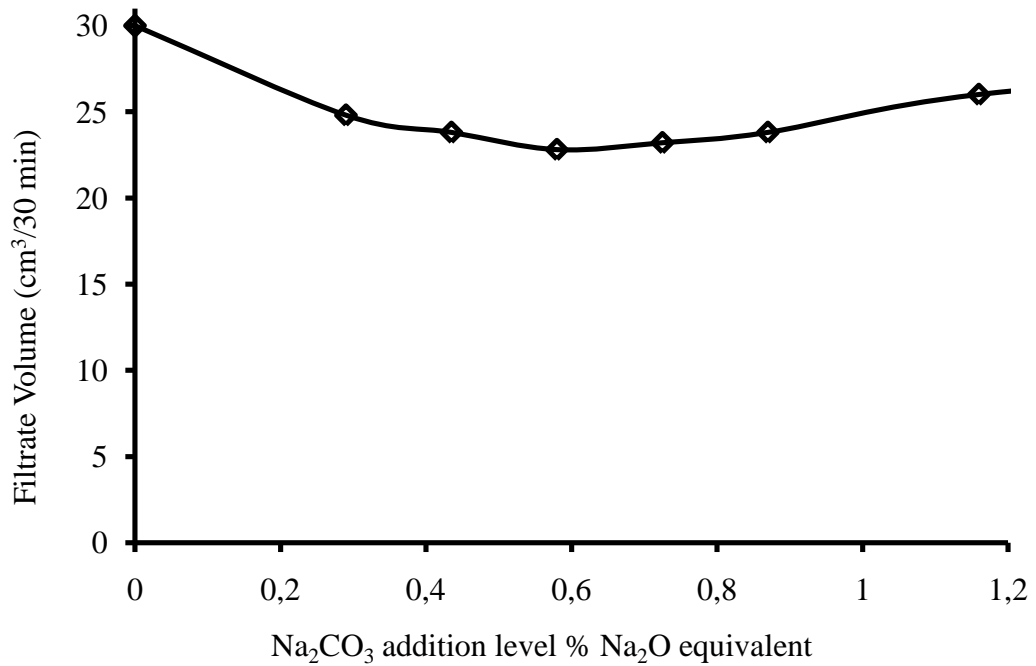


Figure 5-13 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Filtrate Volume of C-Ben



### 5.2.3 The Effect of $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ on the Rheology and Filtrate Properties of E-Ben

Figures 5-14 through 5-16 (see also appendix Table A-8) summarize the effect of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  on the rheological and filtrate properties of bentonite suspension. Incremental addition of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  resulted in a progressive increase in the values of the apparent viscosity (AV) up to  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 3,82%  $\text{Na}_2\text{O}$  equivalent (Figure 5-14). The addition of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  solution more than 3,82%  $\text{Na}_2\text{O}$  also produced a slight drop in viscosity. The reason for increasing and decreasing in rheology is the same as discussed in page 55 and 56. Meanwhile, the results of filter loss values of the activated samples are shown in Figure 5-16. At the 3,82%  $\text{Na}_2\text{O}$  concentration of Na-Borate very high levels of gel strength were observed for E-Ben (Figure 5-15).

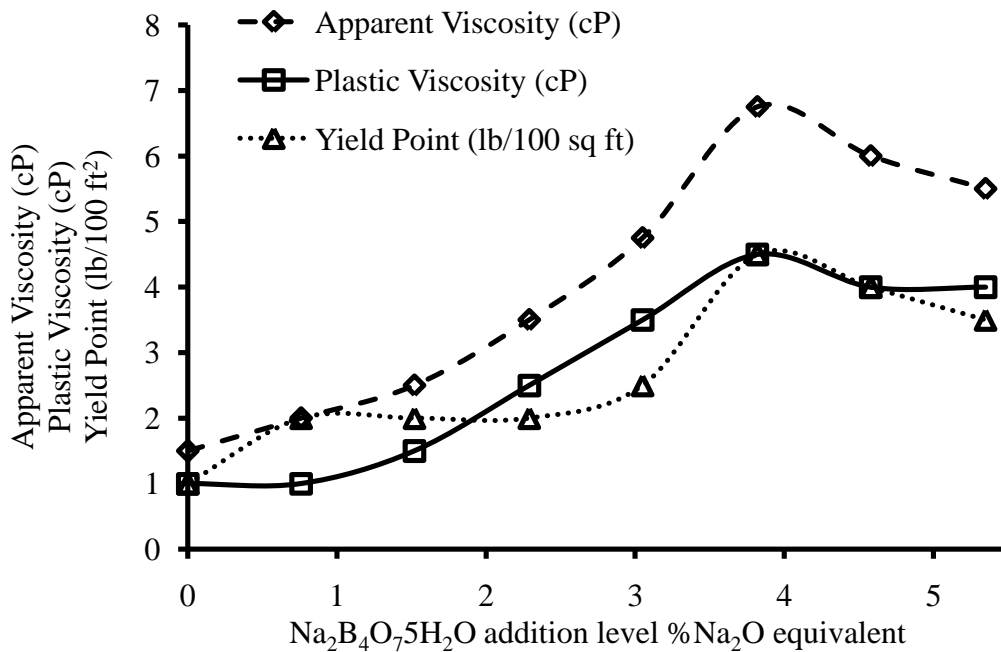


Figure 5-14 Effect of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Dissolved at Room Temperature on the Rheology of E-Ben

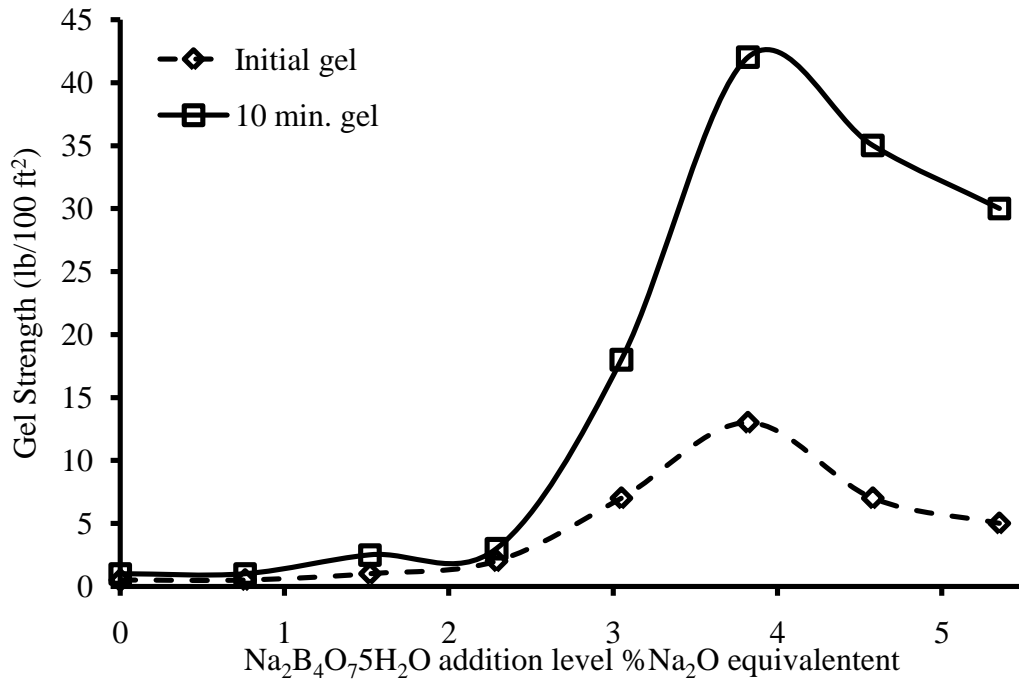


Figure 5-15 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Gel Strength of E-Ben

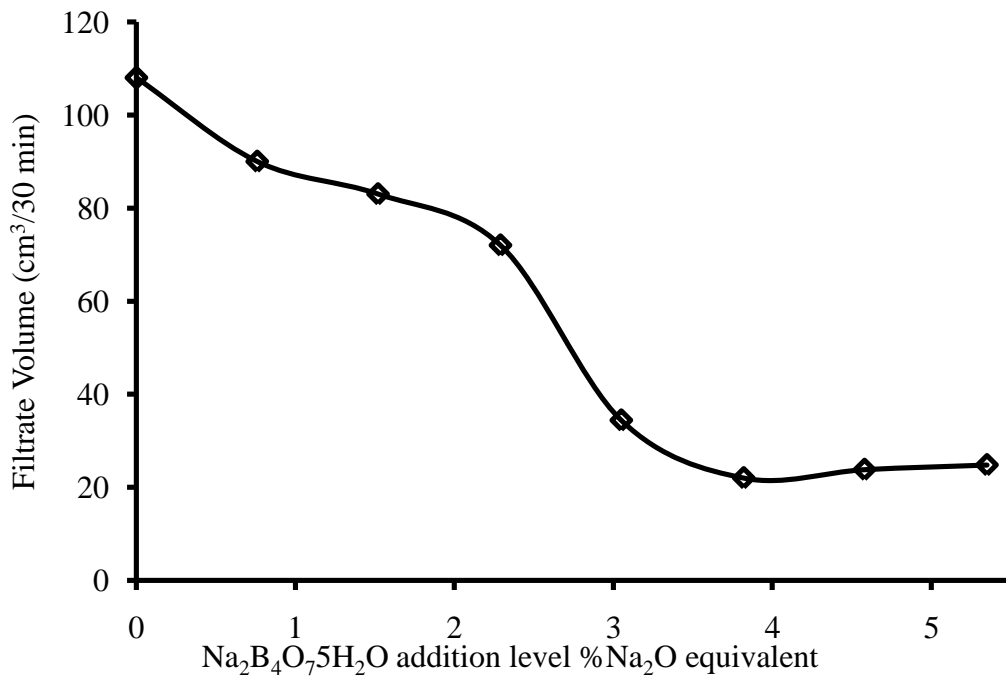


Figure 5-16 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Filtrate Volume of E-Ben

In the previous experiment the water quantity in saturated  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  solution at a room temperature for maximum activation was quite high (5000 ml for 1 kg bentonite). Because of this reason  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  was dissolved at 55 °C for E-Ben activation.

Figures 5-17 through 5-19 (see also appendix Table A-9) show the rheology and filtrate properties of E-Ben by mixing with different amounts of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  dissolved at 55 °C. After adding 3,78%  $\text{Na}_2\text{O}$  the maximum apparent viscosity (4,75 cP) shown in Figure 5-17 and the minimum filtrate volume (27  $\text{cm}^3$ ) shown in Figure 5-19 were achieved.

Figure 5-18 shows that the 10 gel-strength of bentonite increased the maximum level with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 3,78%  $\text{Na}_2\text{O}$  equivalent.

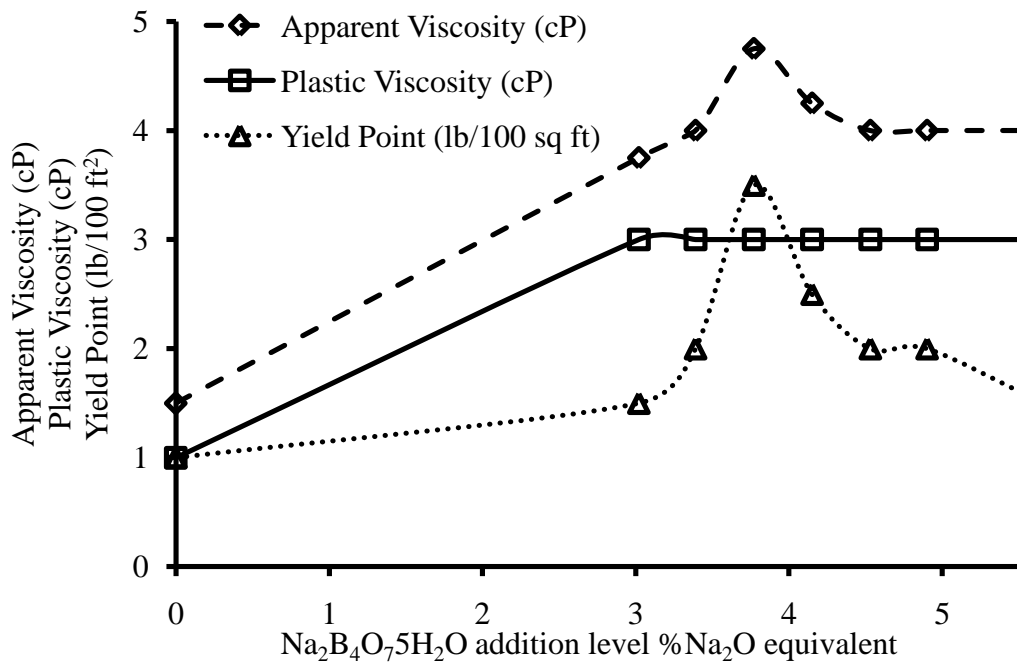


Figure 5-17 Effect of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Dissolved at 55 °C on the Rheology of E-Ben

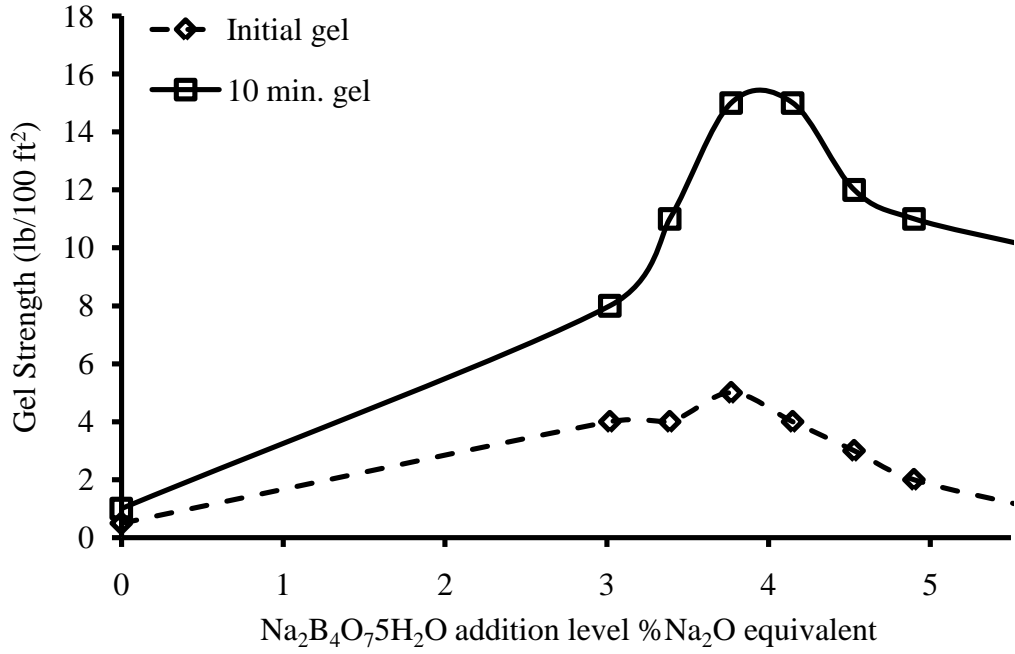


Figure 5-18 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at 55 °C on the Gel-Strength of E-Ben

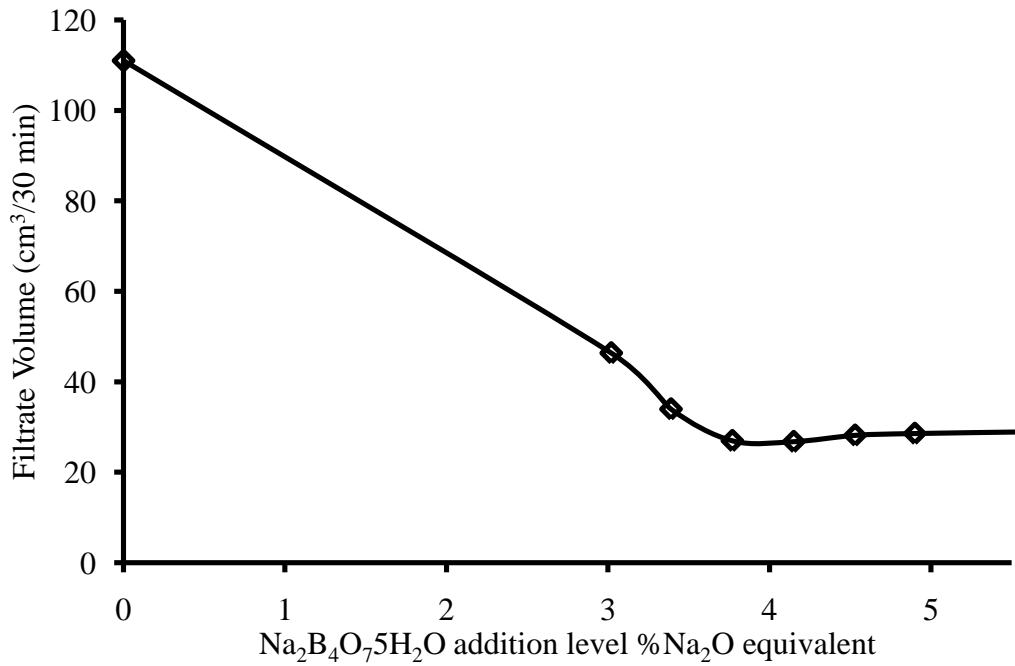


Figure 5-19 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at 55 °C on the Filtrate Volume of E-Ben

### 5.2.4 The Effect of $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ on the Rheology and Filtrate Properties of C-Ben

The results of the rheological and filtrate properties of C-Ben activated with Na-Borate are given in Figures 5-20 through 5-22 (see also appendix Table A-10).

$\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  caused an increase in rheological properties of C-Ben in concentration up to 0,76%  $\text{Na}_2\text{O}$ . The addition of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  more than 0,76%  $\text{Na}_2\text{O}$  also produced a drop in rheology (Figure 5-20). At the same time, the plastic viscosity of the activated C-Ben varied between 3-4 cP.

The 10-gel strength of activated bentonite increased from 9 to 16 lb/100 ft<sup>2</sup> and the filtrate loss of that decreased from 30 to 25 cm<sup>3</sup> by  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,76%  $\text{Na}_2\text{O}$  equivalent.

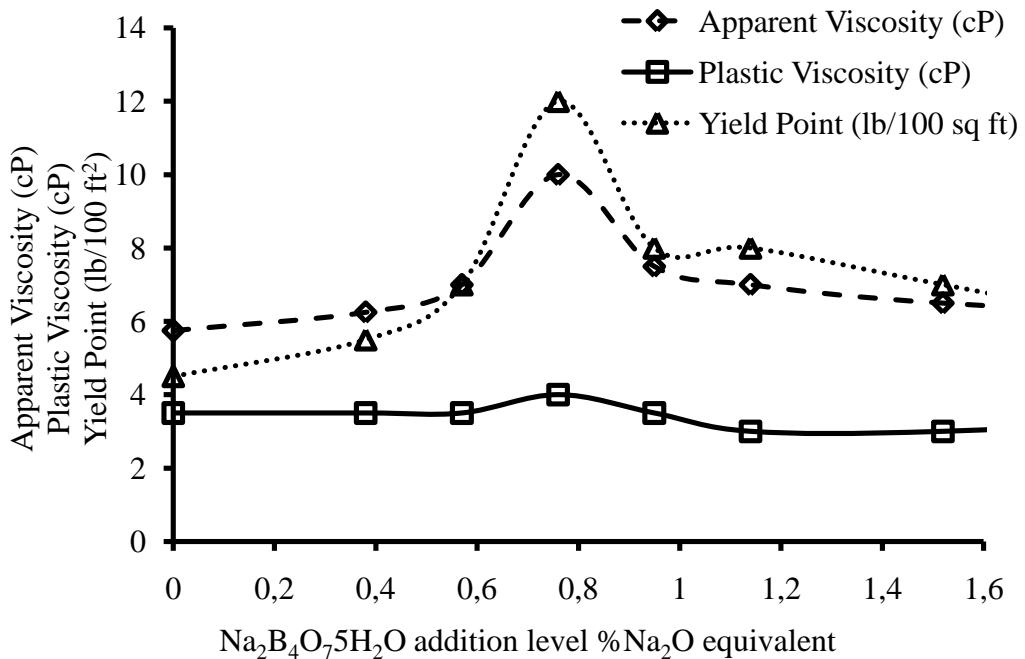


Figure 5-20 Effect of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Dissolved at Room Temperature on the Rheology of C-Ben

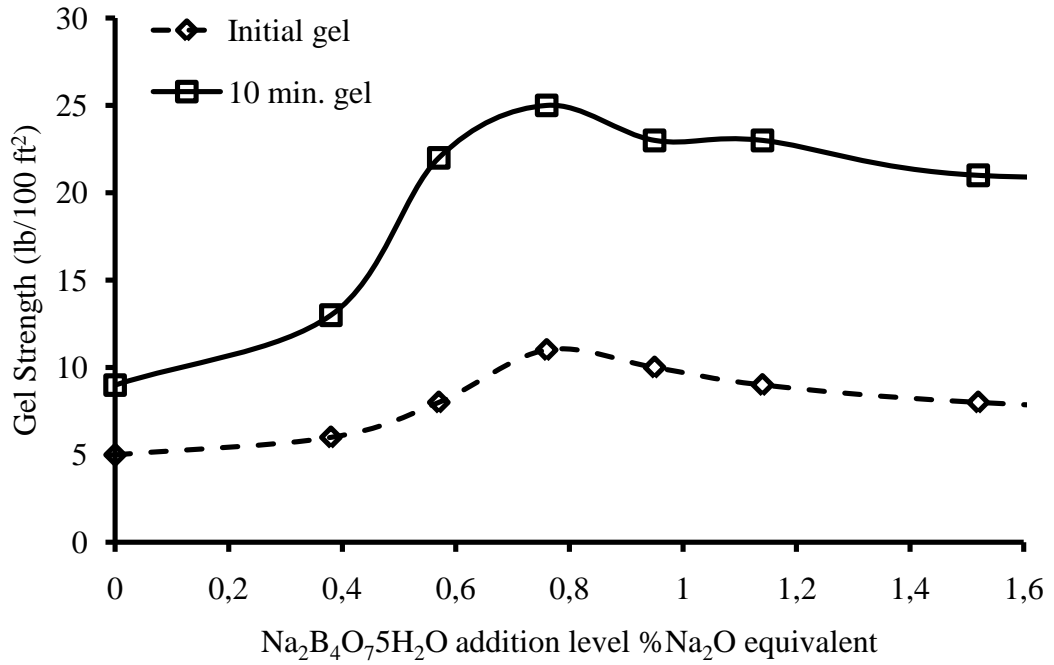


Figure 5-21 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Gel Strength of C-Ben

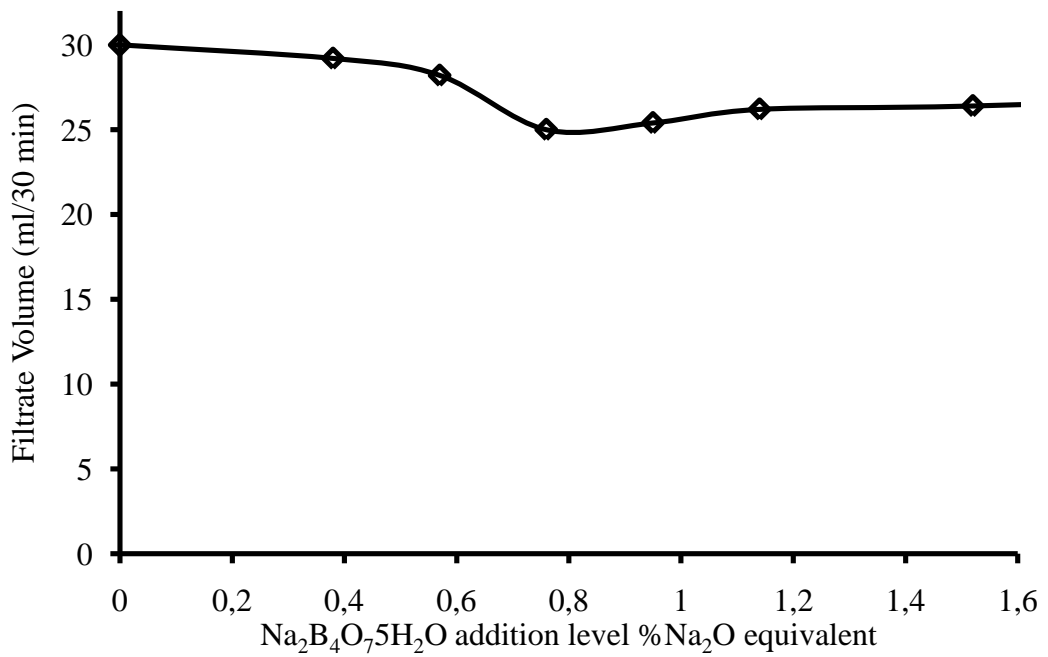


Figure 5-22 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Filtrate Volume of C-Ben

### 5.2.5 Comparison of the Effect of $\text{Na}_2\text{CO}_3$ and $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ on E-Ben and C-Ben

Figures 5-23 through 5-26 summarize the comparative effects of  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  on the apparent viscosity and filtrate volume of E-Ben and C-Ben, respectively. It is seen from these figures that the effect of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  on the rheological behaviours of bentonites gave about the same result with  $\text{Na}_2\text{CO}_3$ . However, the required amount of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; % $\text{Na}_2\text{O}$  equivalent was higher than  $\text{Na}_2\text{CO}_3$  addition level; % $\text{Na}_2\text{O}$  equivalent.

Incremental addition of  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  resulted in a progressive increase in the values of the apparent viscosity and decrease in the values of filtrate volume. But such results were lower for apparent viscosity and higher for filtrate volume than the API requirements (shown as a dash-dot line in the figures) and this was attributed to both relatively low smectite content and the presence of high proportions of non-swelling minerals in these raw samples. The reason for this is that the CEC of E-Ben (66 meq/100g clay) and C-Ben (52 meq/100g clay) are lower than API (90 meq/100g clay) and Reşadiye (78 meq/100g clay) bentonites.

As a result, the best results are achieved with the following activated bentonites.

- i-  $\text{Na}_2\text{CO}_3$  addition level; 2,90%  $\text{Na}_2\text{O}$  equivalent for E-Ben,
- ii-  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  (dissolved at room temperature) addition level; 3,82%  $\text{Na}_2\text{O}$  equivalent for E-Ben,
- iii-  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  (dissolved at 55 °C) addition level; 3,78%  $\text{Na}_2\text{O}$  equivalent for E-Ben,
- iv-  $\text{Na}_2\text{CO}_3$  addition level; 0,58%  $\text{Na}_2\text{O}$  equivalent for C-Ben,
- v-  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,76%  $\text{Na}_2\text{O}$  equivalent for C-Ben.

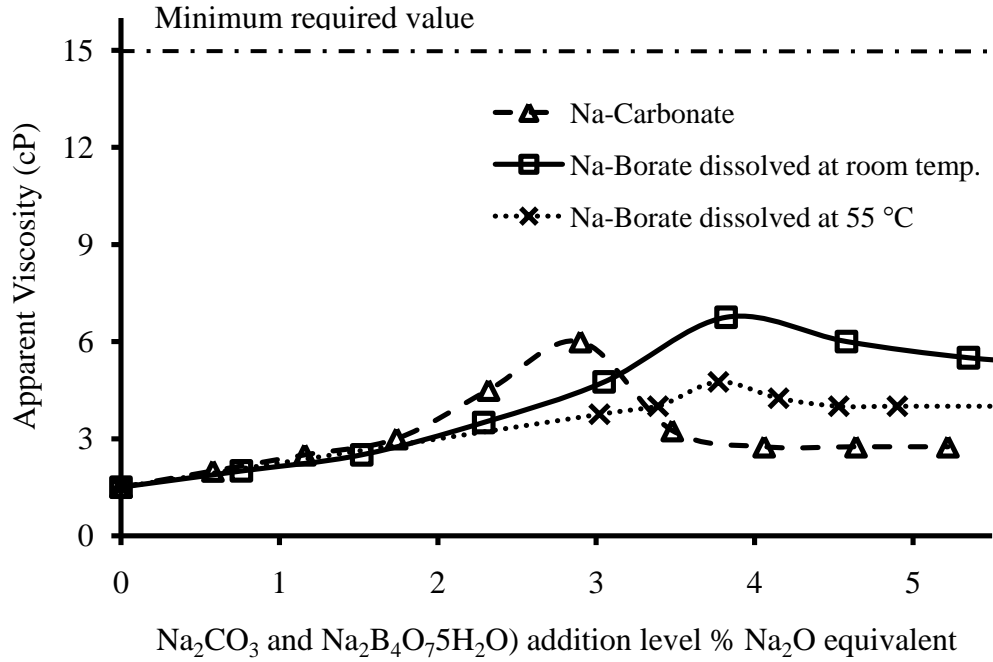


Figure 5-23 Comparison of the Effect of Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O on the Apparent Viscosity of E-Ben

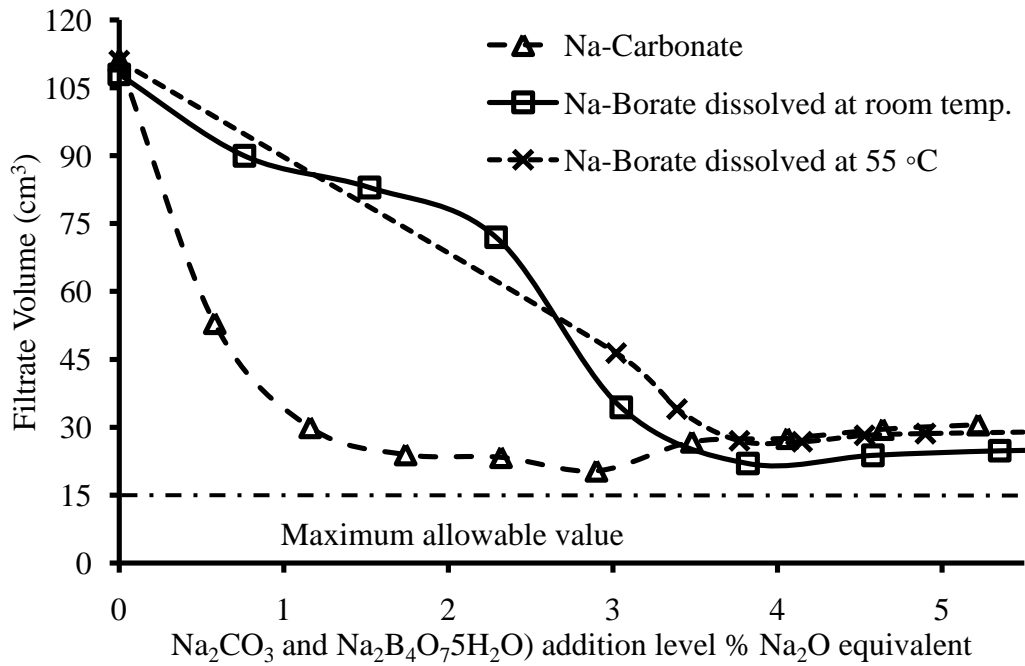


Figure 5-24 Comparison of the Effect of Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O on the Filtrate Volume of E-Ben



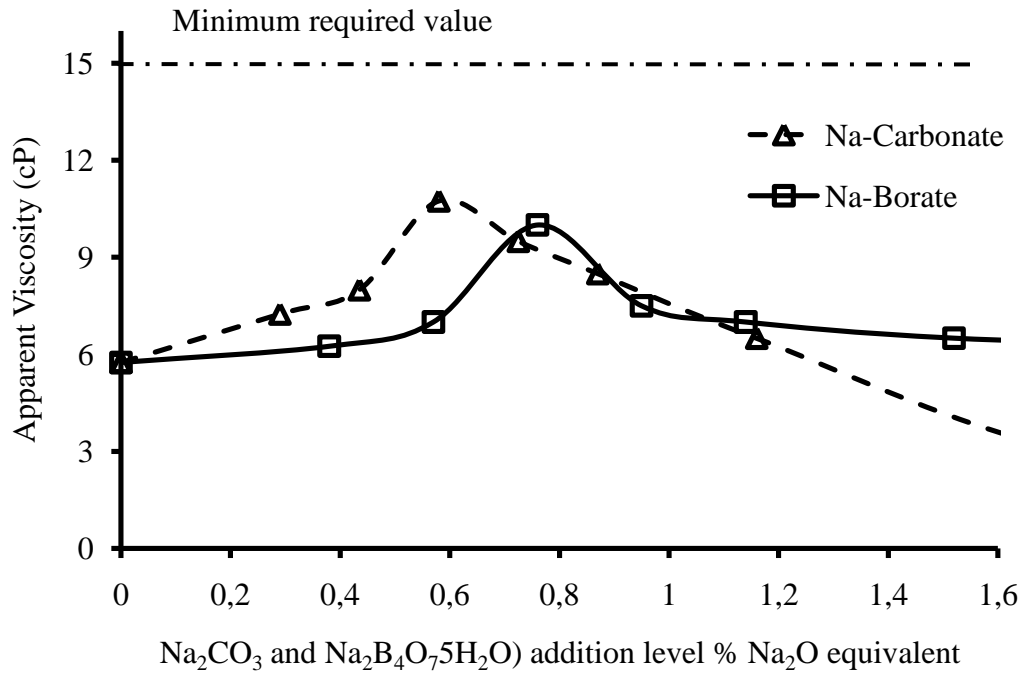


Figure 5-25 Comparison of the Effect of Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O on the Apparent Viscosity of C-Ben

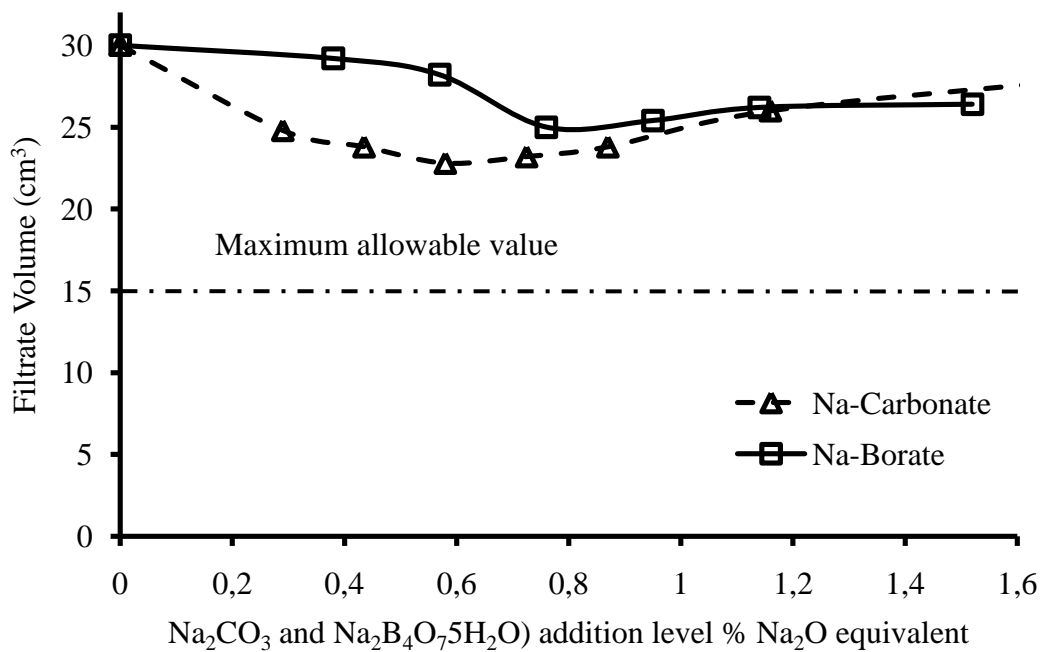


Figure 5-26 Comparison of the Effect of Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O on the Filtrate Volume of C-Ben

### **5.2.6 The Effect of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture on the Rheology and Filtrate Properties of E-Ben and C-Ben**

The effect of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture dissolved at room temperature on the rheology and filtrate properties of E-Ben and C-Ben were also investigated at different ratios (Na<sub>2</sub>CO<sub>3</sub>/Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O addition level % Na<sub>2</sub>O equivalent; 0,73/2,86, 1,45/1,91, 2,18/0,95 for E-Ben, 0,14/0,57, 0,29/0,38, 0,44/0,19 for C-Ben) as shown in Figures 5-27 and 5-28 (see also appendix Tables A-6 and A-7).

Figures 5-27 and 5-28 summarize the results obtained in the rheology and filtrate properties determinations of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture activated bentonites. The minimum required value for apparent viscosity ( $\geq 15$ ) and maximum required value for filtrate volume ( $\leq 15$ ) are shown in the dash-dot line while the maximum required value for YP/PV ( $\leq 3$ ) is shown in the dotted line. According to these results the rheology and filtrate properties were not enough to meet the required API standard.

Na<sub>2</sub>CO<sub>3</sub>/Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O addition level; 2,18/0,95 Na<sub>2</sub>O equivalent mixture for E-Ben and Na<sub>2</sub>CO<sub>3</sub>/Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O addition level; 0,29/0,38 Na<sub>2</sub>O equivalent mixture for C-Ben were chosen for treatment with polymers.

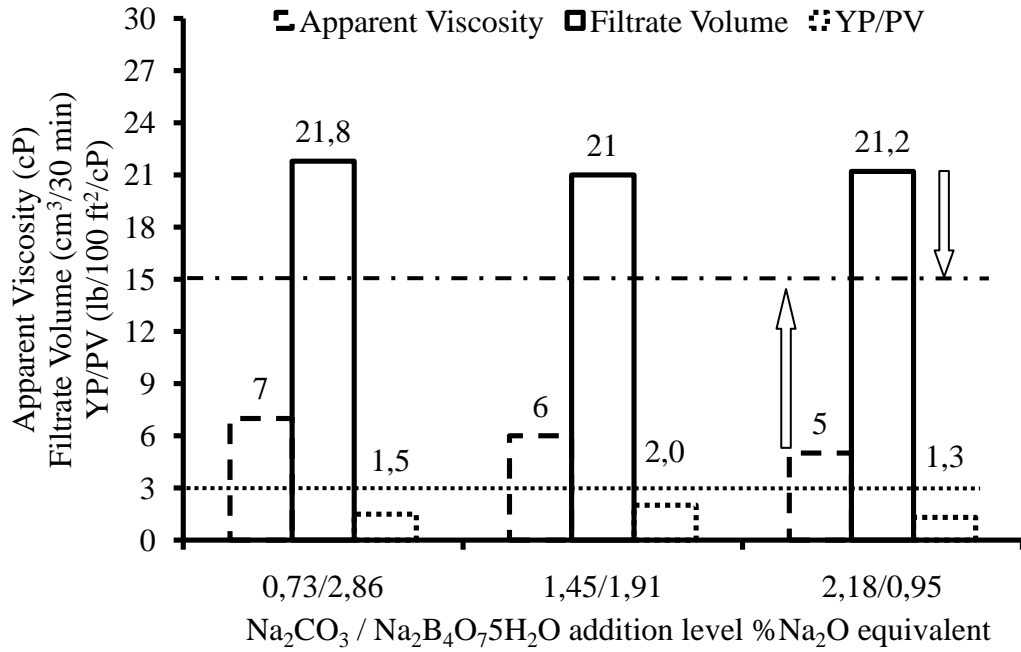


Figure 5-27 Effect of  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture Dissolved at Room Temperature on the Rheology and Filtrate Properties of E-Ben

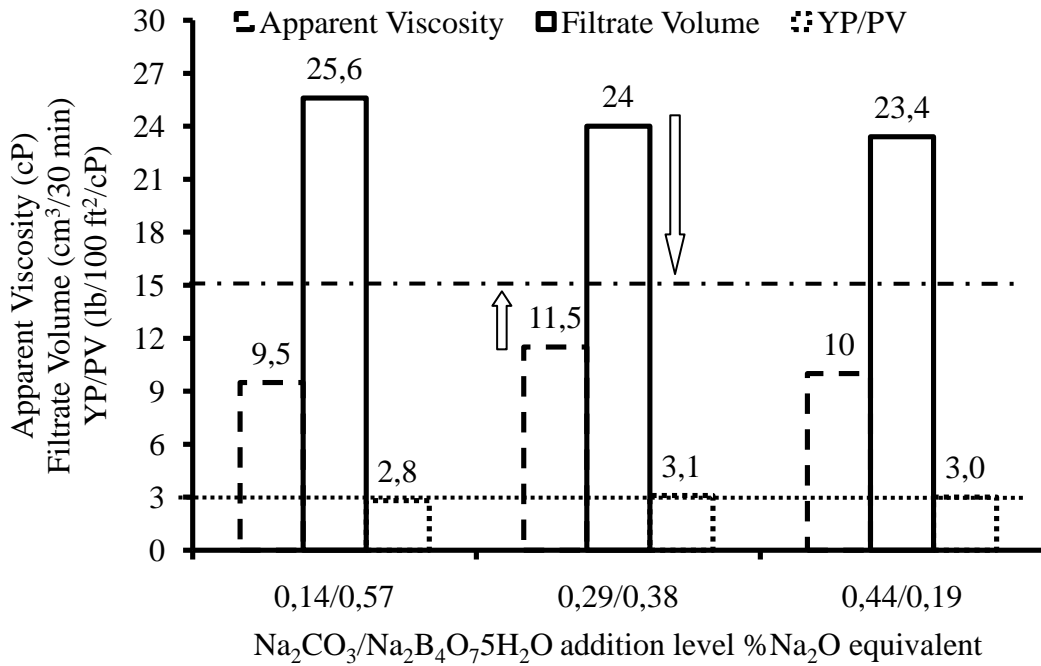


Figure 5-28 Effect of  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture Dissolved at Room Temperature on the Rheology and Filtrate Properties of C-Ben

### 5.3 The Effect of Inorganic Salts on the Chemical Structure of E-Ben and C-Ben

In order to understand whether added  $\text{Na}^+$  ions enter into the interlayer of bentonites or not, XRD analyses of following activated E-Ben and C-Ben were done to measure  $d_{001}$  basal spacing.

- i-  $\text{Na}_2\text{CO}_3$  addition level; 2,90%  $\text{Na}_2\text{O}$  equivalent for E-Ben,
- ii-  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 3,78%  $\text{Na}_2\text{O}$  equivalent for E-Ben,
- iii-  $\text{Na}_2\text{CO}_3$  addition level; 2,18%  $\text{Na}_2\text{O}$  equivalent and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,95%  $\text{Na}_2\text{O}$  equivalent mixture for E-Ben,
- iv-  $\text{Na}_2\text{CO}_3$  addition level; 0,58%  $\text{Na}_2\text{O}$  equivalent for C-Ben,
- v-  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,76%  $\text{Na}_2\text{O}$  equivalent for C-Ben,
- vi-  $\text{Na}_2\text{CO}_3$  addition level; 0,29%  $\text{Na}_2\text{O}$  equivalent and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,38%  $\text{Na}_2\text{O}$  equivalent mixture for C-Ben.

The X-ray diffraction patterns of the  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ , and  $\text{Na}_2\text{CO}_3 - \text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated E-Ben are illustrated in Figures 5-29, 5-30 and 5-31. The interlayer spacing ( $d_{001}$ ) was decreased by  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ , and  $\text{Na}_2\text{CO}_3 - \text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activation for E-Ben from 15,62 to 12,78, 12,77, and 12,82 °A, respectively. Besides, the X-ray diffraction patterns of the  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ , and  $\text{Na}_2\text{CO}_3 - \text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated C-Ben are illustrated in Figures 5-32, 5-33 and 5-34.  $d_{001}$  spacing was decreased by  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ , and  $\text{Na}_2\text{CO}_3 - \text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activation for C-Ben from 13,15 to 12,16, 12,79, and 12,51 °A, respectively.

The sheets in clay structures bound each other with covalent bonds and therefore, their crystal structures are stable. In contrast, the layers bound with Van der Waals bonds exchangeable cation changes the interlayer spacing when introduced in the interlayers. Browning and Perricone (1963) and Yıldız and Kuşcu (2007) stated that the interlayer spacing values close to 12,5 Å, indicate the presence of exchangeable  $\text{Na}^+$  and interlayer spacing of 15,5 Å implies predominance of  $\text{Ca}^{2+}$ . So, the decrease in XRD data revealed that  $\text{Na}^+$  ions replaced  $\text{Ca}^{2+}$  ions in the interlayer and formed Na-bentonite due to ion exchange process.

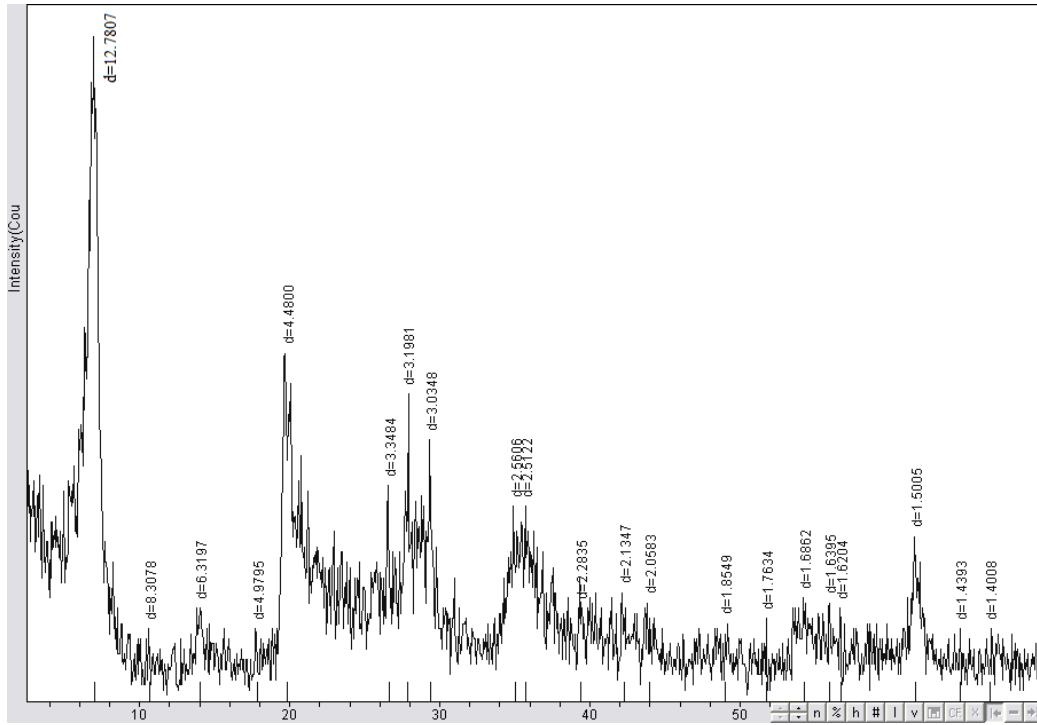


Figure 5-29 XRD of E-Ben Activated with  $\text{Na}_2\text{CO}_3$

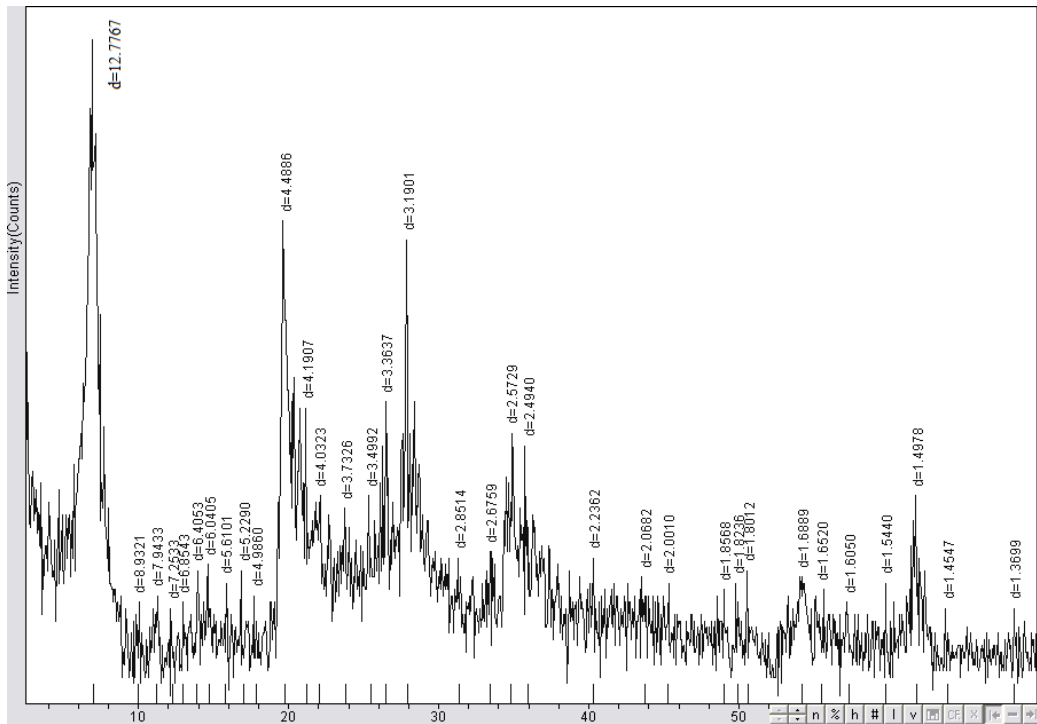


Figure 5-30 XRD of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

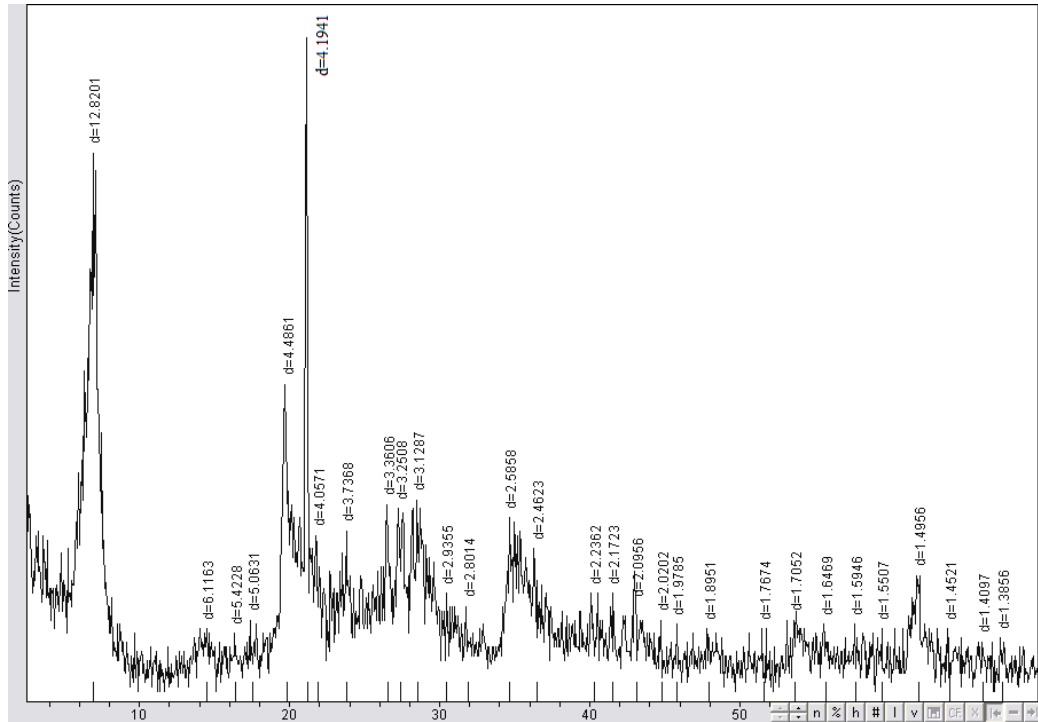


Figure 5-31 XRD of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

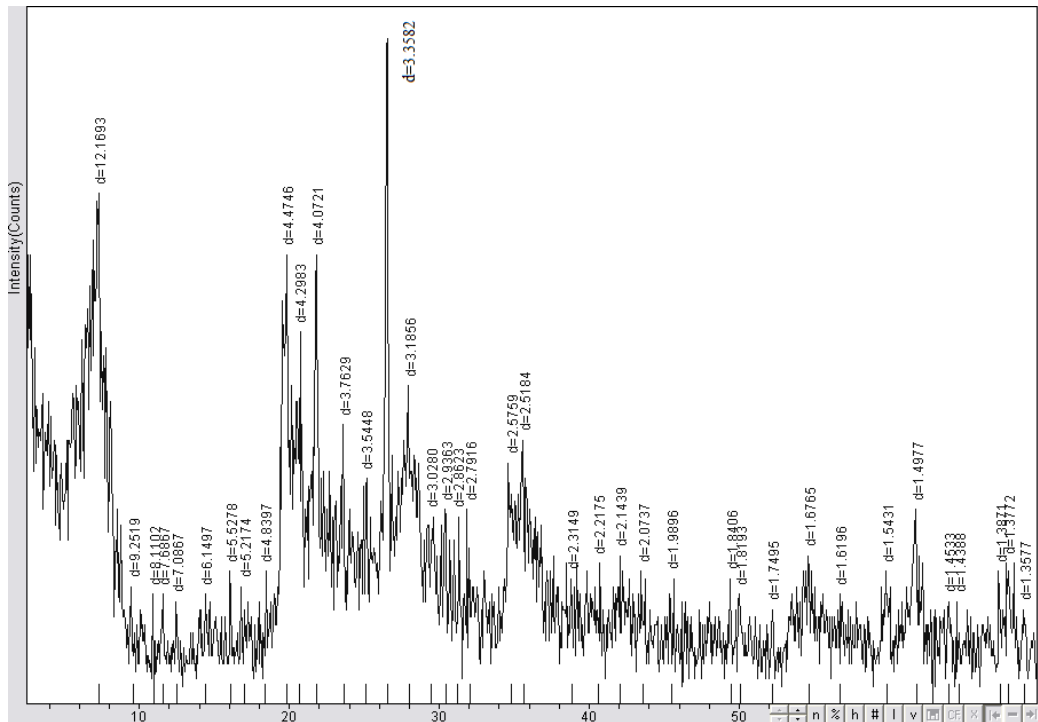


Figure 5-32 XRD of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

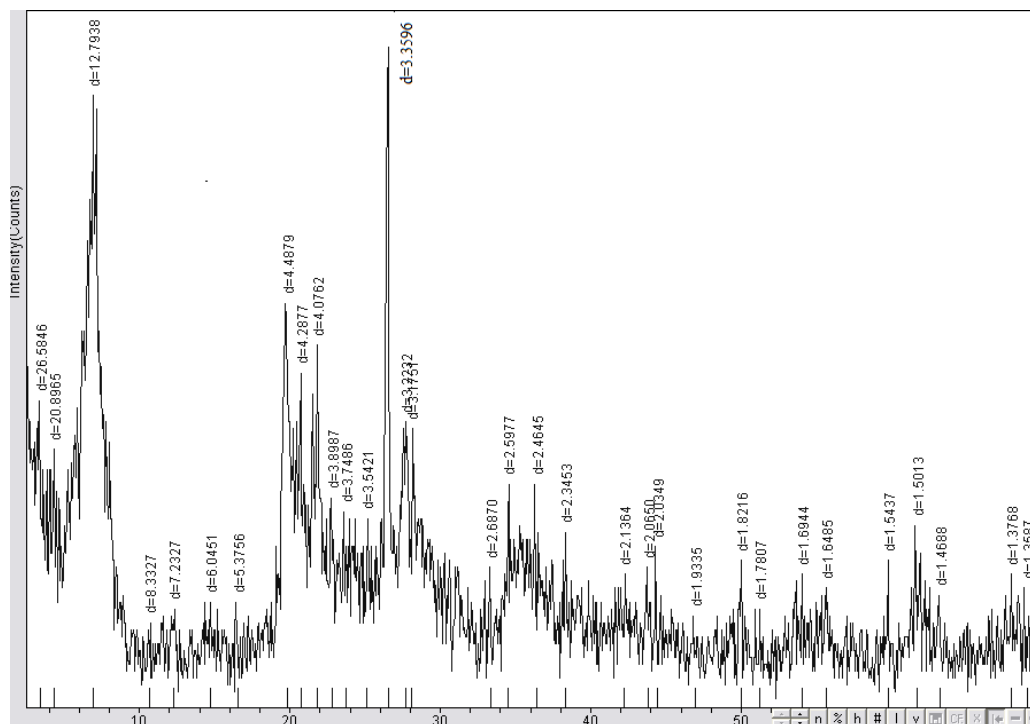


Figure 5-33 XRD of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

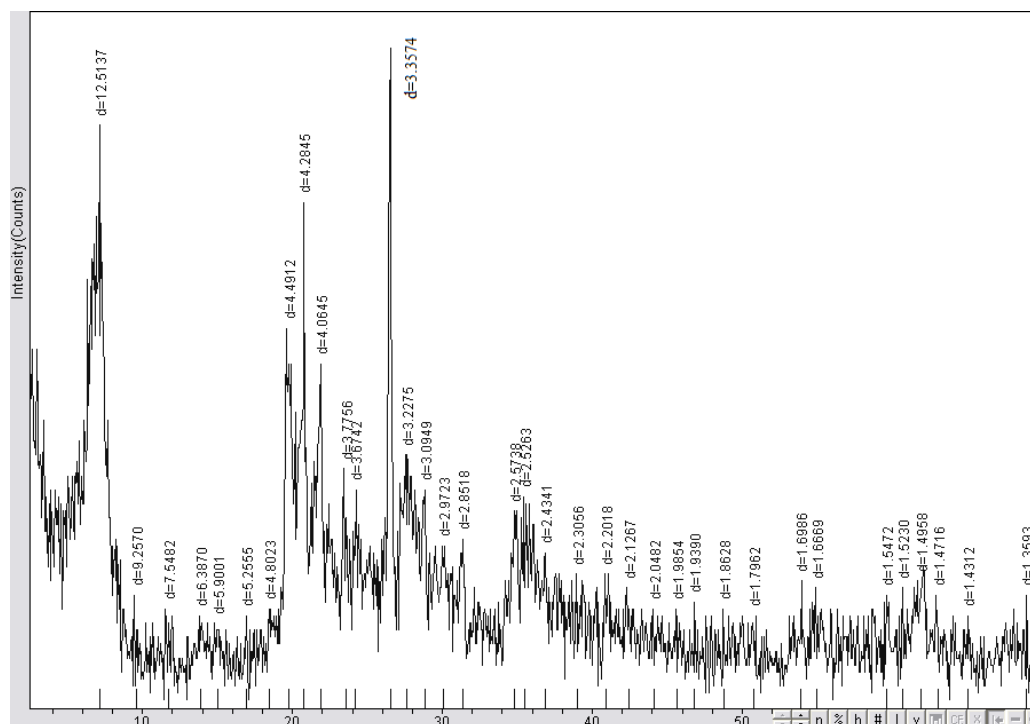


Figure 5-34 XRD of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

## **5.4 The Effect of Polymers on the Rheology and Filtrate Properties of E-Ben and C-Ben**

Since E-Ben and C-Ben activated with inorganic salts failed to meet the API specification they were treated with polymers. In this experiment low-, and high-viscosity samples of reagent grade CMC, PAC, and xanthan gum and dextrid were chosen because they have been used for viscosity and filtration control in drilling application.

In this study bentonite+polymer water suspensions were prepared and rheological properties (apparent viscosity, yield point, initial and 10 minute gel strengths) and filtrate volumes were measured using standard API procedure.

### **5.4.1 The Effect of Polymers on the Rheology and the Filtrate Properties of E-Ben**

As the apparent viscosity (1,5 cP) of E-Ben was very below the minimum required value of 15 cP, the raw bentonite samples were treated with different concentration of the polymers in this study. The effects of HV CMC, PAC R, and xanthan gum on the rheological and filtrate properties of E-Ben are given Figures 5-35 through 5-47 (see also appendix Tables B-1, B-2, and B-3).

It was found that the incremental addition of polymer resulted in a progressive increase in the values of the apparent viscosity (Figures 5-35 through 5-37). Firstly, the filtrate volume of muds decreased very sharply by binding a small amount of polymer onto bentonite. After the sharp decrease a steadily decrease in the filtrate volume was very likely due to multilayer adsorption with increasing concentration of polymer. This adsorption mechanism is either of electrostatic nature between anionic charges of the additives and positively charged clay edges or due to the interactions between the negatively charged clay particles and the polymers by the way of one of the following possibilities; (i) an anionic exchange between the surface hydroxyls of the mineral and the anionic part of the polymer; (ii) the formation of hydrogen bonds between the surface hydroxyl and the polymer chain; (iii) the establishment of bridges involving divalent ions, originated from electrostatic forces (Benchabane and Bekkour, 2006; Yalçın et al., 2002; Güngör and



Karaođlan, 2001). The mechanism for fluid loss control in drilling muds might be as the following; firstly, polymers adsorbed onto the particles are used to form a regular impermeable filter cake and secondly, the free polymers in solution are located in the pores of the filter cake and are plugging the pore spaces (Audibert et al., 1999; Plank and Hamberger, 1988).

The rheological and filtrate properties of polymers used in the treatment of bentonites are given in Figure 5- 38 (see also appendix Table B-4). It can be seen in the figure that PAC R did not form any impermeable cake, thereby increasing the filtration loss. This figure and the previous figures show that the decrease in API filtrate volume due to increase in polymer concentration and improvement of mudcake vary depending on the composition and physico-chemical nature of polymers.

Meanwhile an extra indication of extension was given by plastic viscosity, and yield point. Figures 5-39, 5-40 and 5-41 indicate that polymer adsorption by bentonite increased plastic viscosity and yield point and xanthan gum treated mud showed much better yield points than CMC and PAC treated mud. The values of YP/PV (Figures 5-42 through 5-44) ratios remained relatively constant except for xanthan gum treated mud. Figures 5-45 through 5-47, a plot of 0/10 gel strengths, showed that the xanthan gum treated mud imparted much higher gel strengths than the other polymer treated muds.

The maximum allowable values for filtrate volume and minimum allowable value for apparent viscosity are shown as a dash-dot line in the figures. As a result, 7,5% HV CMC, 3,75% PAC R and 7,5% xanthan gum treated E-Ben met the API specification required for application in drilling mud.

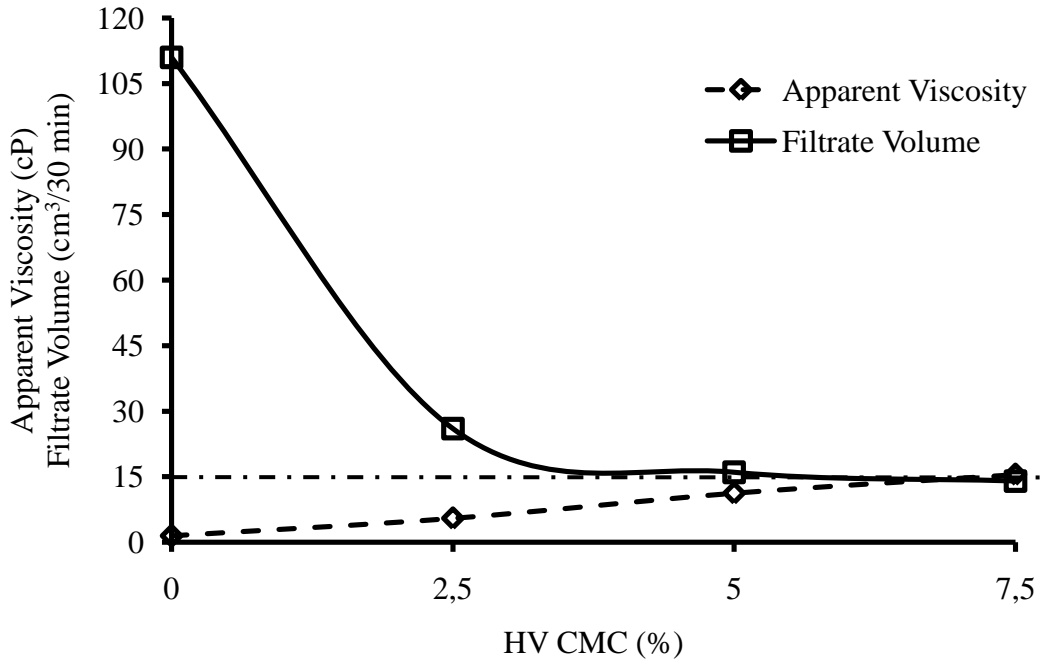


Figure 5-35 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of E-Ben

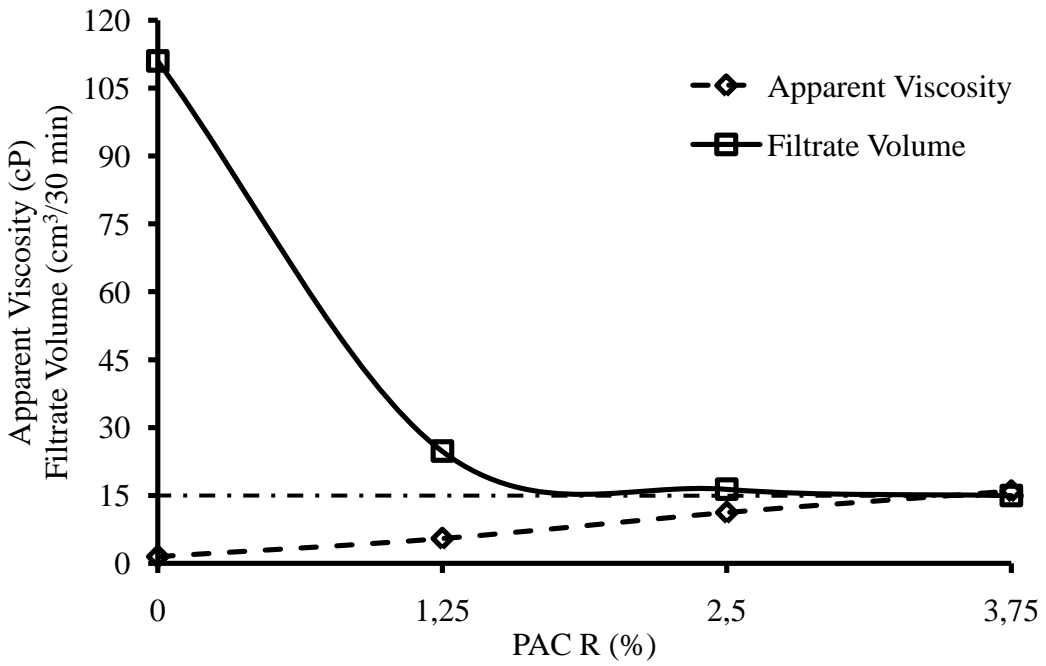


Figure 5-36 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of E-Ben

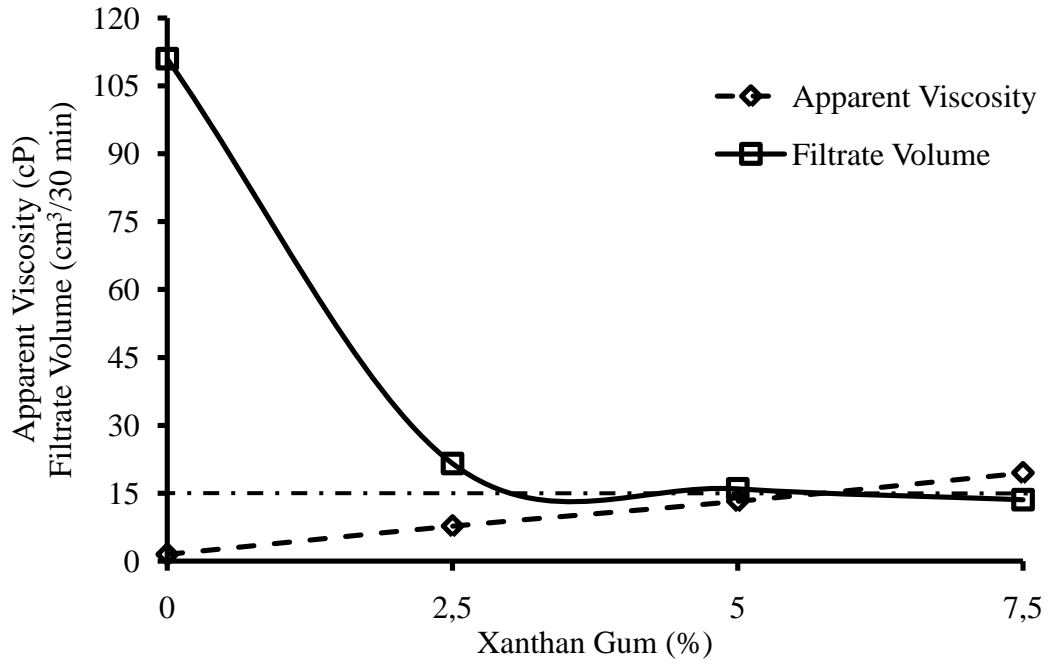


Figure 5-37 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of E-Ben

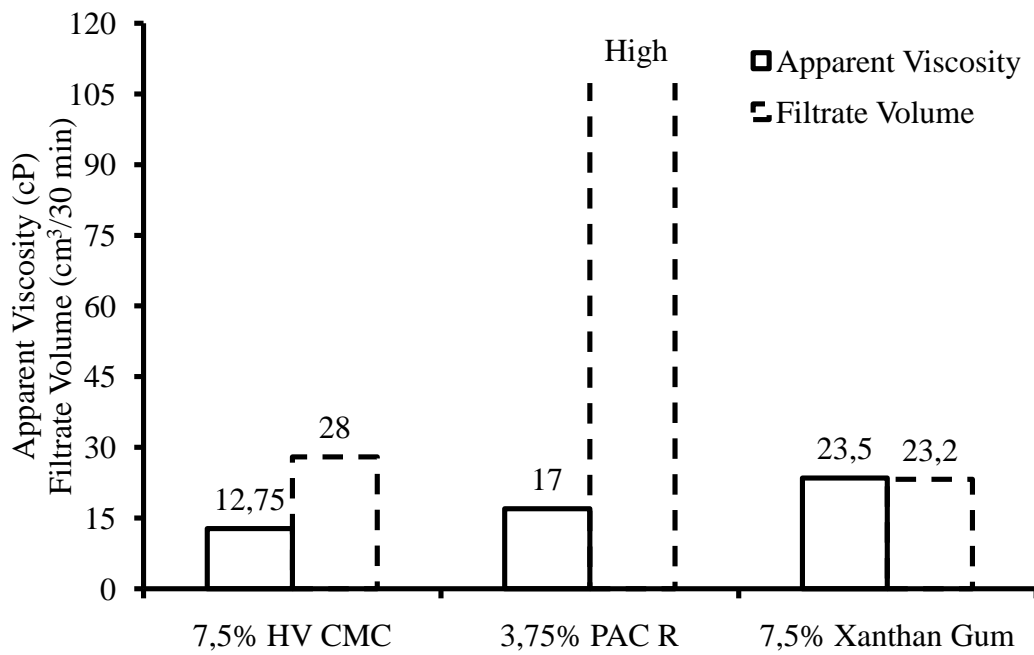


Figure 5-38 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of E-Ben

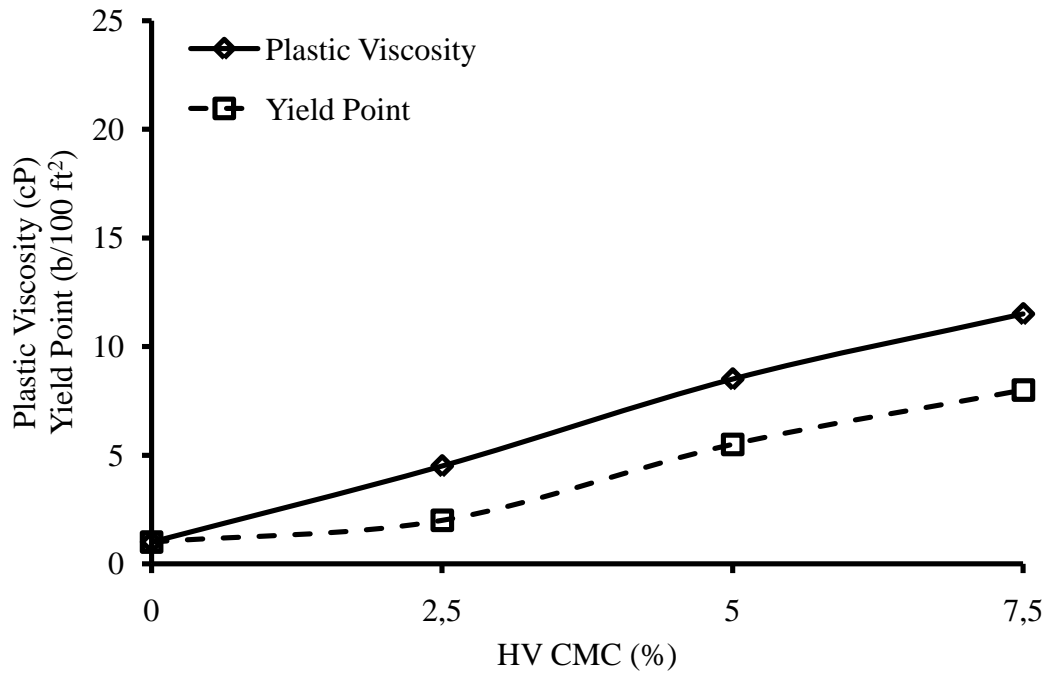


Figure 5-39 Effect of HV CMC on the Plastic Viscosity and Yield Point of E-Ben

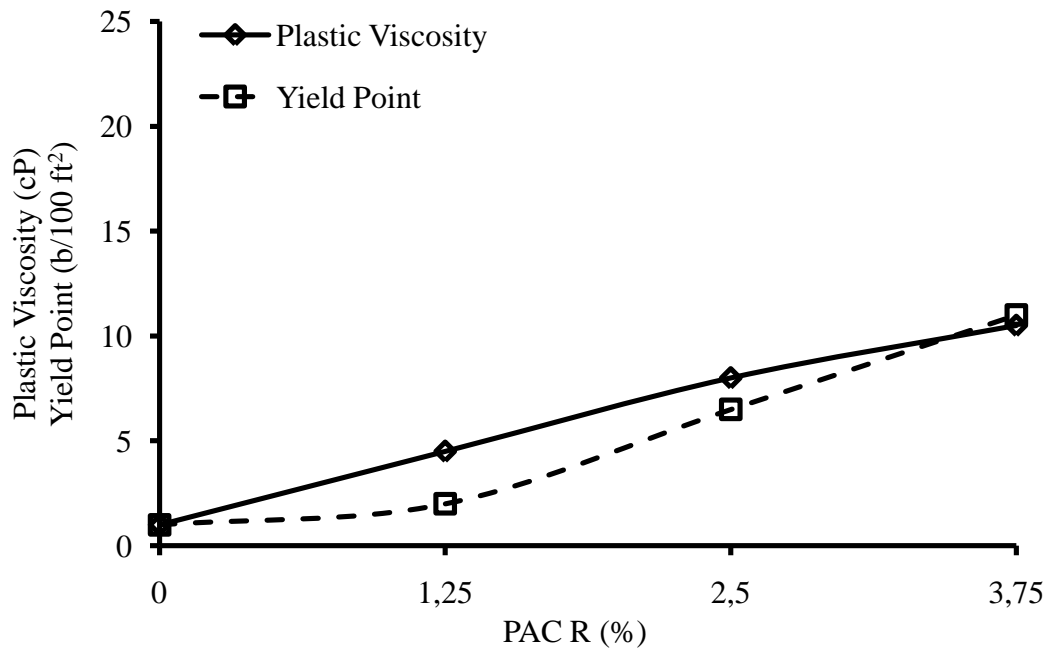


Figure 5-40 Effect of PAC R on the Plastic Viscosity and Yield Point of E-Ben

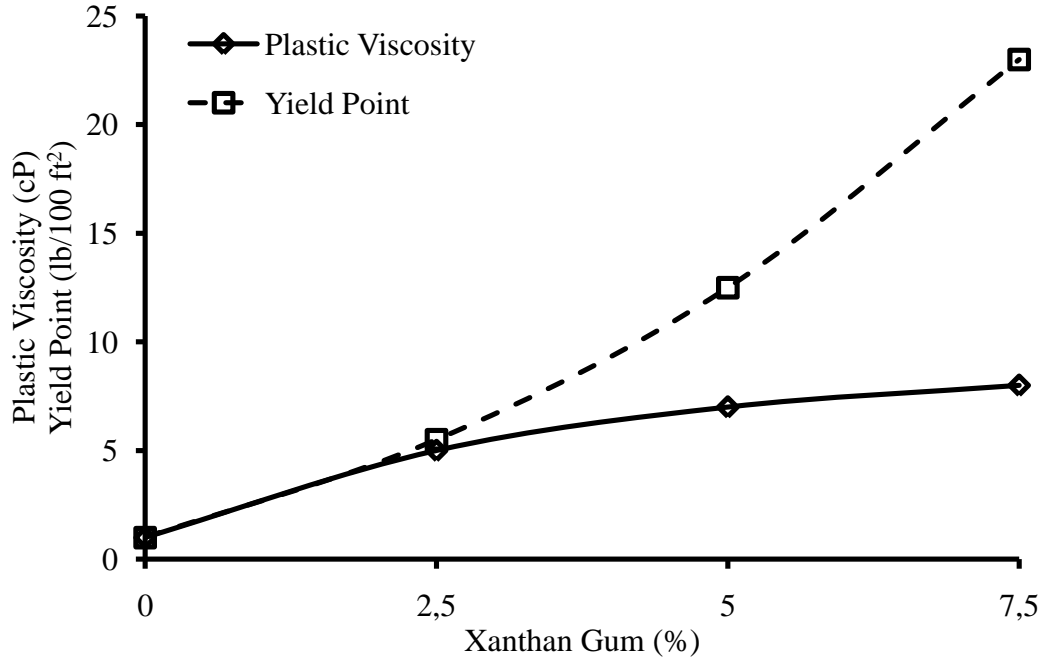


Figure 5-41 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of E-Ben

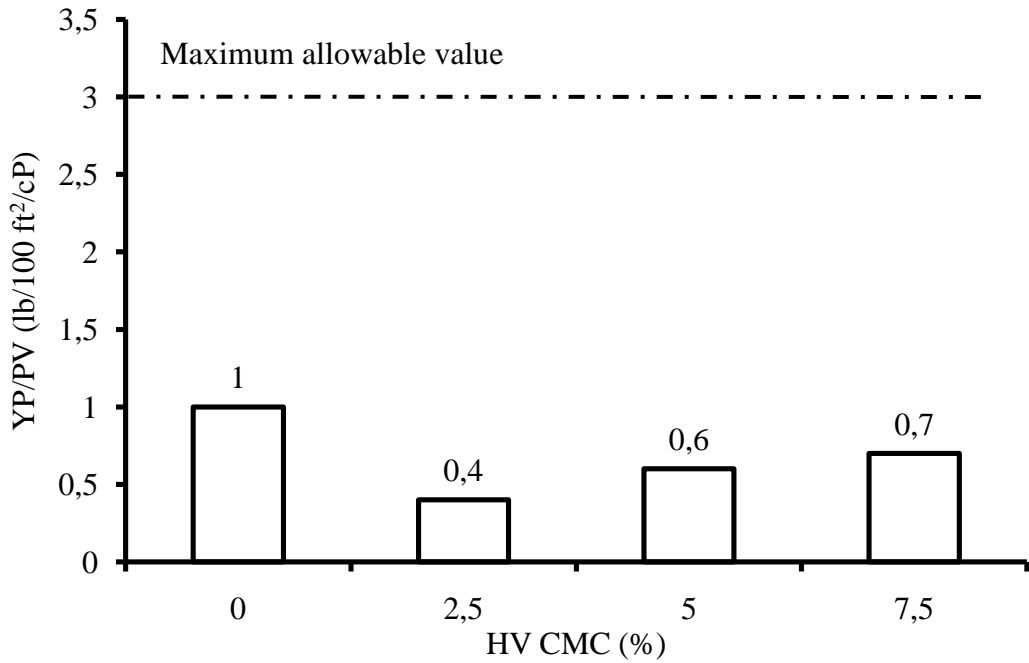


Figure 5-42 Effect of HV CMC on the YP/PV of E-Ben

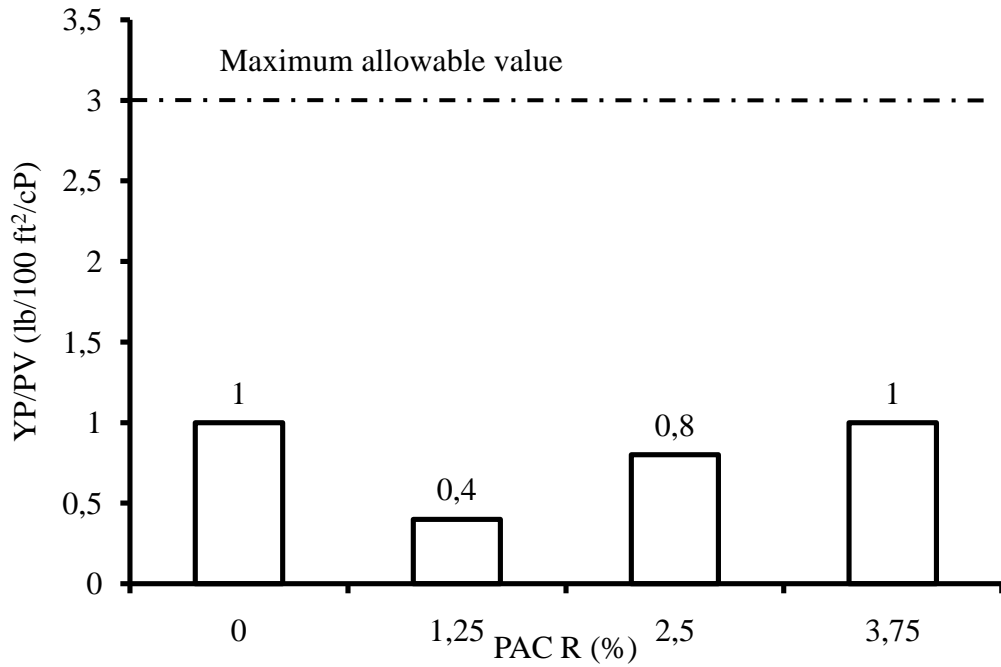


Figure 5-43 Effect of PAC R on the YP/PV of E-Ben

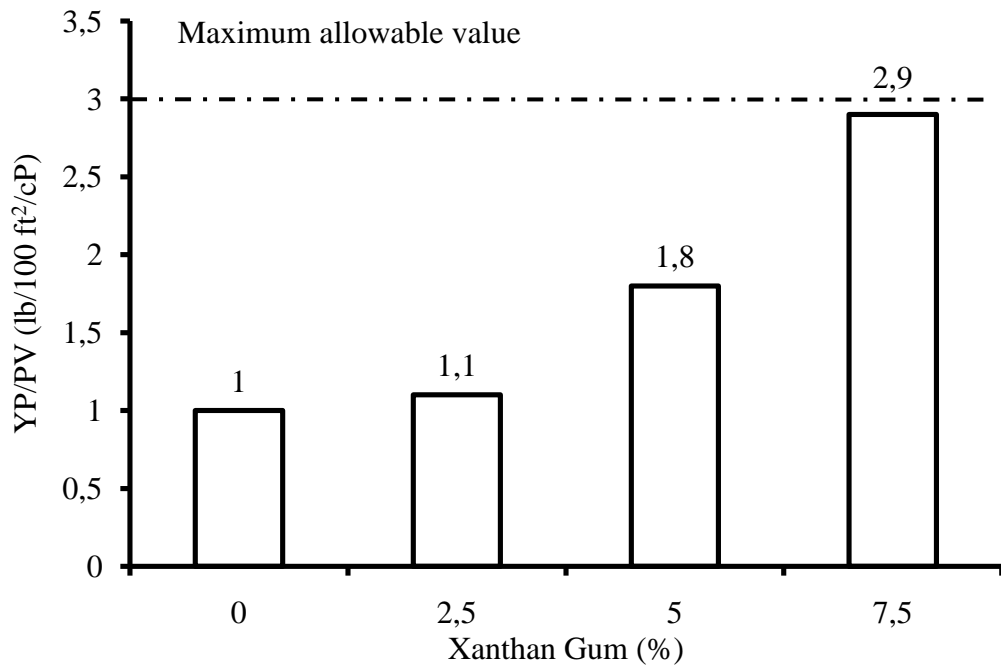


Figure 5-44 Effect of Xanthan Gum on the YP/PV of E-Ben

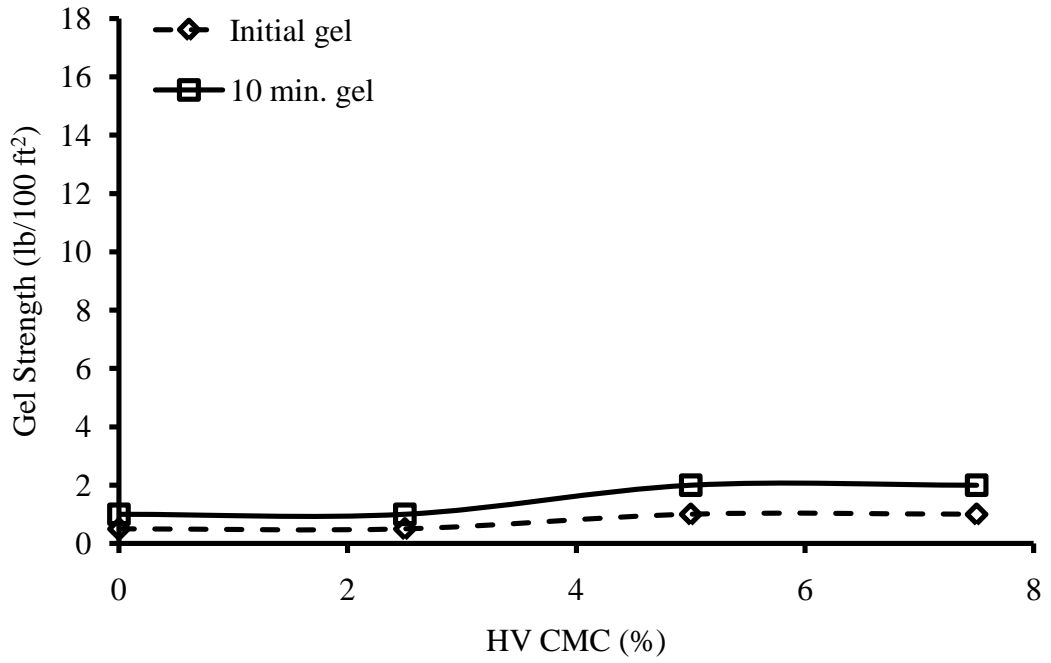


Figure 5-45 Effect of HV CMC on the Gel Strength of E-Ben

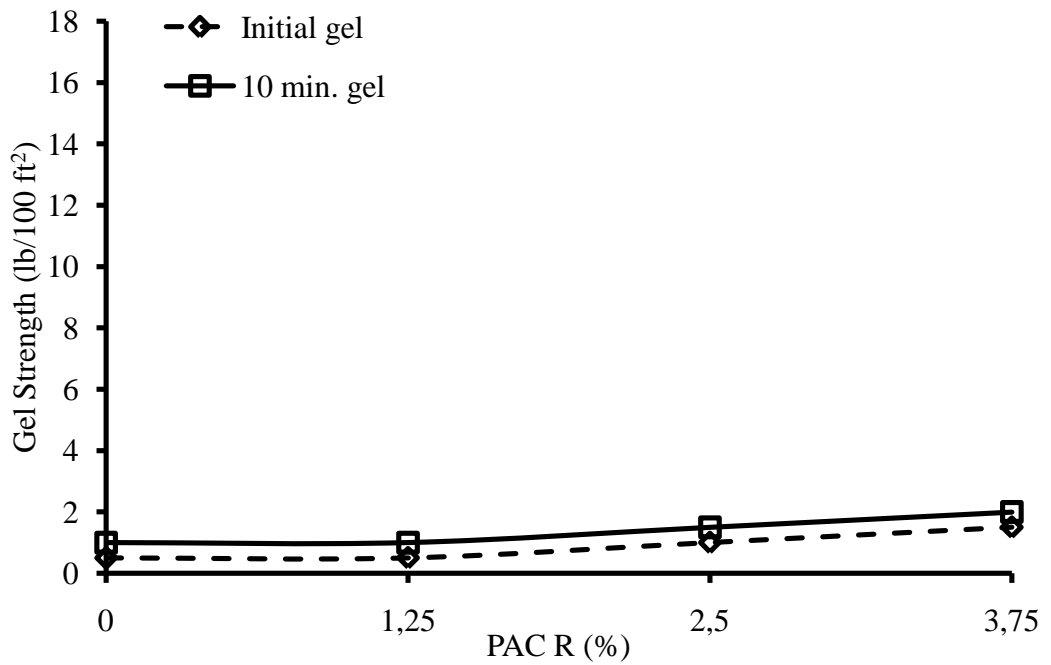


Figure 5-46 Effect of PAC R on the Gel Strength of E-Ben

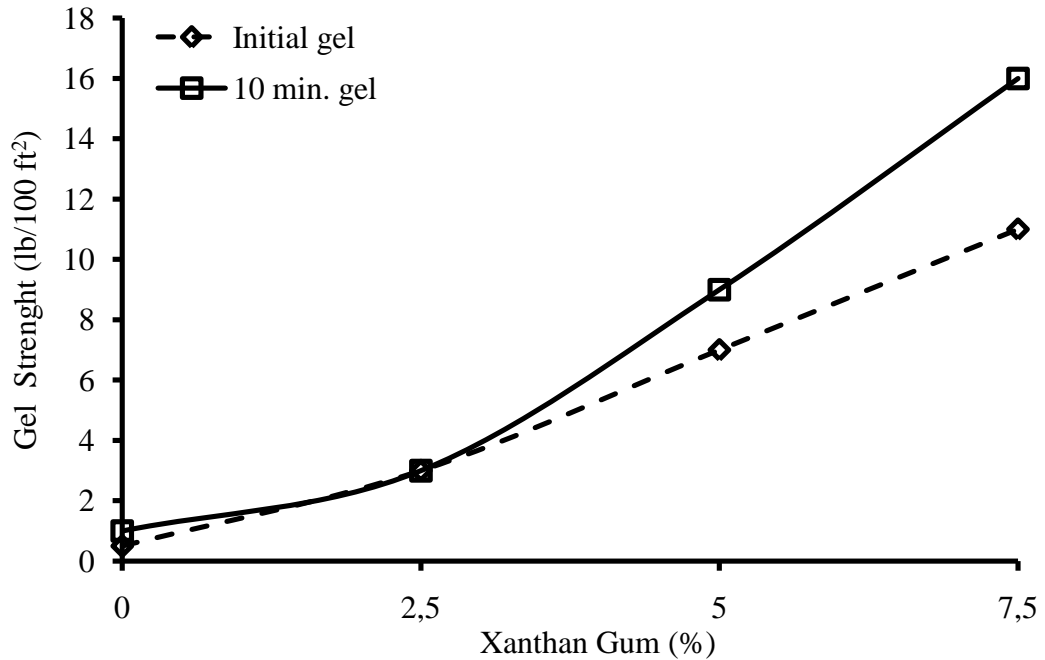


Figure 5-47 Effect of Xanthan Gum on the Gel Strength of E-Ben

#### 5.4.2 The Effect of Polymers on the Rheology and Filtrate Properties of E-Ben Activated with $\text{Na}_2\text{CO}_3$

The rheological properties and filtrate volumes of muds consisting of  $\text{Na}_2\text{CO}_3$  ( $\text{Na}_2\text{CO}_3$  addition level; 2,90%  $\text{Na}_2\text{O}$  equivalent) activated E-Ben and treated with different concentration of HV CMC, PAC R and xanthan gum are given in Figures 5-48 through 5-60 (see also appendix Tables B-5, B-6, and B-7).

Figures 5-48, 5-49 and 5-50 show that the effect of polymers on the rheology and filtrate properties of bentonite suspension were found to be steadily increase and decrease respectively with the increasing of polymer concentration. The steadily increase of viscosity and decrease of filtrate volume showed completely the binding of the polymer onto bentonite with 1,2% HV CMC, %1 PAC-R and 3% xanthan gum concentration. The rheological and filtrate properties of polymers used in the treatment of E-Ben activated with  $\text{Na}_2\text{CO}_3$  are given in Figure 5-51 (see also appendix Table B-8).



The increase in yield points (Figures 5-52 through 5-54) was observed with the increasing polymer concentrations. Figures 5-55 through 5-57 show the ratios of YP's and PV's under various polymers concentrations. All of the muds composition had a YP/PV ratios lower than 3 lb/100 ft<sup>2</sup>/cP (the maximum required value). On the other hand polymer treated raw bentonite, the 10-gel strengths of muds consisting of Na<sub>2</sub>CO<sub>3</sub> activated bentonite and CMC or PAC (Figures 5-58 through 5-60) increased with the increasing polymer concentration.

The results of apparent viscosity, filtrate volume and YP/PV values of muds of Na<sub>2</sub>CO<sub>3</sub> activated bentonite treated with polymer are within the standard limit. The high concentration 7,5% of CMC, 3,75% PAC R, and 7,5% xanthan gum required for treatment of unactivated bentonite mud reduced to 1,2% HV CMC, 1,0% PAC R and %3 xanthan gum, respectively for Na<sub>2</sub>CO<sub>3</sub> activated E-Ben mud. As a result the rheological properties are dependent on polymers types and quantities and as well as the type of the clay.

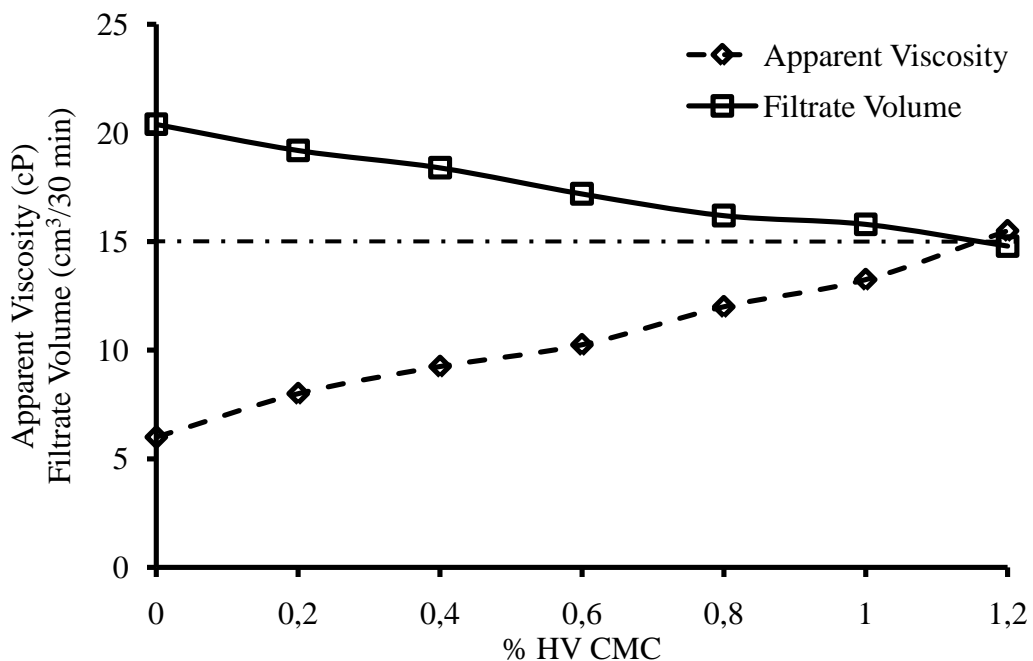


Figure 5-48 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

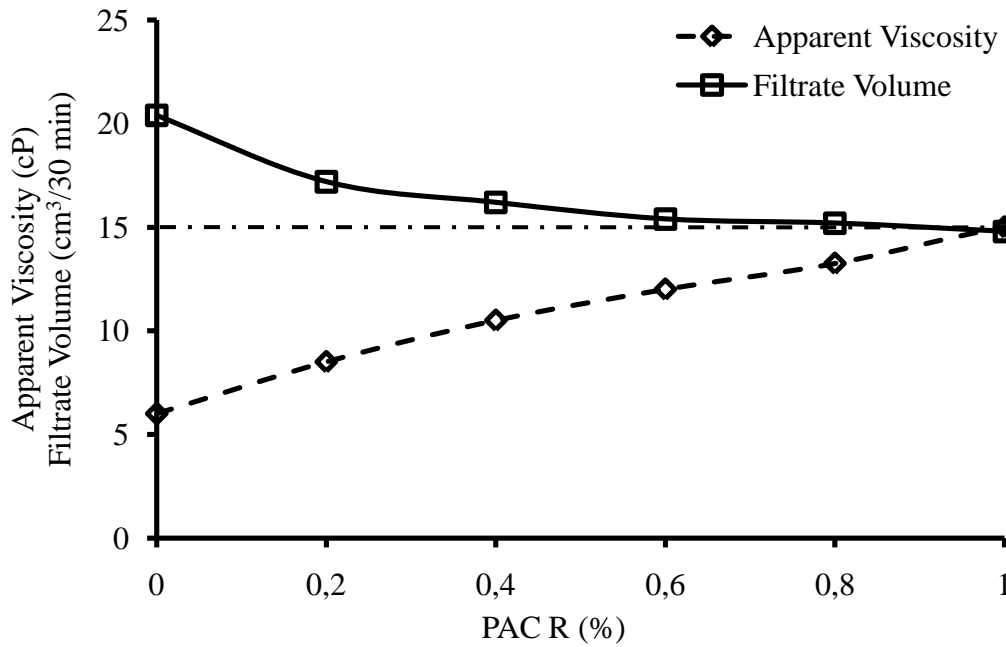


Figure 5-49 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{CO}_3$

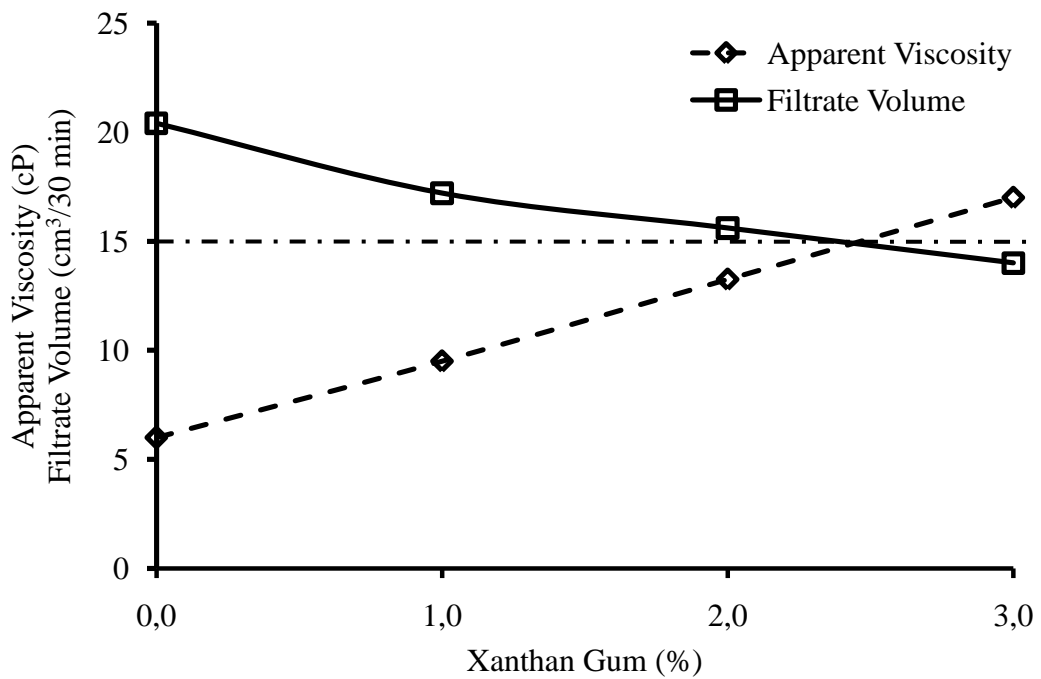


Figure 5-50 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{CO}_3$

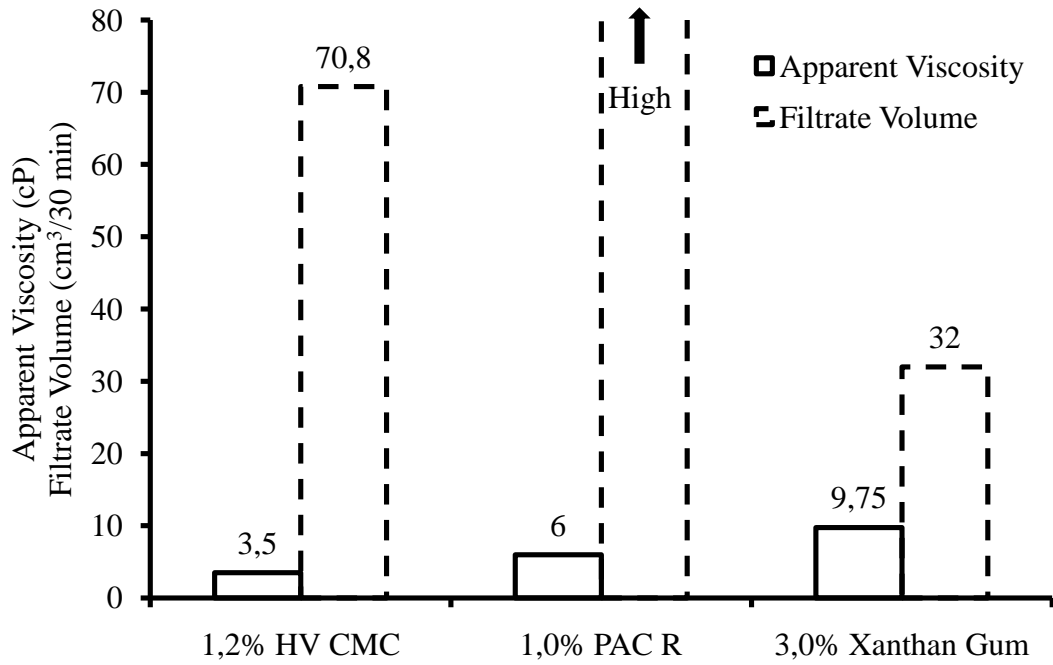


Figure 5-51 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of E-Ben Activated with  $\text{Na}_2\text{CO}_3$

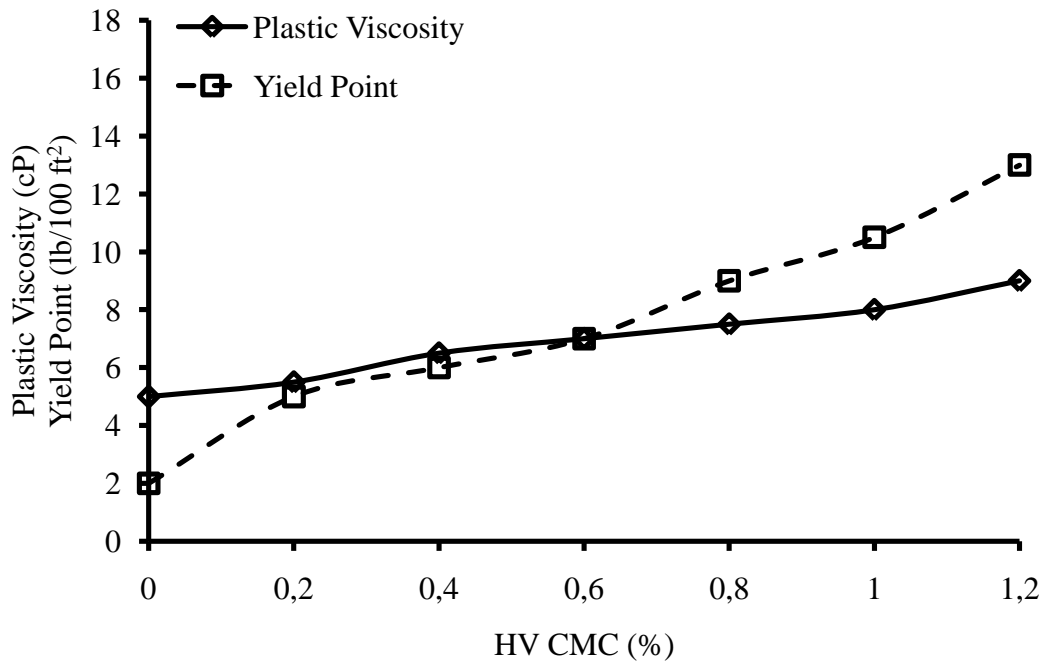


Figure 5-52 Effect of HV CMC on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{CO}_3$

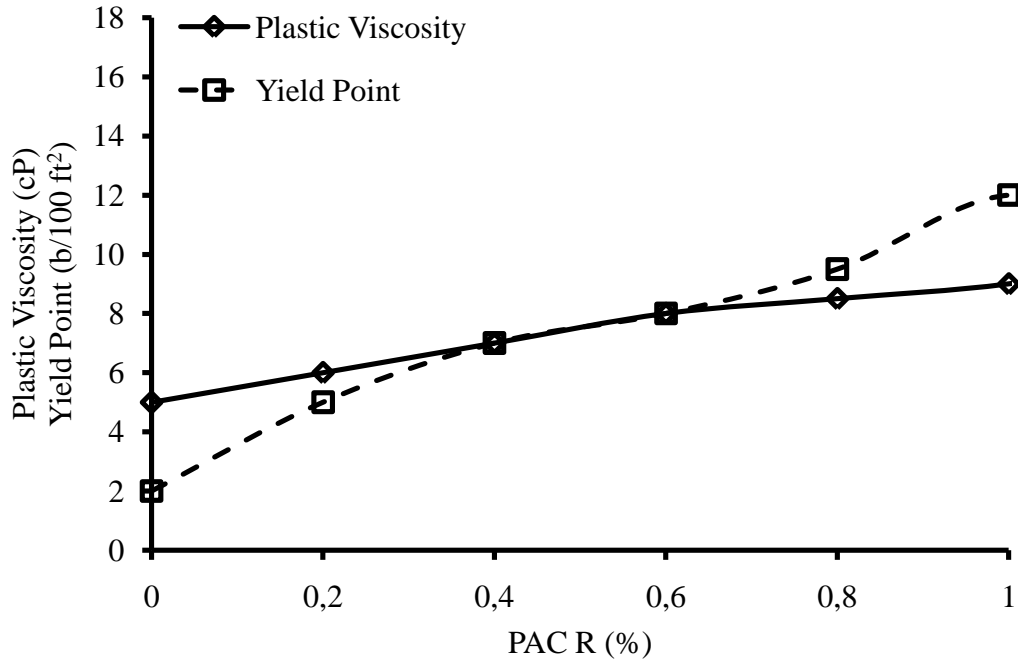


Figure 5-53 Effect of PAC R on the Plastic Viscosity and Yield Point of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

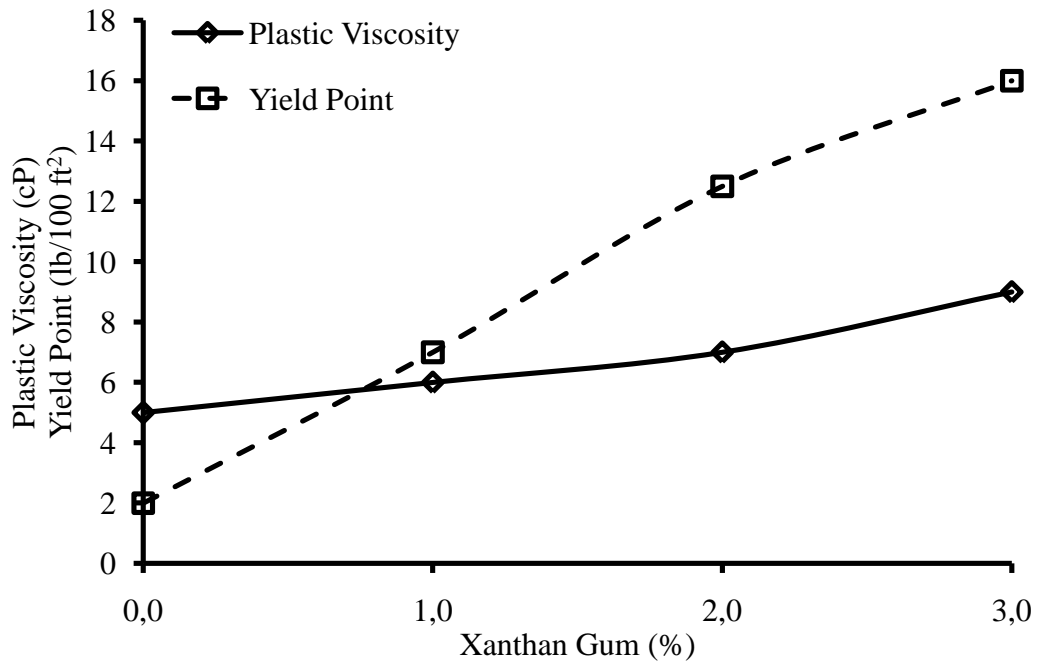


Figure 5-54 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

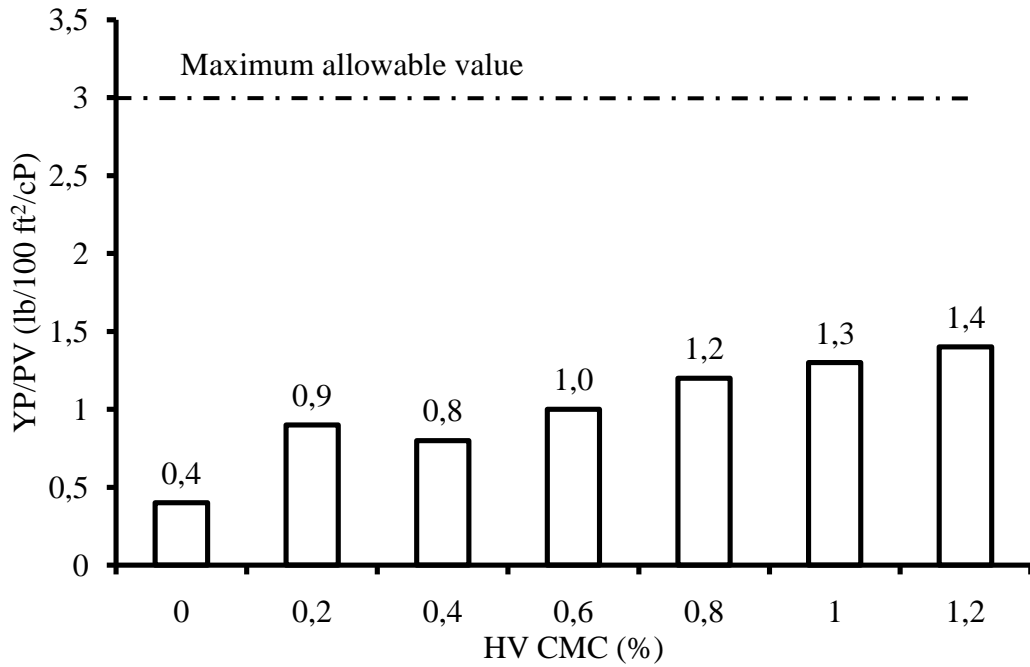


Figure 5-55 Effect of HV CMC on the YP/PV of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

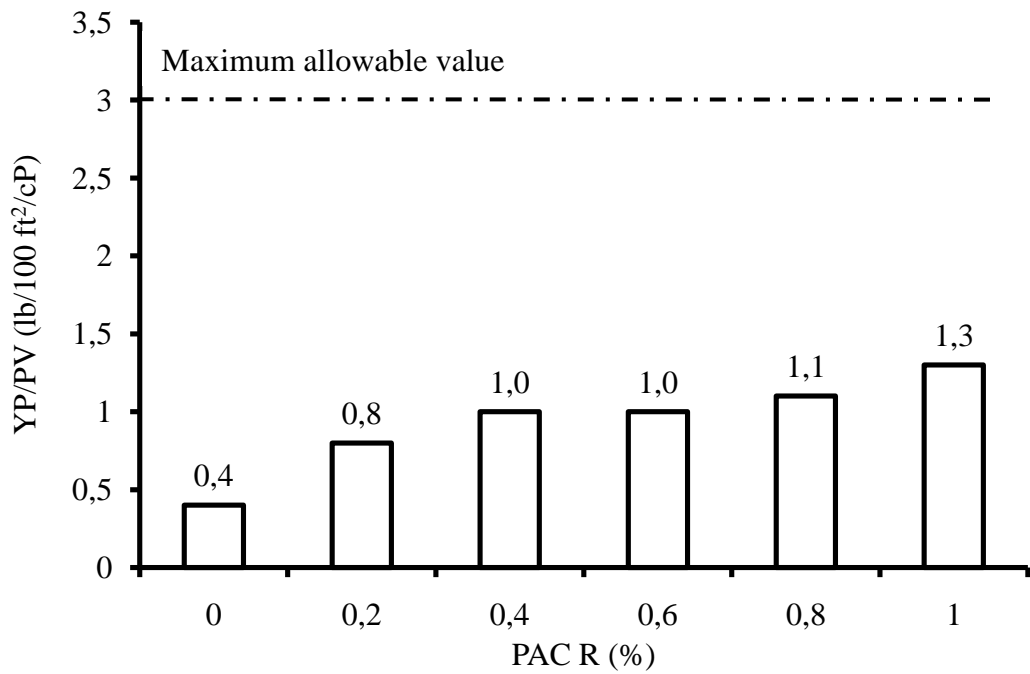


Figure 5-56 Effect of PAC R on the YP/PV of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

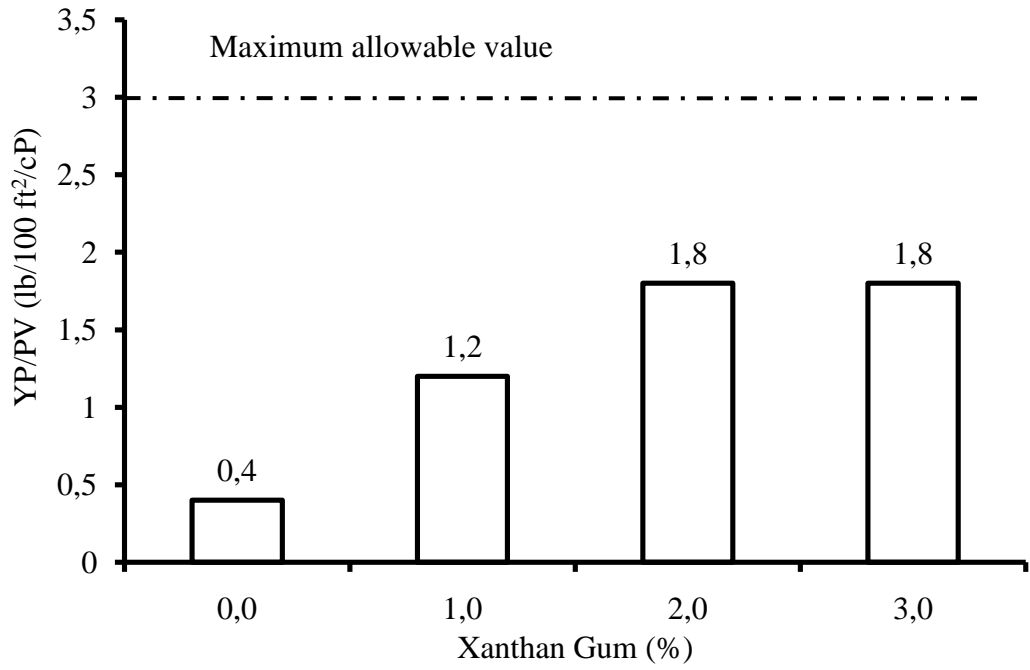


Figure 5-57 Effect of Xanthan Gum on the YP/PV of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

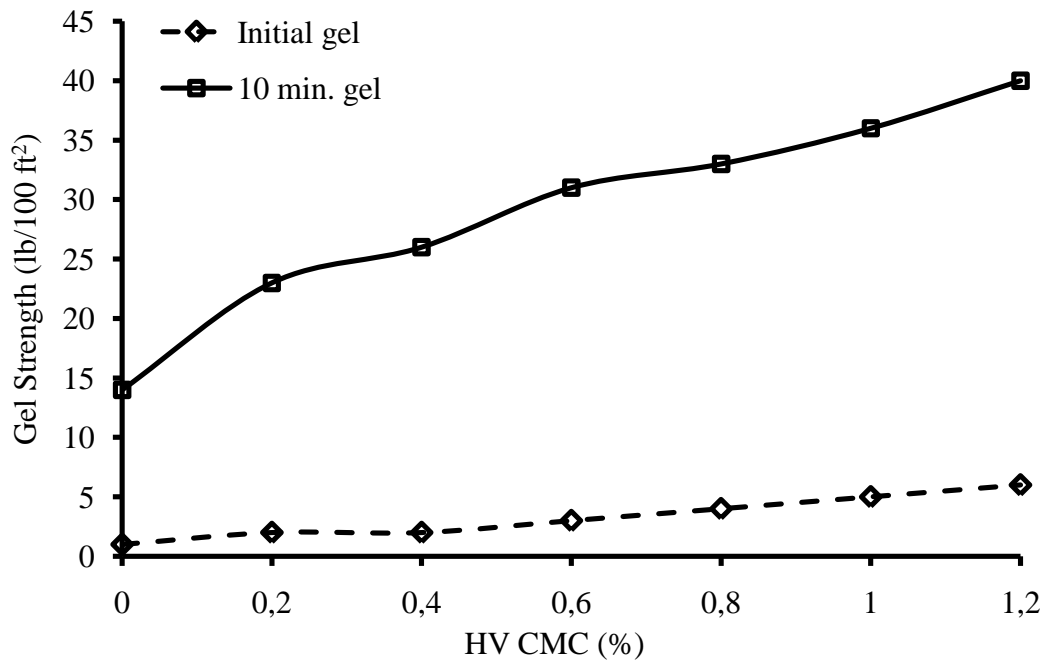


Figure 5-58 Effect of HV CMC on the Gel Strength of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

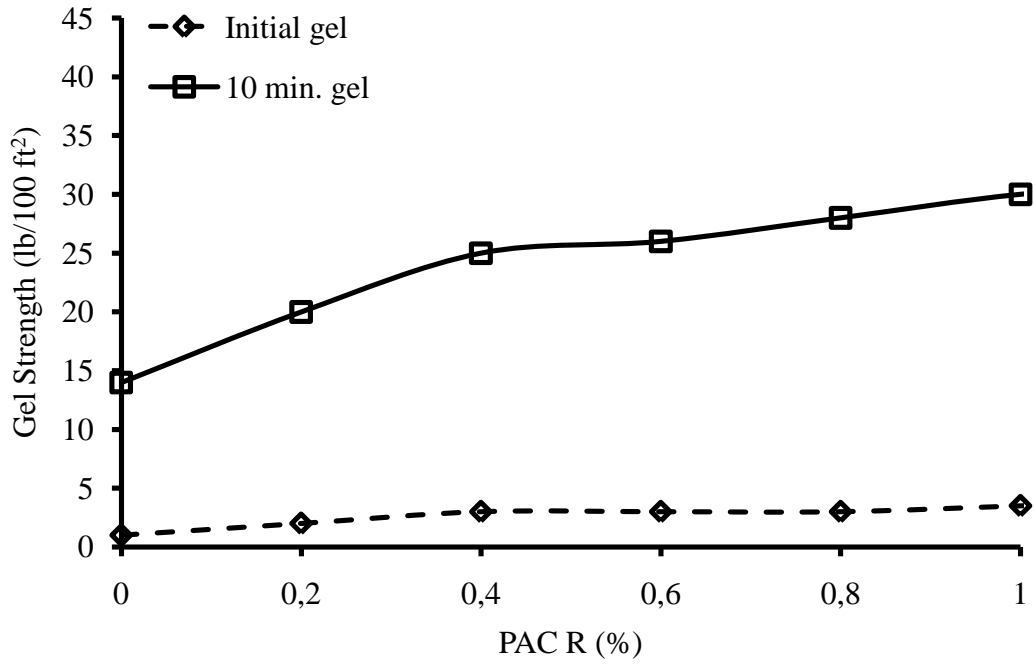


Figure 5-59 Effect of PAC R on the Gel Strength of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

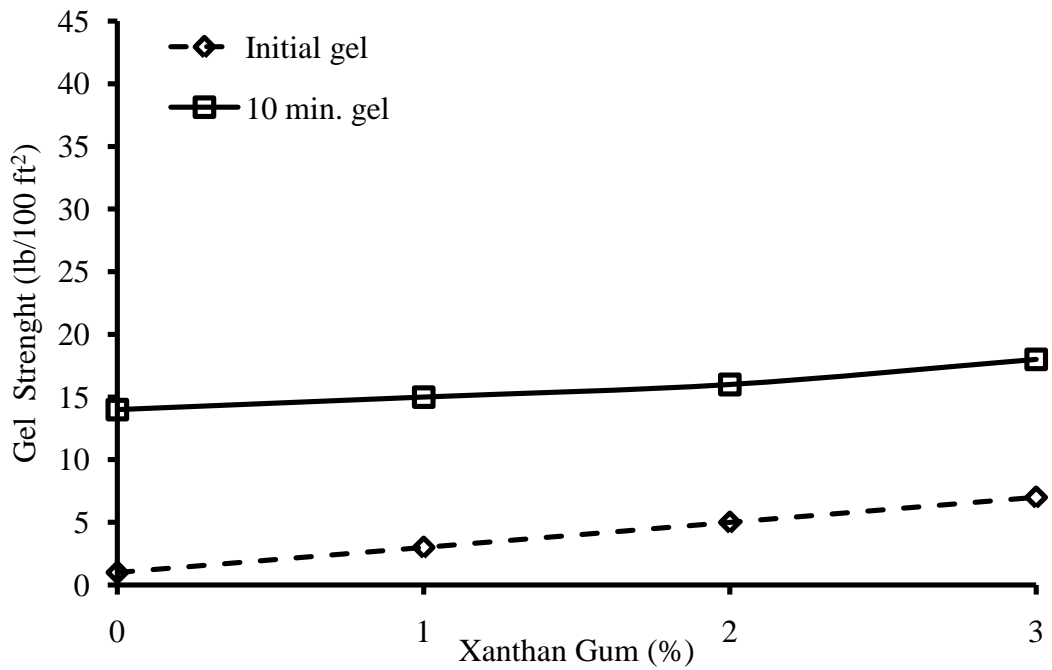


Figure 5-60 Effect of Xanthan Gum on the Gel Strength of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

### **5.4.3 The Effect of Polymers on the Rheology and Filtrate Properties of E-Ben Activated with $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$**

The effects of polymers on the rheological and filtrate properties of E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  ( $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 3,78%  $\text{Na}_2\text{O}$  equivalent) are given in Figures 5-61 through 5-73 (see also appendix Tables B-9, B-10, and B-11).

Figures 5-61, 5-62 and 5-63 show the polymer effects on the filtration rates and viscosity of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  activated mud. It was noted that the increase in the viscosity caused the expected decrease in filtration rate. The rheological and filtrate properties of polymers used in the treatment of E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  are given in Figure 5-64 (see also appendix Table B-12).

Figures 5-65 through 5-67 show that the effect of polymer on the plastic viscosity and yield point resulted in a steadily increase with increasing polymer concentration. Besides, the YP/PV ratios were within the desirable limit (Figures 5-68 through 5-70).

Figures 5-71 through 5-73, a plot of gel strengths, showed that the polymer treated mud caused higher gel strength with the increasing polymer concentration.



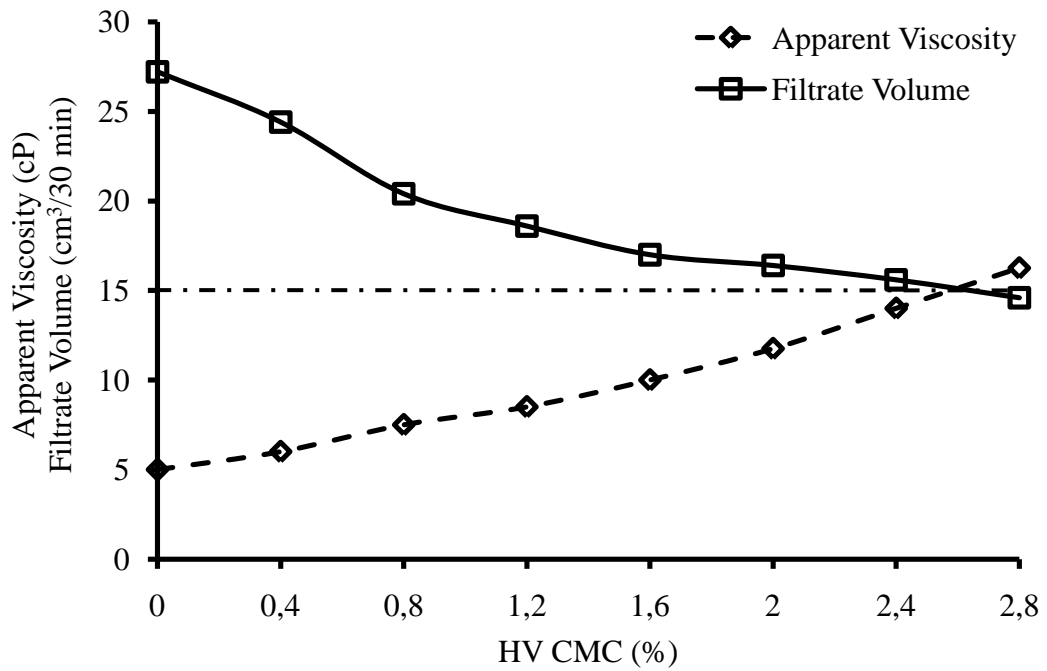


Figure 5-61 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

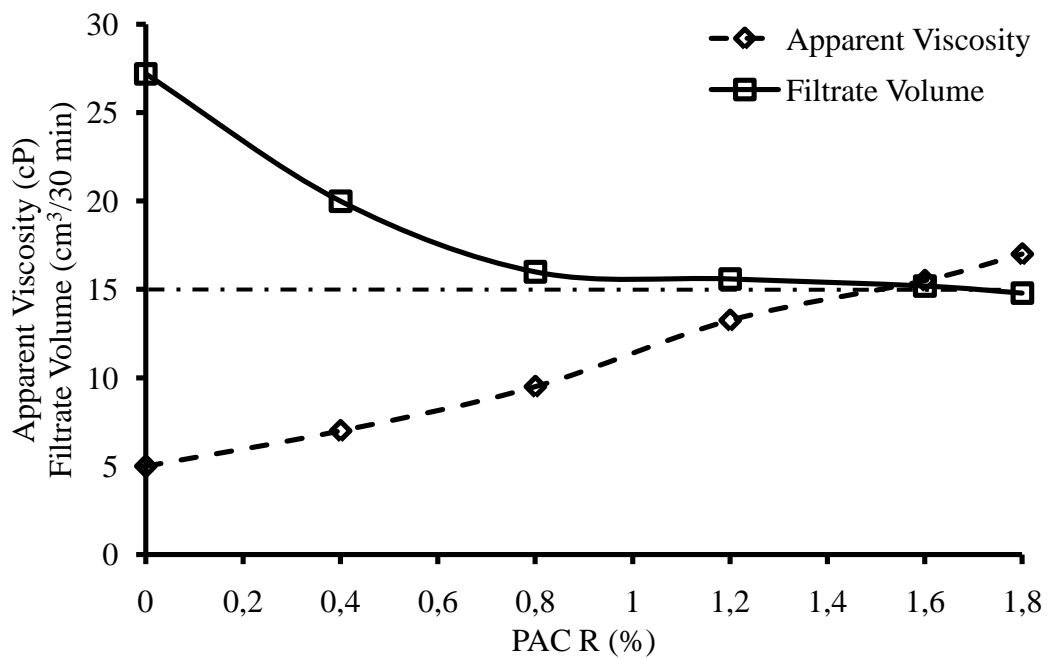


Figure 5-62 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

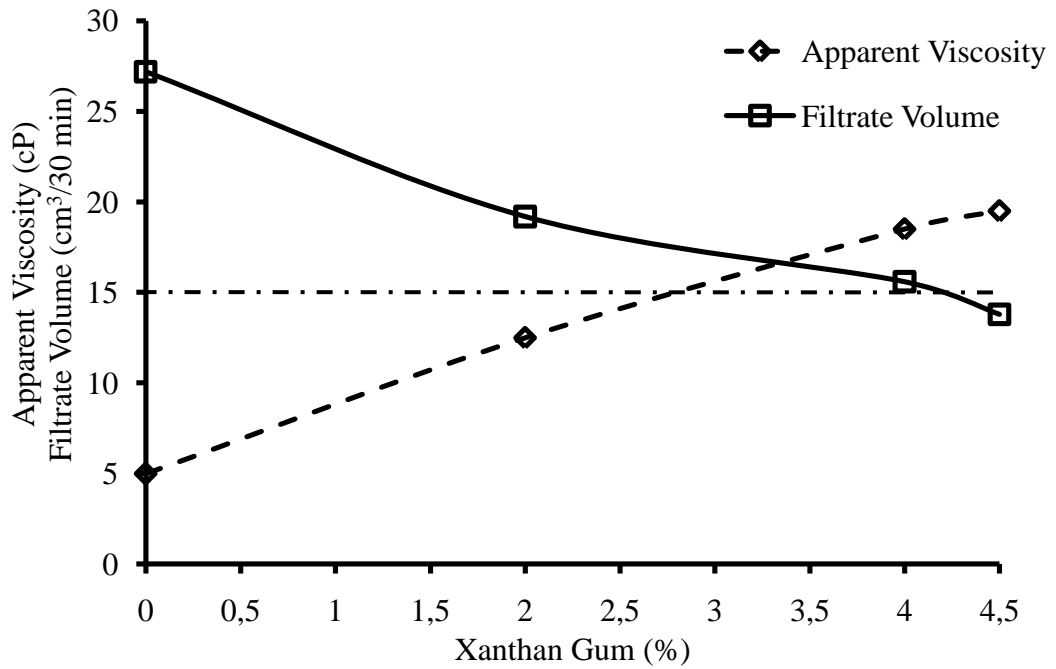


Figure 5-63 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

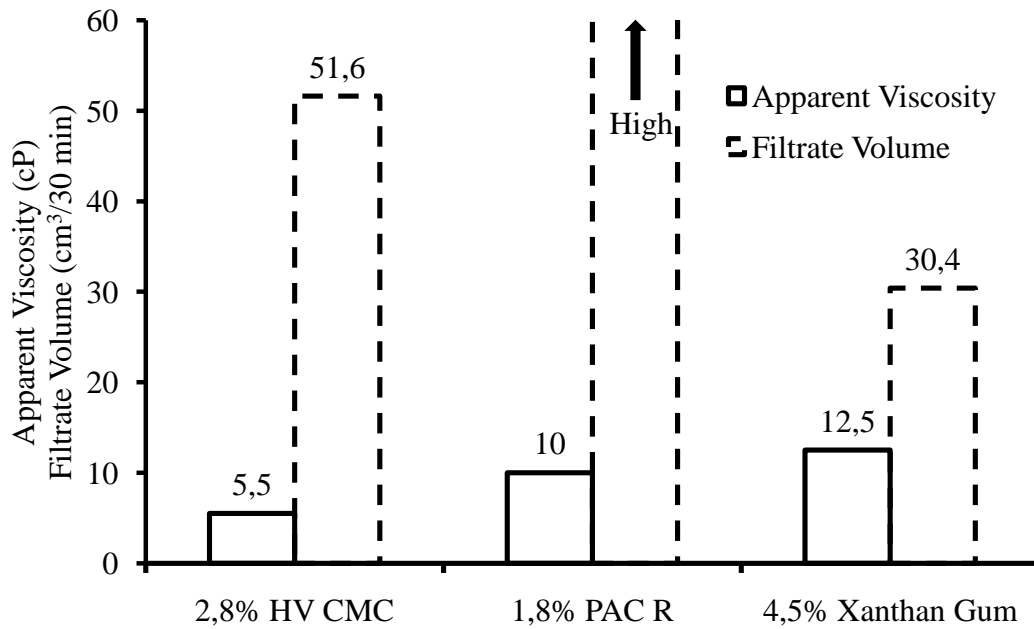


Figure 5-64 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

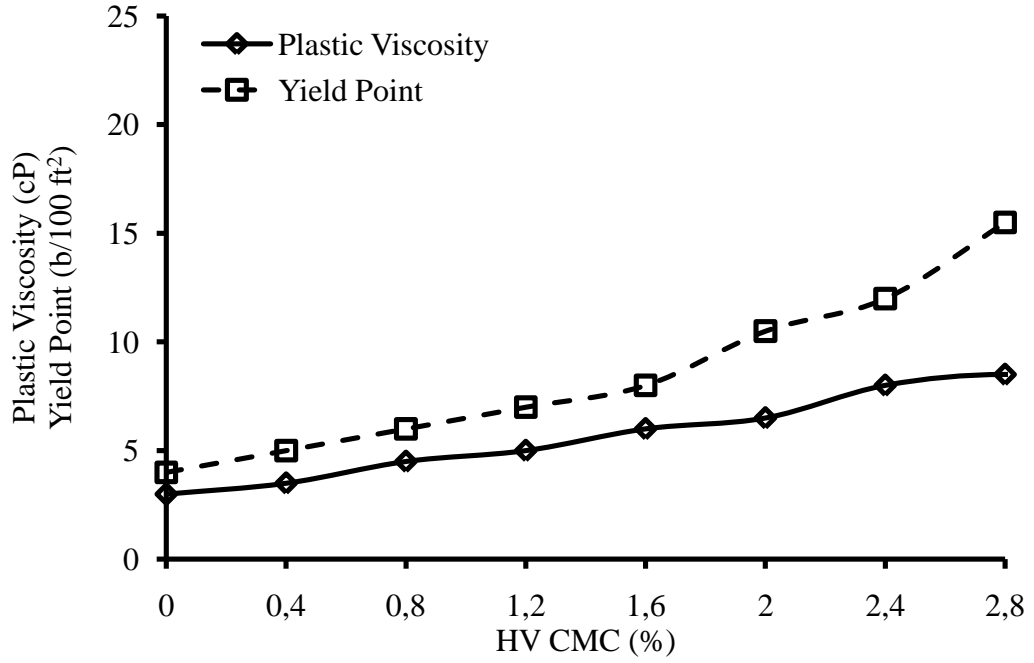


Figure 5-65 Effect of HV CMC on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

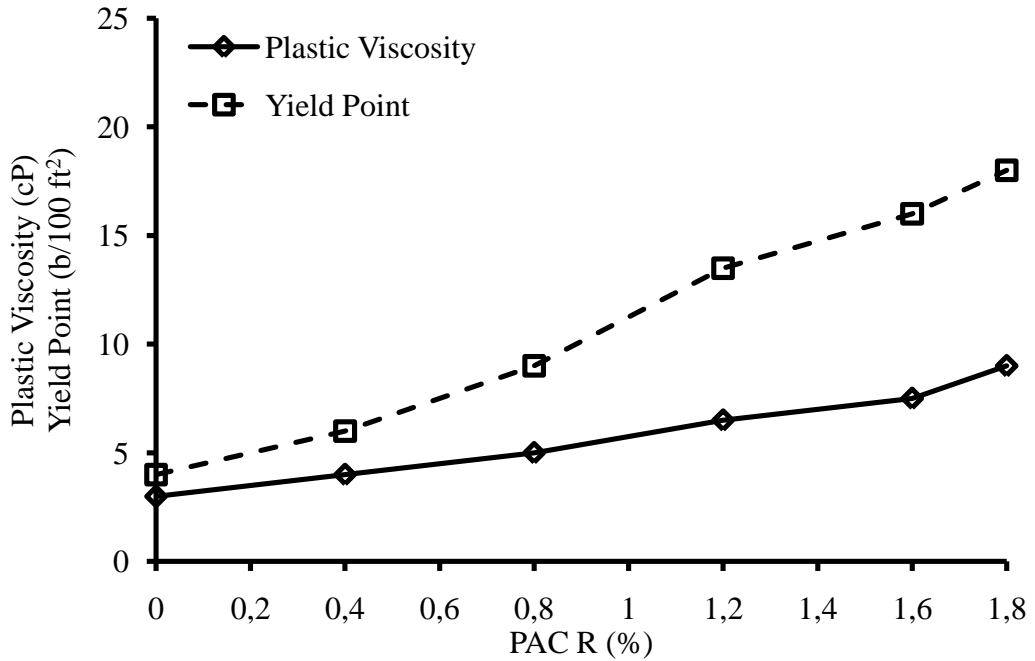


Figure 5-66 Effect of PAC R on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

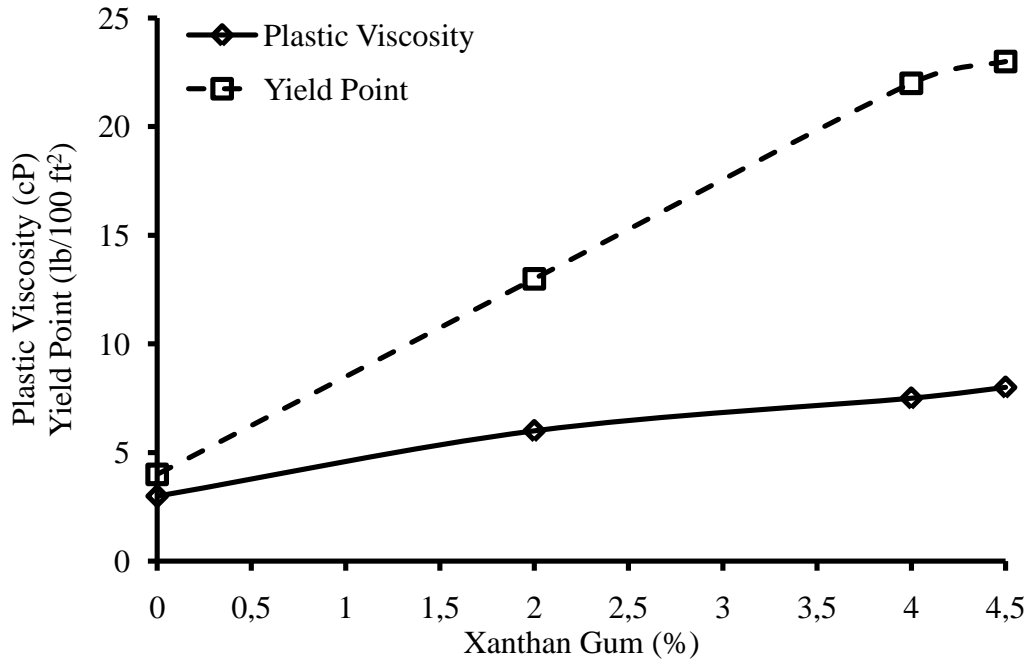


Figure 5-67 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

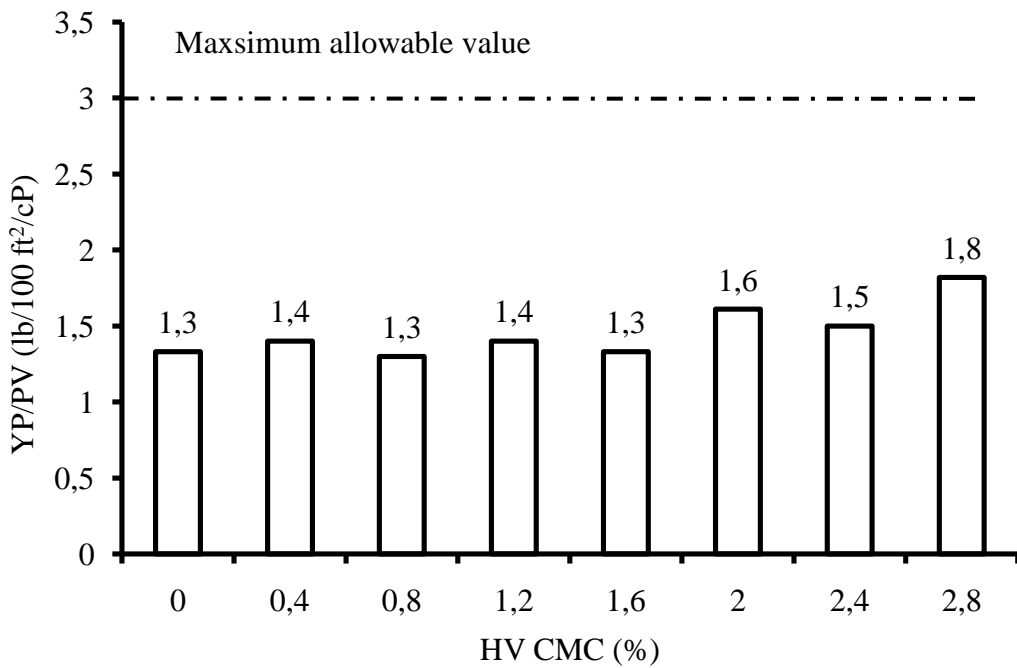


Figure 5-68 Effect of HV CMC on the YP/PV of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

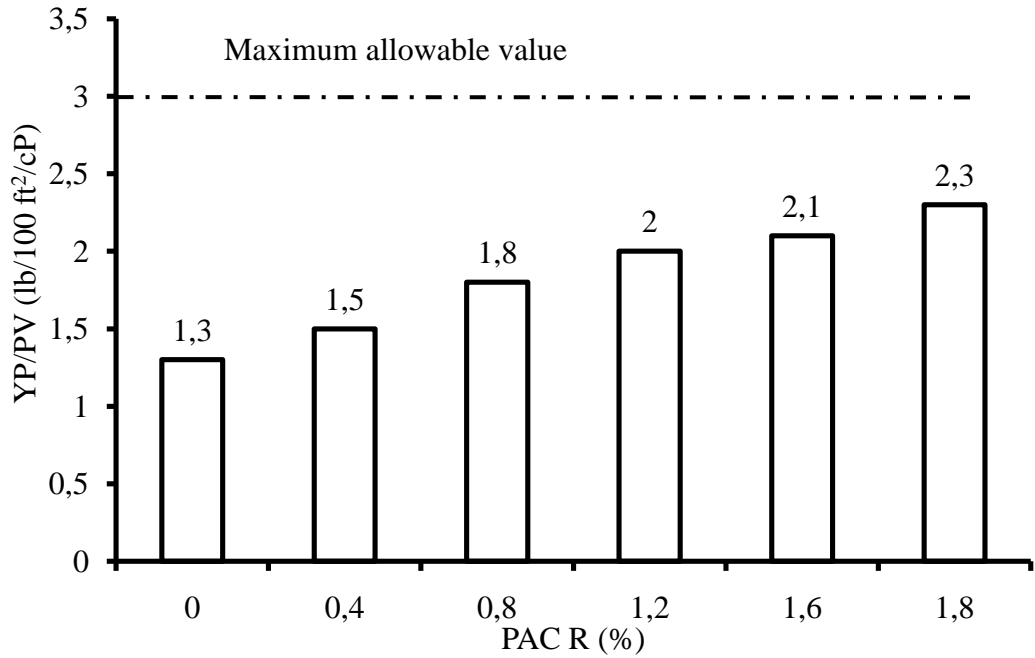


Figure 5-69 Effect of PAC R on the YP/PV of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

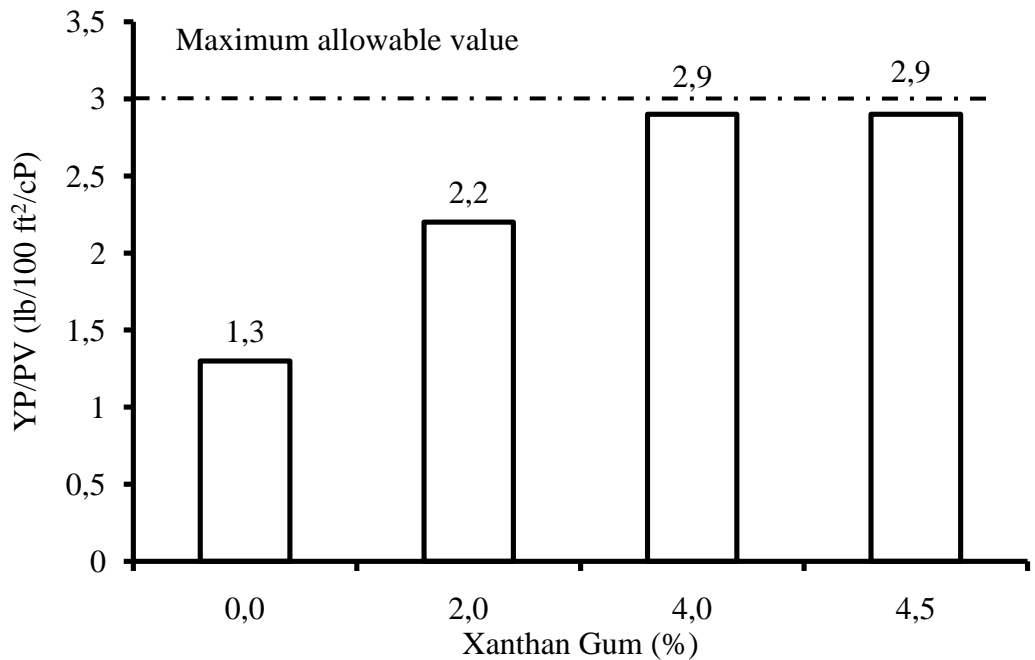


Figure 5-70 Effect of Xanthan Gum on the YP/PV of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

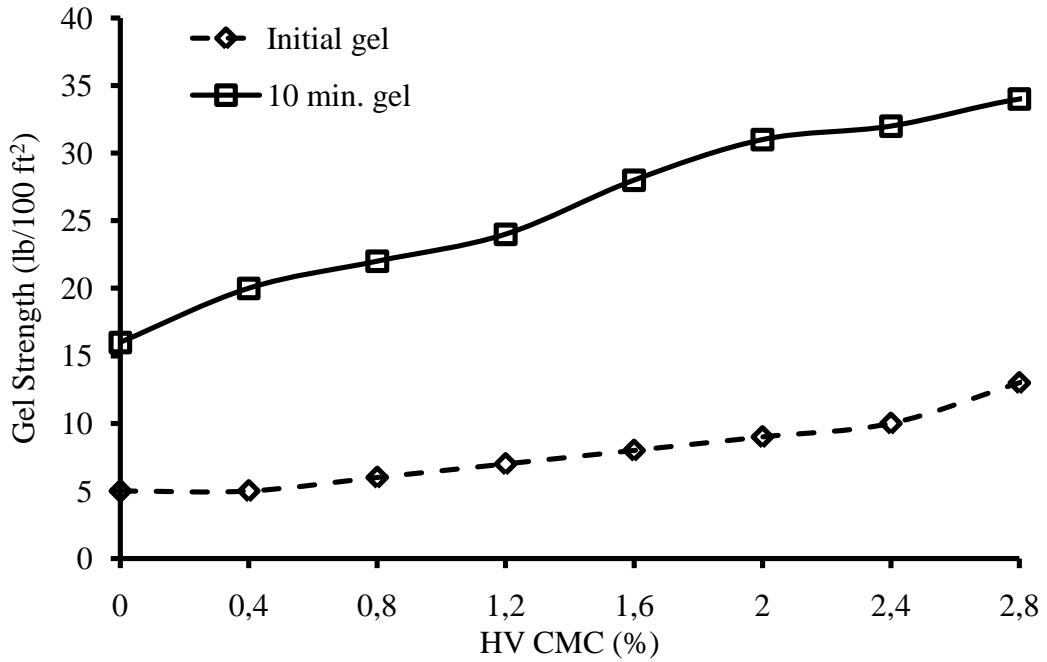


Figure 5-71 Effect of HV CMC on the Gel Strength of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

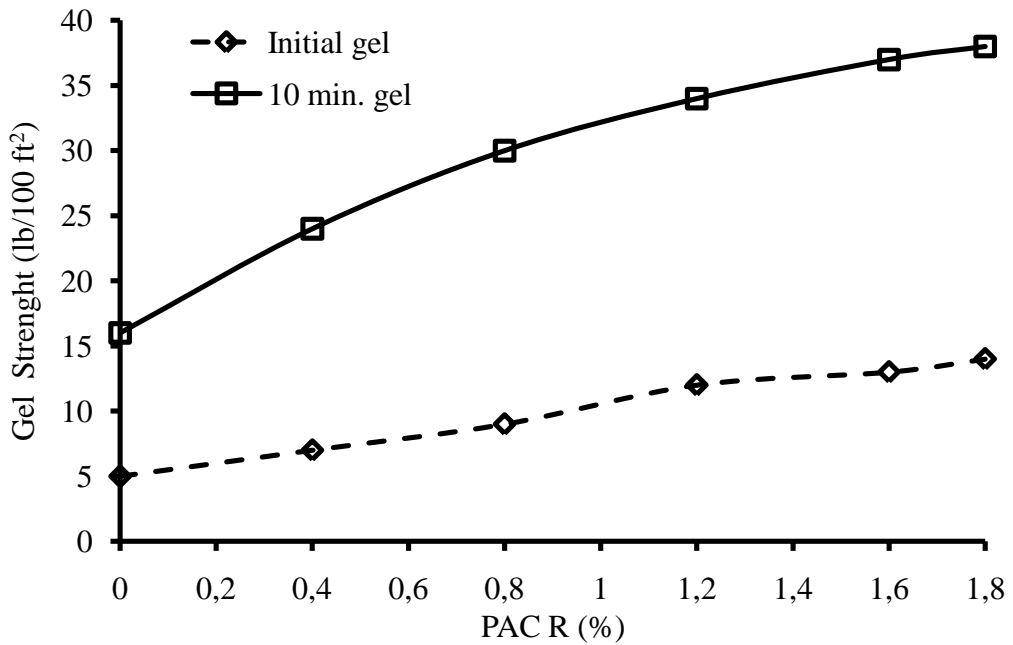


Figure 5-72 Effect of PAC R on the Gel Strength of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

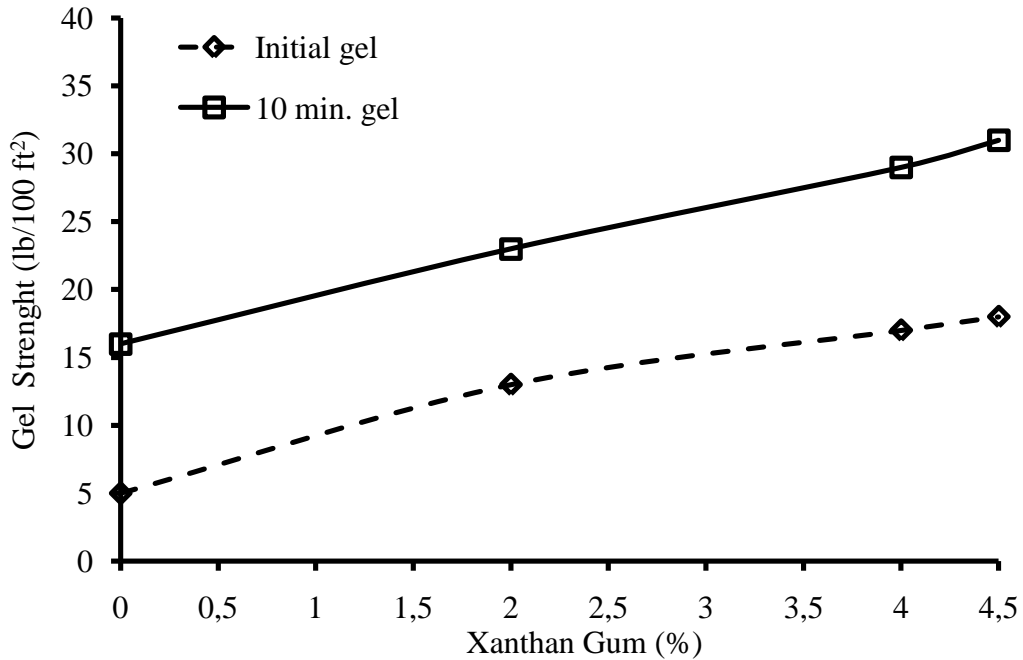


Figure 5-73 Effect of Xanthan Gum on the Gel Strength of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

#### 5.4.4 The Effect of Polymers on the Rheology and Filtrate Properties of E-Ben Activated with $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ Mixture

E-Ben which is activated with a  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture ( $\text{Na}_2\text{CO}_3$  addition level; 2,18%  $\text{Na}_2\text{O}$  equivalent and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,95%  $\text{Na}_2\text{O}$  equivalent) was developed by treatment with polymers to control the rheology and filtrate loss for water-based drilling fluids. The results of investigation are given in Figures 5-74 through 5-86 (see also appendix Tables B-13, B-14, and B-15).

The apparent viscosity of activated bentonite suspension changed with CMC and PAC and xanthan gum, reaching a required minimum value (15 cP) at a polymer concentration of 2,0% CMC, 1,5% PAC, and 3,5% xanthan gum, respectively. Filtrate volumes were also reduced to the required value ( $\leq 15$  ml) (Figures 5-74 through 5-76). The rheological and filtrate properties of polymers used in the treatment of E-Ben activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture are given in Figure 5-77 (see also appendix Table B-16).

The effects of polymers on the yield point and plastic viscosity of bentonite suspension were found to have increased with the increasing polymer concentrations (Figures 5-78 through 5-80). The results of YP/PV values of treated bentonite given in Figures 5-81 through 5-83 were within maximum allowable limit.

From Figures 5-84, 5-85, and 5-86 incremental addition of polymers resulted in a steadily increase in the values of the gel strength.

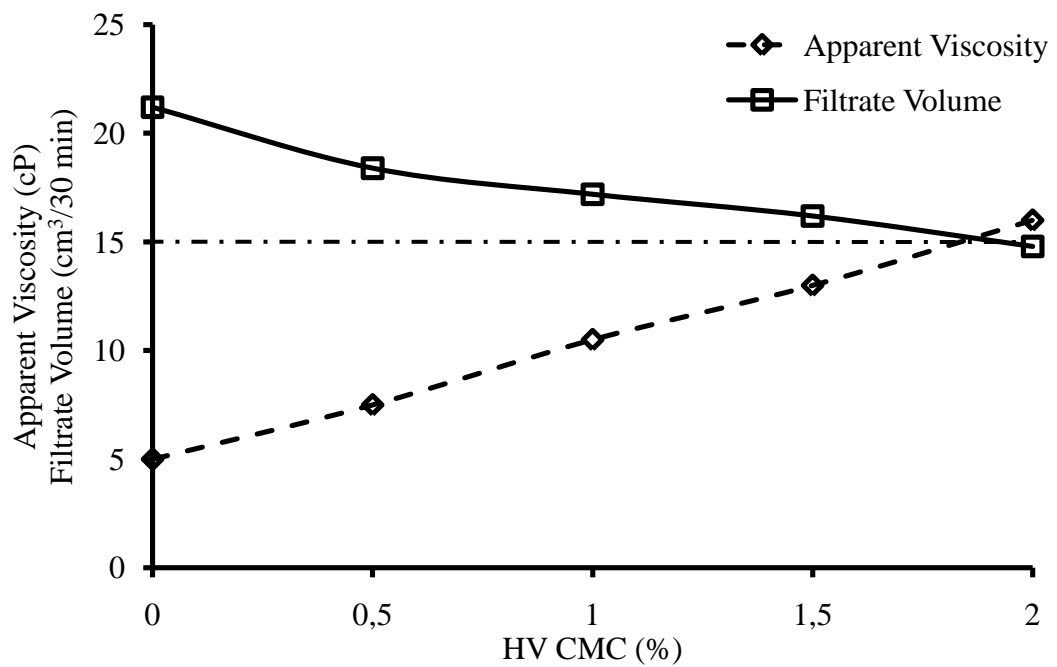


Figure 5-74 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture



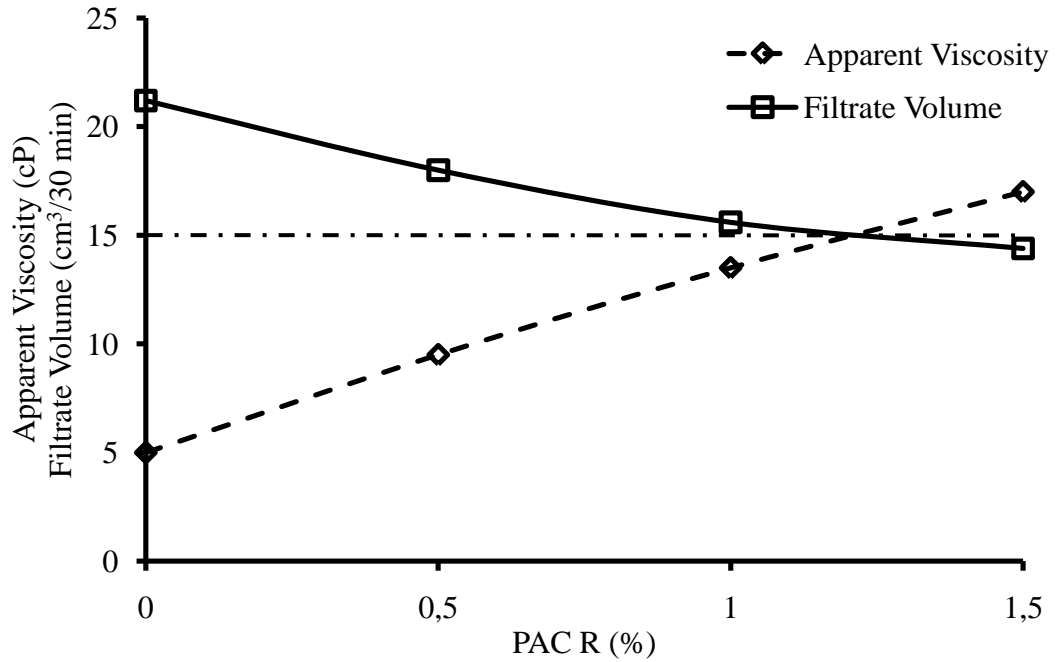


Figure 5-75 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

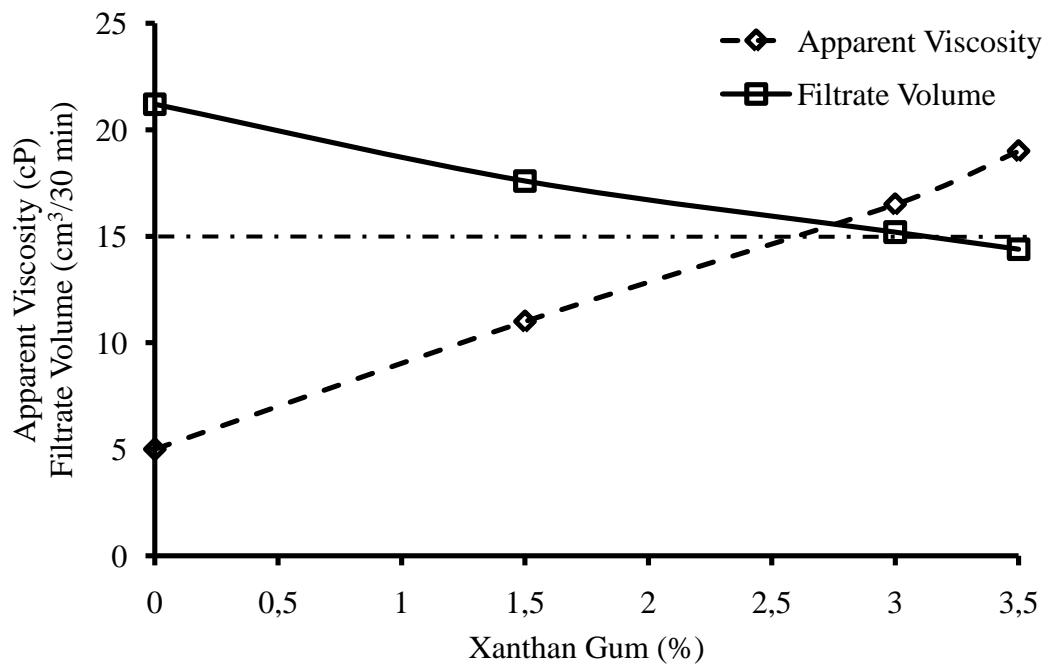


Figure 5-76 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

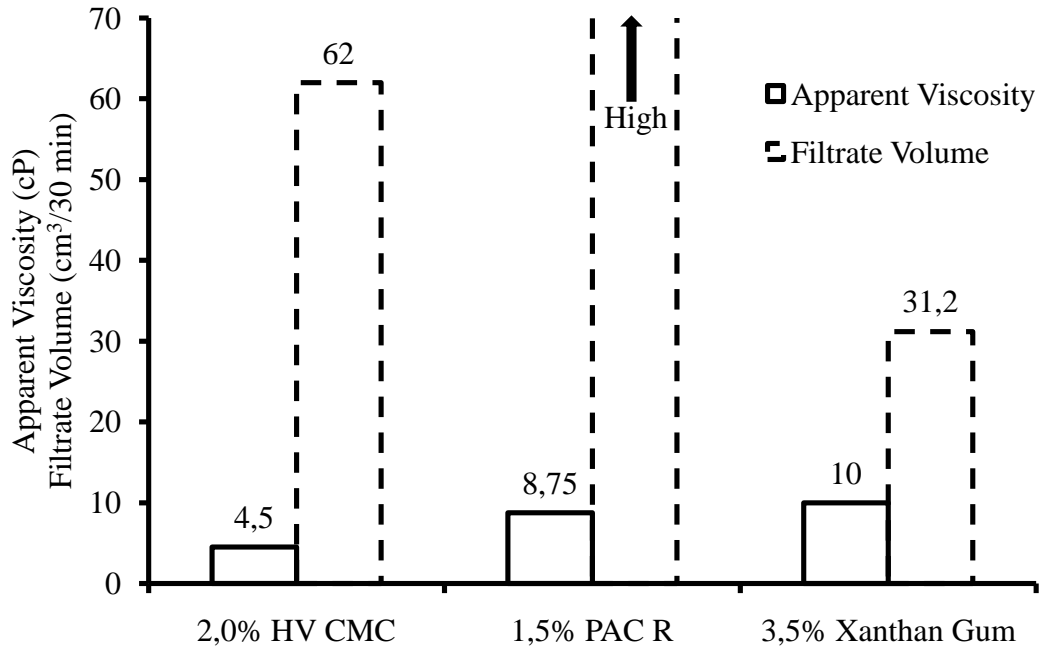


Figure 5-77 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture

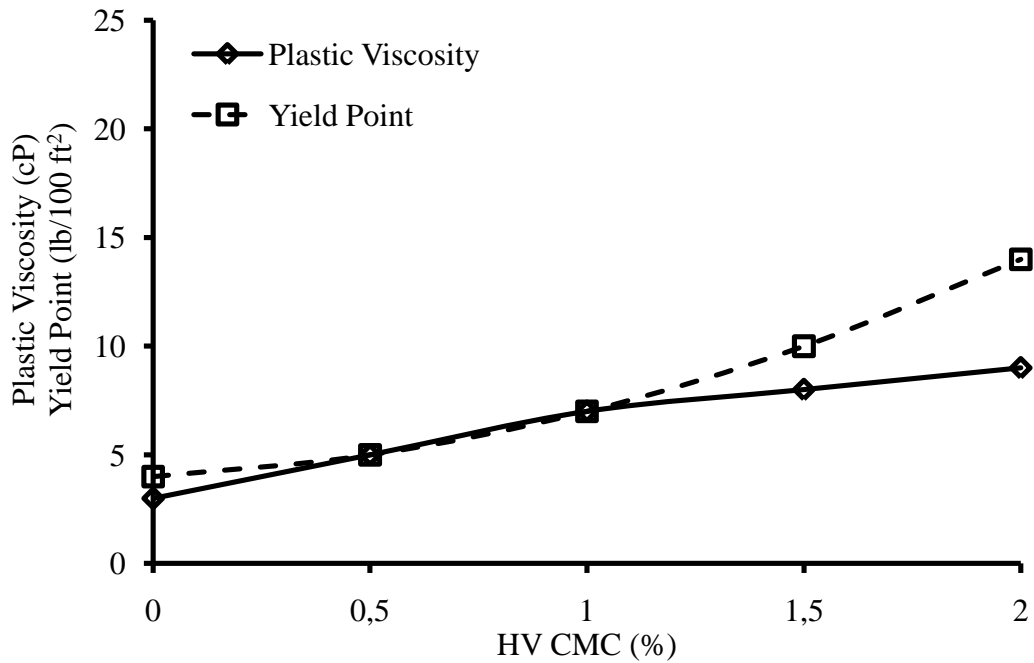


Figure 5-78 Effect of HV CMC on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

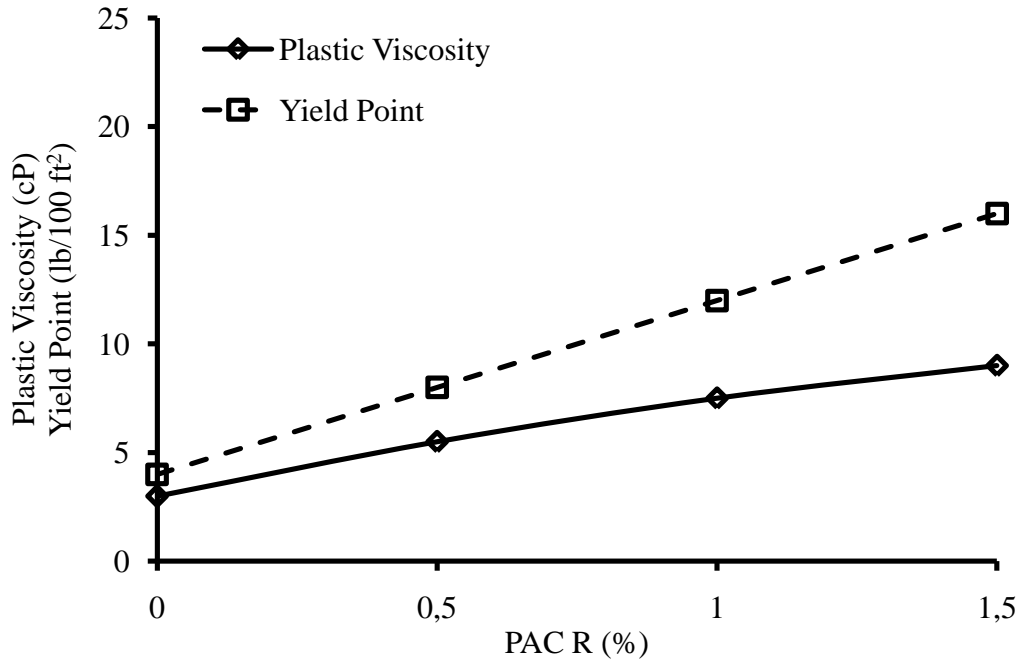


Figure 5-79 Effect of PAC R on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

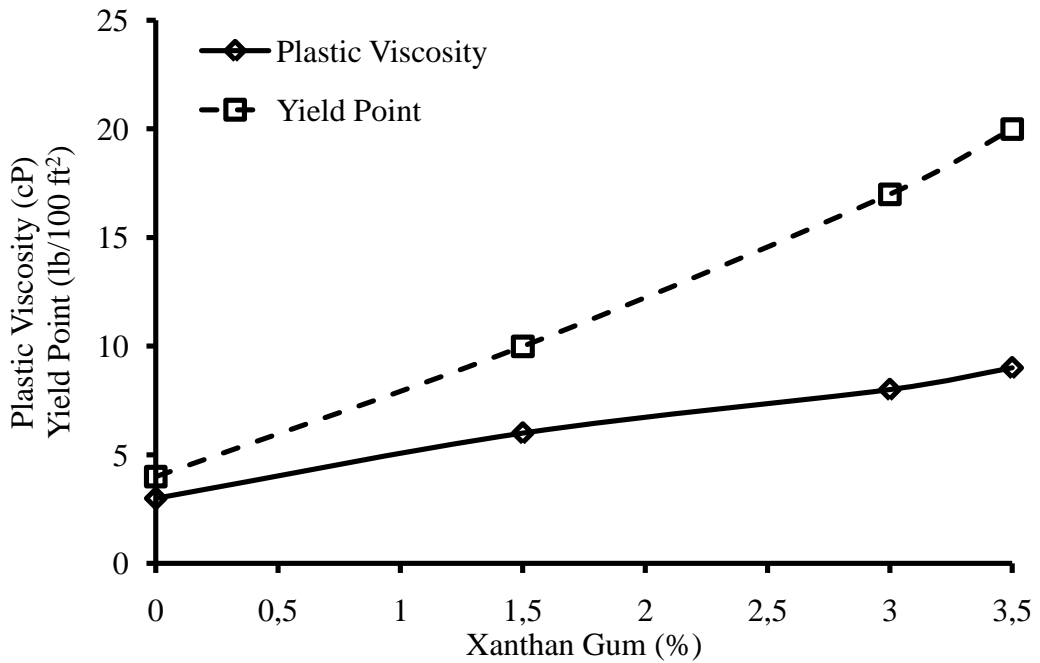


Figure 5-80 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

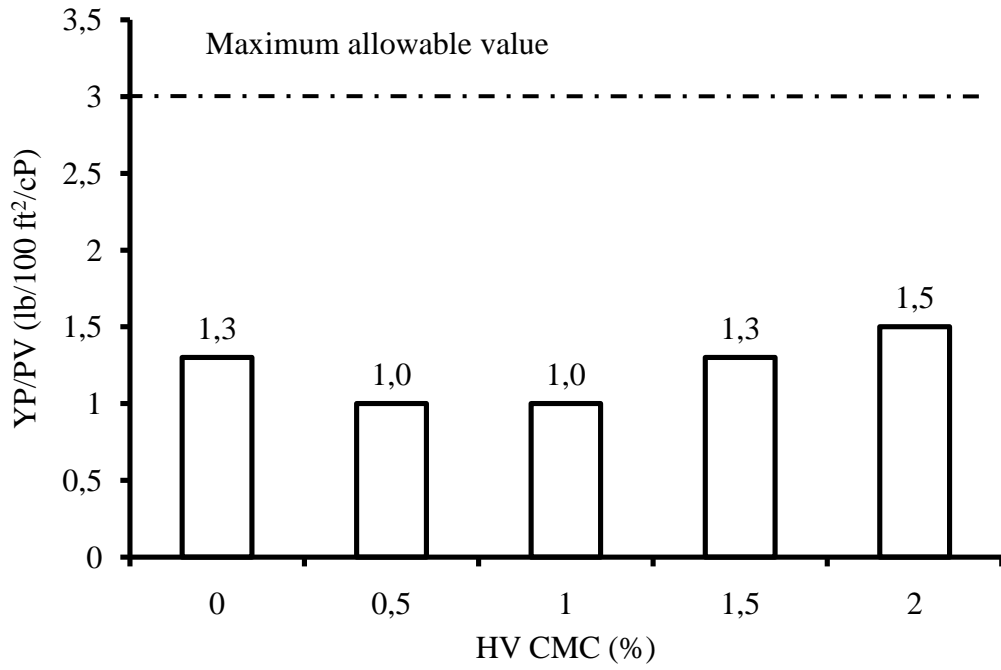


Figure 5-81 Effect of HV CMC on the YP/PV of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

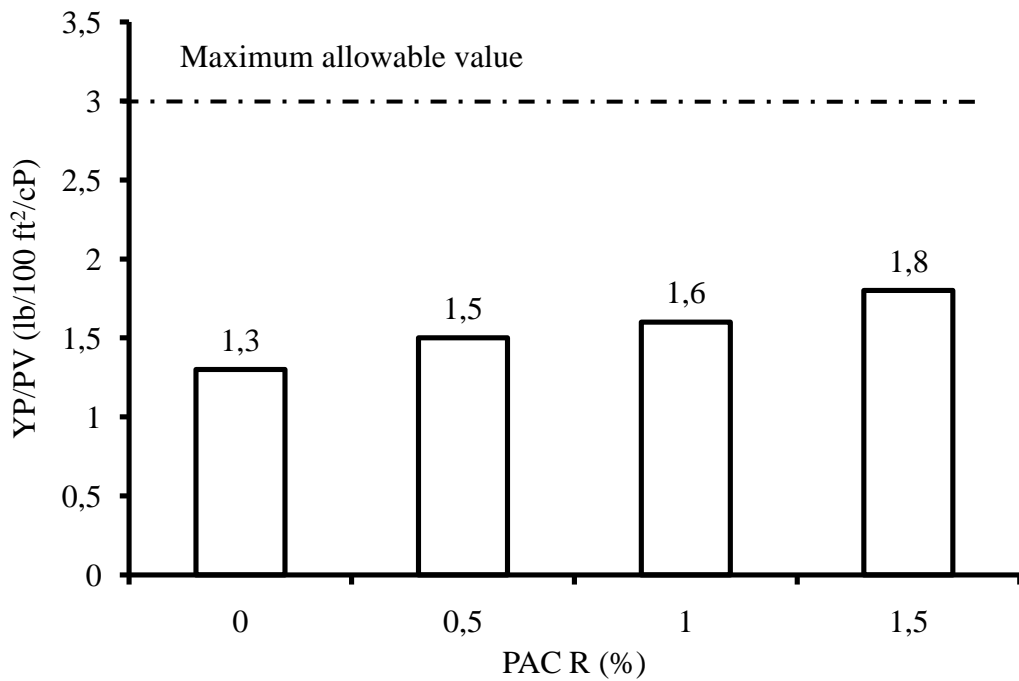


Figure 5-82 Effect of PAC R on the YP/PV of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

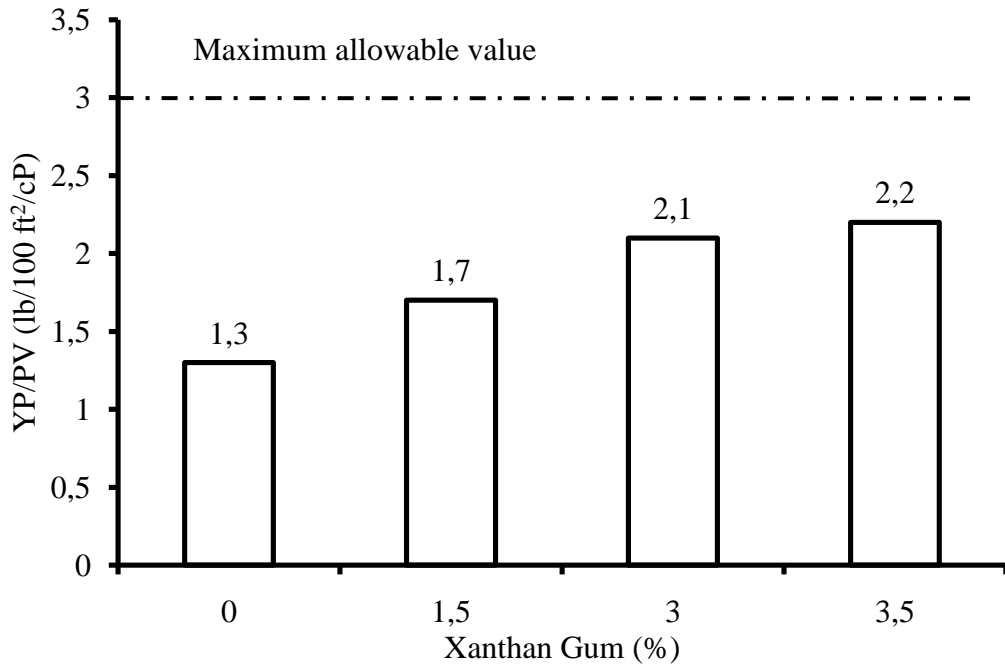


Figure 5-83 Effect of Xanthan Gum on the YP/PV of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

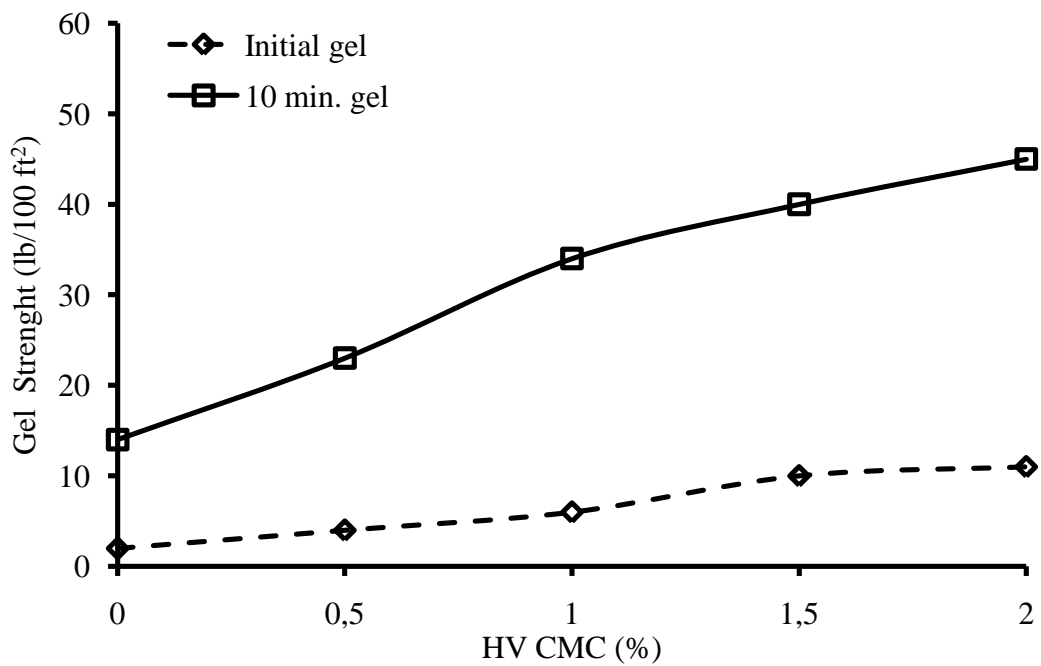


Figure 5-84 Effect of HV CMC on the Gel Strength of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

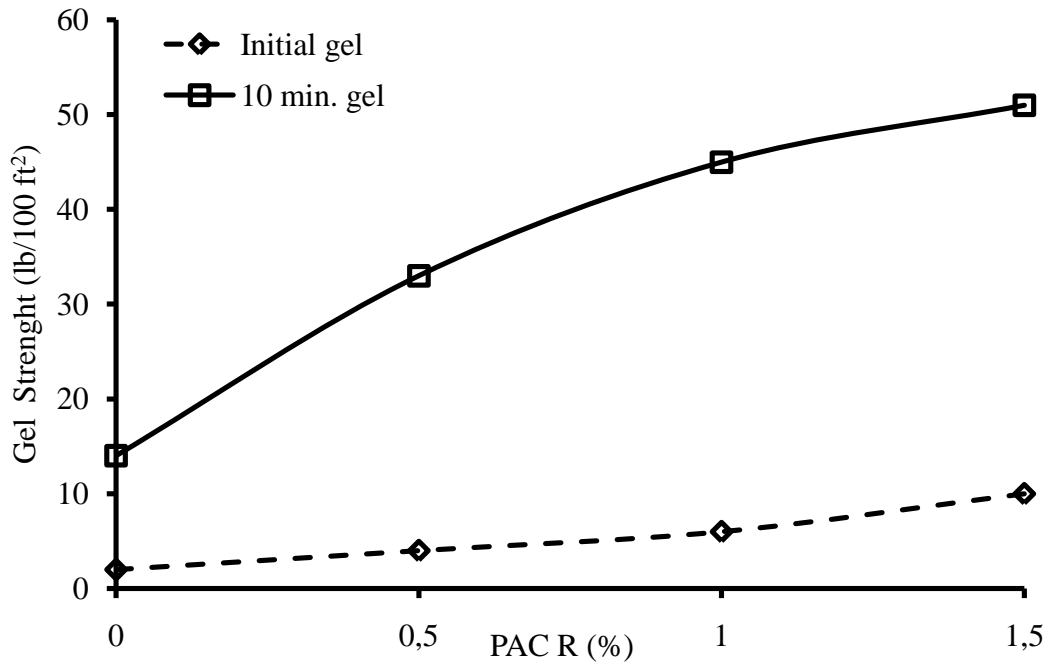


Figure 5-85 Effect of PAC R on the Gel Strength of E-Ben Activated with  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture

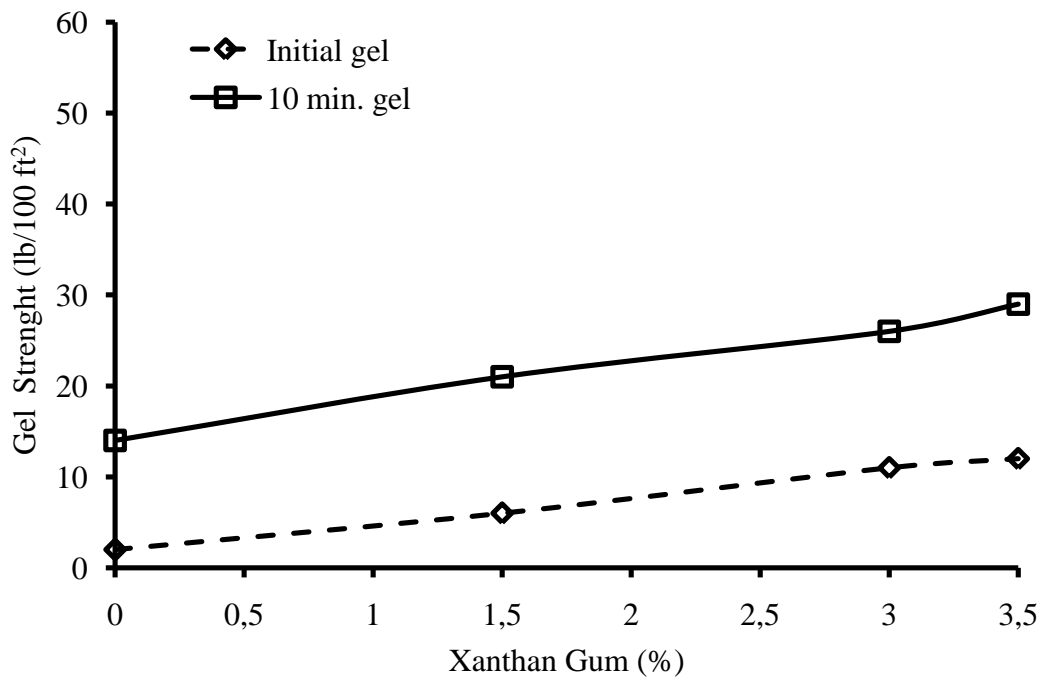


Figure 5-86 Effect of Xanthan Gum on the Gel Strength of E-Ben Activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture

#### **5.4.5 The Effect of Polymers on the Rheology and Filtrate Properties of C-Ben**

As the apparent viscosity of the raw sample was 6,75 cP which is below the minimum required value of 15 cP, the sample were treated with different concentration of polymers. The results of the study are shown in Figures 5-87 through 5-99 (see also appendix Tables B-17, B-18, and B-19).

It was found that the incremental addition of polymer resulted in an increase in the values of the apparent viscosity (Figures 5-87 through 5-89). It was shown that polymers such as CMC, PAC and xanthan gum used as a viscosifier in the mud had effect on filtration reduction by plugging the pores of the cake network. The rheological and filtrate properties of polymers used in the treatment of C-Ben are given in Figure 5-90 (see also appendix Table B-20). Moreover, the values of plastic viscosity and yield point were increased with increasing the concentration of polymer (Figures 5-91 through 5-93). The increase in rheological properties showed that flocculation occurred when the polymer molecule bonded to two or more clay particles.

The results of YP/PV values of treated bentonite given in Figures 5-94 through 5-96 were within the desirable limit expect xanthan gum. Xanthan gum met the OCMA type bentonite requirement (it can be seen page 2) for YP/PV ratio ( $<6 \text{ lb}/100 \text{ ft}^2/\text{cP}$ ). 3% HV CMC, 2% PAC R, and 2,5% xanthan gum treated C-Ben met the API specification required for application in drilling mud.

From Figures 5-97 through 5-99 the addition of polymers resulted in a steadily increase in the values of gel strength.

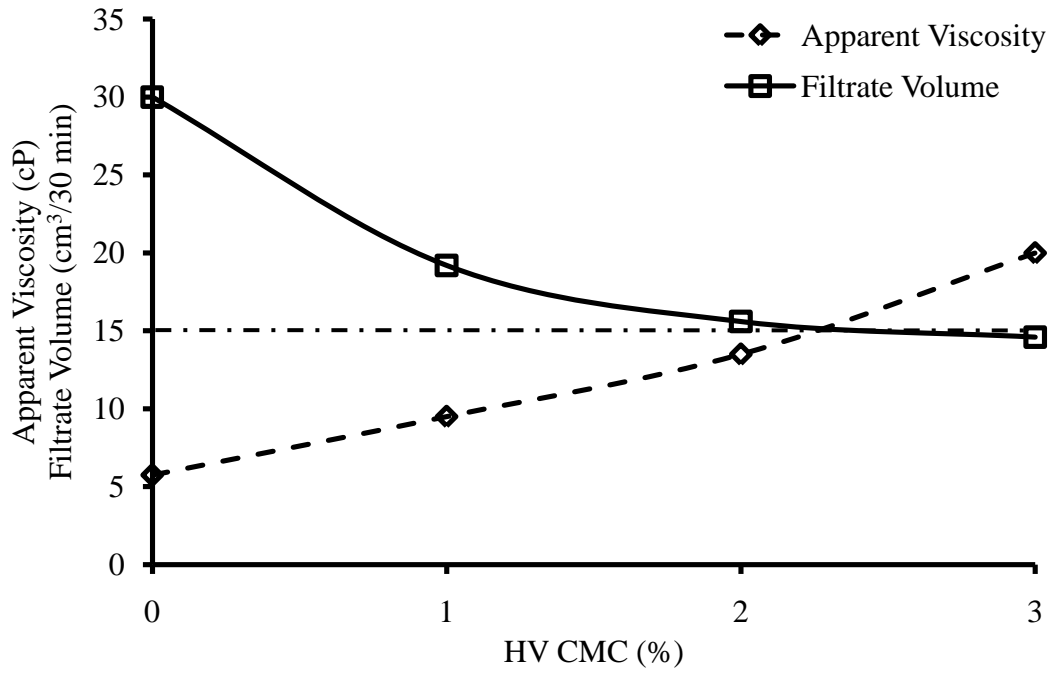


Figure 5-87 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of C-Ben

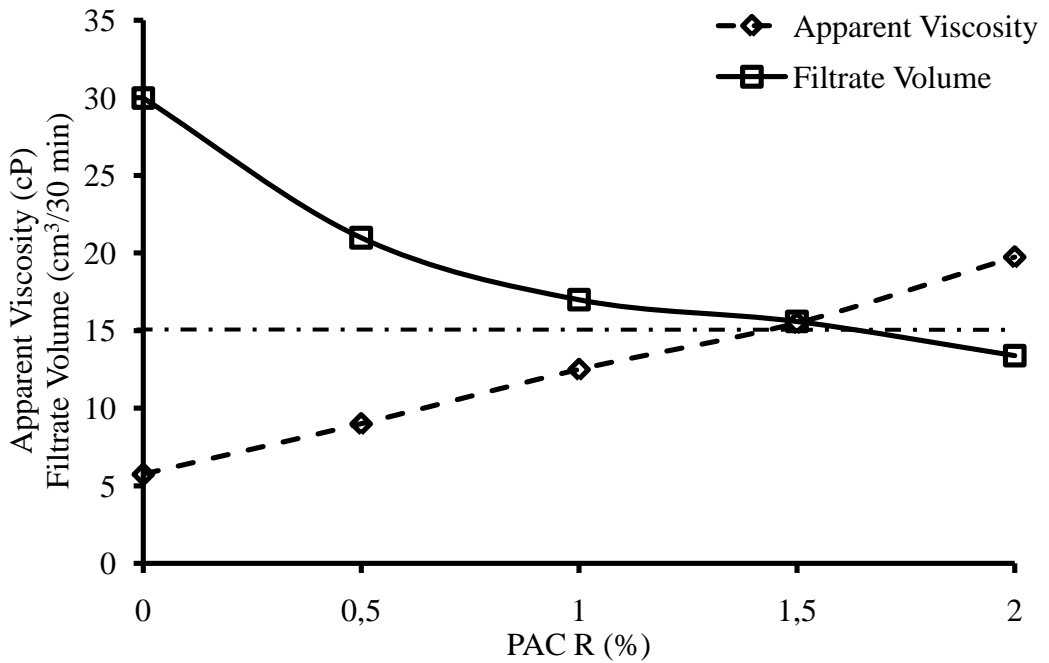


Figure 5-88 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of C-Ben



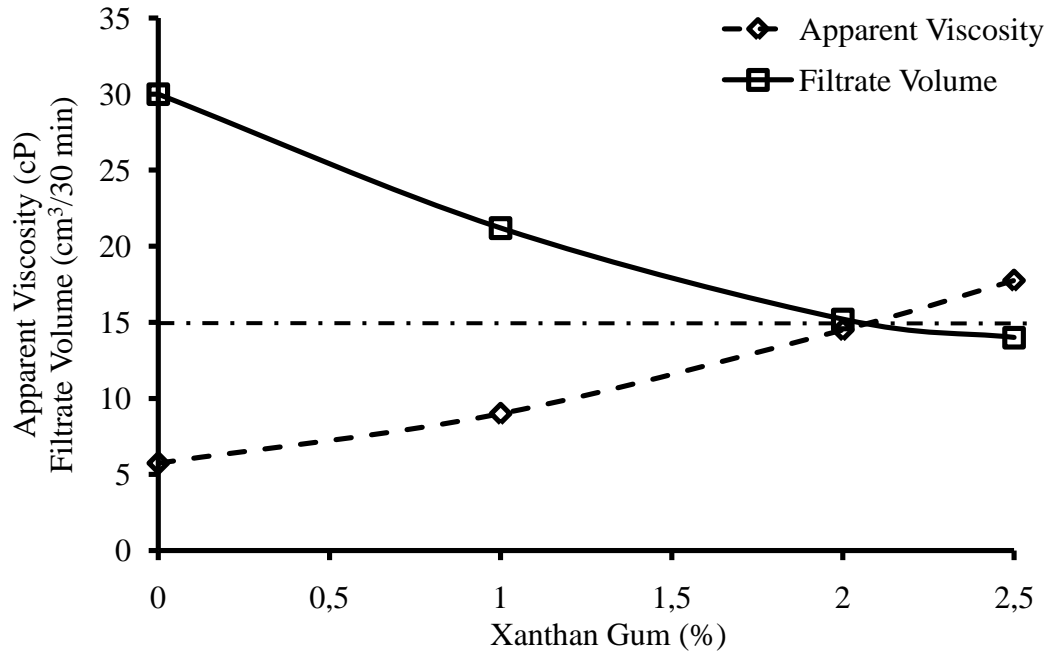


Figure 5-89 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of C-Ben

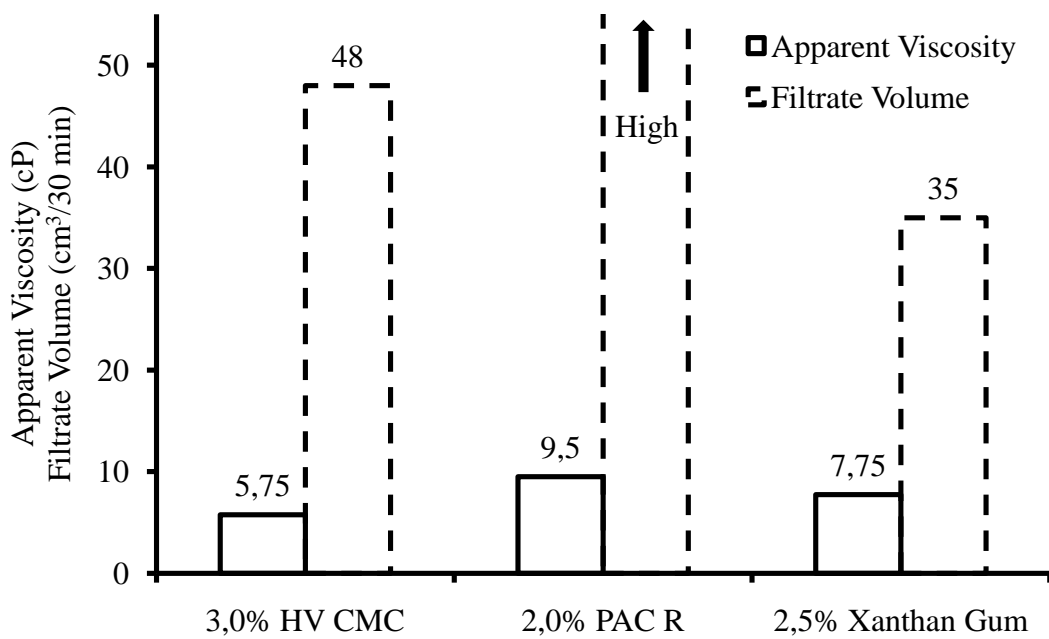


Figure 5-90 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of C-Ben

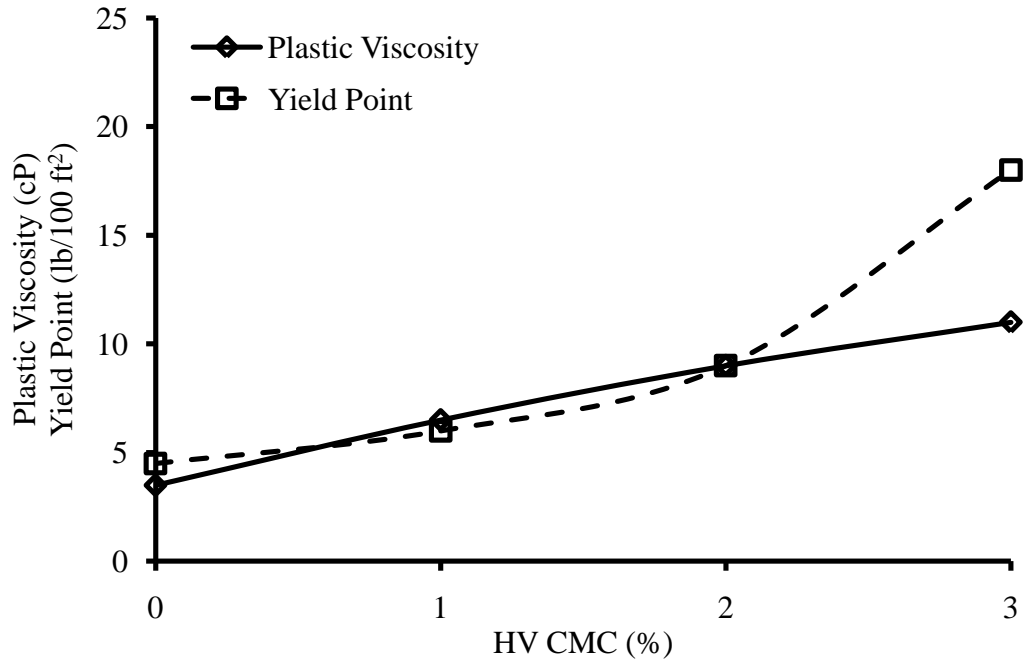


Figure 5-91 Effect of HV CMC on the Plastic Viscosity and Yield Point of C-Ben

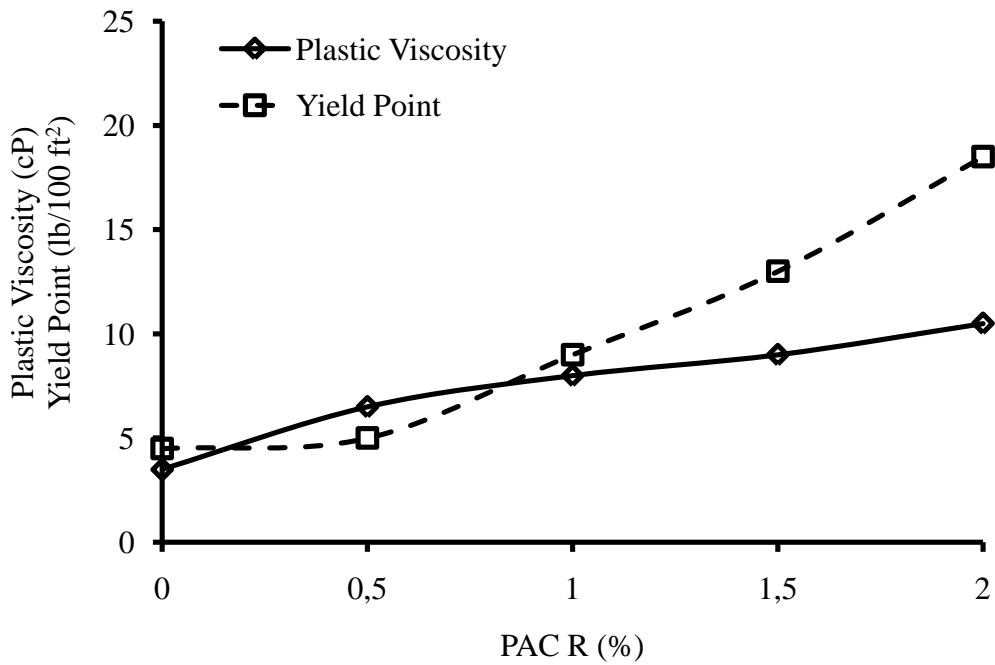


Figure 5-92 Effect of PAC R on the Plastic Viscosity and Yield Point of C-Ben

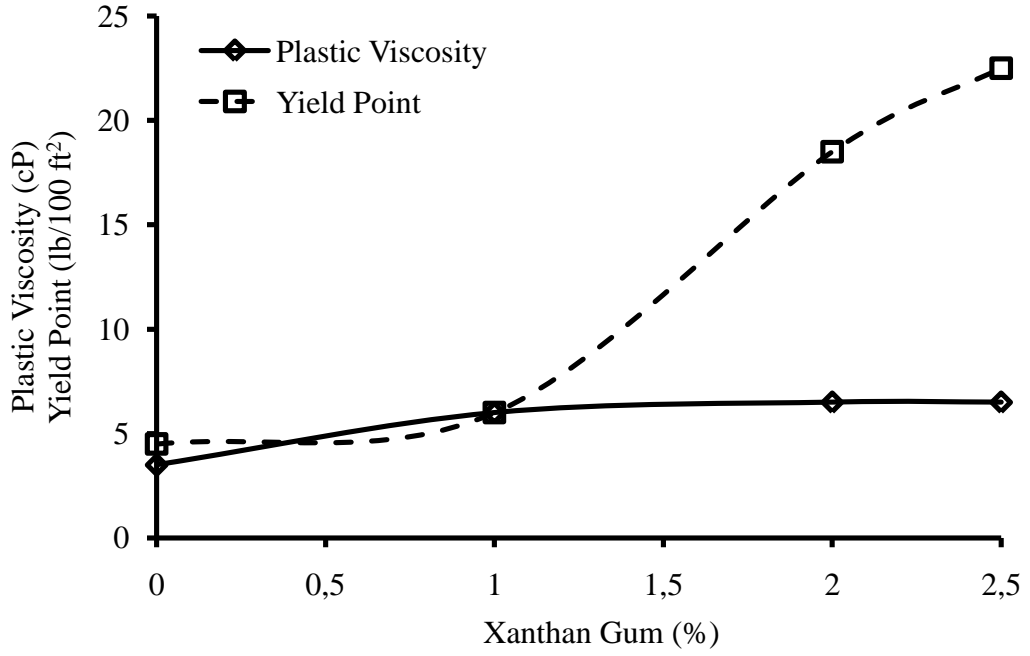


Figure 5-93 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of C-Ben

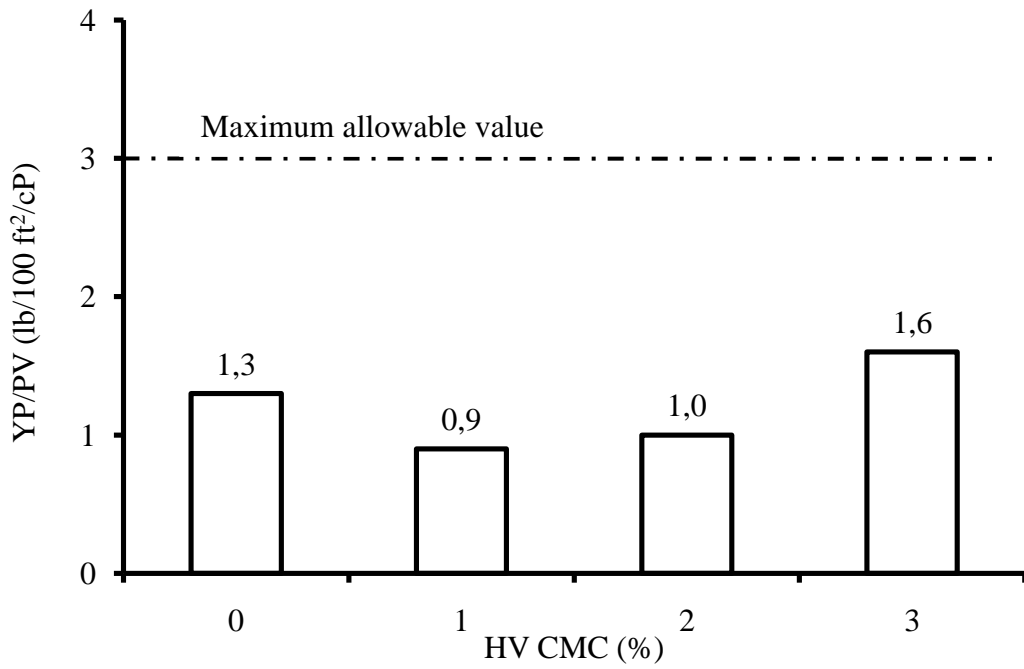


Figure 5-94 Effect of HV CMC on the YP/PV of C-Ben

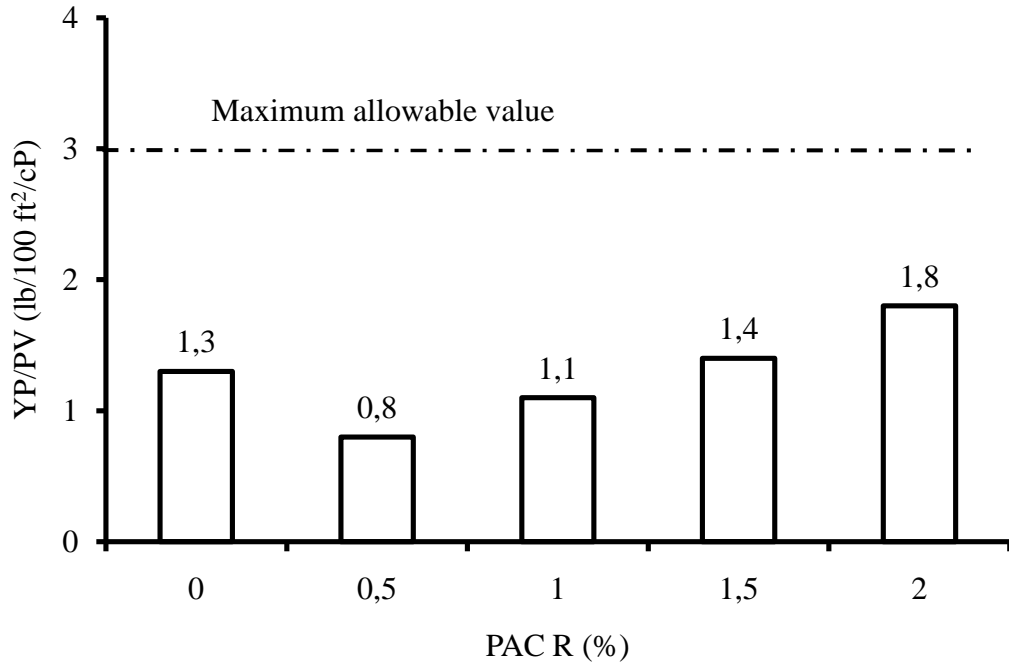


Figure 5-95 Effect of PAC R on the YP/PV of C-Ben

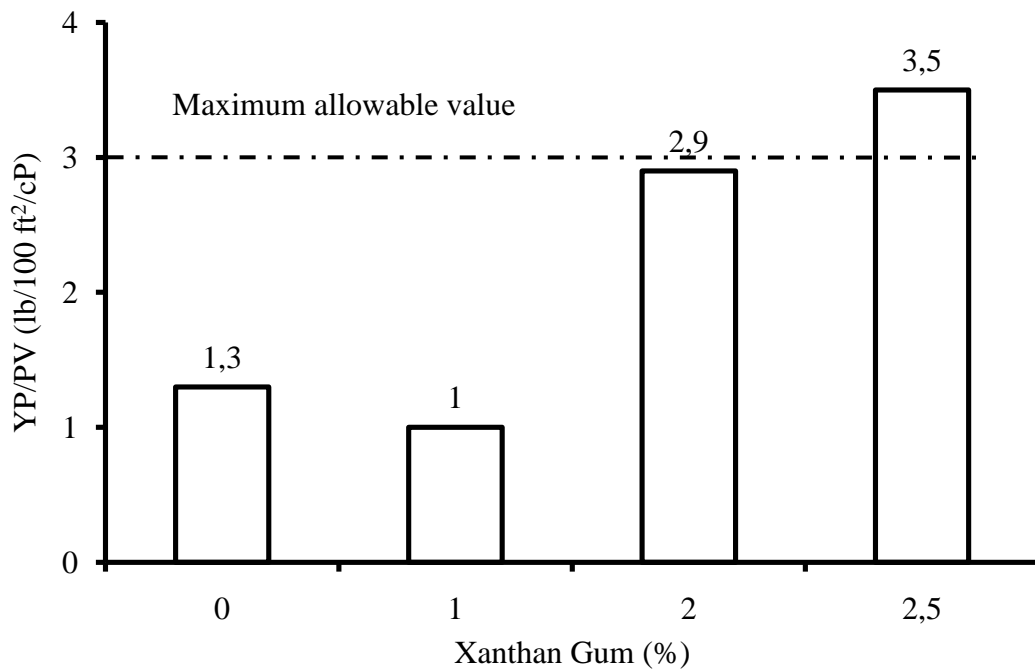


Figure 5-96 Effect of Xanthan Gum on the YP/PV of C-Ben

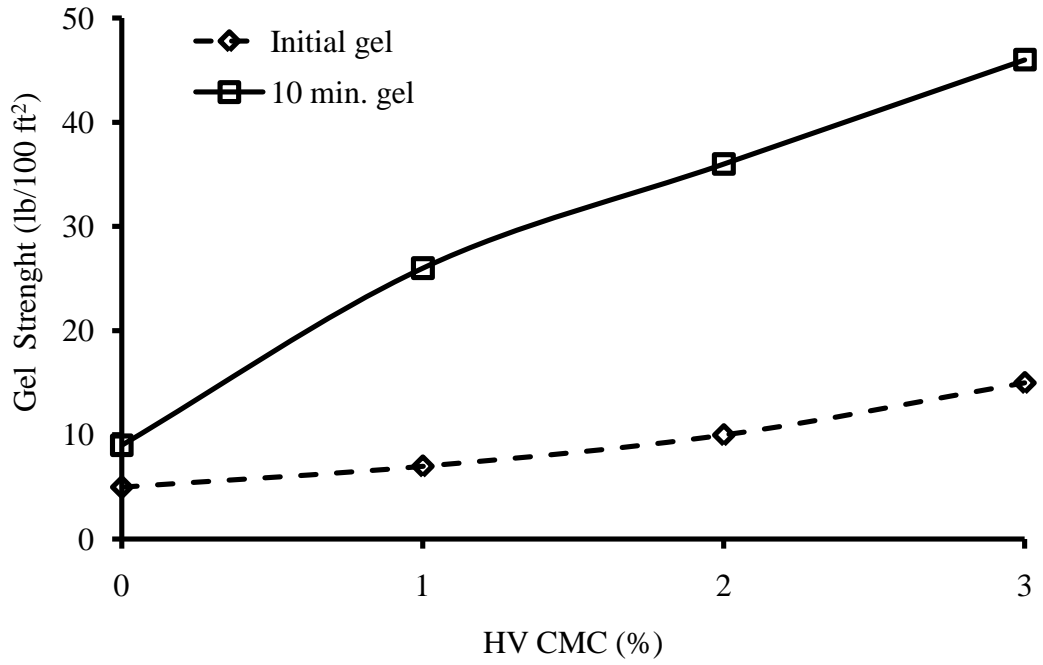


Figure 5-97 Effect of HV CMC on the Gel Strength of C-Ben

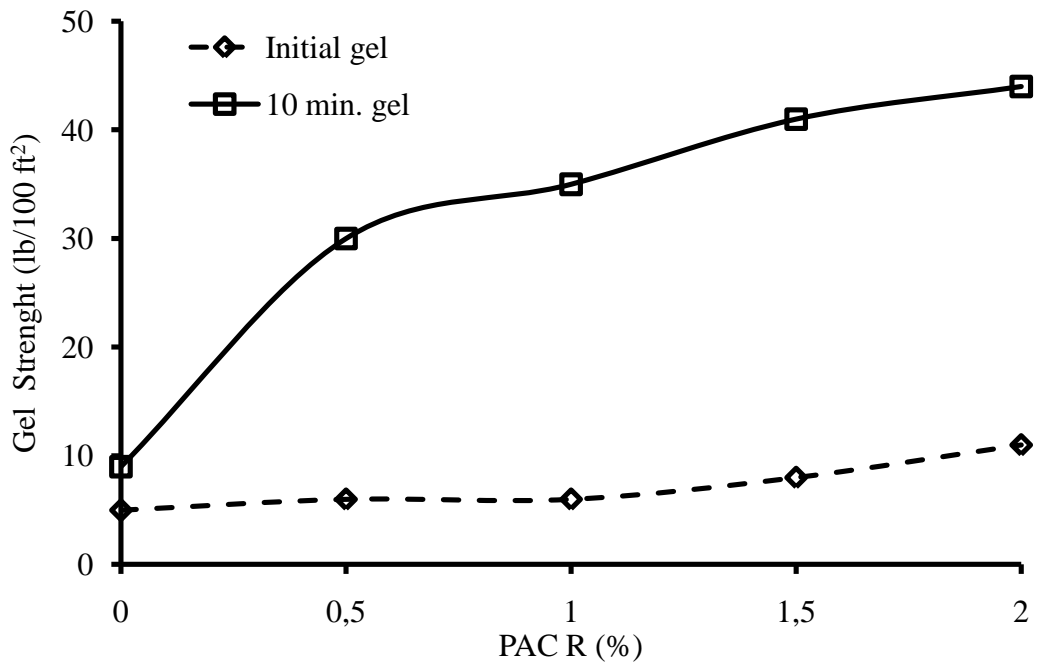


Figure 5-98 Effect of PAC R on the Gel Strength of C-Ben

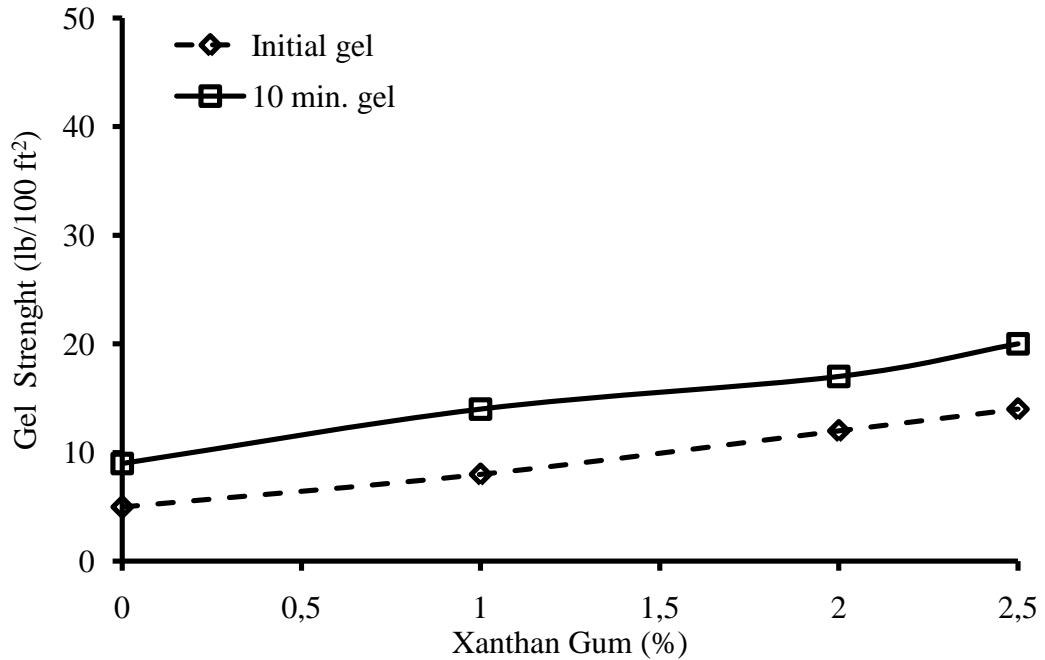


Figure 5-99 Effect of Xanthan Gum on the Gel Strength of C-Ben

#### 5.4.6 The Effect of Polymers on the Rheology and Filtrate Properties of C-Ben Activated with $\text{Na}_2\text{CO}_3$

In this experiment C-Ben activated with  $\text{Na}_2\text{CO}_3$  ( $\text{Na}_2\text{CO}_3$  addition level; 0,58%  $\text{Na}_2\text{O}$  equivalent) was developed by treating with polymers to control the rheology and filtrate loss for water-based drilling fluids. The effects of polymers (HV CMC, PAC R, and xanthan gum) on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{CO}_3$  are given in Figures 5-100 through 5-111 (also the appendix in Table B-21, B-22, and B-23).

With the HV CMC, PAC R, and xanthan gum treated muds, the increased polymer concentration resulted in a decrease in fluid loss and increase in apparent viscosity. But an acceptable value 15 ml for filtrate volume (shown as a dash-dot line) was not obtained with increasing polymer concentrations (Figures 5-100 through 5-102). Yield point increased by increasing polymer concentration whereas the plastic viscosity of the suspension in the presence of polymers was close to that of the suspension with the clay alone (Figures 5-103 through 5-105). Because of the

different behaviours between yield point and plastic viscosity, YP/PV values of treated bentonite were higher than the standard limit (Figures 5-106 through 5-108). The maximum allowable value is given as a dash-dot line.

It is interesting note to that the 10 minute gel strength abruptly decreased with the addition of very small amount of HV CMC and PAC R (Figures 5-109 and 5-110) except for xanthan gum (Figure 5-111). This small amount of polymer may become adsorbed on the clay edges and/or surfaces. Recently, Benchabane and Bekkour (2006) showed that  $d_{001}$  spacing remains unchanged after addition of CMC, the polymers do not penetrate the interlayer space, but act only on the external surface of the clay particles by coating them, hence preventing further particles interaction. The reason for decreasing of 10-gel strength of clay-water suspension by the addition of CMC and PAC may be due to the changes in surface charges and electrical double layers and consequently, the interaction among clay particles.

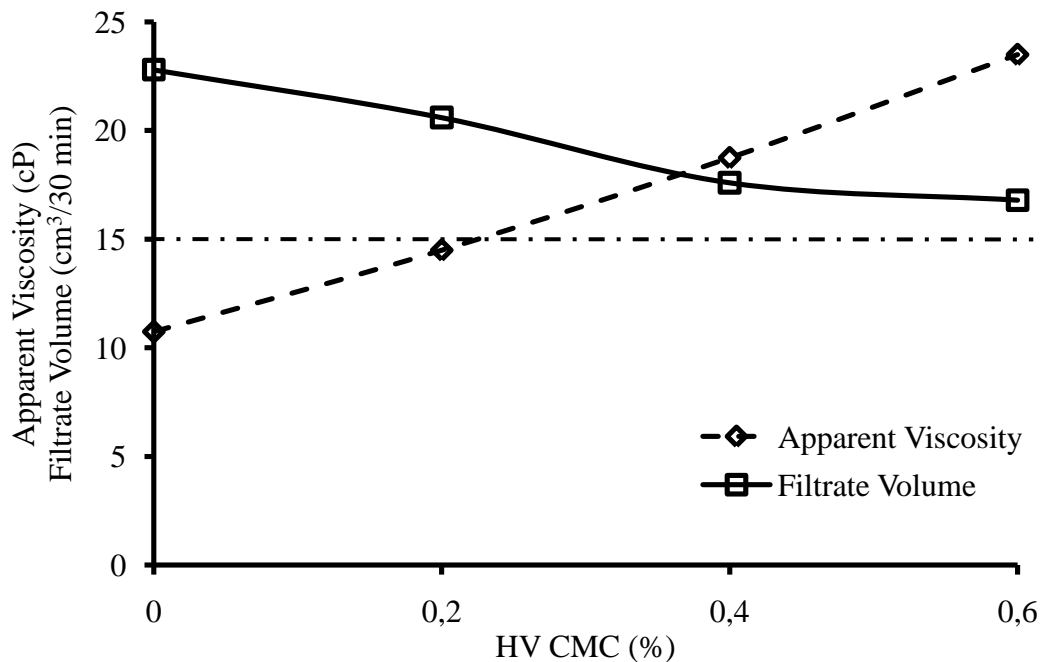


Figure 5-100 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

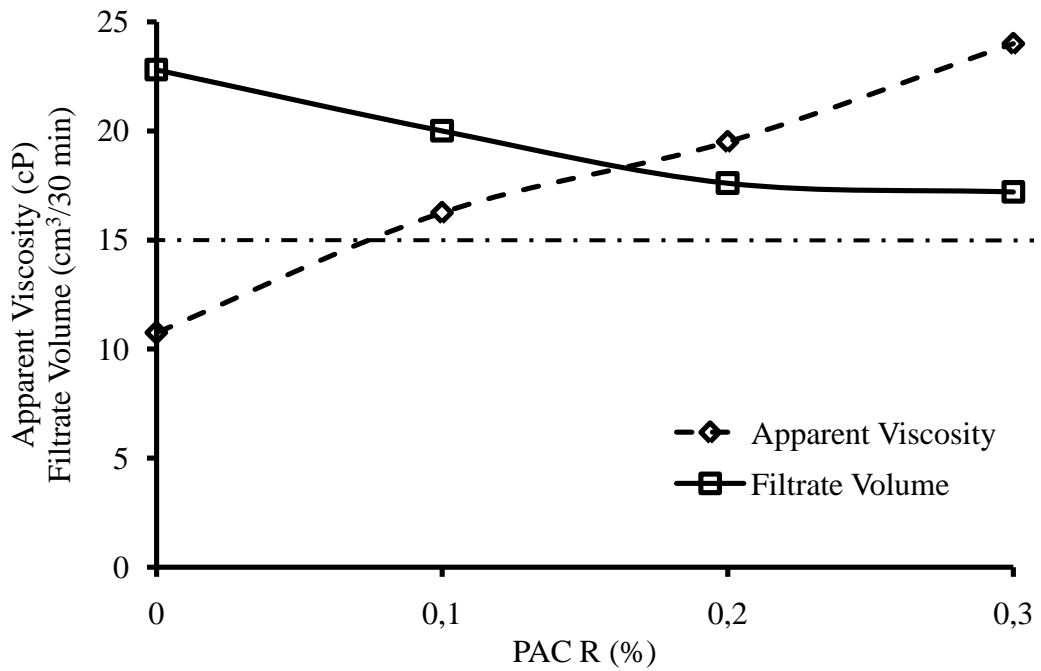


Figure 5-101 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

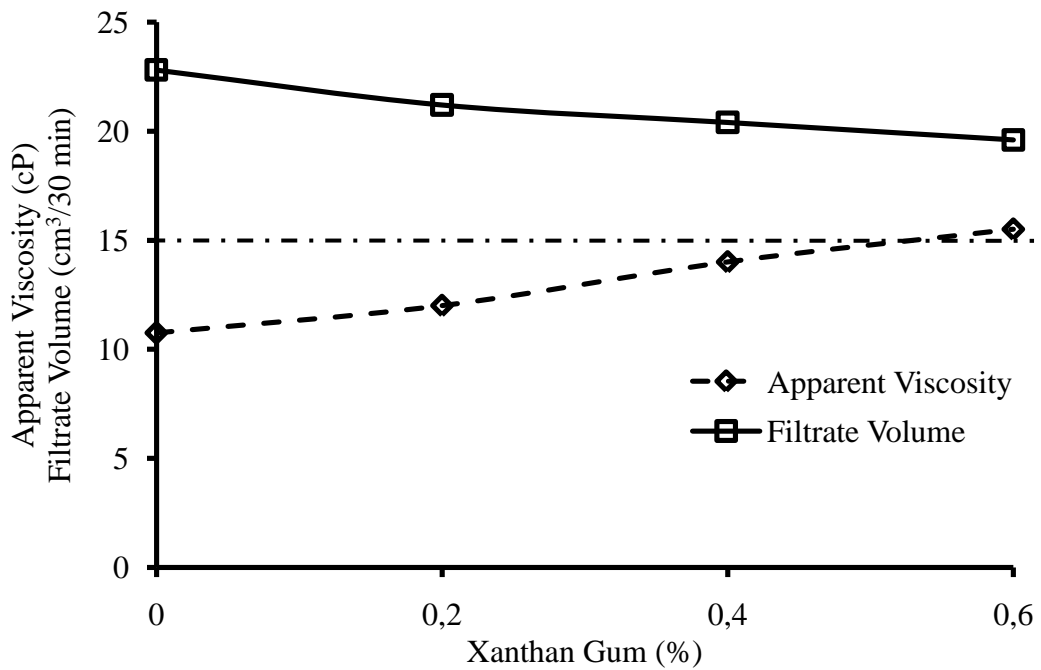


Figure 5-102 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{CO}_3$



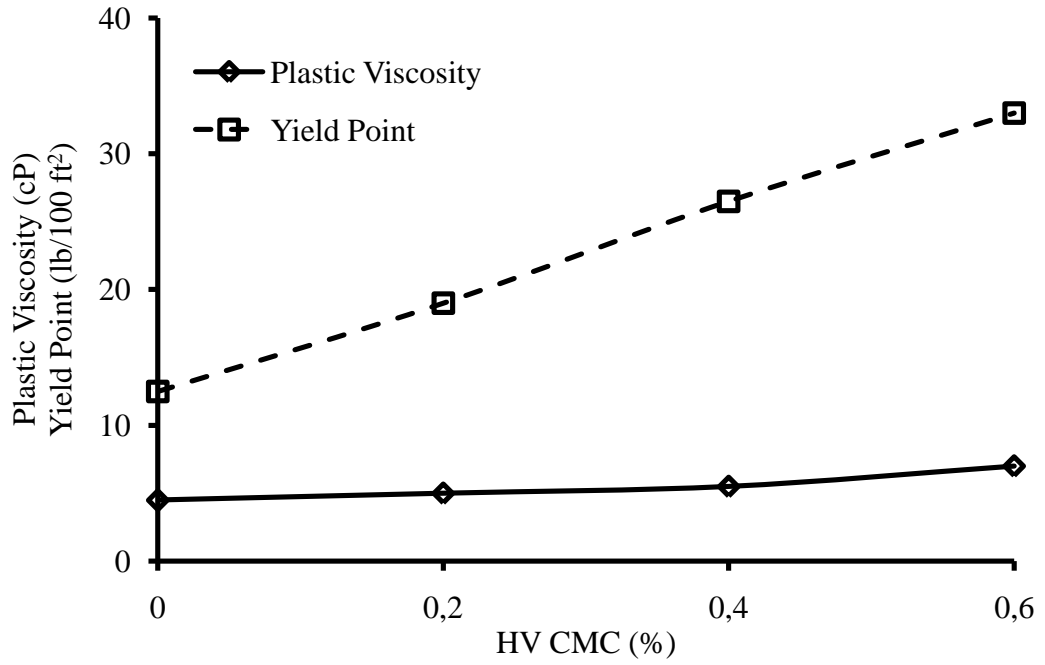


Figure 5-103 Effect of HV CMC on the Plastic Viscosity and Yield Point of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

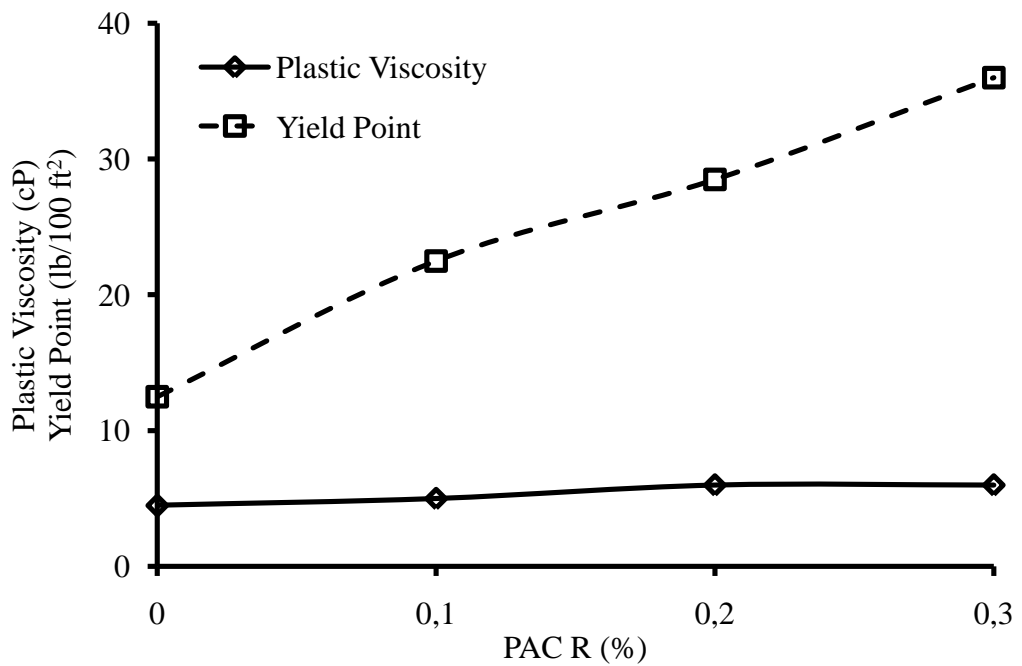


Figure 5-104 Effect of PAC R on the Plastic Viscosity and Yield Point of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

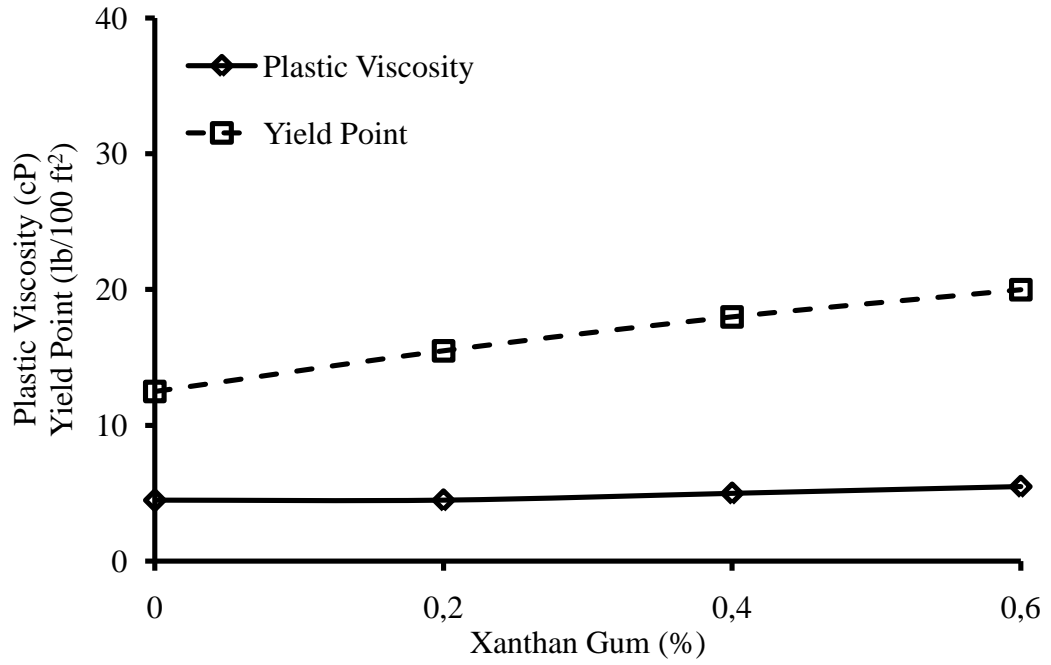


Figure 5-105 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

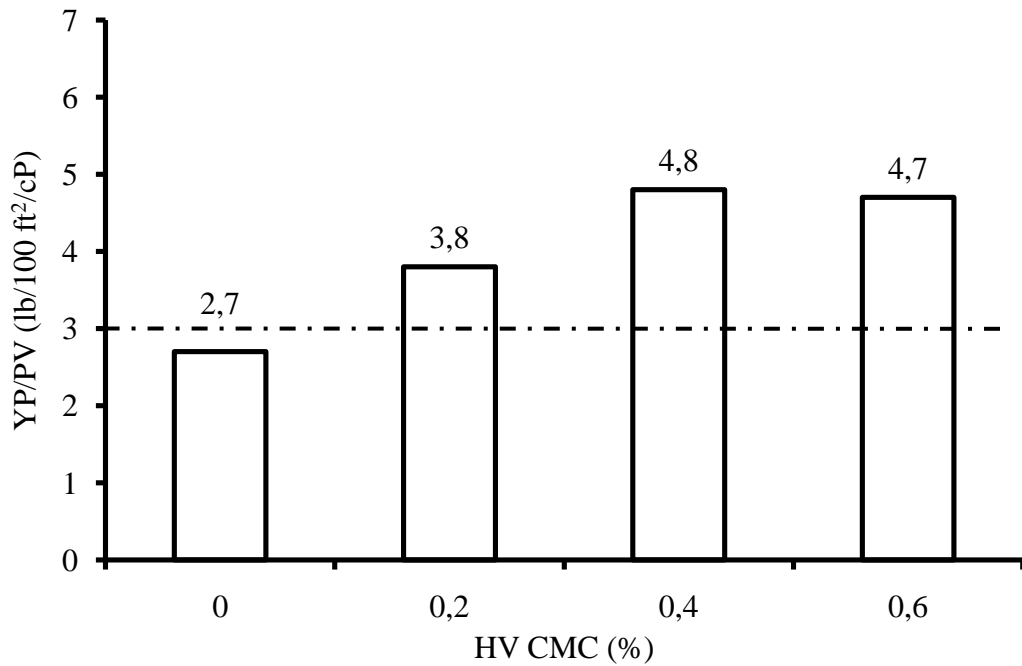


Figure 5-106 Effect of HV CMC on the YP/PV of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

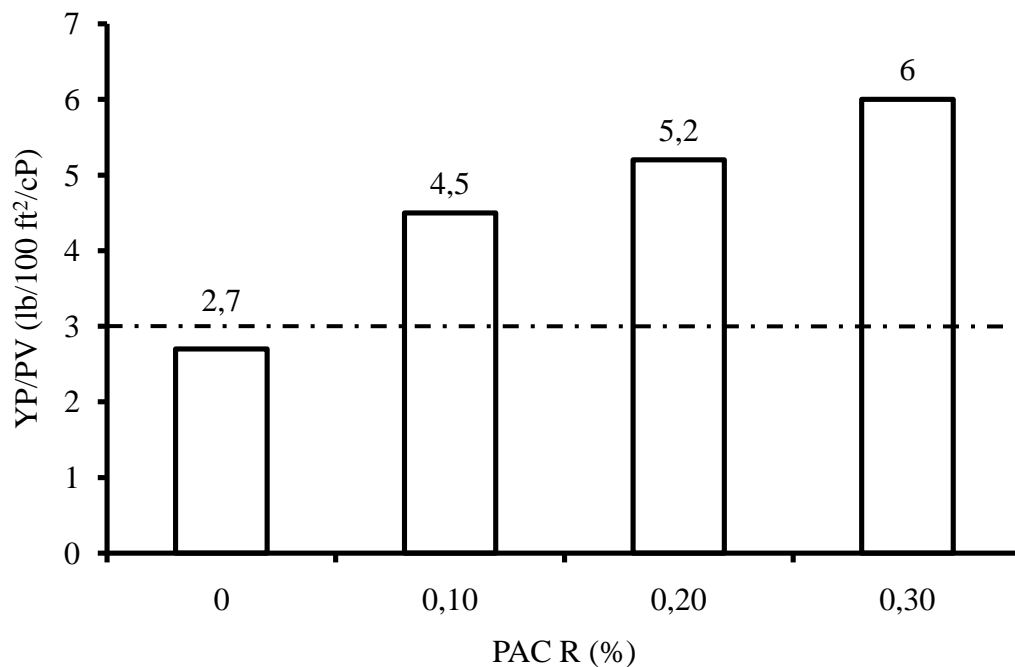


Figure 5-107 Effect of PAC R on the YP/PV of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

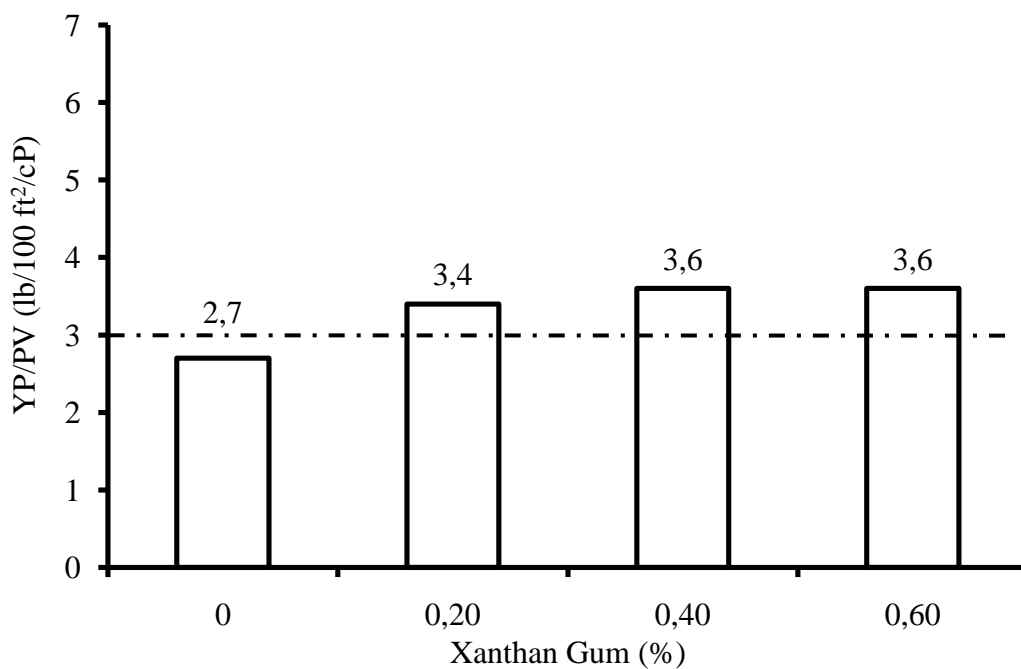


Figure 5-108 Effect of Xanthan Gum on the YP/PV of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

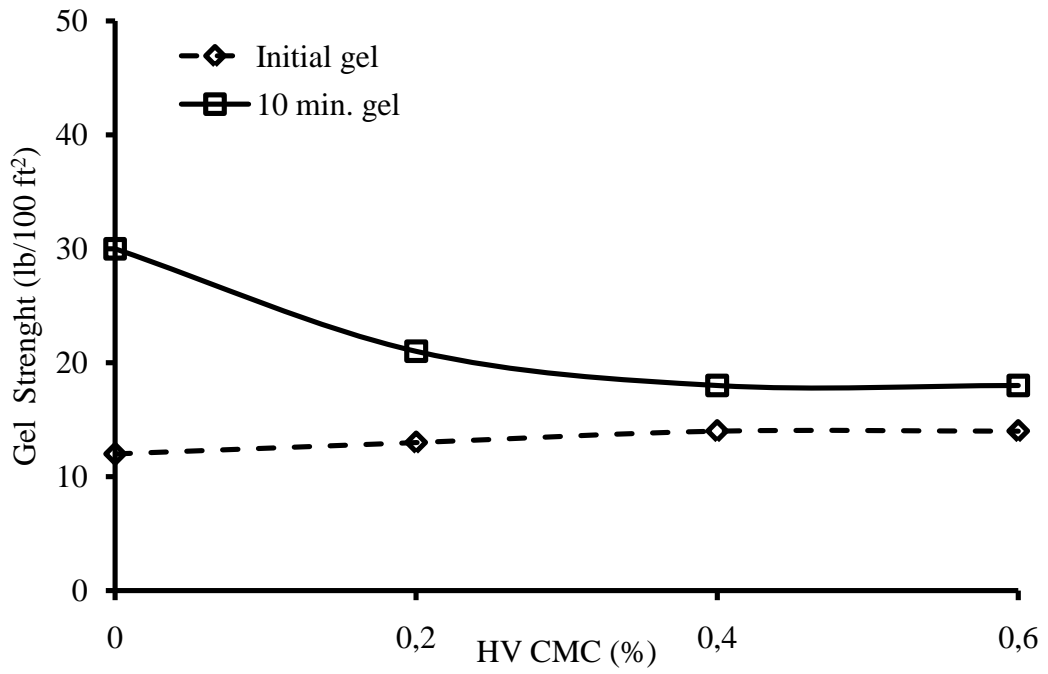


Figure 5-109 Effect of HV CMC on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

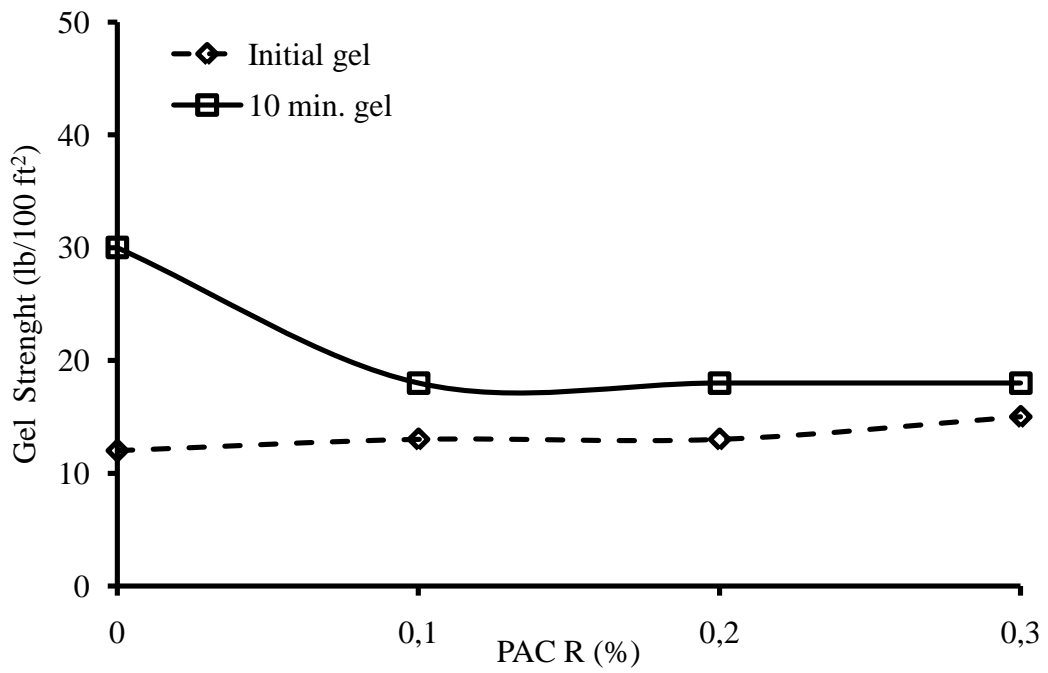


Figure 5-110 Effect of PAC R on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

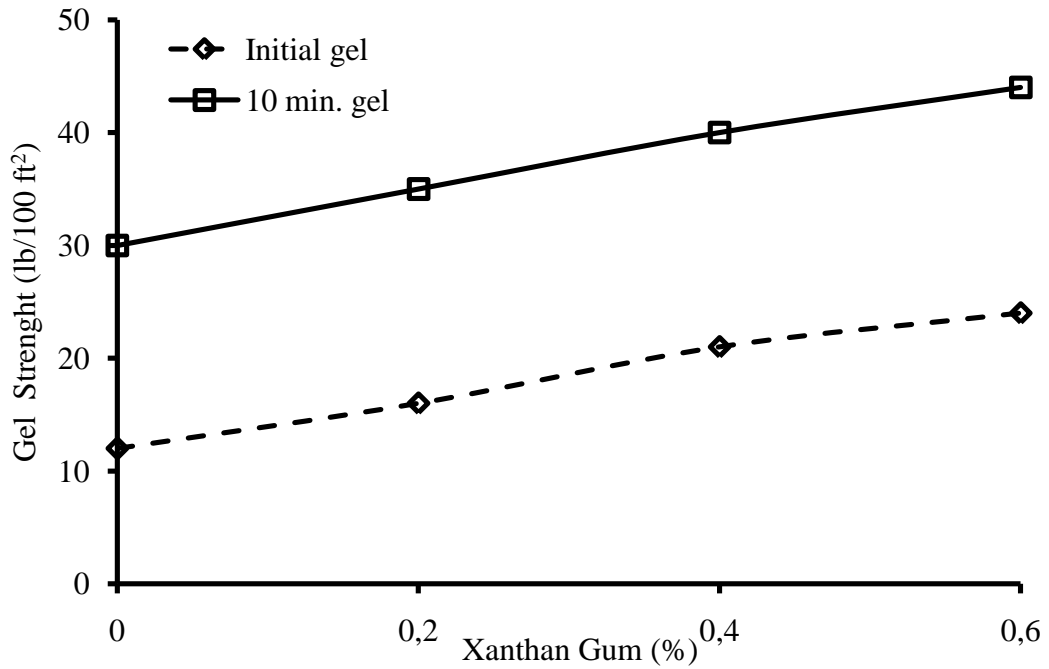


Figure 5-111 Effect of Xanthan Gum on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

Because of high filtrate volume of bentonite treated with HV CMC and PAC R and xanthan gum muds, the bentonite was treated with HV CMC and LV CMC mixture, PAC R and PAC L mixture, and xanthan gum and dextrid mixture, separately.

The bentonite treated with various ratios of high and low viscosity grade polymers mixture to meet the API requirements. The concentrations of polymers mixture used in the treatment of bentonite are given in Table 5-1.

Table 5-1 The concentrations of polymers mixture used in the treatment of Na<sub>2</sub>CO<sub>3</sub> activated C- Ben

Concentration (%)		Concentration (%)		Concentration (%)	
HV CMC	LV CMC	PAC R	PAC L	Xant. Gum	Dextrid
1,25	0,75	0,70	0,85	0,40	1,60
0,80	0,40	0,50	0,30	0,40	2,00
0,70	0,35	0,40	0,30	0,80	2,20
0,60	0,30	0,40	0,20	0,80	2,40

The effect of HV CMC and LV CMC mixture, used as viscosifier and filtrate loss control respectively, on the rheology and filtrate properties of C-Ben activated with Na<sub>2</sub>CO<sub>3</sub> is given in Figure 5-112 (see also appendix Table B-24). It can be seen in figure an acceptable value (15 cp) for apparent viscosity and (15 cm<sup>3</sup>) for filtrate volume were obtained with 0,70% HV CMC and 0,35% LV CMC mixture This is the smallest amount of polymer mixture to meet API requirements. But a 0,80% HV CMC and 0,40% LV CMC mixture was chosen for aging test to compare with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite. Because this amount of polymer is the smallest amount of polymer to meet the API standard for Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite.

The effect of PAC R and PAC L, used as viscosifier and filtrate loss control respectively, on the rheology and filtrate properties of C-Ben activated with Na<sub>2</sub>CO<sub>3</sub> is given in Figure 5-113 (see also appendix Table B-25). As it can be seen in figure an acceptable value (15 cp) for apparent viscosity and (15 cm<sup>3</sup>) for filtrate volume were obtained with 0,40% PAC R and 0,30% PAC L (the smallest amount of polymer to meet API standard). But 0,50% PAC R and 0,30% PAC L mixture was chosen for aging test to compare with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite because of the same reason.

It is noted that the minimum required value for apparent viscosity and maximum allowable value for filtrate volume were showed as a dash-dot line and the maximum allowable value for YP/PV ratio was showed as a dotted line in the figures.

The effect of xanthan gum and dextrid mixture, used as viscosifier and filtrate loss control respectively, on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{CO}_3$  is given in Figure 5-114 (see also appendix Table B-26). It can be seen in figure that an acceptable value (15 cp) for apparent viscosity and (16  $\text{cm}^3$ ), the maximum required value for OCMA type bentonite) for filtrate volume were obtained with a 0,80% xanthan gum and 2,40% dextrid mixture. The physical properties of  $\text{Na}_2\text{CO}_3$  activated bentonite is close to OCMA type bentonite requirements. The requirement for filtrate volume is shown as a straight line and that of viscosity is shown as a dashed line in the figures. The dotted line is used to show the maximum allowable value for YP/PV (6, lb/100  $\text{ft}^2/\text{cP}$ ).

The rheological and filtrate properties of polymers used in the treatment of C-Ben activated with  $\text{Na}_2\text{CO}_3$  are given in Figure 5-115 (see also appendix Table B-27).

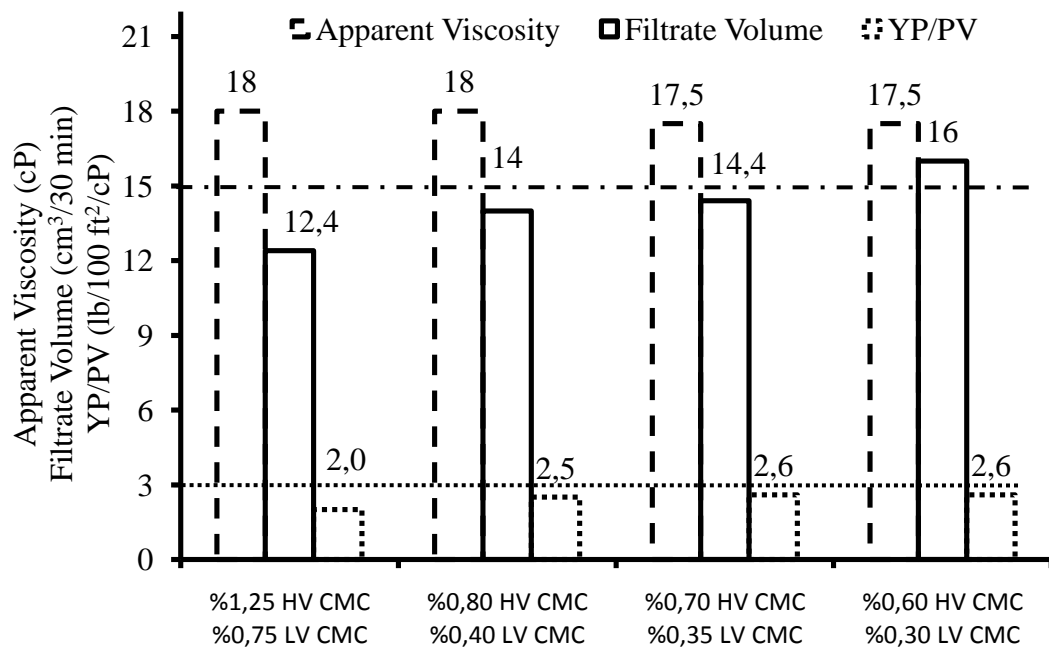


Figure 5-112 Effect of the Mixture of HV CMC and LV CMC on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

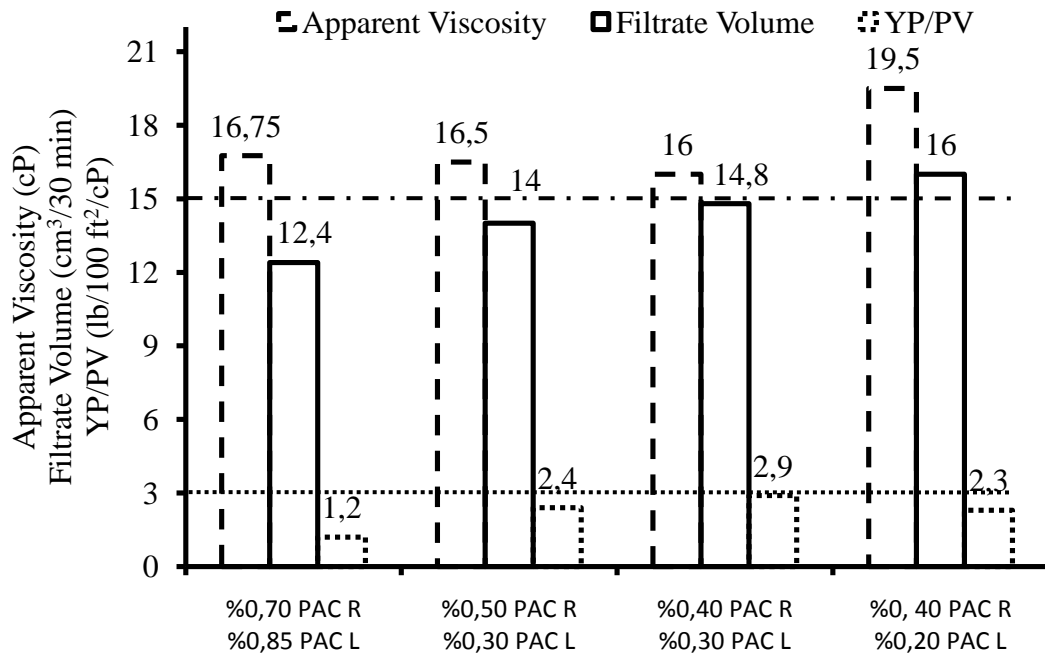


Figure 5-113 Effect of the Mixture of PAC R and PAC L on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

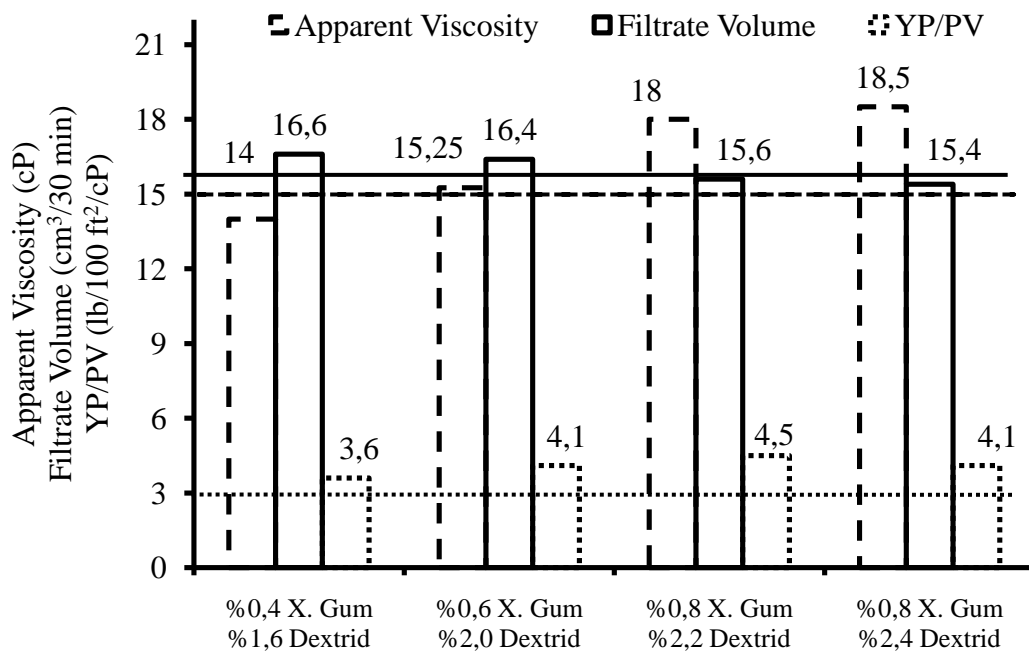


Figure 5-114 Effect of the Mixture of Xanthan Gum and Dextrid on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>



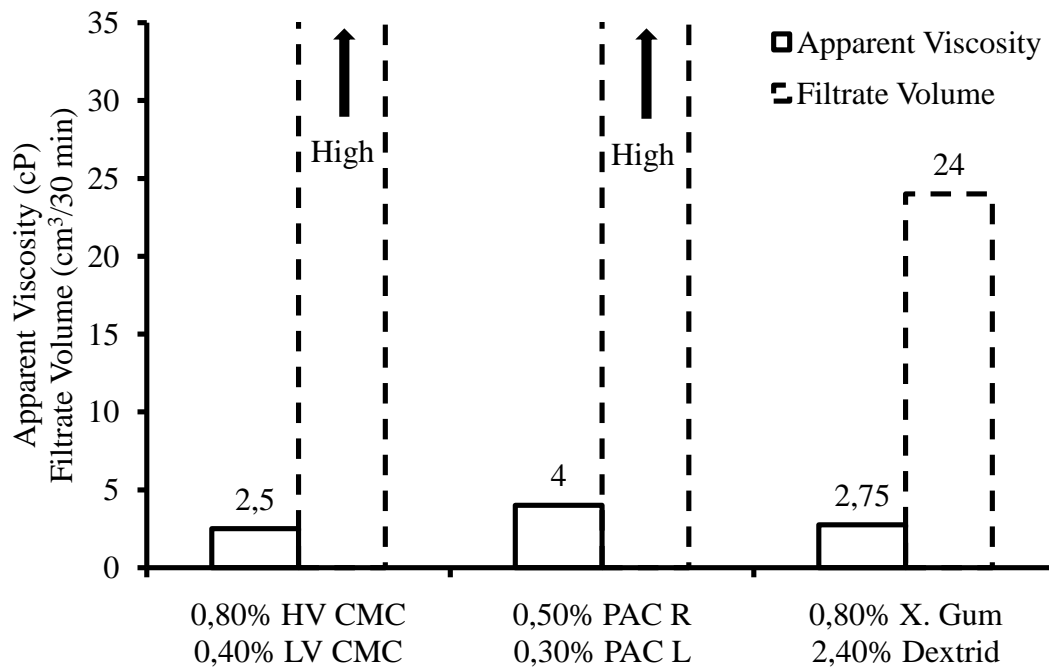


Figure 5-115 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of C-Ben Activated with  $\text{Na}_2\text{CO}_3$

#### 5.4.7 The Effect of Polymers on the Rheology and Filtrate Properties of C-Ben Activated with $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

In this experiment C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  addition level; 0,76%  $\text{Na}_2\text{O}$  equivalent) was obtained by treatment with polymers to control the rheology and filtrate loss for water-based drilling fluids. The effects of polymers (HV CMC, PAC R and xanthan gum) on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  are given in Figures 5-116 through 5-127 (see also appendix Table B-28, B-29 and B-30).

The addition of 0,6% HV CMC increased the apparent viscosity of mud from 10 to 17,5 cP and reduced the fluid loss from 25 to 17,4  $\text{cm}^3$  (Figure 5-116) while the addition of 0,3% PAC R increased the apparent viscosity of mud from 10 to 20,5 cP and reduced the fluid loss from 25 to 17,2  $\text{cm}^3$  (Figure 5-117). Moreover the addition of 0,9% xanthan gum increased the apparent viscosity of mud from 10 to 16 cP and reduced the fluid loss from 25 to 18,8  $\text{cm}^3$  (Figure 5-118). It was seen that

the acceptable value of 15 ml for filtrate volume was not obtained by increasing concentration of polymer. Examination of the graphs revealed that this viscosity changes were brought about by increasing the yield point. Little or no changes were experienced in plastic viscosity (Figures 5-119 through 5-121). Due to the different behaviour between plastic viscosity and filtrate volume, YP/PV values of treated bentonite were higher than the standard limit (Figures 5-122 through 5-124).

The addition of HV CMC and PAC R decreased the 10 minute gel strengths from 25 down to 20 and 16, respectively (Figures 5-125 and 5-126). In contrast, the addition of xanthan gum increased the gel strengths (Figure 5-127)

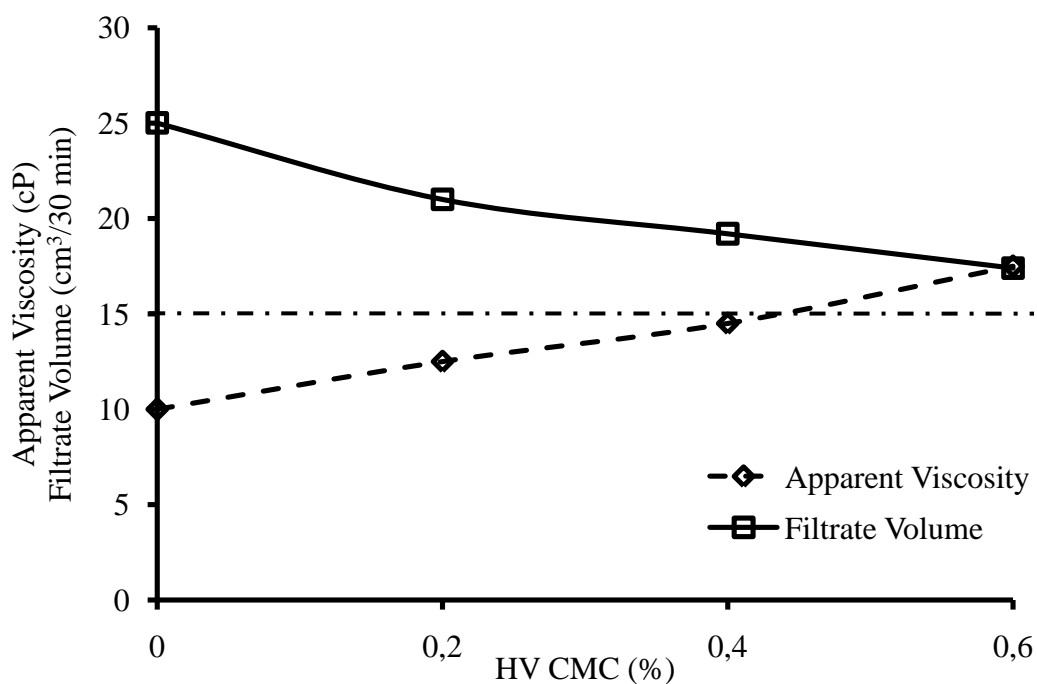


Figure 5-116 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

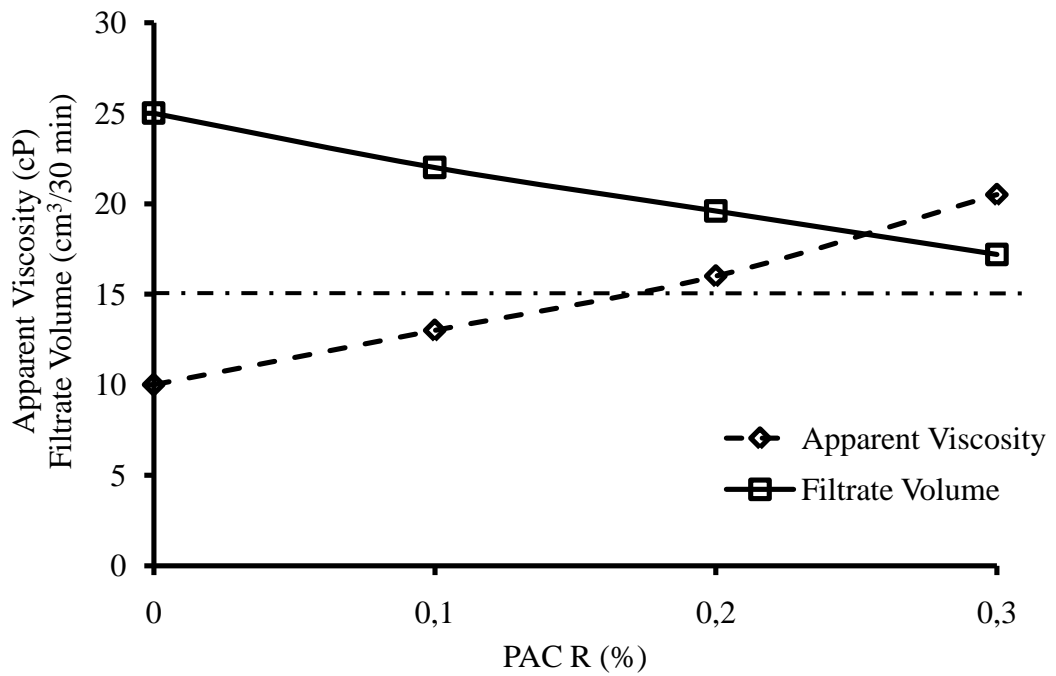


Figure 5-117 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

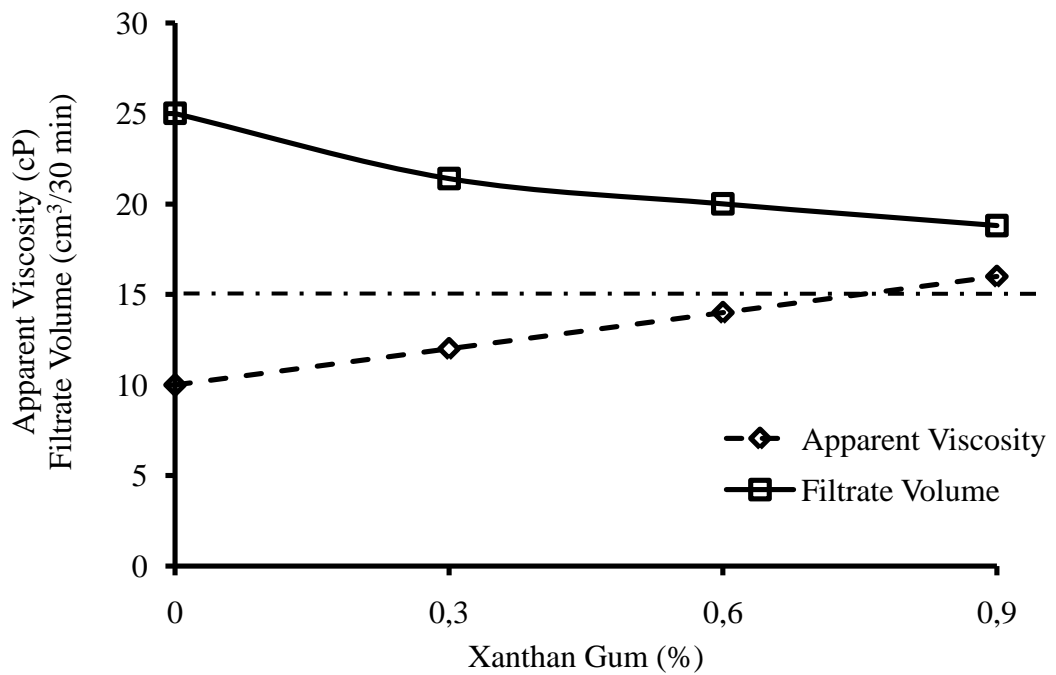


Figure 5-118 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

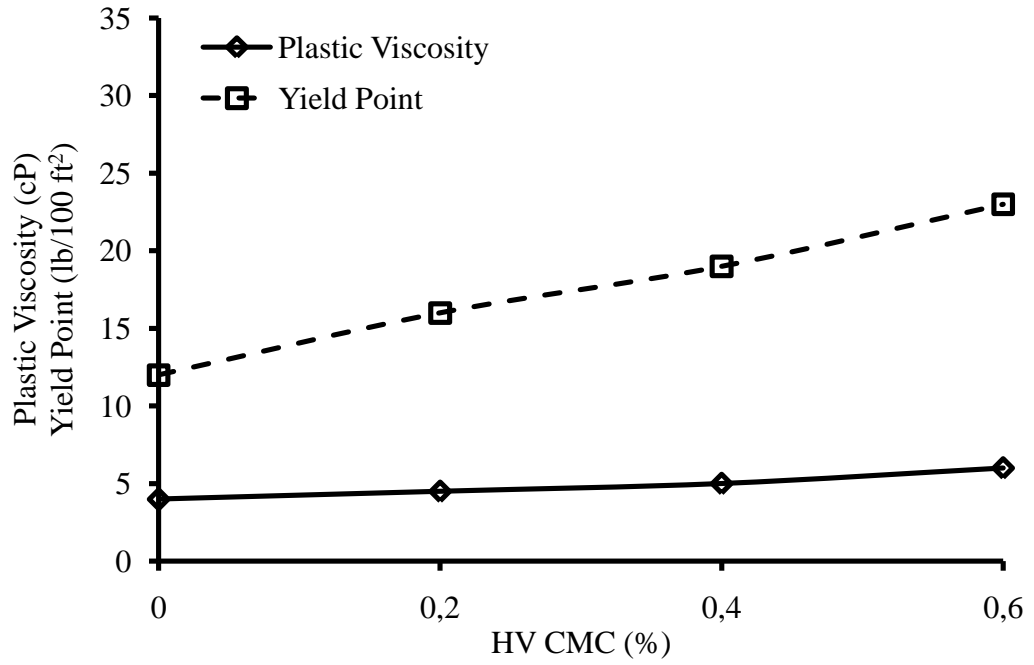


Figure 5-119 Effect of HV CMC on the Plastic Viscosity and Yield Point of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

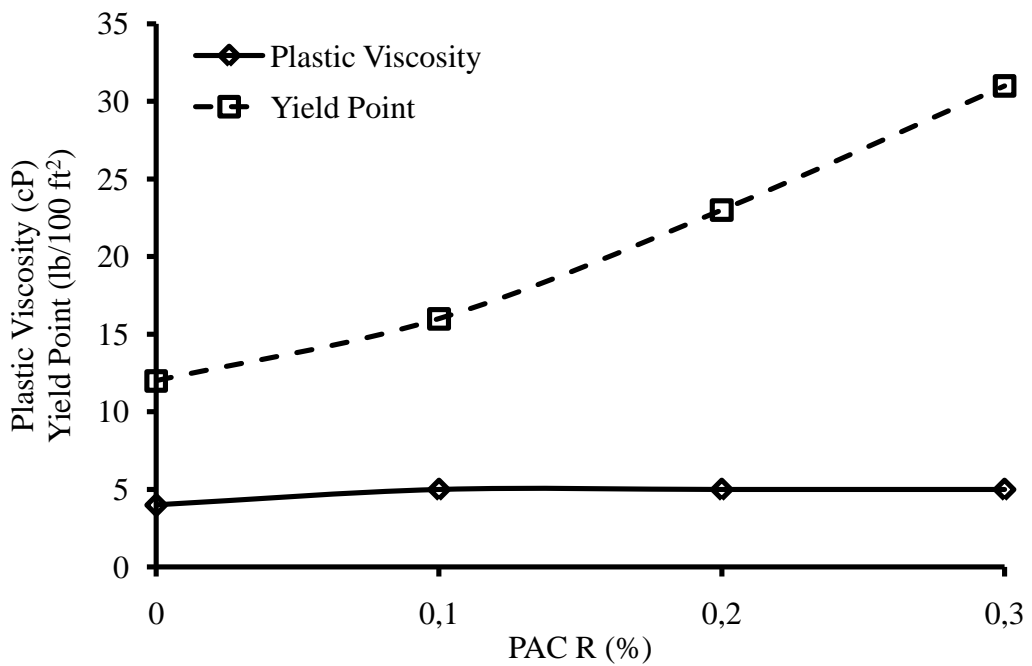


Figure 5-120 Effect of PAC R on the Plastic Viscosity and Yield Point of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

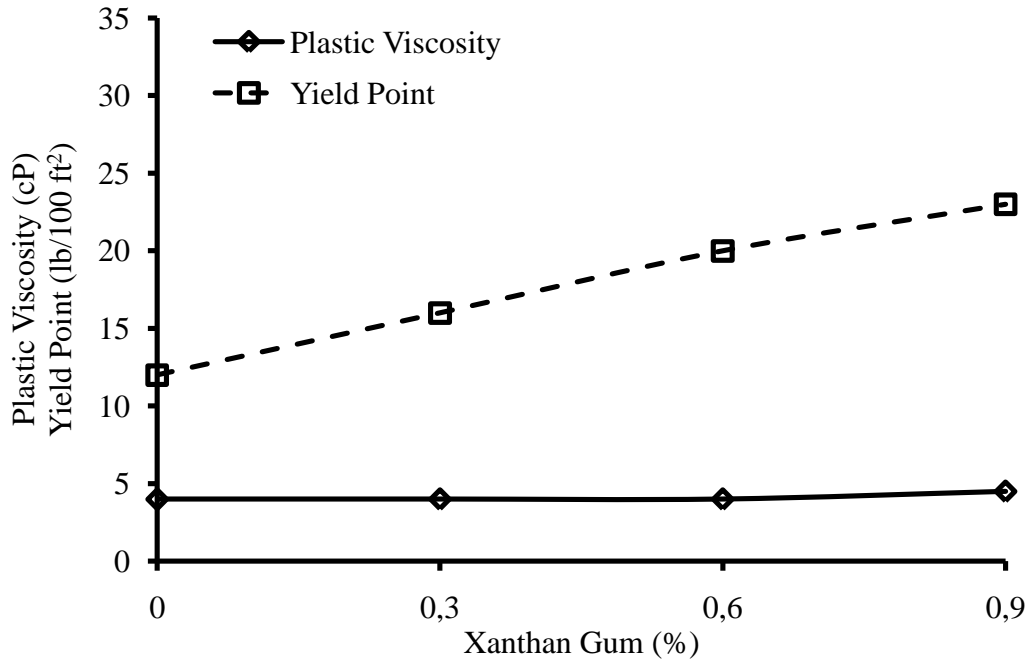


Figure 5-121 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

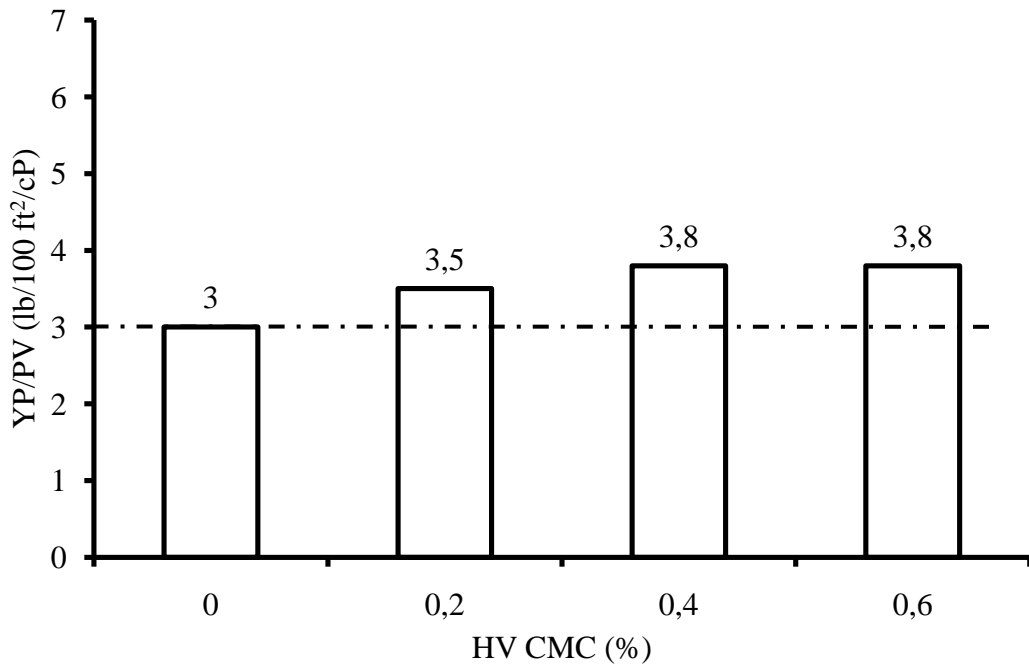


Figure 5-122 Effect of HV CMC on the YP/PV of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

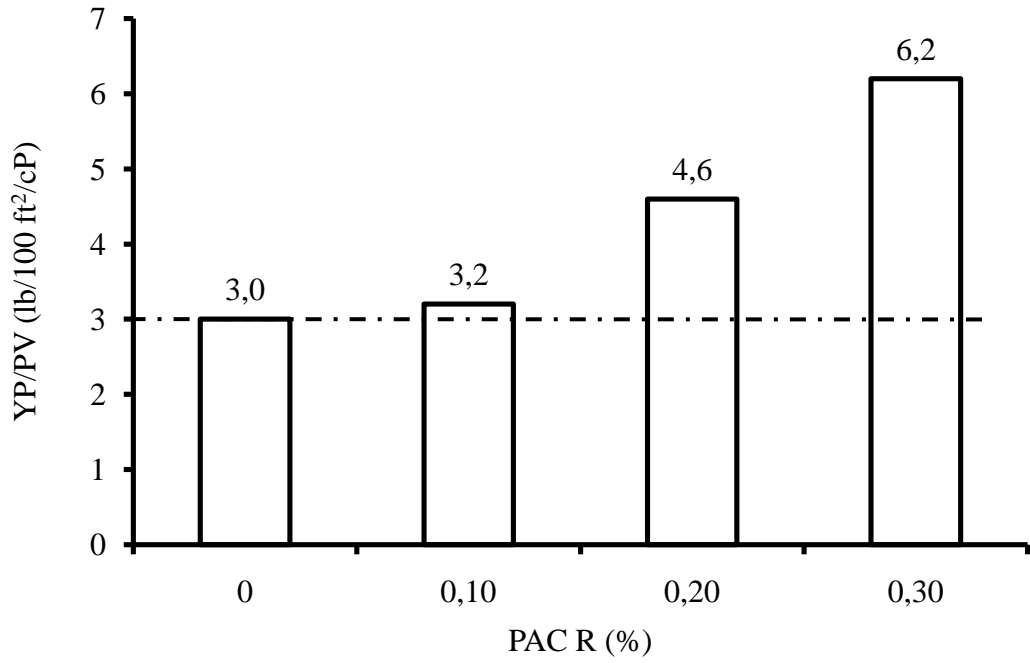


Figure 5-123 Effect of PAC R on the YP/PV of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

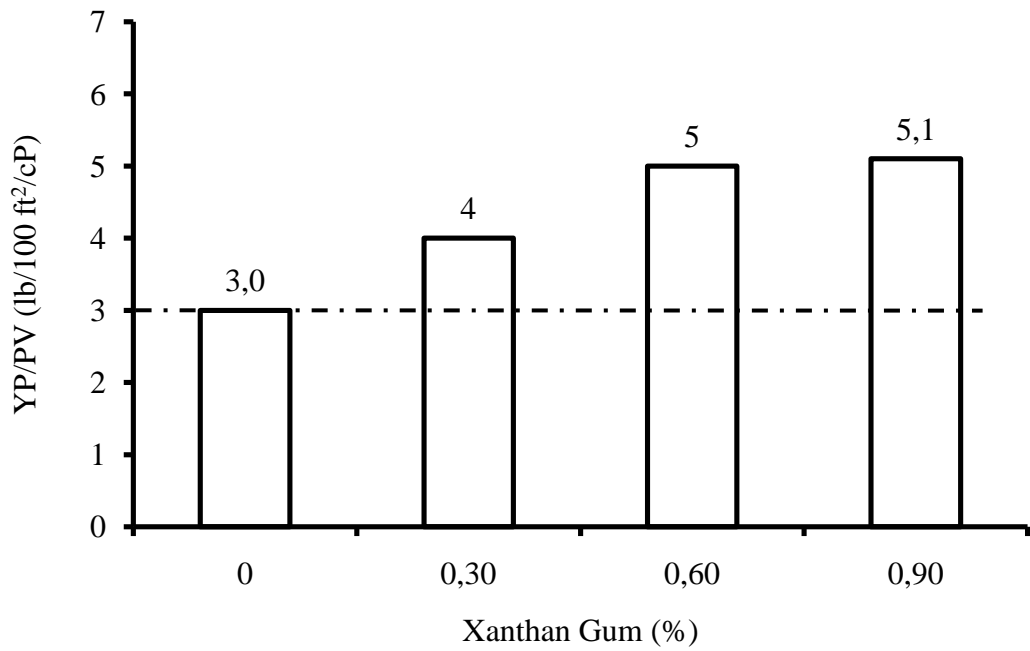


Figure 5-124 Effect of Xanthan Gum on the YP/PV of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

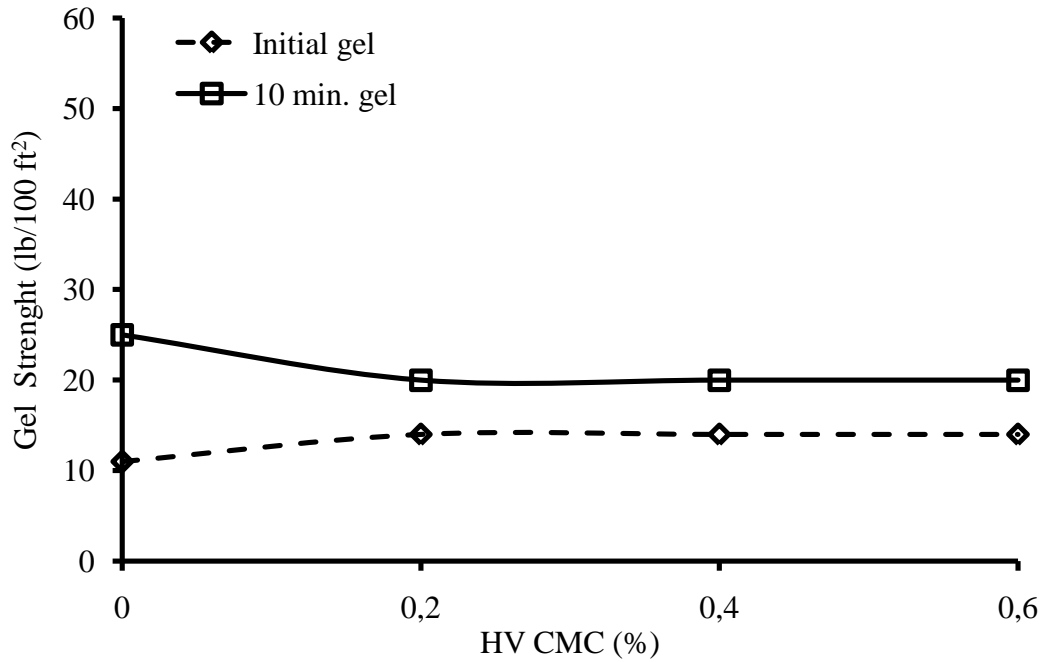


Figure 5-125 Effect of HV CMC on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

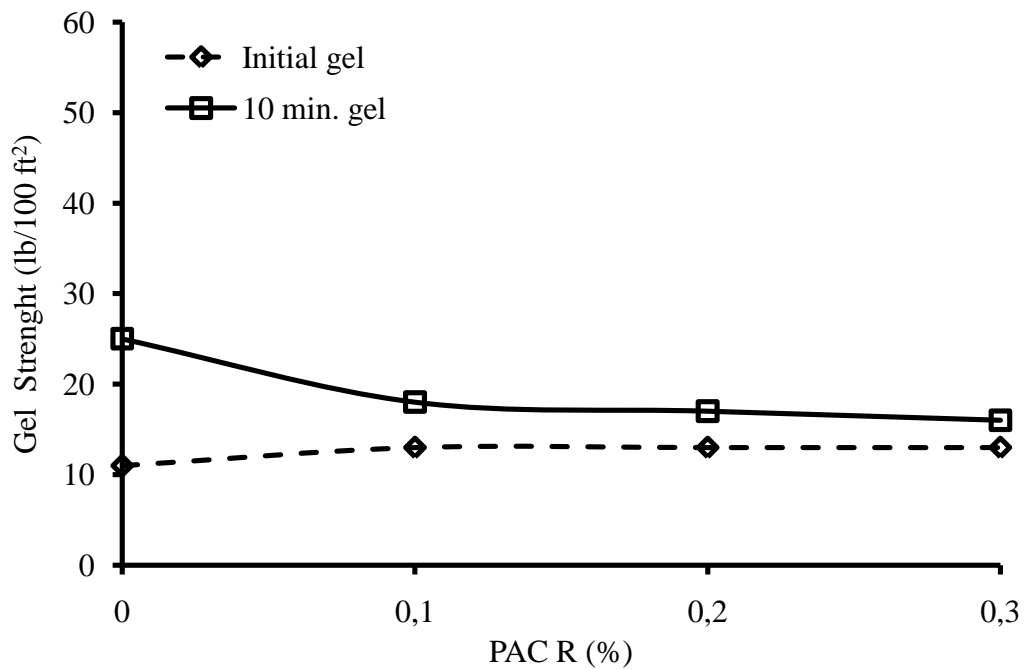


Figure 5-126 Effect of PAC R on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

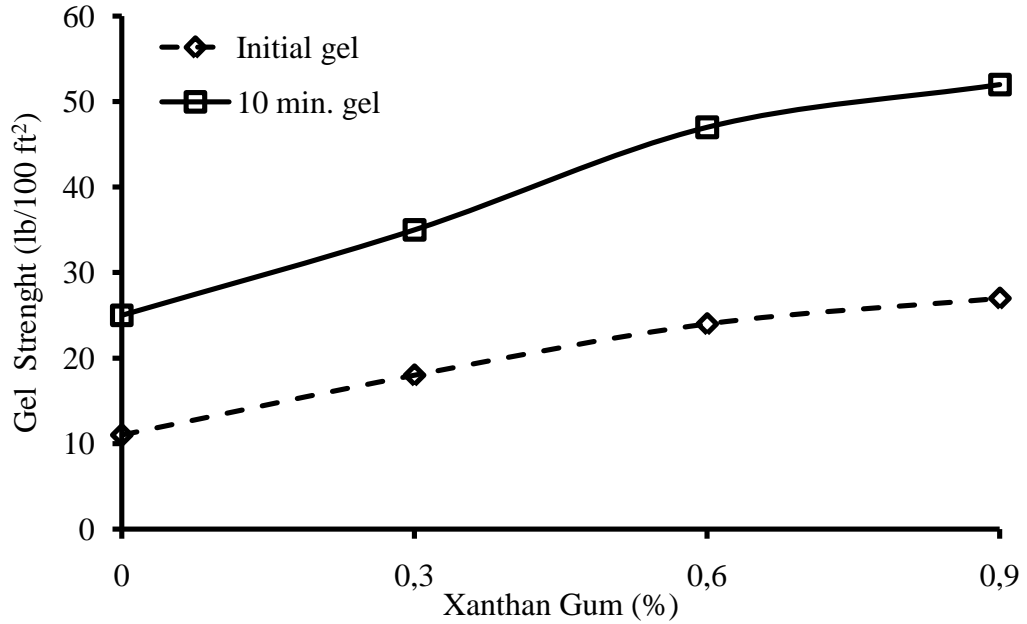


Figure 5-127 Effect of Xanthan Gum on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

Because of high filtrate volume and high YP/PV ratio, the bentonite was treated with a HV CMC and LV CMC mixture, PAC R and PAC L mixture, and xanthan gum and Dextrid LT, separately. The concentrations of polymers mixture used in the treatment of bentonite are given in Table 5-2.

Table 5-2 The concentrations of polymers mixture used in the treatment of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  activated C- Ben

Concentration (%)		Concentration (%)		Concentration (%)	
HV CMC	LV CMC	PAC R	PAC L	Xant. Gum	Dextrid
1,30	0,50	0,85	0,60	0,40	1,60
1,00	0,40	0,60	0,30	0,40	2,00
0,70	0,30	0,50	0,30	0,80	2,20
0,80	0,40	0,40	0,25	0,80	2,40



The effect of HV CMC and LV CMC mixture on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  is given in Figure 5-128 (see also appendix Table B-31). As it can be seen in figure a minimum acceptable value (15 cP) for apparent viscosity and maximum acceptable value ( $15 \text{ cm}^3$ ) for filtrate volume (shown as a dash-dot line) were obtained with 0,80% HV CMC and 0,40% LV CMC.

The effect of PAC R and PAC L mixture on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  is given in Figure 5-129 (see also appendix Table B-32). It can be seen in figure an acceptable value (15 cp) for apparent viscosity and ( $15 \text{ cm}^3$ ) for filtrate volume (shown as a dash-dot line) were obtained with 0,50% PAC R and 0,30% PAC L.

The effect of xanthan gum and dextrans mixture on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{CO}_3$  is given in Figure 5-130 (see also appendix Table B-33). The physical properties of  $\text{Na}_2\text{CO}_3$  activated bentonite treated with the mixture of xanthan gum and dextrans are related to OCMA type bentonite requirements. It can be seen in figure that an acceptable value (15 cP) for apparent viscosity and ( $16 \text{ cm}^3$ ) for filtrate volume were obtained with 0,80% xanthan gum and 2,40% dextrans mixture.

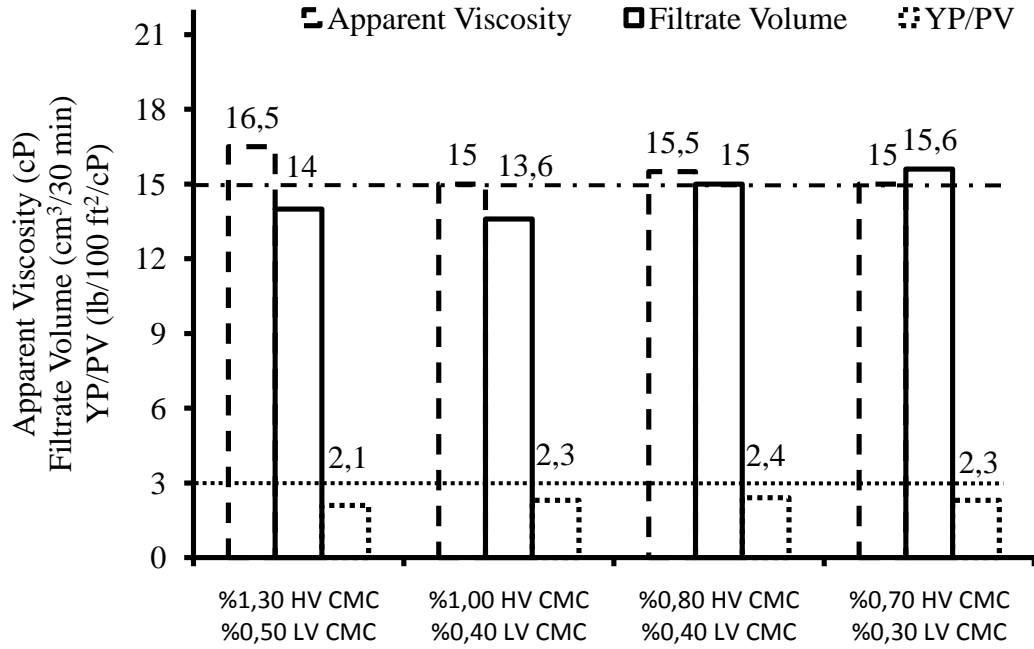


Figure 5-128 Effect of the Mixture of HV CMC and LV CMC on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O

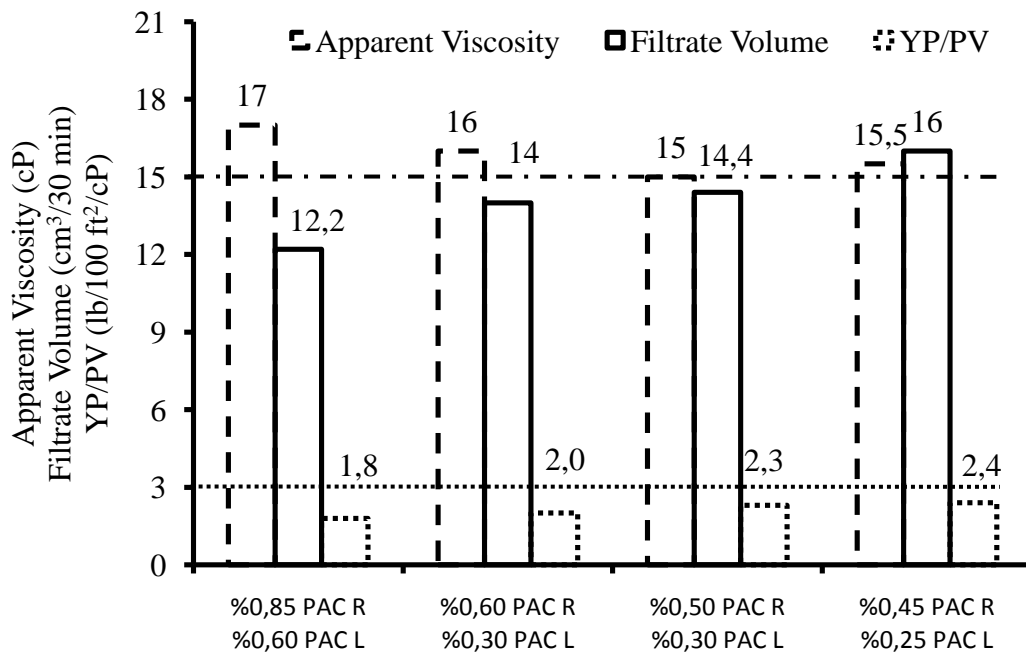


Figure 5-129 Effect of the Mixture of PAC R and PAC L on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O

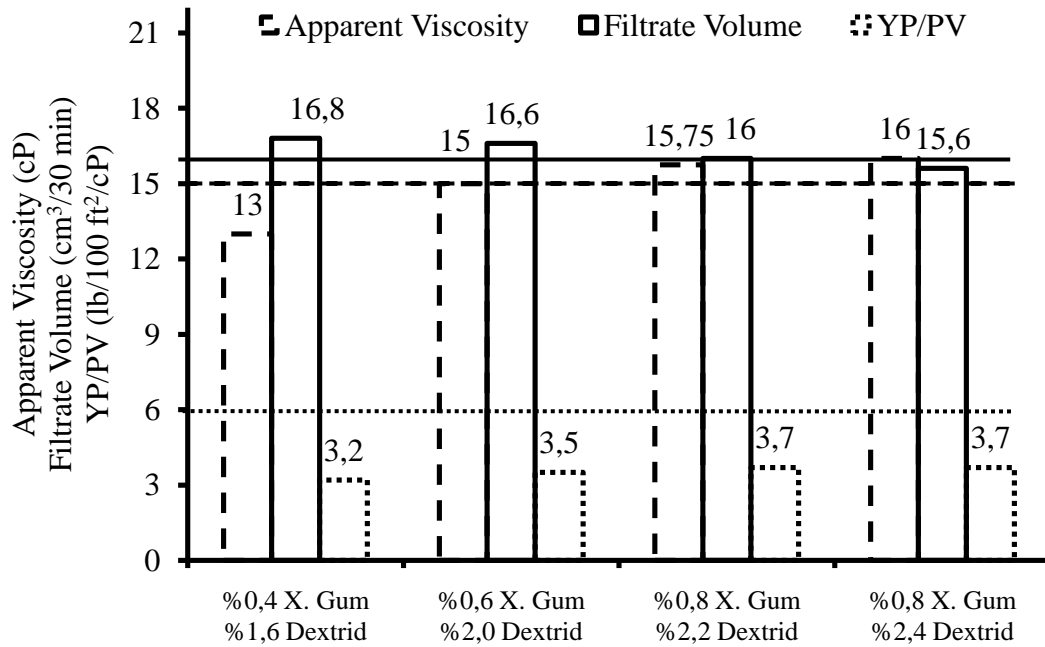


Figure 5-130 Effect of the Mixture of Xanthan Gum and Dextrid on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

#### 5.4.8 The Effect of Polymers on the Rheology and Filtrate Properties of C-Ben Activated with $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ Mixture

In this experiment C-Ben which is activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture ( $\text{Na}_2\text{CO}_3$  addition level; 0,29%  $\text{Na}_2\text{O}$  equivalent and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  addition level; 0,38%  $\text{Na}_2\text{O}$  equivalent) was developed by treating polymers to control the rheology and filtrate loss for water-based drilling fluids. The effects of polymers (HV CMC, PAC R, and xanthan gum) on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{CO}_3$  are given in Figures 5-131 through 5-142 (see also appendix Table B-34, B-35, and B-36).

With the HV CMC, PAC R, and xanthan gum treated muds, the increased polymer concentration resulted in a decrease in fluid loss and increase in apparent viscosity. But the acceptable value of 15 ml for filtrate volume was not obtained with increasing polymer concentrations (Figures 5-131 through 5-133). Figures 5-134 through 5-136 show that the increased polymer concentration resulted in an increase

in yield point and little or no changes were experienced in plastic viscosity. YP/PV values of treated bentonite were higher than the standard limit (Figures 5-137 through 5-139). Gel strengths are given in Figures 5-140, 5-141 and 5-142.

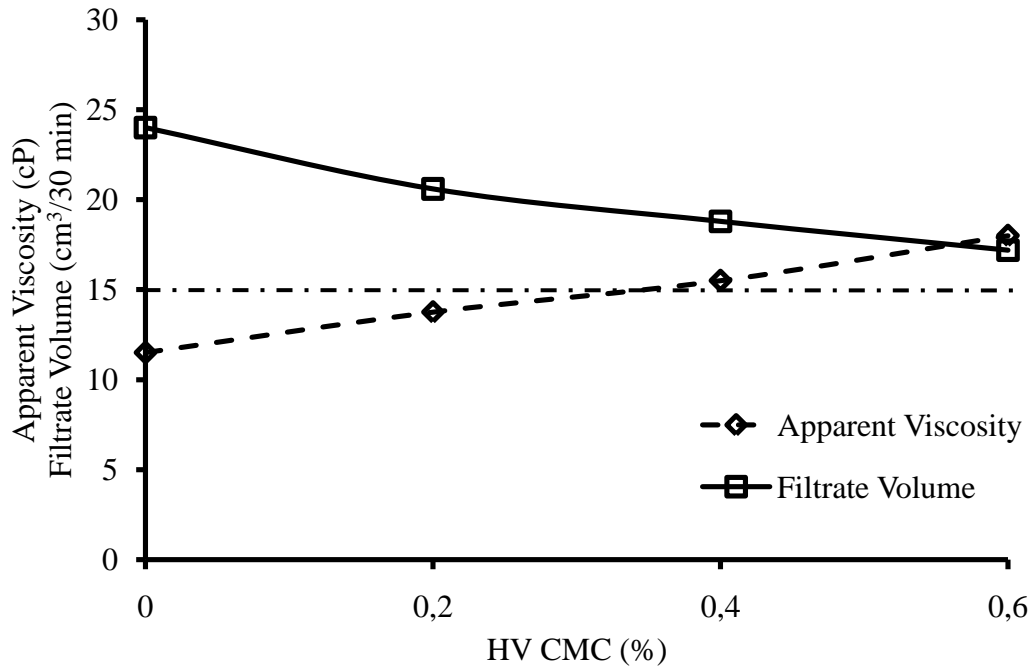


Figure 5-131 Effect of HV CMC on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture

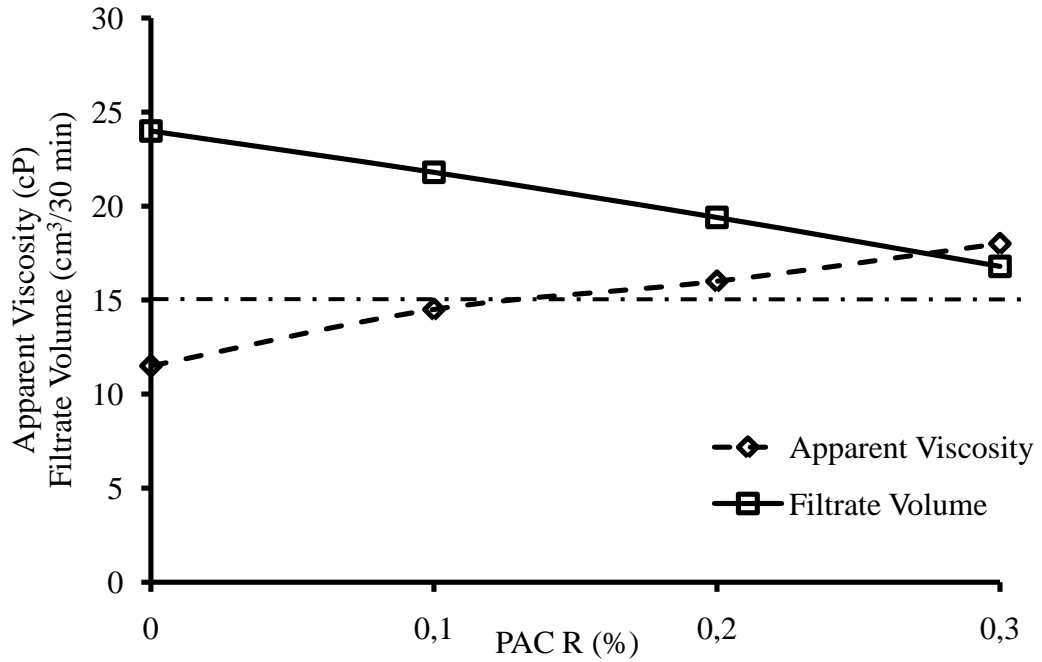


Figure 5-132 Effect of PAC R on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

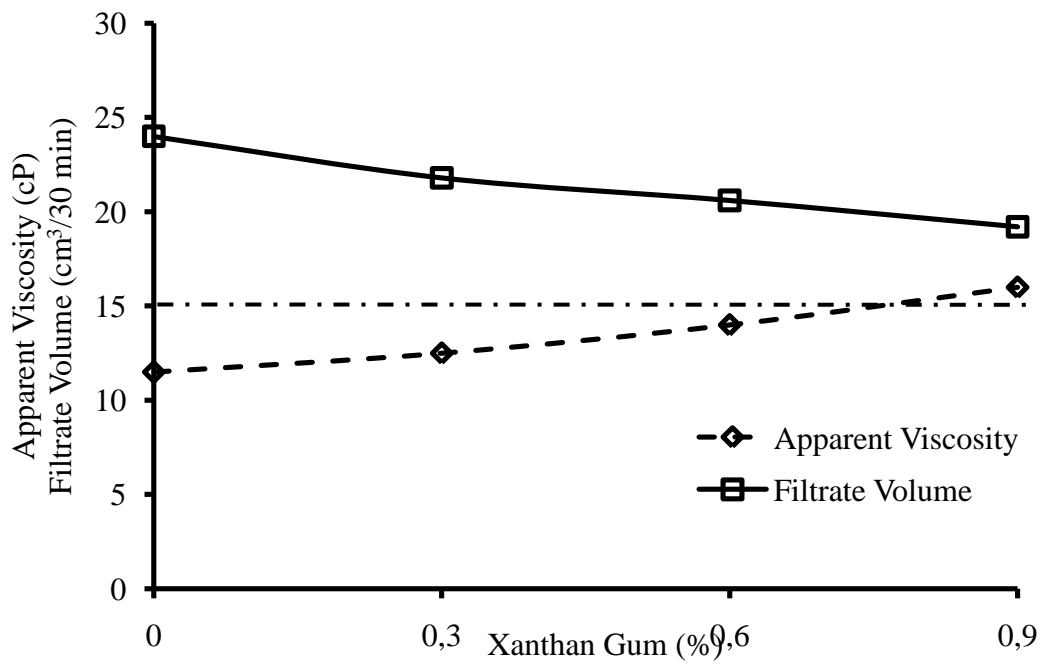


Figure 5-133 Effect of Xanthan Gum on the Apparent Viscosity and Filtrate Volume of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

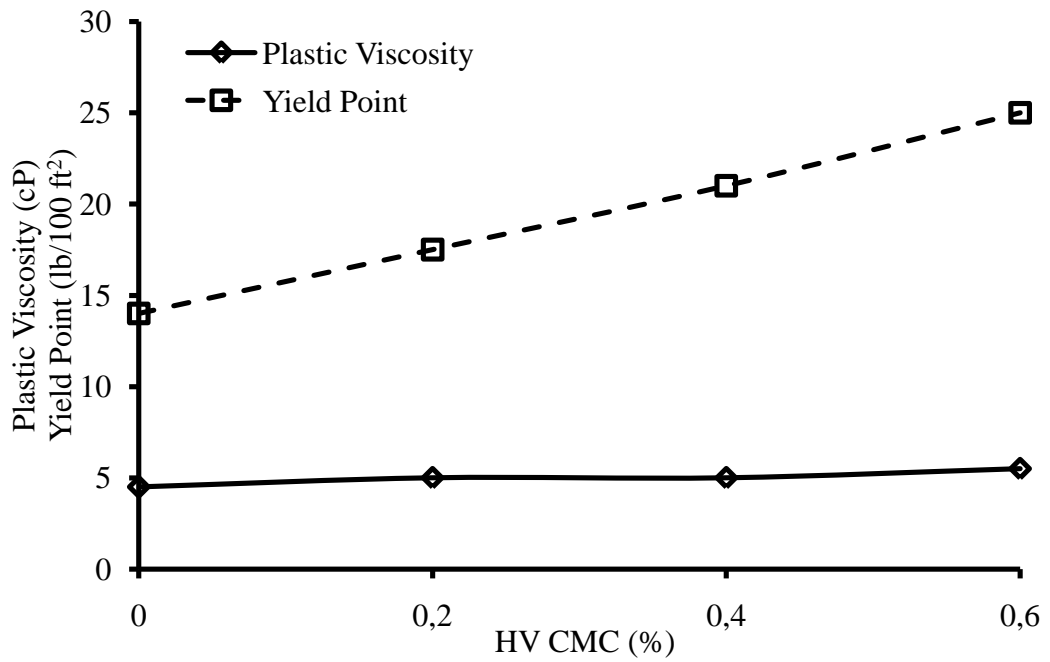


Figure 5-134 Effect of HV CMC on the Plastic Viscosity and Yield Point of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

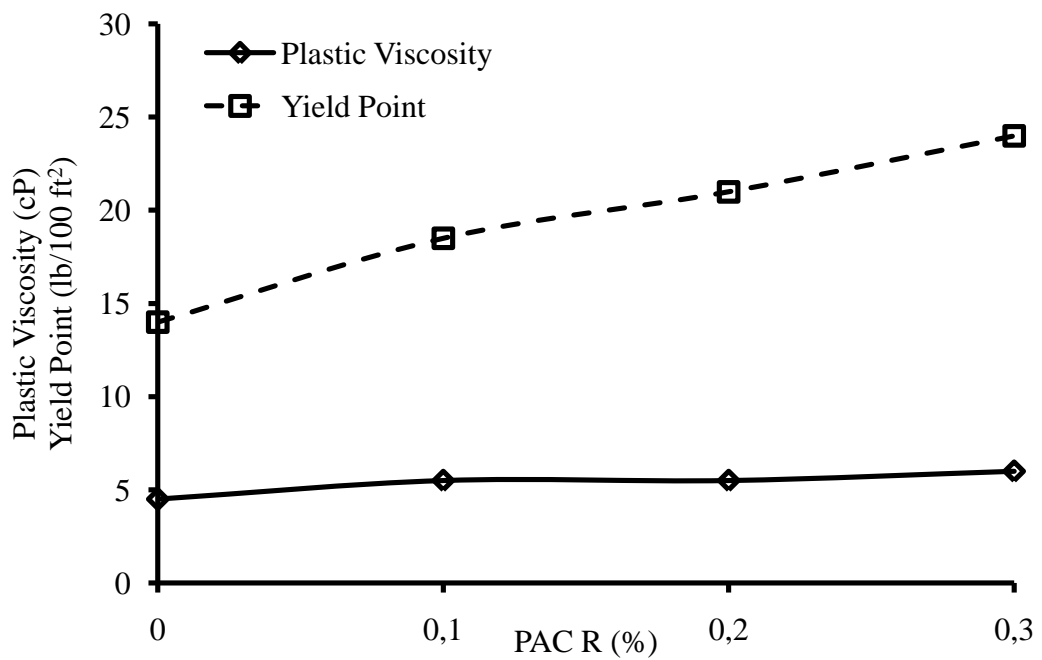


Figure 5-135 Effect of PAC R on the Plastic Viscosity and Yield Point of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

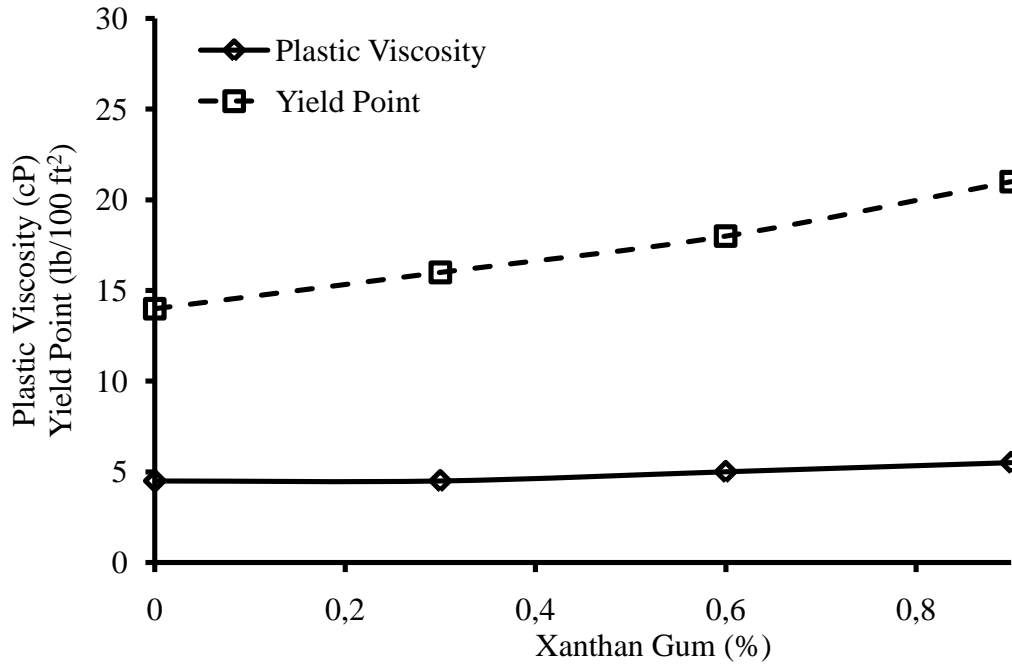


Figure 5-136 Effect of Xanthan Gum on the Plastic Viscosity and Yield Point of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

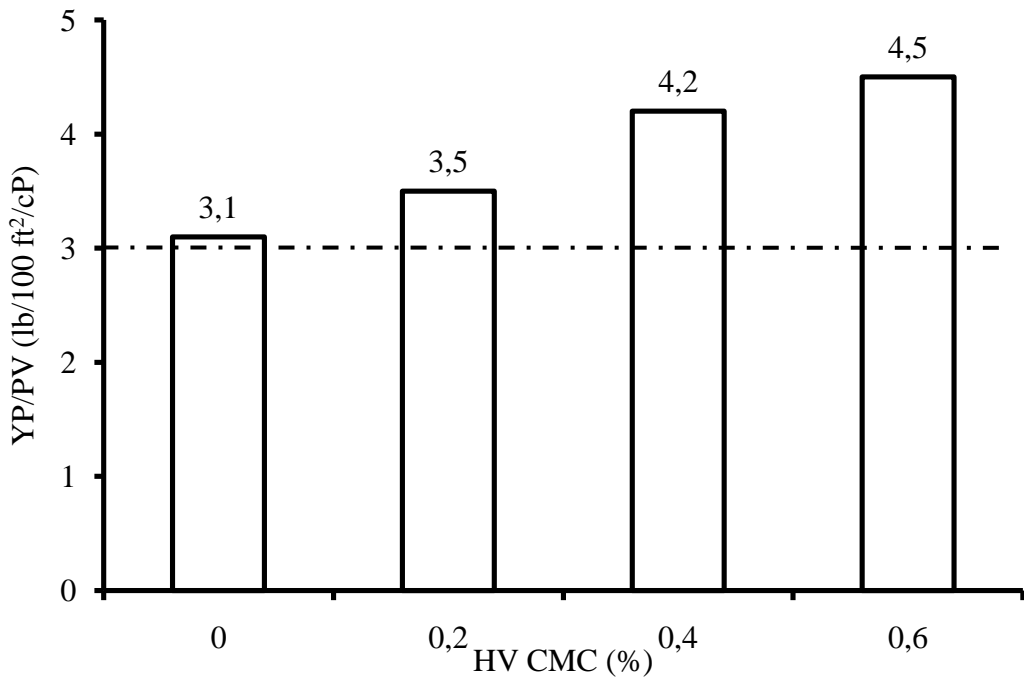


Figure 5-137 Effect of HV CMC on the YP/PV of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

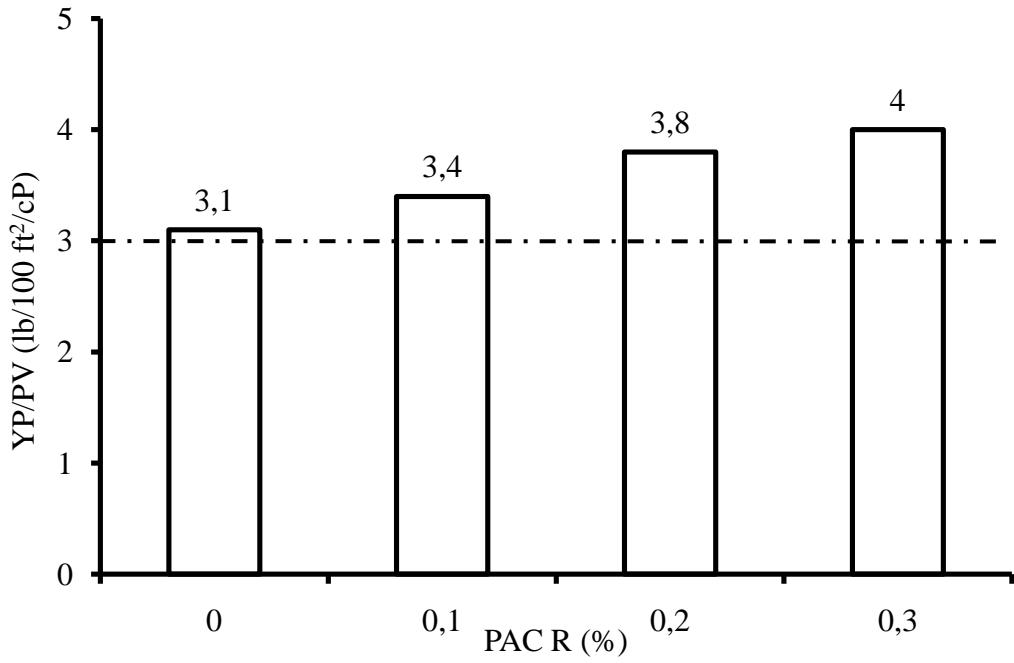


Figure 5-138 Effect of PAC R on the YP/PV of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

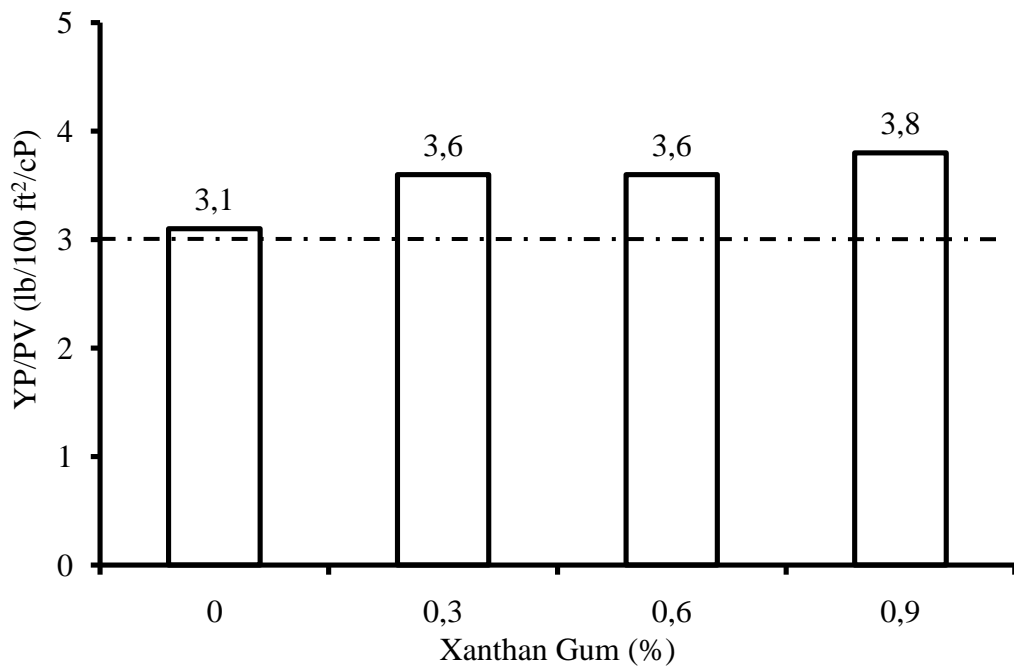


Figure 5-139 Effect of Xanthan Gum on the YP/PV of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture



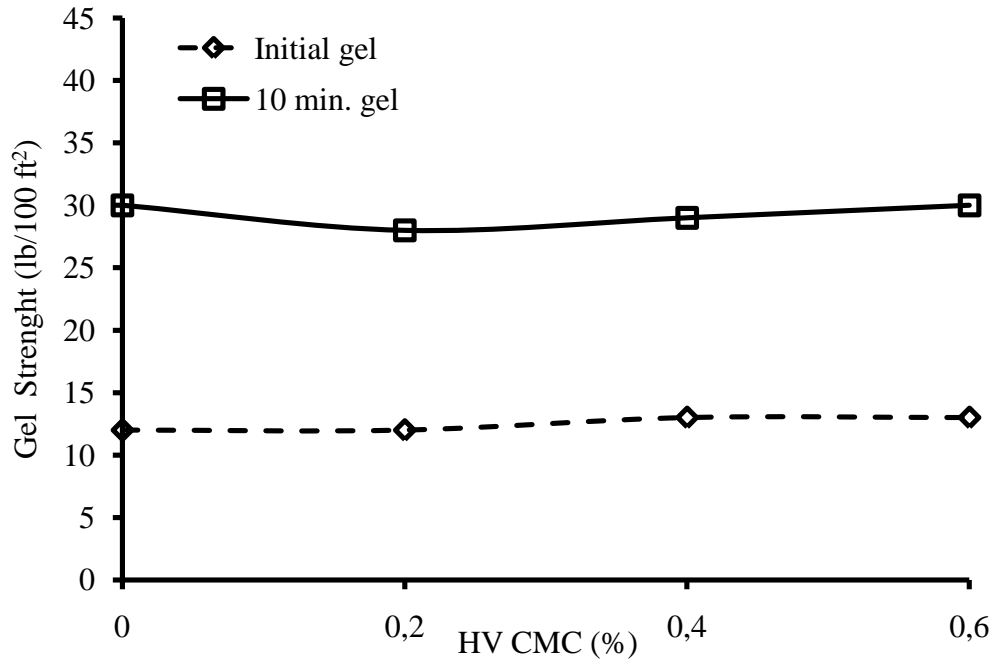


Figure 5-140 Effect of HV CMC on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

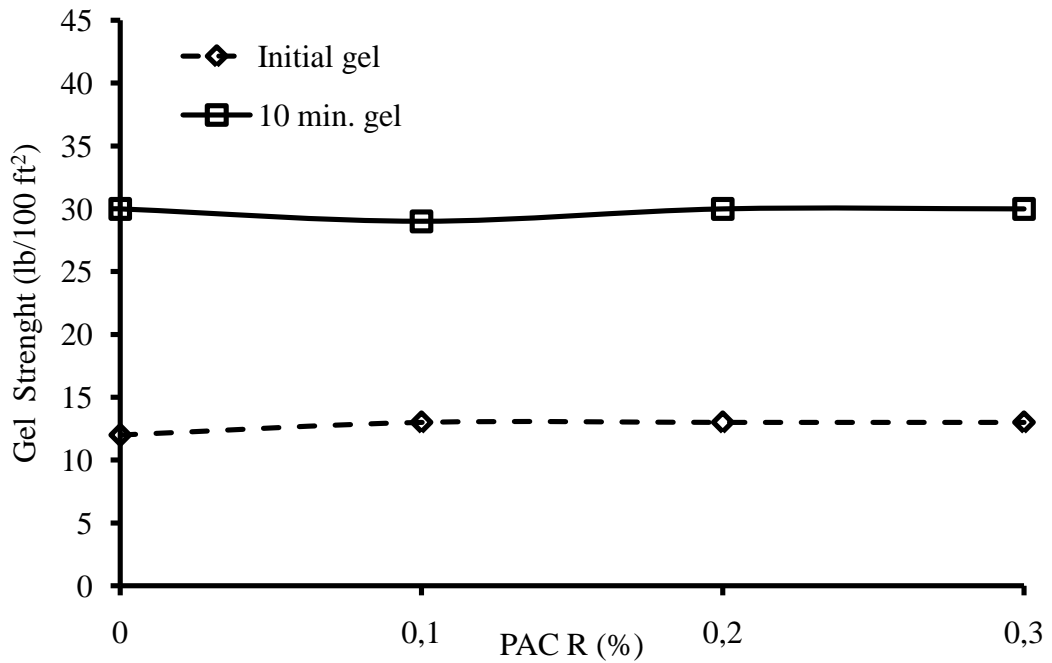


Figure 5-141 Effect of PAC R on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

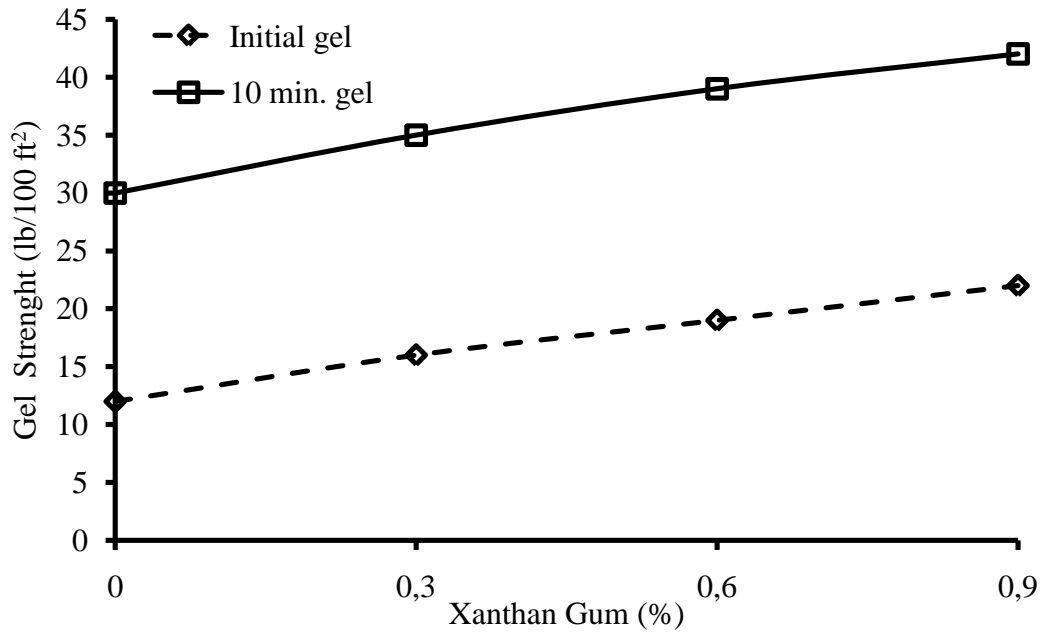


Figure 5-142 Effect of Xanthan Gum on the Gel Strength of C-Ben Activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

Because of the high filtrate volume of bentonite treated with polymers muds, the bentonite was treated with high viscosity and low viscosity polymers mixture. The concentrations of polymers mixture used in the treatment of bentonite are given in Table 5-3.

Table 5-3 The concentrations of polymers mixture used in the treatment of  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated C- Ben

Concentration (%)		Concentration (%)		Concentration (%)	
HV CMC	LV CMC	PAC R	PAC L	Xant. Gum	Dextrid
0,70	0,50	0,50	0,30	0,40	1,60
0,80	0,50	0,50	0,40	0,40	2,00
0,90	0,60	0,60	0,50	0,80	2,20
1,00	0,70	0,70	0,50	0,80	2,40

The effect of HV CMC and LV CMC on the rheology and filtrate properties of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture is given in Figure 5-143 (see also appendix Table B-37). As it can be seen in figure an acceptable value for apparent viscosity and for filtrate volume were obtained with 1,00% HV CMC and 0,70% LV CMC.

The effect of PAC R and PAC L used to control on the rheology and filtrate loss for the mud of C-Ben activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture is given in Figure 5-144 (see also appendix Table B-38). As it can be seen in figure the fluid loss reduced the API standard from high values by adding various combinations of polymers (0,60% PAC R and 0,50% PAC L).

The effect of xanthan gum and dextrim mixture on the rheology and filtrate properties of C-Ben activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture is given in Figure 5-145 (see also appendix Table B-39). It can be seen in figure the fluid loss reduced the API standard from high values by adding various combinations of xanthan gum and dextrim mixture. The physical properties of this type of mud are close to OCMA type bentonite requirements.

The rheological and filtrate properties of polymers used in the treatment of C-Ben activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture are given in Figure 5-146 (see also appendix Table B-40).

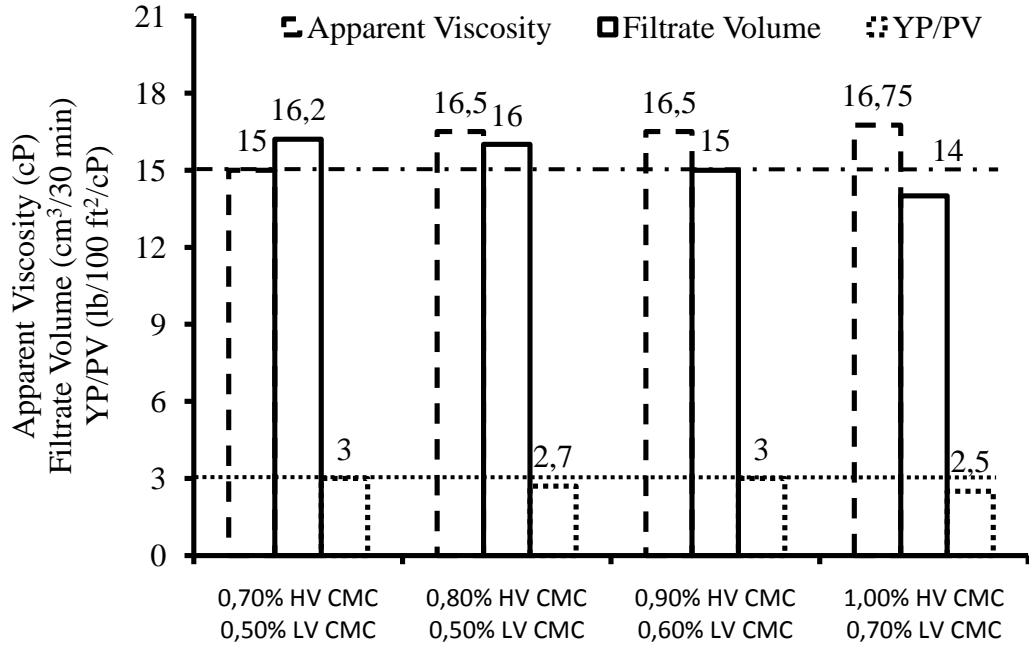


Figure 5-143 Effect of the Mixture of HV CMC and LV CMC on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

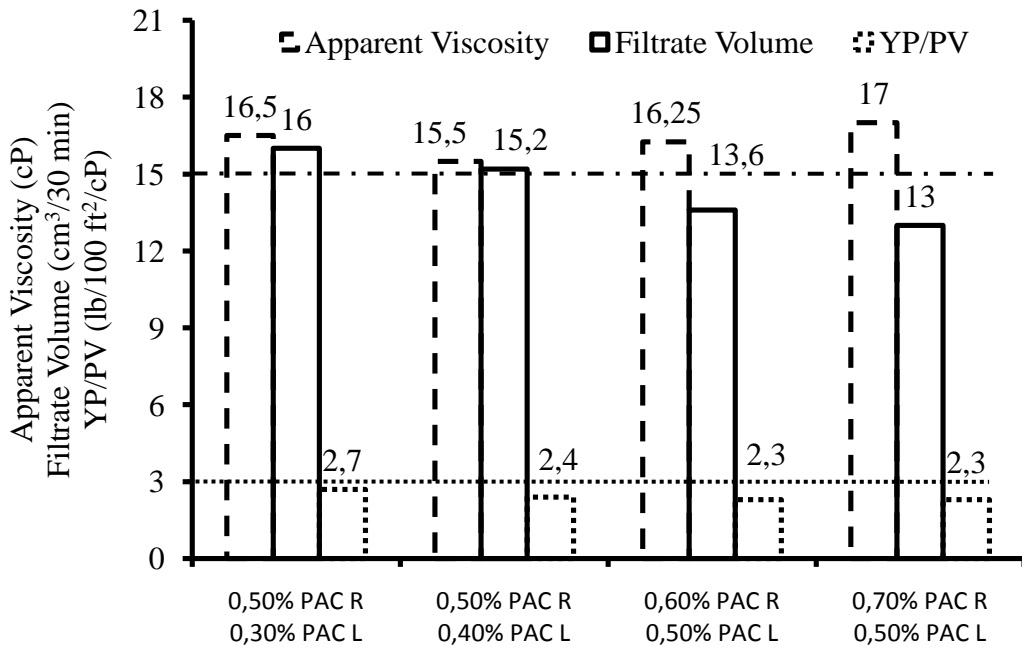


Figure 5-144 Effect of the Mixture of PAC R and PAC L on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

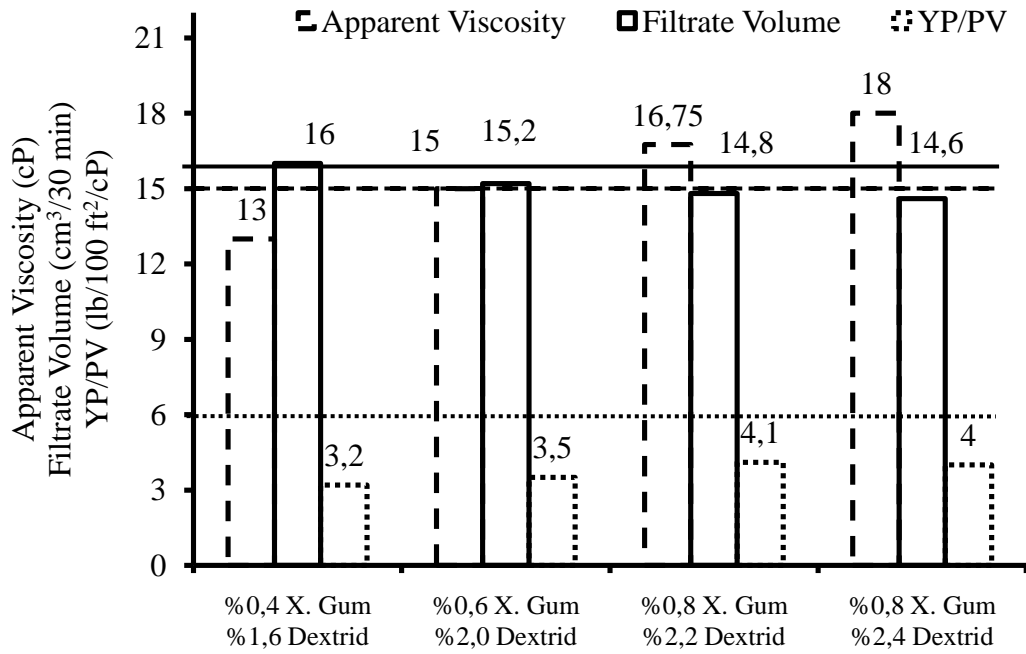


Figure 5-145 Effect of the Mixture of Xanthan Gum and Dextrid on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

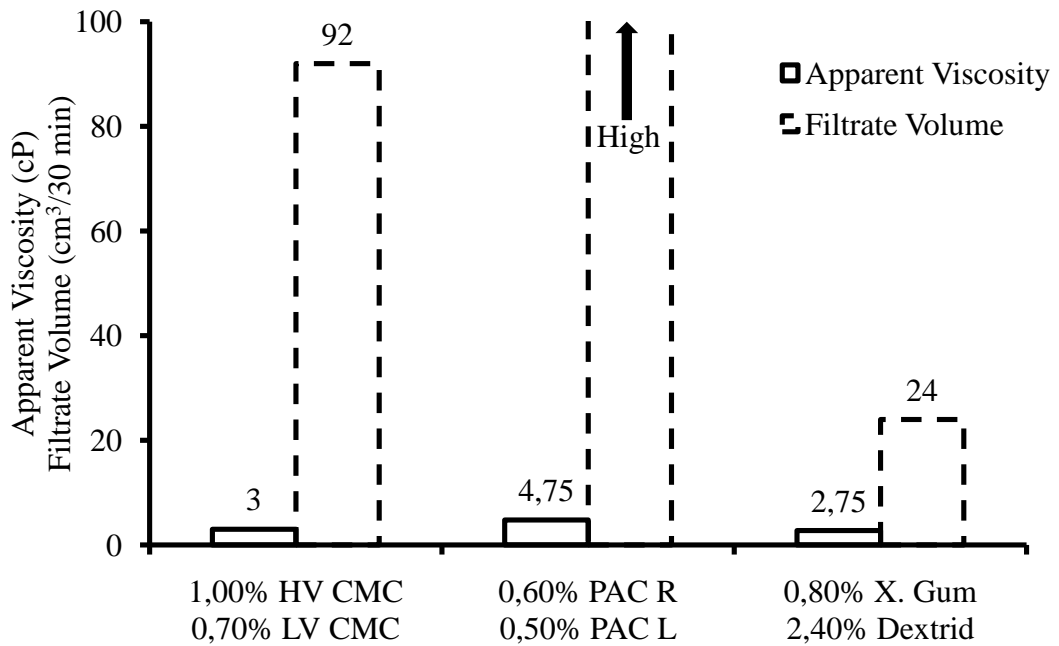


Figure 5-146 Apparent Viscosity and Filtrate Volume of Polymers Used in the Treatment of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

## **5.5 Aging of Bentonite Muds for a 21-day period at 36 °C**

Drilling fluid property changes which due to elevated temperature and aging time frequently cause problems in the drilling wells. One of the major problems in drilling wells with water-base drilling fluids has been the bacterial degradation of the chemical components. Fundamental changes in rheological properties and filtration characteristics of polymers occur with aging time.

This experiment included the aging time of 1 day, 4 days, 8 days, 15 days and 21 days in an orbital incubator at 36 °C and the effect of various chemicals (inorganic salts and polymers) used to control rheological properties and filtration characteristics was determined. Values of apparent and plastic viscosity, yield point, gel strengths (initial and 10 min.), filtrate volume and pH as a function of aging time are given in Appendix C.

### **5.5.1 Aging of Nontreated Bentonite**

API Standard Bentonite and Reşadiye Bentonite were aged to compare with the aging of E-Ben and C-Ben activated with the inorganic salts and treated with the polymers.

Based on the data in Figures 5-147 and 5-148 (see also appendix Table C-1 and C-2) it was possible to suggest that the aging of nontreated bentonite muds was quite stable at 36 °C for a 21-day period.

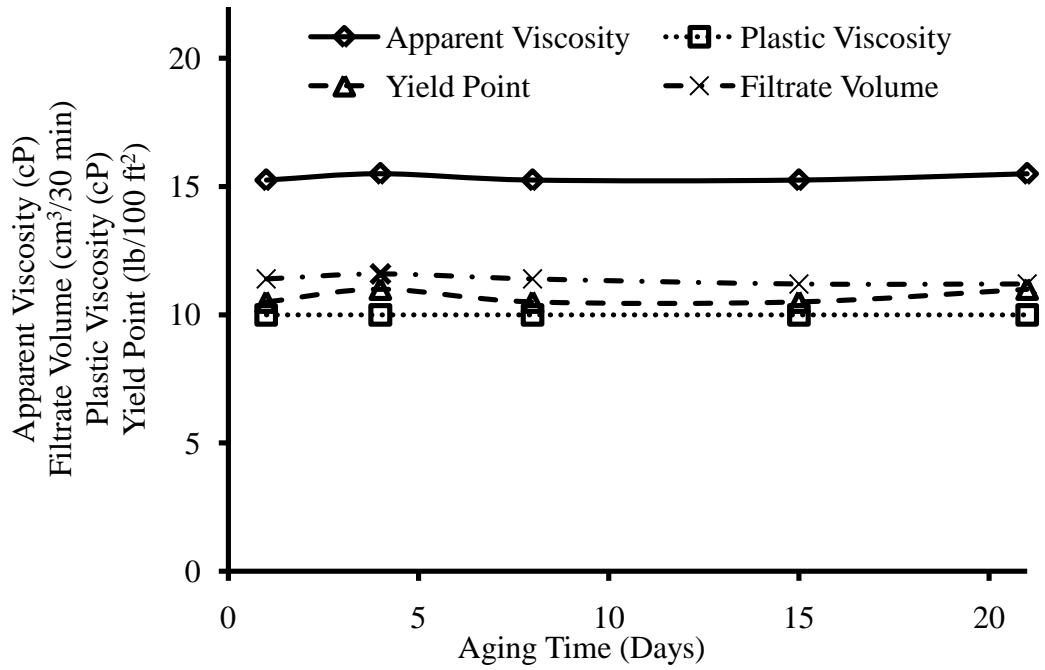


Figure 5-147 Aging of API Standard Bentonite for a 21-day period

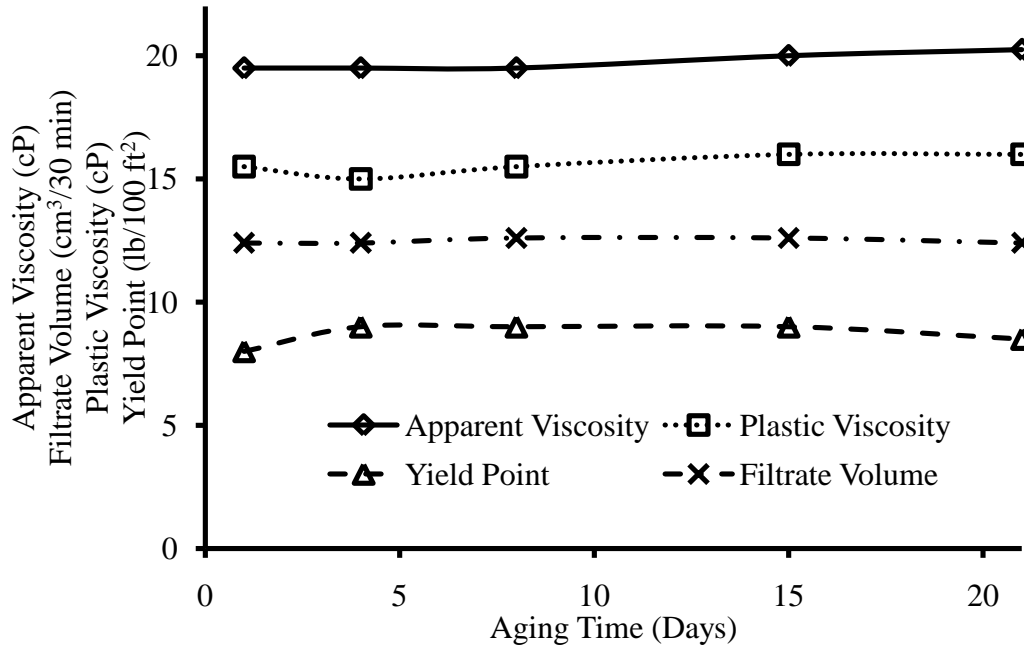


Figure 5-148 Aging of Reşadiye Bentonite for a 21-day period

### 5.5.2 Aging of Polymers

Data are presented in Figures 5-142 through 5-145 (see also appendix Tables C-3 through C-10) to illustrate the extent of degradation of the different amounts of CMC, PAC and xanthan gum used in the muds when aged for a 21-day period at 36 °C.

These results show the stability of the CMC (Figure 5-149) and PAC (Figure 5-150) used in the treatment of E-Ben. This is an expected result because CMC and PAC are resistant to bacterial attack.

Figure 5-151 and 5-152 show that xanthan gum used in the treatment of E-Ben and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated E-Ben was susceptible to bacterial attack. Bacterial action on xanthan gum caused an increase in the filtrate loss and a decrease in the rheological properties with the aging time. Another way to monitor xanthan gum degradation in a fluid was to monitor the pH; a significant drop in pH from 6,0 to 4,7 was a clear indication of polymer degradation.

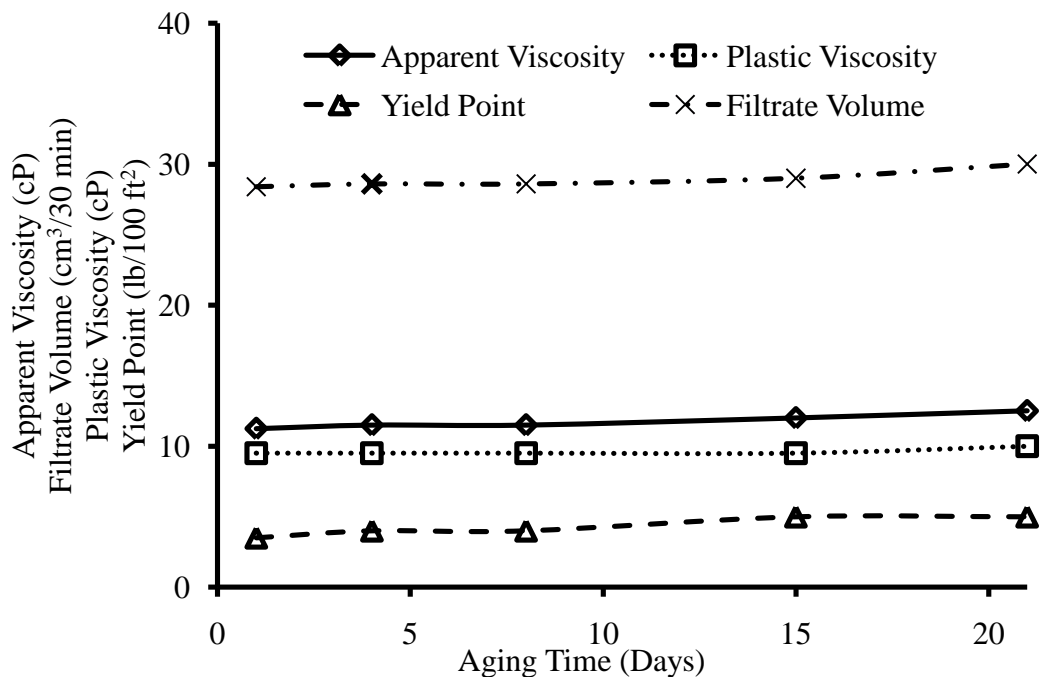


Figure 5-149 Aging of CMC Used in the Treatment of E-Ben for a 21-day period



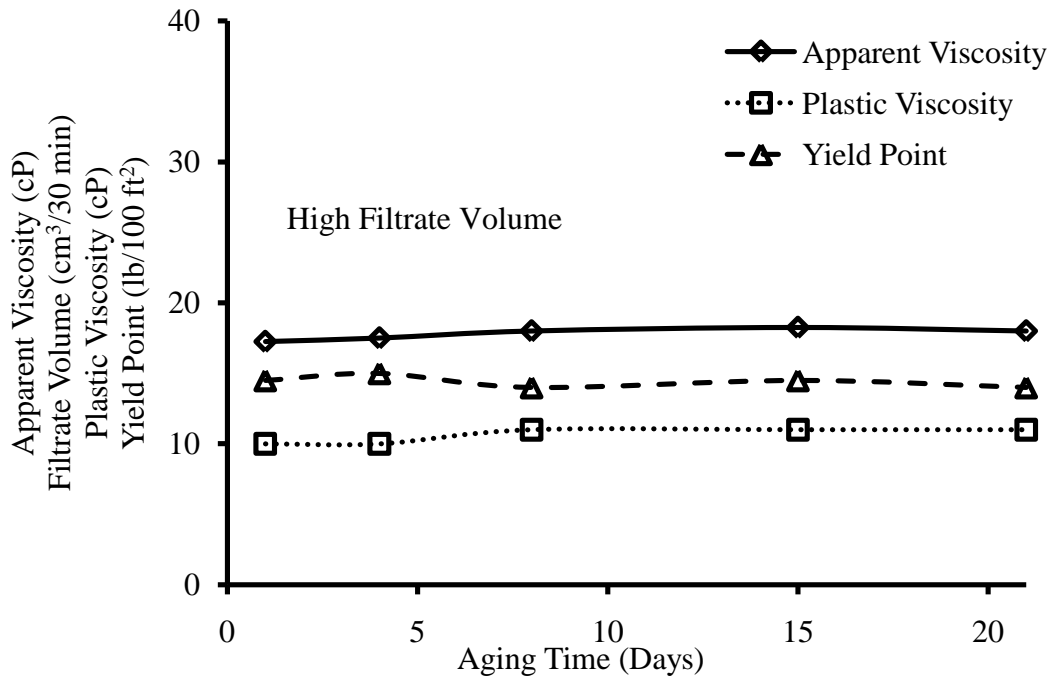


Figure 5-150 Aging of PAC Used in the Treatment of E-Ben for a 21-day period

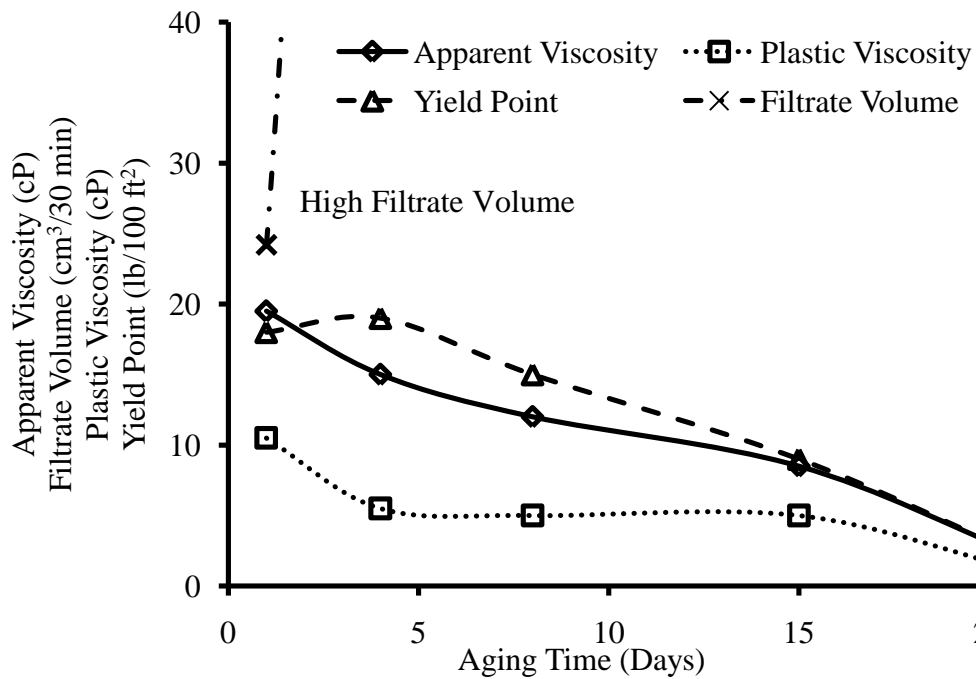


Figure 5-151 Aging of Xanthan Gum Used in the Treatment E-Ben for a 21-day period

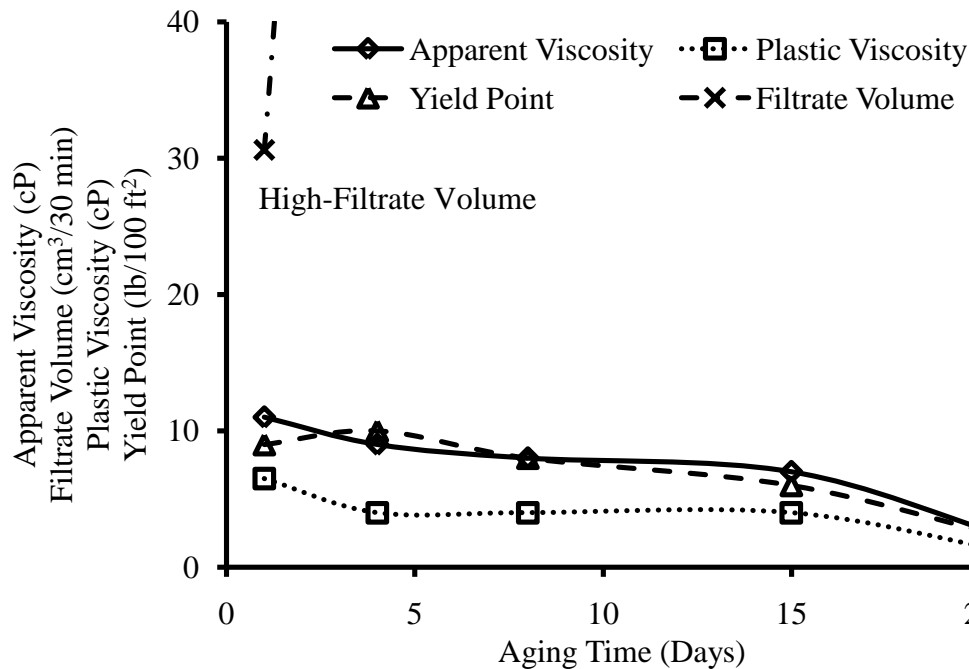


Figure 5-152 Aging of Xanthan Gum Used in the Treatment of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Activated E-Ben for a 21-day period

### 5.5.3 Aging of Unactivated Bentonites and Polymer Treated Muds

Optimum concentrations of bentonites treated with polymer muds were aged to monitor bacterial degradation. These are;

- E-Ben treated with 7,5% HV CMC mud,
- C-Ben treated with 3% HV CMC mud,
- E-Ben treated with 3,75% PAC R mud,
- C-Ben treated with 2% PAC R mud,
- E-Ben treated with 7,5% xanthan gum mud,
- C-Ben treated with 2,5% xanthan gum mud.

In Figures 5-153 through 5-158 (see also appendix Table C-11 through C-16) are listed the rheological properties of the muds aged for a 21-day period at 36 °C.

In the muds containing E-Ben or C-Ben treated with polymers (CMC and PAC), the rheological and filtrate properties were not changed very much with the aging time (Figures 5-153, 5-154, 5-155 and 5-156).

Xanthan gum deterioration in a mud was monitored as a increase in filtrate volume from 13,6 to 17,6 for E-Ben mud (Figure 5-157) and from 15 to 29,2 for C-Ben mud (Figure 5-158). Furthermore, the rheological properties of the sample treated with xanthan gum decreased. Besides, a colour change from yellow to black was seen in C-Ben mud. As a result, these figures show that xanthan gum was attacked by bacteria, reducing its ability to control viscosity and water loss.

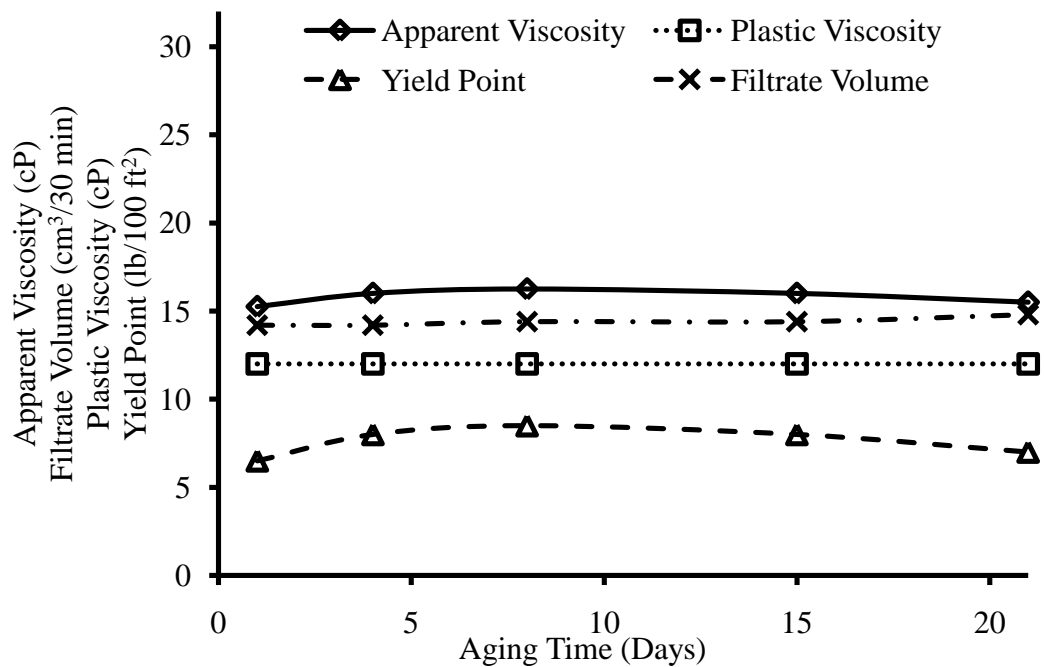


Figure 5-153 Aging of E-Ben & HV CMC Mud for a 21-day period

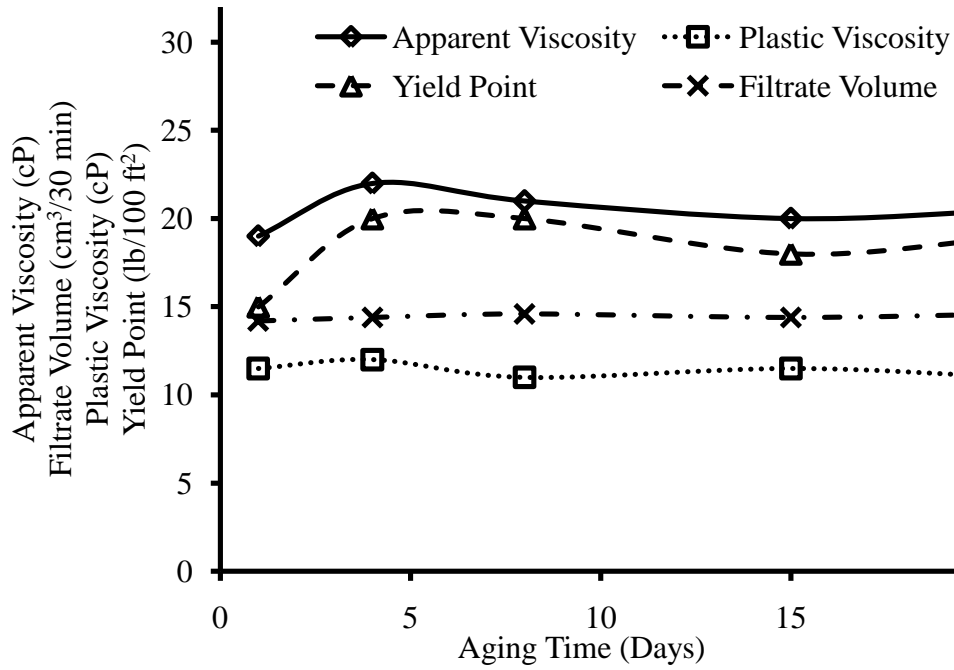


Figure 5-154 Aging of C-Ben & HV CMC Mud for a 21-day period

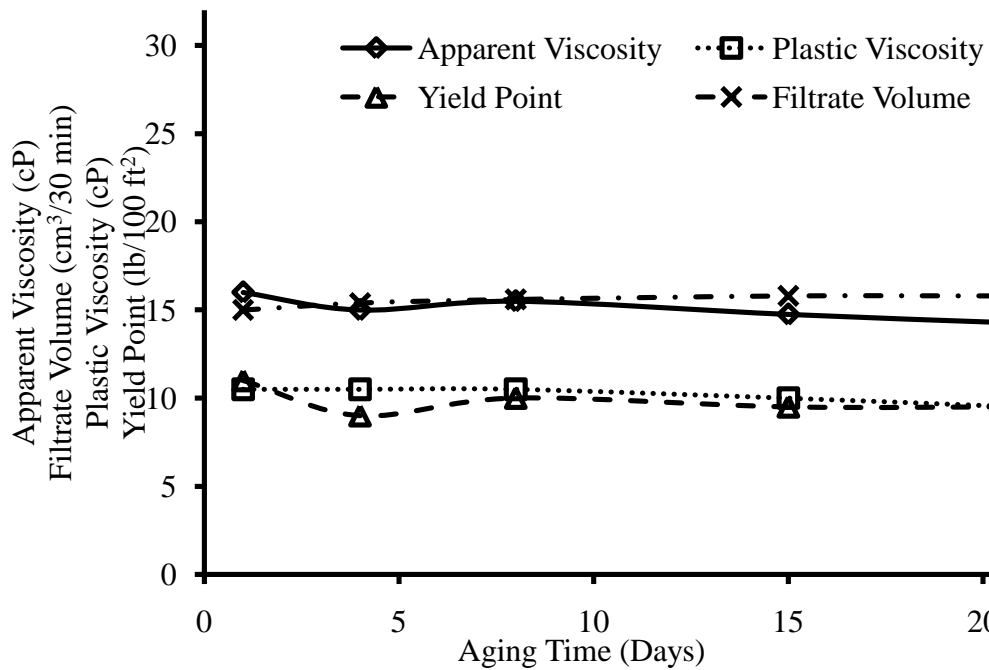


Figure 5-155 Aging of E-Ben & PAC R Mud for a 21-day period

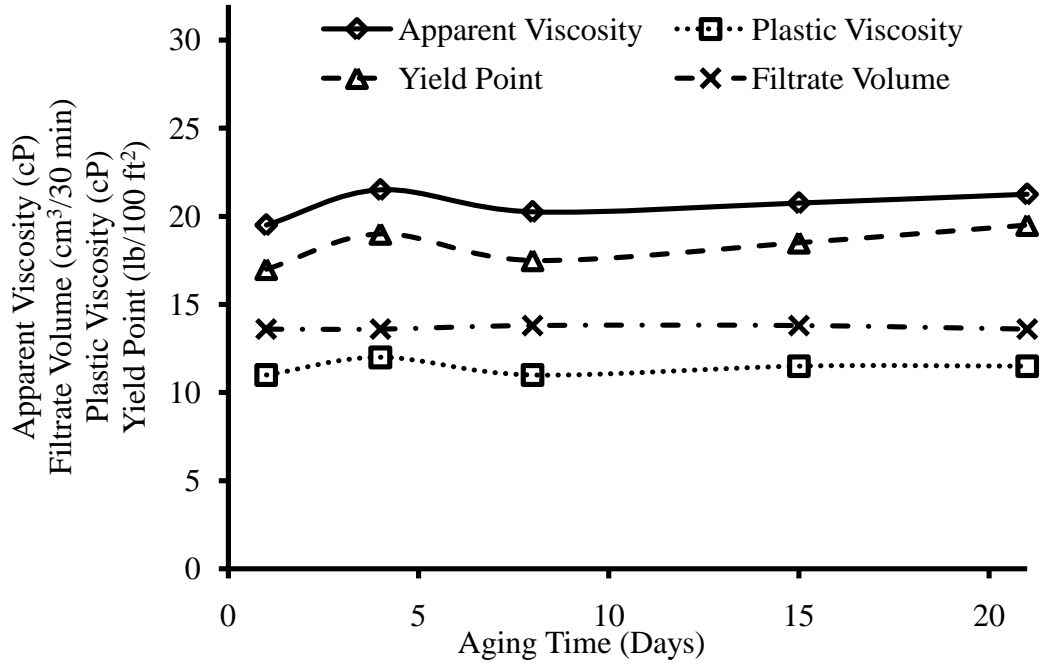


Figure 5-156 Aging of C-Ben & PAC R Mud for a 21-day period

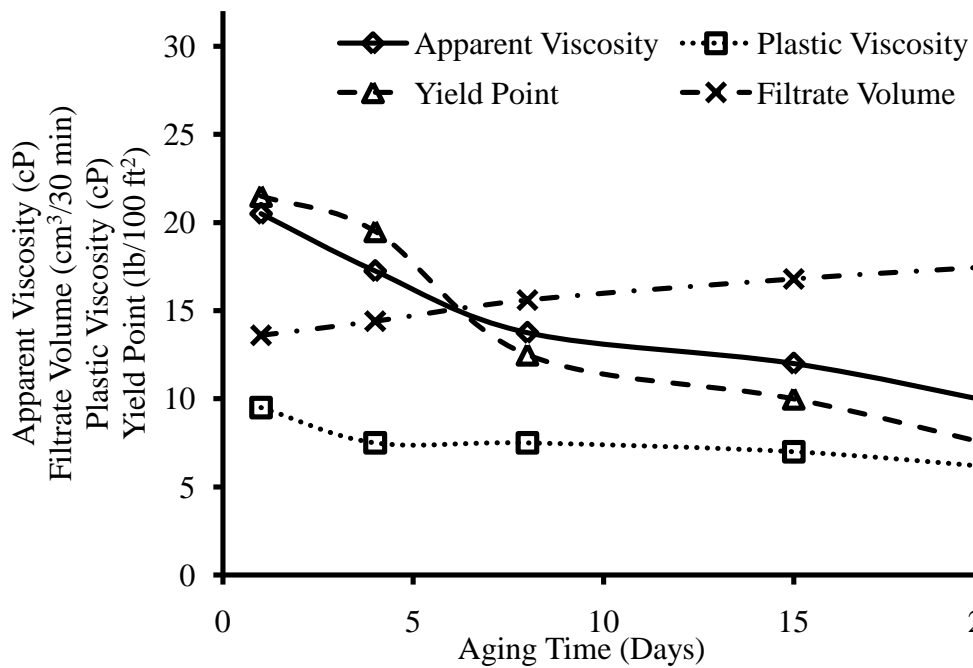


Figure 5-157 Aging of E-Ben & Xanthan Gum Mud for a 21-day period

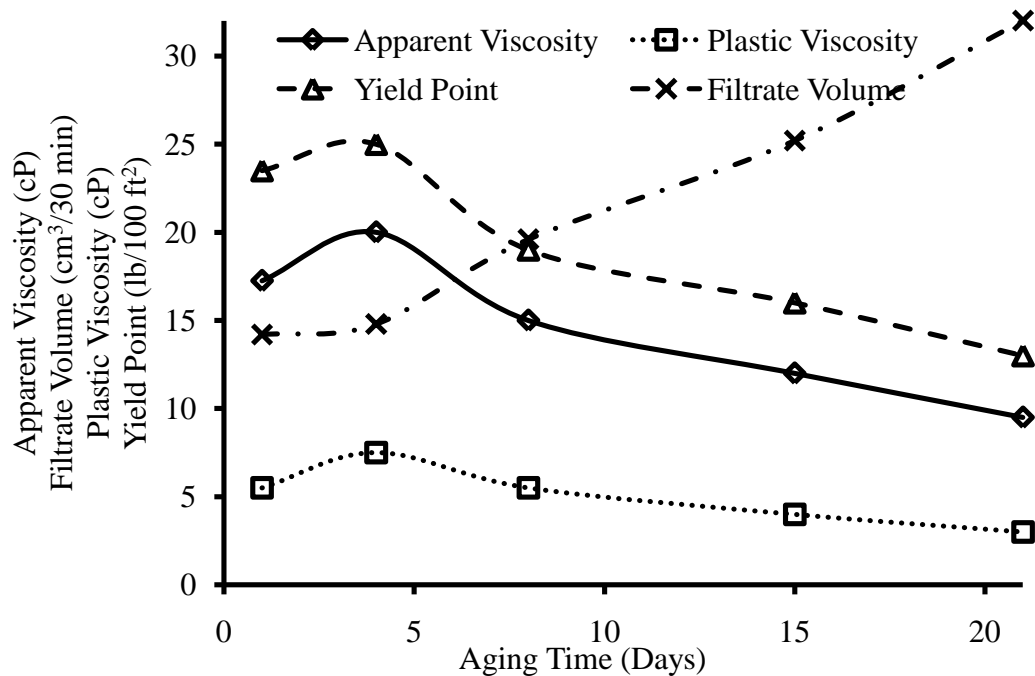


Figure 5-158 Aging of C-Ben & Xanthan Gum Mud for a 21-day period

#### 5.5.4 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated Bentonite and Polymers Treated Muds

Optimum concentrations of Na<sub>2</sub>CO<sub>3</sub> (Na<sub>2</sub>CO<sub>3</sub> addition level; 2,90% Na<sub>2</sub>O equivalent for E-Ben and 0,58% Na<sub>2</sub>O equivalent for C-Ben) activated bentonites treated with polymers muds were aged to monitor bacterial degradation. These are;

- E-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with 1,2% HV CMC,
- C-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with 0,8% HV CMC and 0,4% LV CMC mixture,
- E-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with 1,0% PAC R,
- C-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with 0,50% PAC R and 0,30% PAC L mixture,
- E-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with 3,0% xanthan gum,
- C-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with 0,80% xanthan gum and 2,4% dextrid mixture.

The prepared samples were aged in an incubator for a 21-day period at 36 °C. The results of rheology measurements of Na<sub>2</sub>CO<sub>3</sub> activated E-Ben and C-Ben treated

with polymer muds are shown in Figures 5-159 through 5-164 (see also appendix Tables C-17 through C-22).

As it is seen in Figures 5-159, 5-160, 5-161 and 5-162 both E-Ben and C-Ben activated with  $\text{Na}_2\text{CO}_3$  and treated with CMC and PAC muds showed a slight viscosity increase with the aging time. Besides, the aging effect was diminished with the increase in aging time.

$\text{Na}_2\text{CO}_3$  activated E-Ben and xanthan gum treated mud showed an increase at apparent viscosity and yield point with aging time (Figure 5-163). But the aging effect was diminished with the increase in aging time after 8 days.

$\text{Na}_2\text{CO}_3$  activated C-Ben and xanthan gum treated mud underwent a radical change in the physical and chemical properties with the aging time at 36 °C (Figure 5-164). In contrast to raw bentonite treated with xanthan gum mud, bacterial activity in  $\text{Na}_2\text{CO}_3$  activated bentonite and xanthan gum treated mud caused an increase in the plastic viscosity and yield point in 4 days. Besides, a decrease in plastic viscosity and a colour change in the mud were observed. These all served to unbalance the mud which caused increased filtration loss.

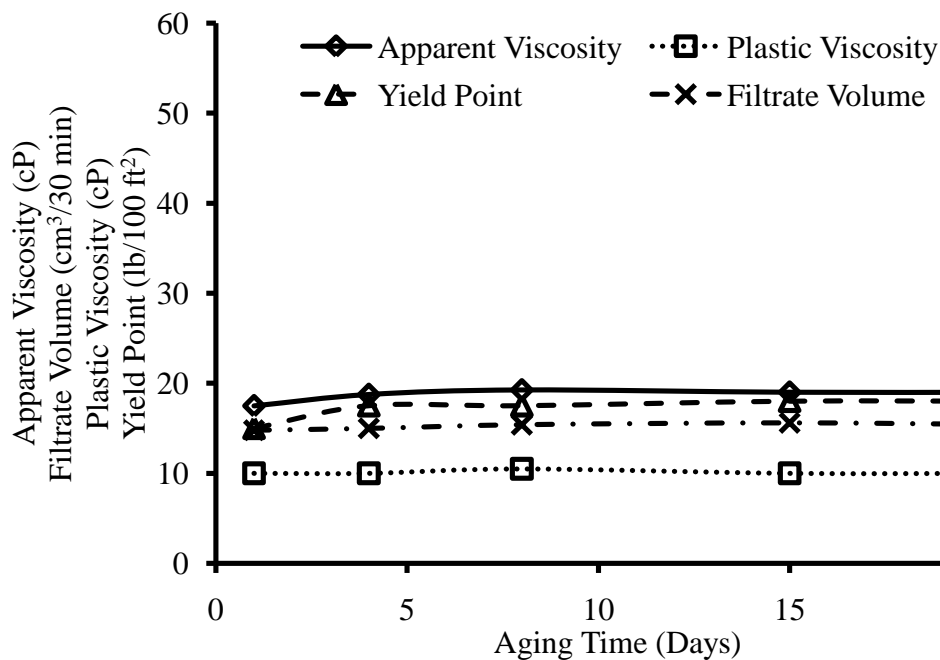


Figure 5-159 Aging of E-Ben Activated with  $\text{Na}_2\text{CO}_3$  & CMC Mud for a 21-day period

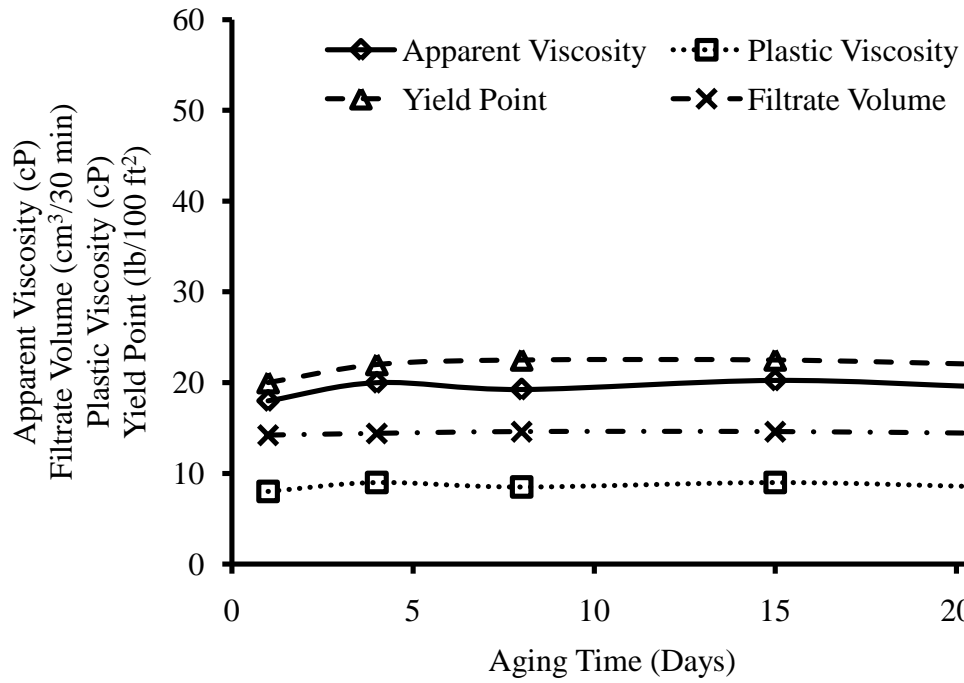


Figure 5-160 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & HV CMC and LV CMC Mud for a 21-day period

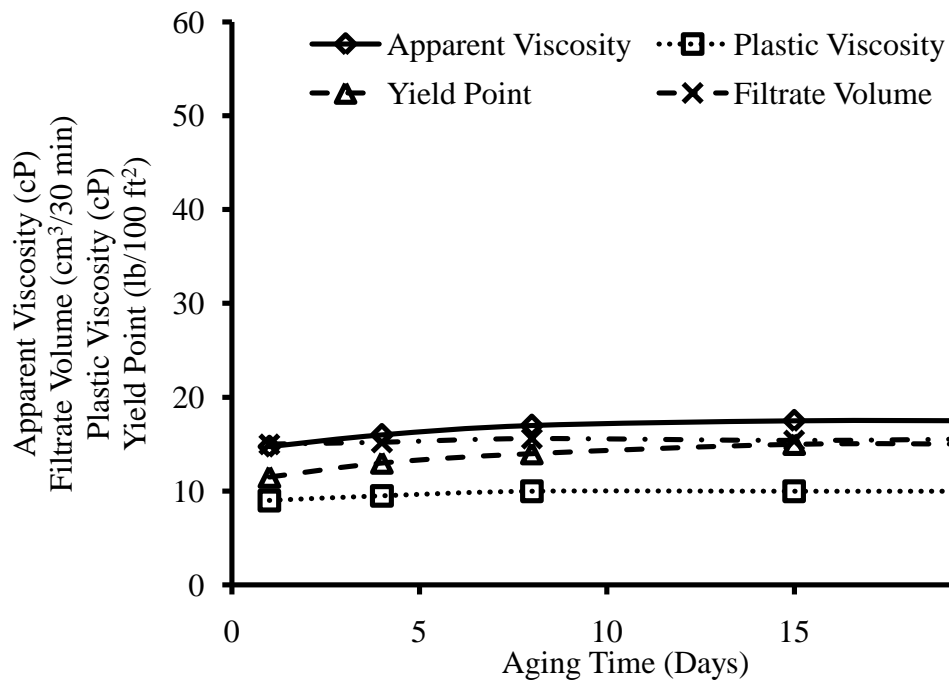


Figure 5-161 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & PAC R Mud for a 21-day period



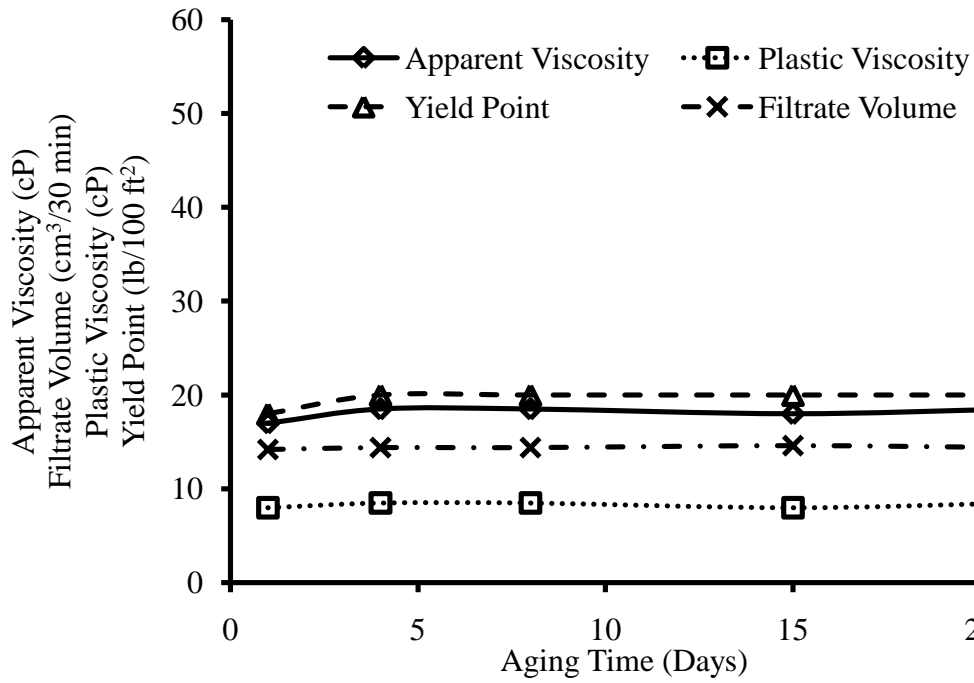


Figure 5-162 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & PAC R and PAC L Mud for a 21-day period

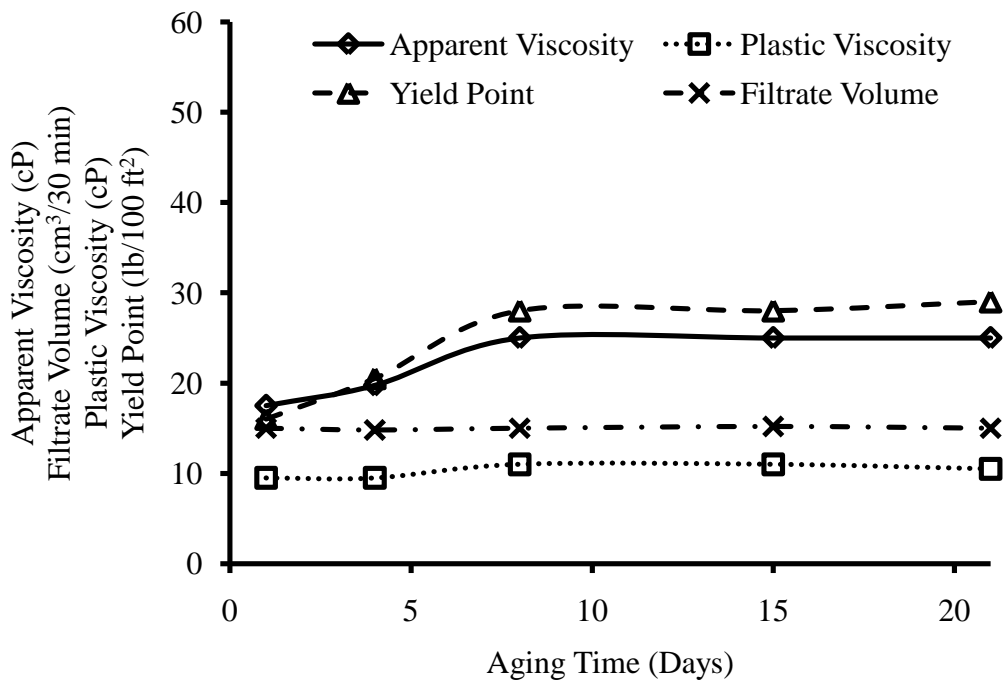


Figure 5-163 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & Xanthan Gum Mud for a 21-day period

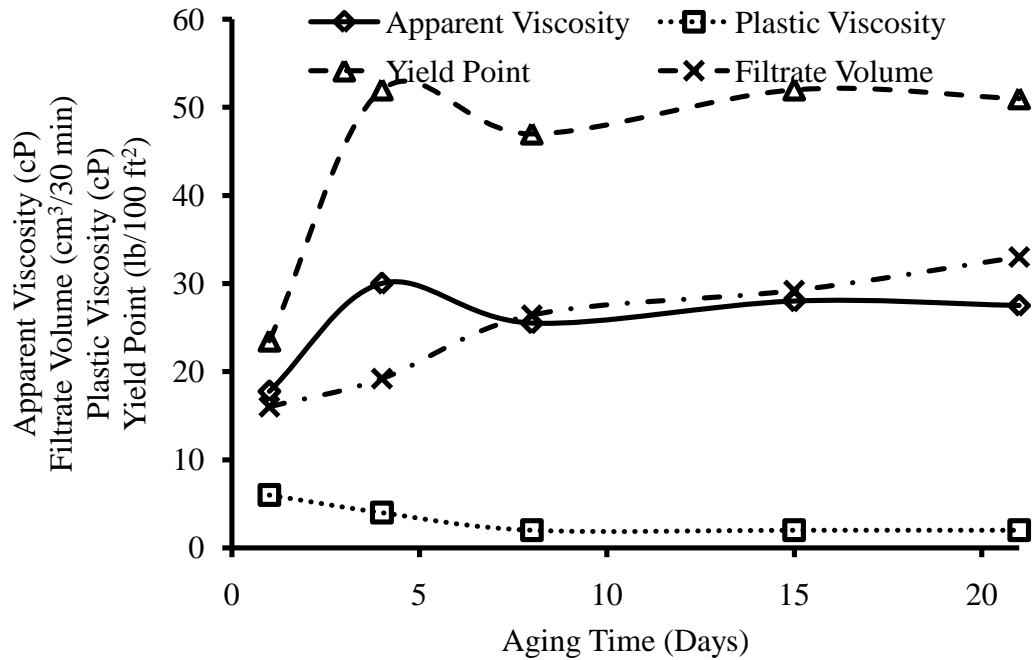


Figure 5-164 Aging of C-Ben Activated with  $\text{Na}_2\text{CO}_3$  & Xanthan Gum and Dextrid Mud for a 21-day period

### 5.5.5 Aging of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ Activated Bentonite and Polymers Treated Muds

Optimum concentration of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  addition level; 3,78%  $\text{Na}_2\text{O}$  equivalent for E-Ben and 0,76%  $\text{Na}_2\text{O}$  equivalent for C-Ben) activated bentonite and treated with polymers muds were determined for aging. These are;

- E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and treated with 2,8% HV CMC,
- C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and treated with 0,8% HV CMC and 0,4% LV CMC mixture,
- E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and treated with 1,8% PAC R,
- C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and treated with 0,50% PAC R and 0,30% PAC L mixture,
- E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and treated with 4,5% xanthan gum,
- C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and treated with 0,80% xanthan Gum and 2,4% dextrid mixture.

Data are presented in Figures 5-165 through 5-170 (see also appendix Tables C-23 through C-28) to illustrate the extent of degradation of E-Ben and C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  treated with polymers when aged for 21-day period at 36 °C in an incubator.

In contrast to  $\text{Na}_2\text{CO}_3$  activated bentonite mud, E-Ben and C-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  and treated with CMC and PAC muds showed a slight viscosity decrease with the aging time (Figure 5-165 through 5-168). With the increase in aging time the aging effect diminished.

Besides, E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  and treated with xanthan gum muds showed a stable viscosity with the aging time, i.e.,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  activated E-Ben treated with xanthan gum mud was not susceptible to bacterial attack (Figure 5-169). This finding shows that  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  can be used for the prevention of bacterial degradation of E-Ben.

While  $\text{Na}_2\text{CO}_3$  activated C-Ben and xanthan gum (Figure 5-164) treated mud led to erratic, unpredictable bentonite performance in 4 days, the rheological and filtrate properties of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  activated C-Ben and xanthan gum treated mud changed after 4 days (Figure 5-170).

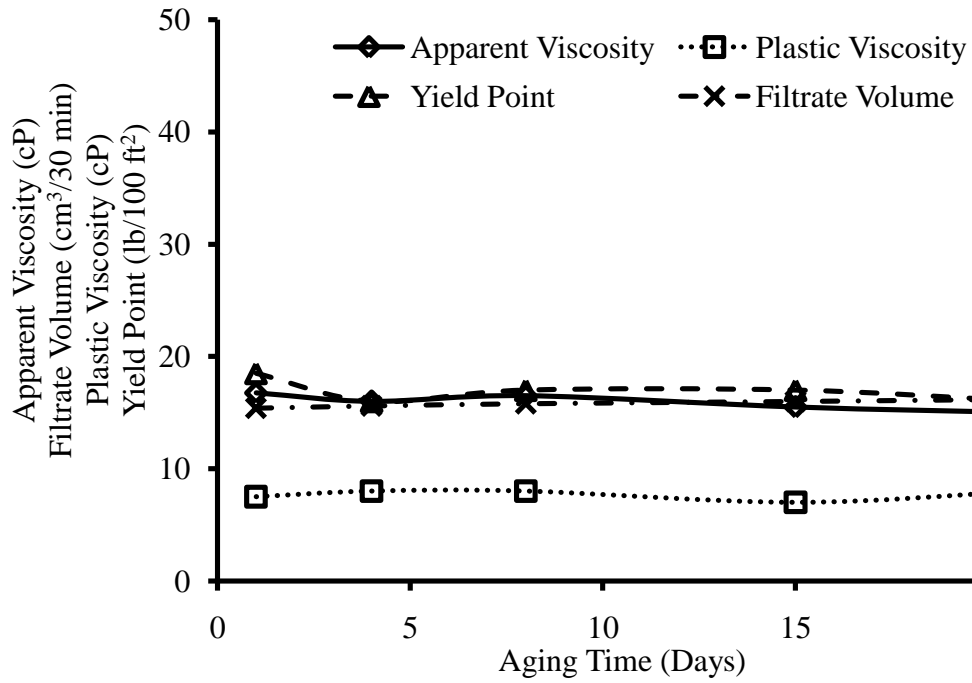


Figure 5-165 Aging of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  & CMC Mud for a 21-day period

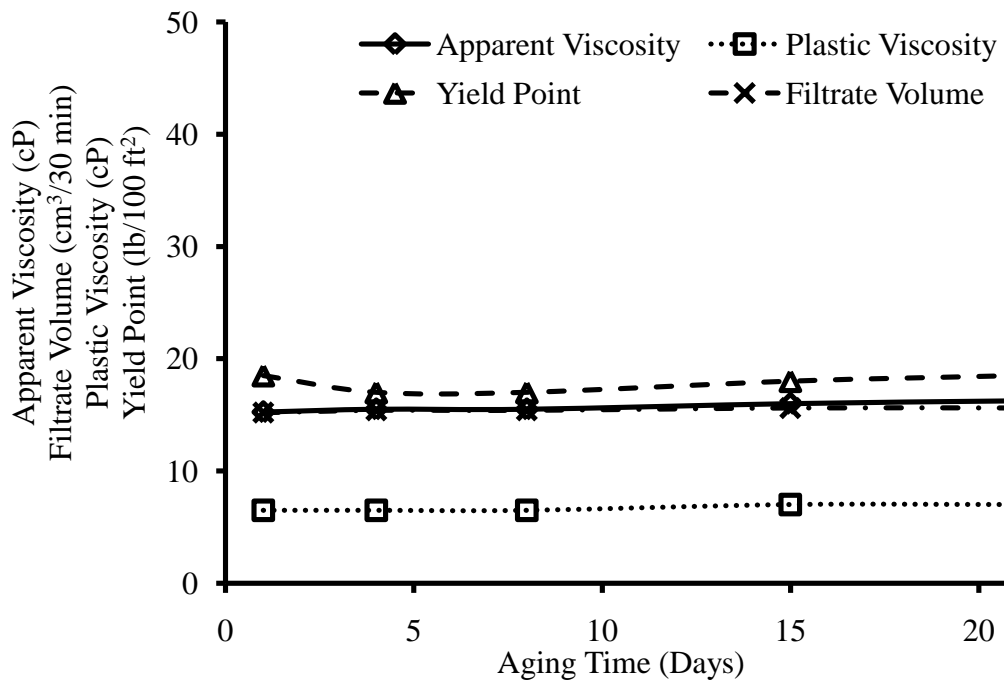


Figure 5-166 Aging of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  & HV CMC and LV CMC Mud for a 21-day period

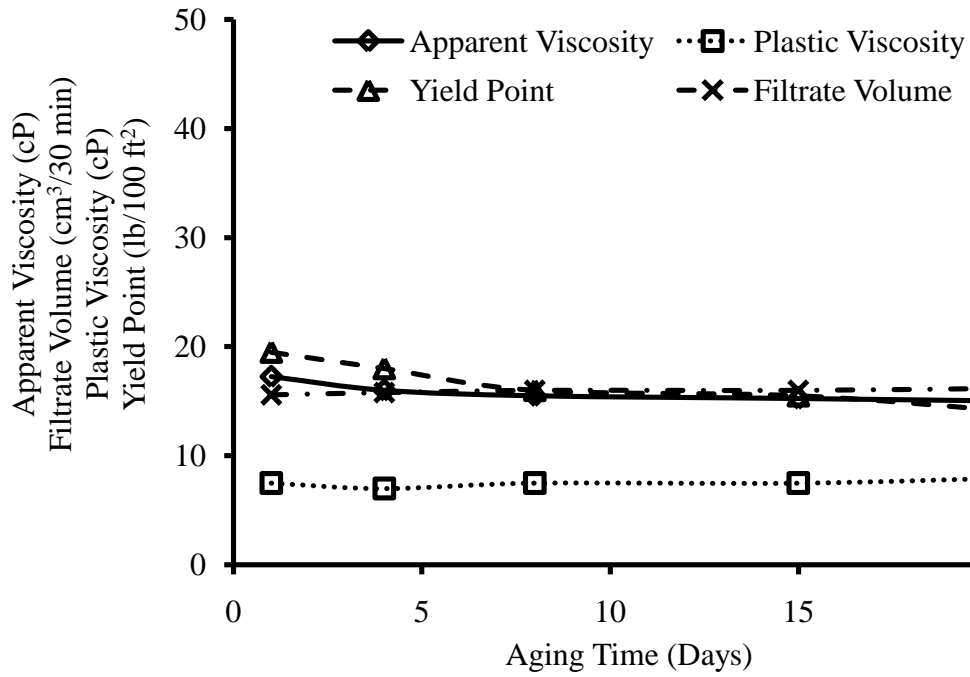


Figure 5-167 Aging of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  & PAC R Mud for a 21-day period

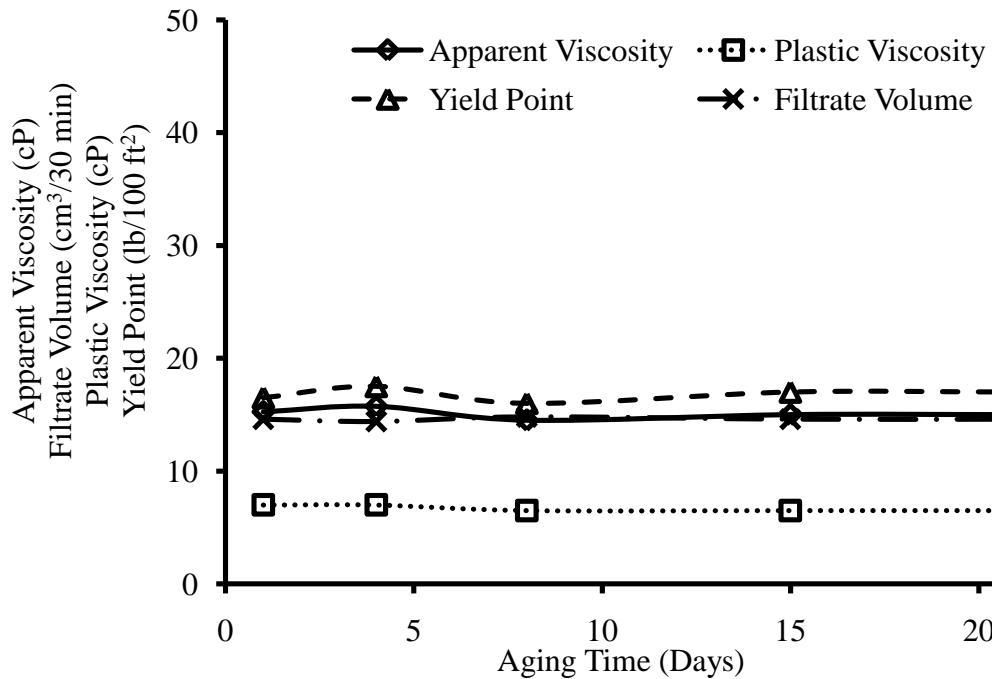


Figure 5-168 Aging of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  & PAC R and PAC L Mud for a 21-day period

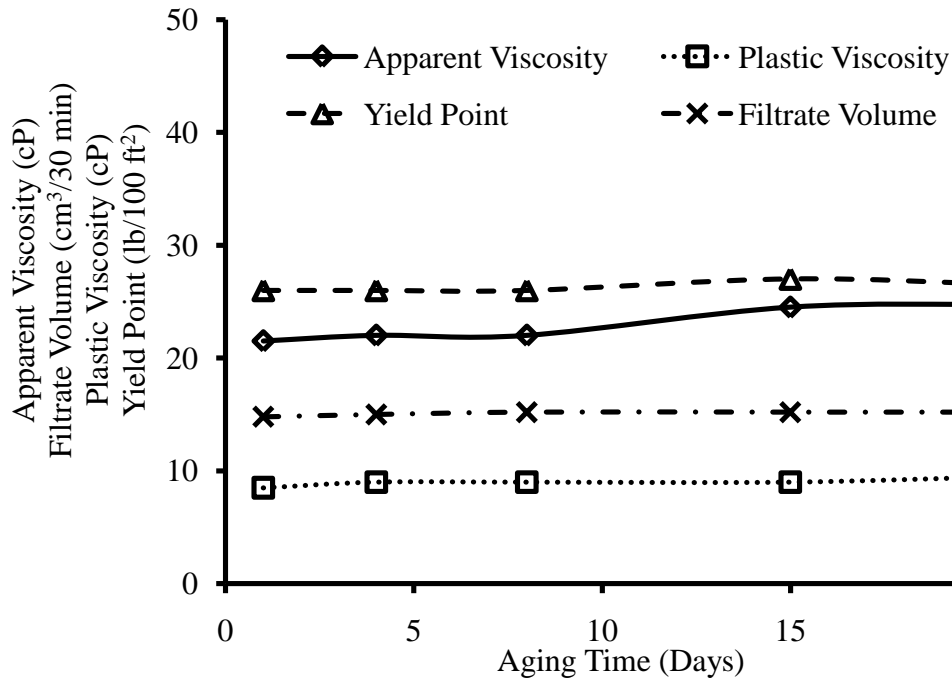


Figure 5-169 Aging of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  & Xanthan Gum Mud for a 21-day period

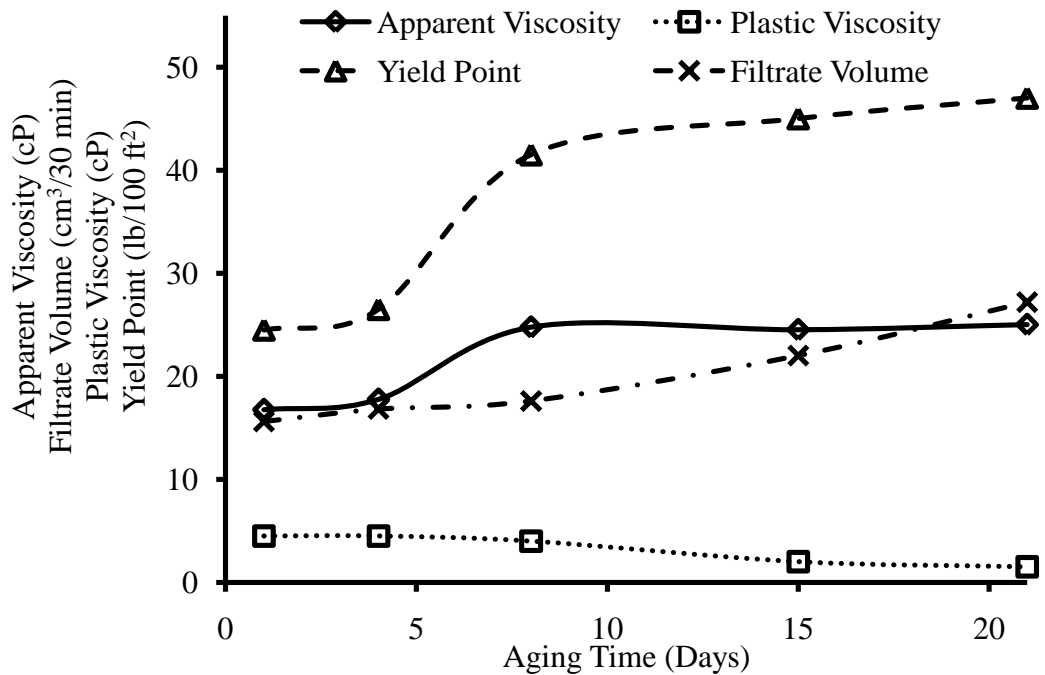


Figure 5-170 Aging of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  & Xanthan Gum and Dextrid Mud for a 21-day period

### 5.5.6 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated Bentonites and Polymers Treated Muds

Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture (Na<sub>2</sub>CO<sub>3</sub> addition level; 2,18% Na<sub>2</sub>O equivalent and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O addition level; 0,95% Na<sub>2</sub>O equivalent mixture for E-Ben, and Na<sub>2</sub>CO<sub>3</sub> addition level; 0,29% Na<sub>2</sub>O equivalent and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O addition level; 0,38% Na<sub>2</sub>O equivalent mixture for C-Ben) activated bentonite and polymers treated muds were aged for a 21-day period at 36 °C. These are;

- E-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with 2% HV CMC,
- C-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with 1,0% HV CMC and 0,7% LV CMC mixture,
- E-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with 1,5% PAC R,
- C-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with 0,6% PAC R and 0,5% PAC L mixture,
- E-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with 3,5% xanthan gum,
- C-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with 0,8% xanthan gum and 2,4% dextrid mixture.

The rheological and filtration properties of E-Ben and C-Ben treated with polymer studied at 36 °C for a 21-day period are given in Figures 5-171 through 5-176 (see also appendix Tables C-29 through C-34).

The effect of different extenders on the rheology and filtrate properties of bentonite muds are shown in Figures 5-171 through 5-175. These figures clearly indicate that the presence of the Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O-Na<sub>2</sub>CO<sub>3</sub> mixture activated bentonite mud gave a stable fluid by treatment with polymer with aging time except xanthan gum treated C-Ben. Figure 5-176 shows that C-Ben activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture and treated with xanthan gum was susceptible to bacterial attack with aging time at 36 °C, reducing the control of rheological and filtrate volume properties.

In contrast to E-Ben activated with Na<sub>2</sub>CO<sub>3</sub> and treated with xanthan gum, Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O-Na<sub>2</sub>CO<sub>3</sub> mixture activated E-Ben and xanthan gum treated mud

showed a stable viscosity like E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and xanthan gum treated mud i.e.,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  activated E-Ben and  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  mixture activated E-Ben showed better rheological properties than  $\text{Na}_2\text{CO}_3$  activated E-Ben. Consequently  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  and  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ - $\text{Na}_2\text{CO}_3$  mixture can be used as a bactericide in the activation of E-Ben.

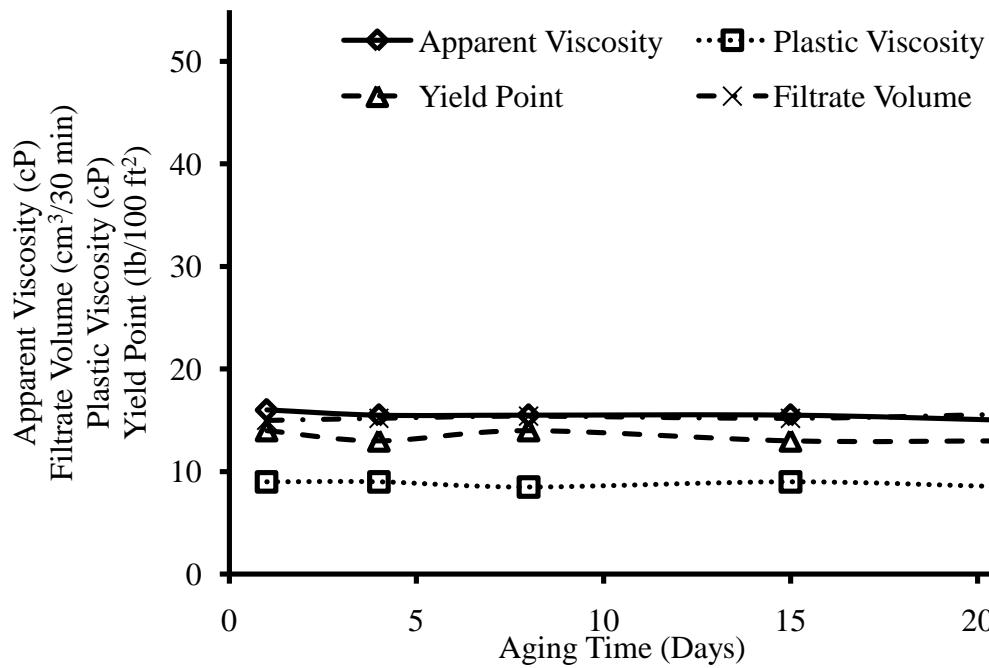


Figure 5-171 Aging of E-Ben Activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture & CMC Mud for a 21-day period



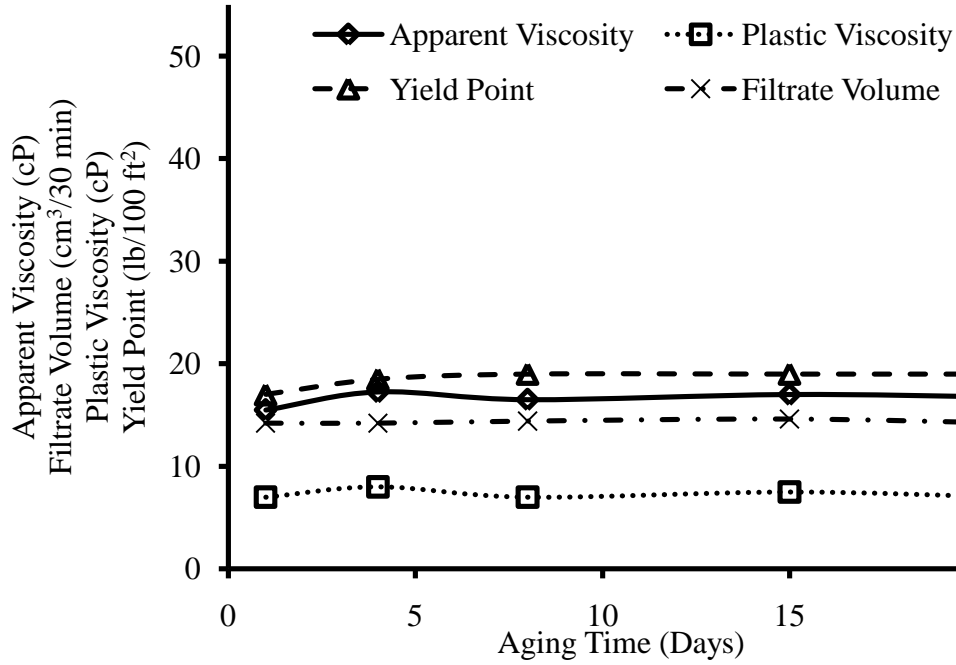


Figure 5-172 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & HV CMC and LV CMC Mud for a 21-day period

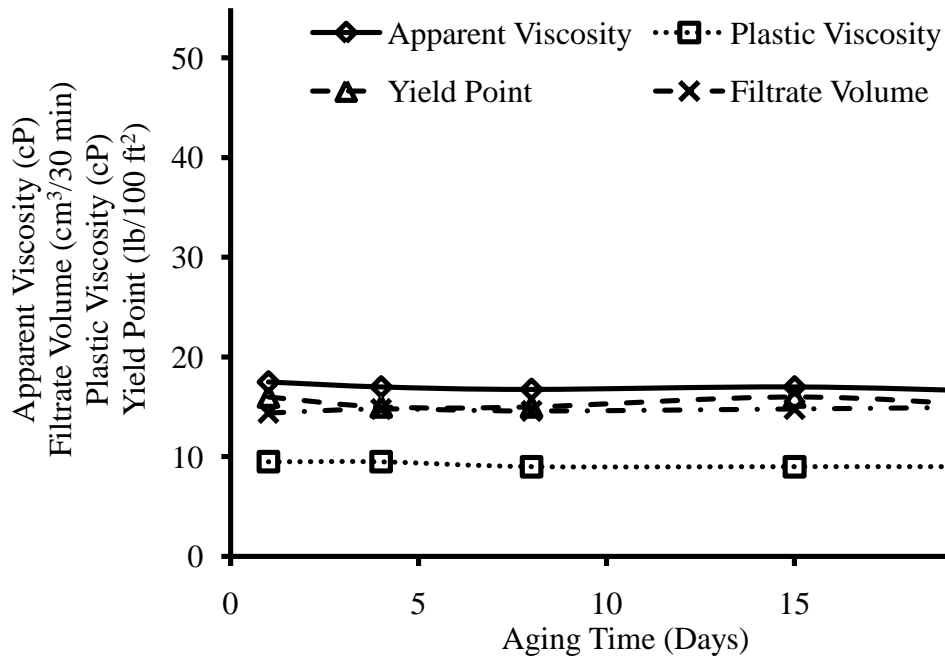


Figure 5-173 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & PAC R Mud for a 21-day period

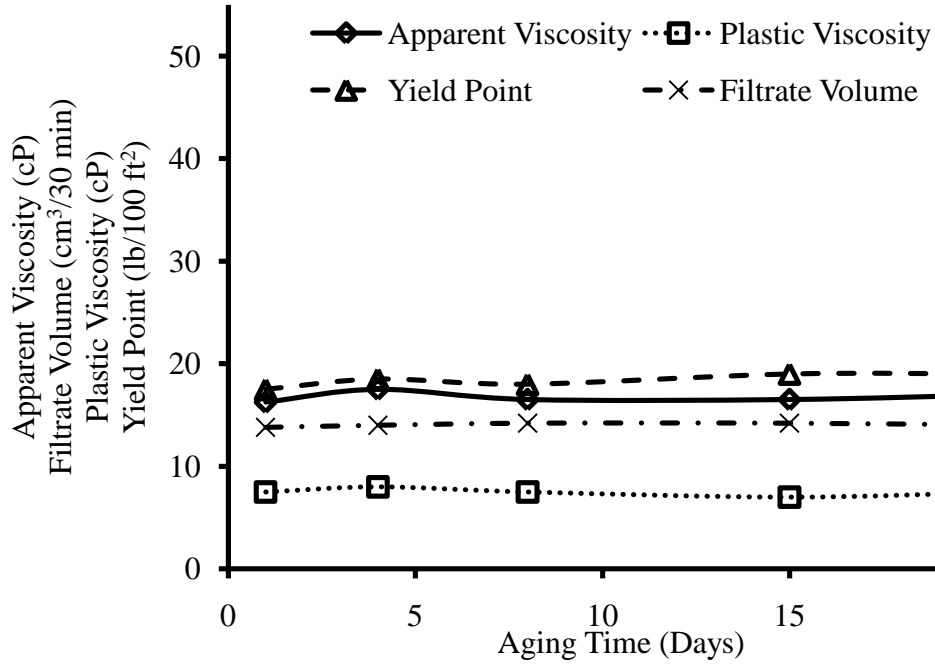


Figure 5-174 Aging of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture & PAC R and PAC L Mud for a 21-day period

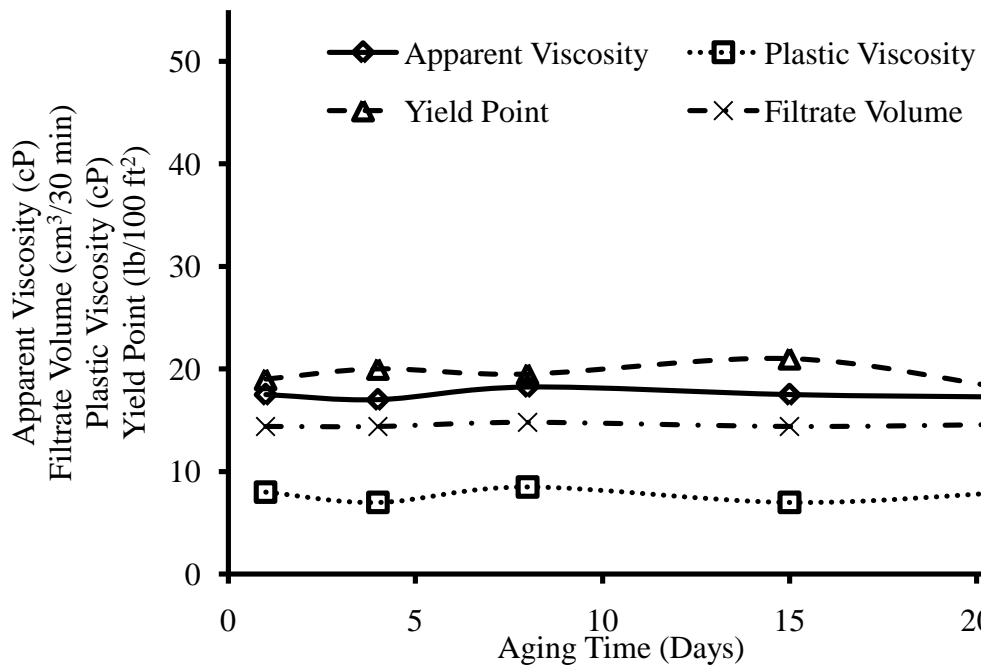


Figure 5-175 Aging of E-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Mixture & Xanthan Gum Mud for a 21-day period

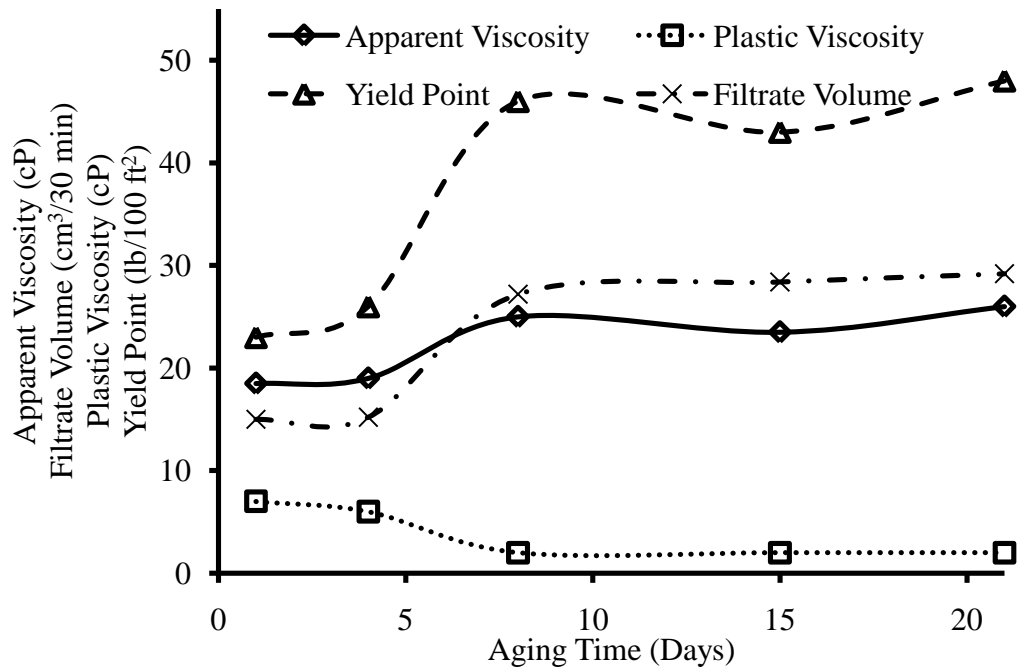


Figure 5-176 Aging of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture & Xanthan Gum and Dextrid Mud for a 21-day period

### 5.6 Aging of Bentonite Muds at High Temperature

A major problem in drilling deep, hot wells with water-base drilling fluids has been thermal degradation of the chemical components. High temperature deteriorates their structure. Partial dehydration and face-to-face attachment of the bentonite particles result in losses of gel structure and thixotropy. Filter-cake porosity increases with the coagulation of clay platelets, resulting in poor filtration control (Plank and Gossen, 1991). An increase in temperature also reduces the effectiveness of most drilling fluid additives because (a) some mud additives degrade at high temperature, and (b) chemical reactions that may affect the performance of drilling mud additives are accelerated at higher temperatures.

Under higher temperatures, the Brownian motion intensifies further; the molecules collide with each other with greater thermo-mechanical energy and thus undergo serious oxidation and hydrolysis reactions leading to the breakage of polymers i.e., thermal degradation of the polymer products (Amanullah, 2004). The

rate of degradation depends on the fluid, dissolved oxygen, pH of the mud and time of exposure to the elevated temperature (Perricone et al., 1986). Thomas (1982) recommends that carboxymethyl cellulose-based products are not used routinely above 149 °C (300 °F). Correspondingly, starch-based polymers should not be used above 107 °C (225 °F).

In this experiment the effect of heat was determined by heating the muds at high temperature (149 °C for CMC and PAC and 121 °C for xanthan gum) for 16 hours in a roller oven. After cooling to room temperature, the fluid properties such as apparent and plastic viscosities, gel strengths (10 seconds/10 minutes), yield points and filtrate volumes were determined.

### **5.6.1 Aging of Nontreated Bentonite**

API Standard Bentonite and Reşadiye Bentonite were aged at 149 °C to compare the aging of E-Ben and C-Ben activated with inorganic salts and treated with polymers.

The results of the rheological and filtrate properties of prepared drilling muds measured at room temperature after aging at 149 °C are given in Figures 5-177 and 5-178 (see also appendix Table D-1 and D-2).

The above mentioned figures show the rheological properties measured at room temperature were affected after aging the bentonite suspension at high temperature. The rheological properties (apparent viscosity, plastic viscosity, yield point and gel strengths) of bentonites suspension increased during the process at 149 °C which were reflected in the slight increase in fluid loss. The increase in yield point was attributed to swelling of the particles at higher temperatures, and to the dissolution of Na<sup>+</sup> cations from the clay surface into the medium (Rossi et al., 1999).

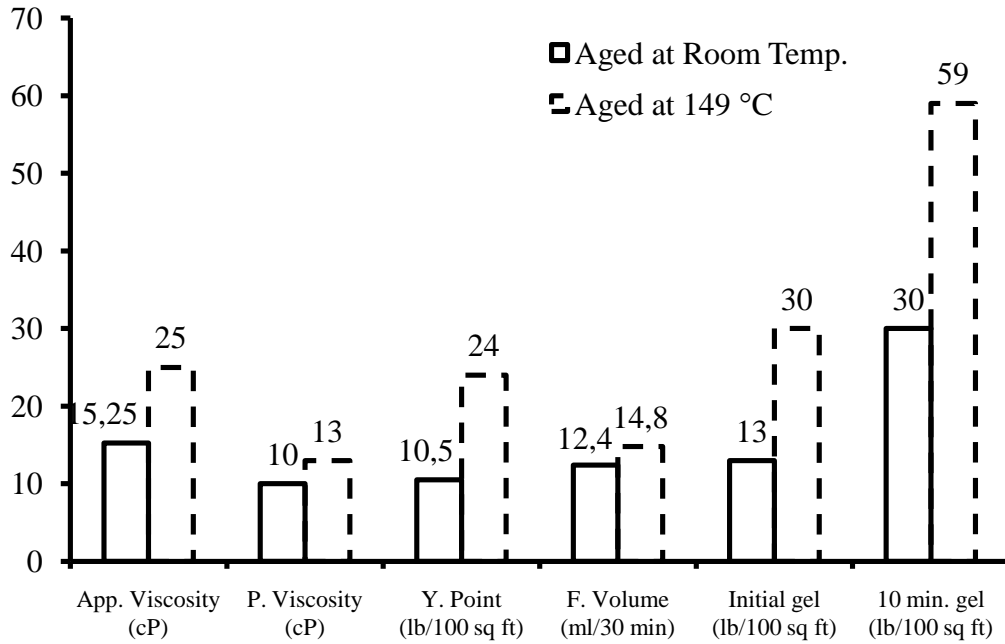


Figure 5-177 Aging of API Bentonite at 149 °C

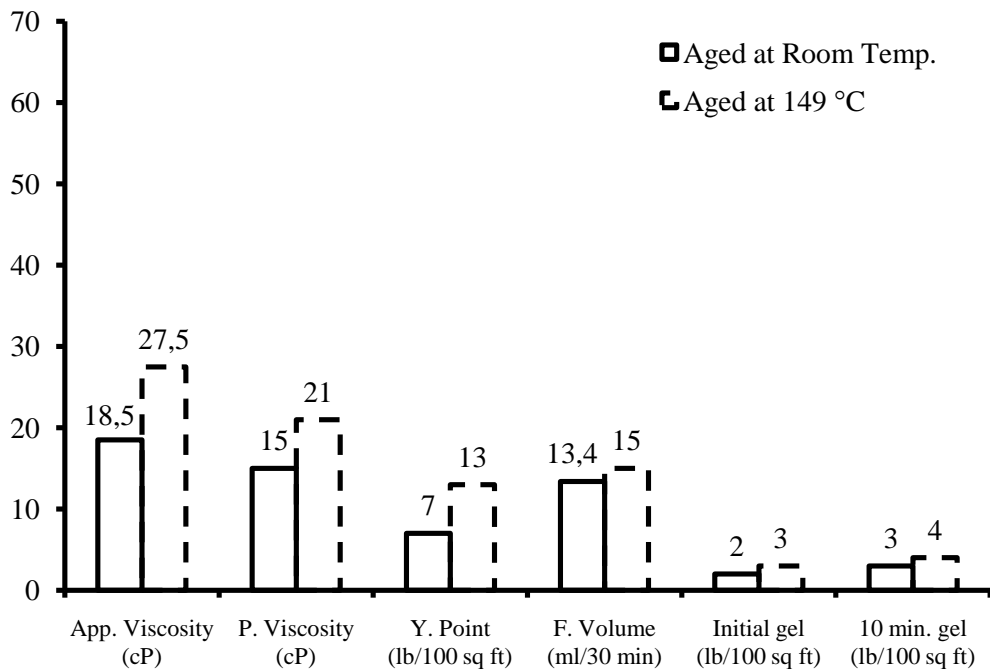


Figure 5-178 Aging of Reşadiye Bentonite at 149 °C

### 5.6.2 Aging of Unactivated Bentonites and Polymers Treated Muds

Water-based drilling fluids containing each of the following composition were prepared by API Standard and aged at high temperature.

- Mud composed of E-Ben & 7,5% HV CMC,
- Mud composed of E-Ben & 3,75% PAC R,
- Mud composed of E-Ben & 7,5% xanthan gum,
- Mud composed of C-Ben & 3% HV CMC,
- Mud composed of C-Ben & 2% PAC,
- Mud composed of C-Ben & 2,5% xanthan gum.

The rheological and filtration loss characteristics values of these mud compositions measured at room temperature are given in Figures 5-179 through 5-184 (see also appendix Tables D-3 through D-8). These test results, summarized in figures, showed that the performance of polymers treated muds was adversely affected by heat i.e., all the muds underwent serious thermal degradation at 149 °C. After aging at 149 °C, the cellulose-treated mud exhibited the highest filtration rate. Moreover, the apparent viscosity of all of the mud compositions decreased to approximately previous values before treatment with polymers. This is not an expected result because it is recommended that the cellulosic materials not be used above 149 °C. As a result, the polymers in unactivated bentonite muds were entirely degraded.

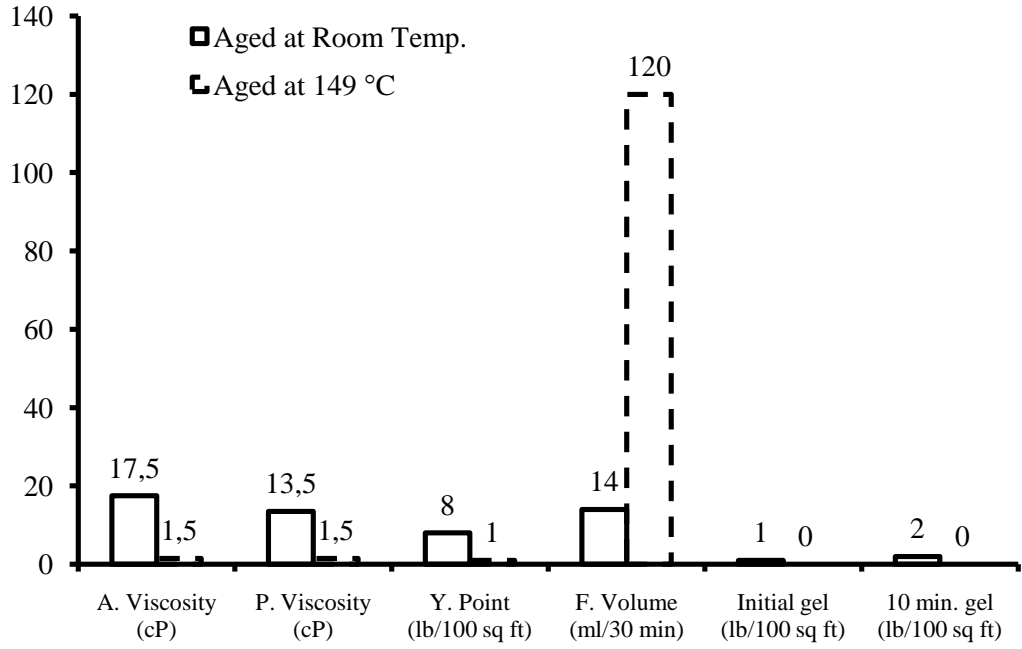


Figure 5-179 Aging of E-Ben & CMC Mud at 149 °C

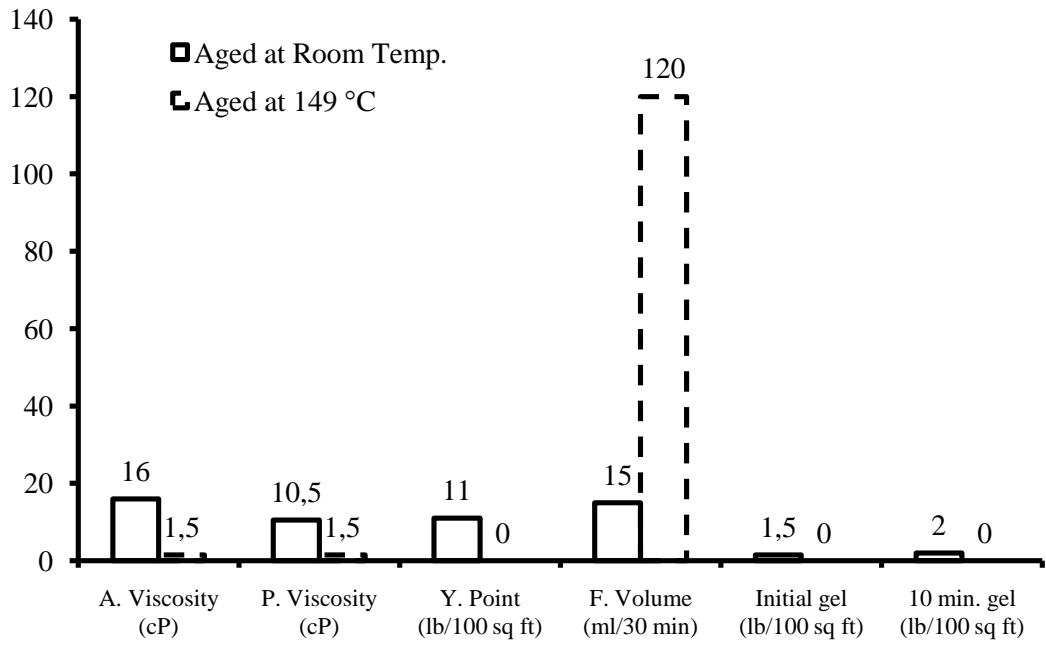


Figure 5-180 Aging of E-Ben & PAC Mud at 149 °C

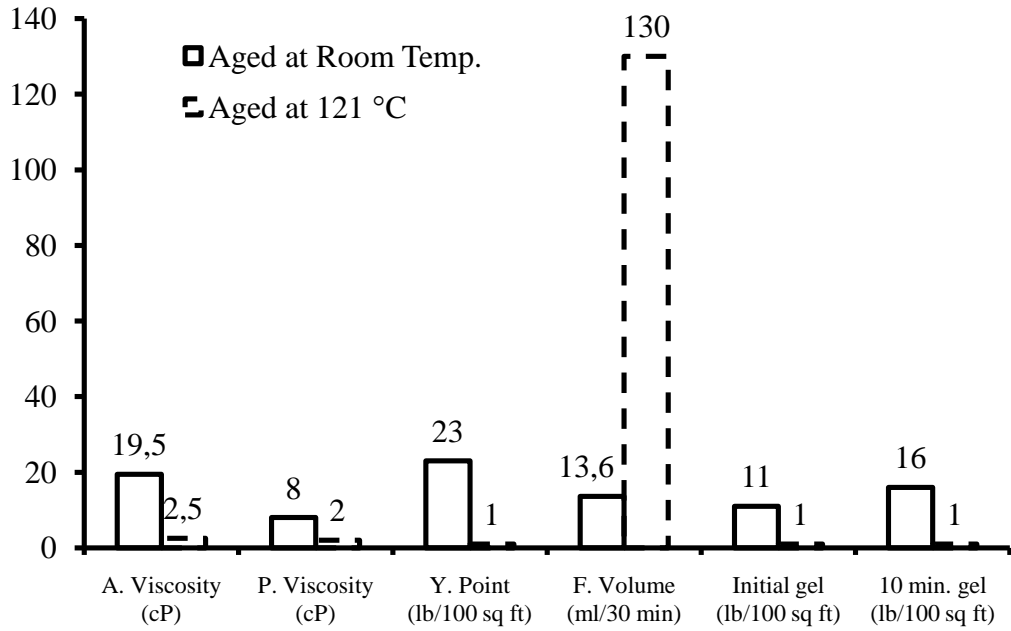


Figure 5-181 Aging of E-Ben & Xanthan Gum Mud at 121 °C

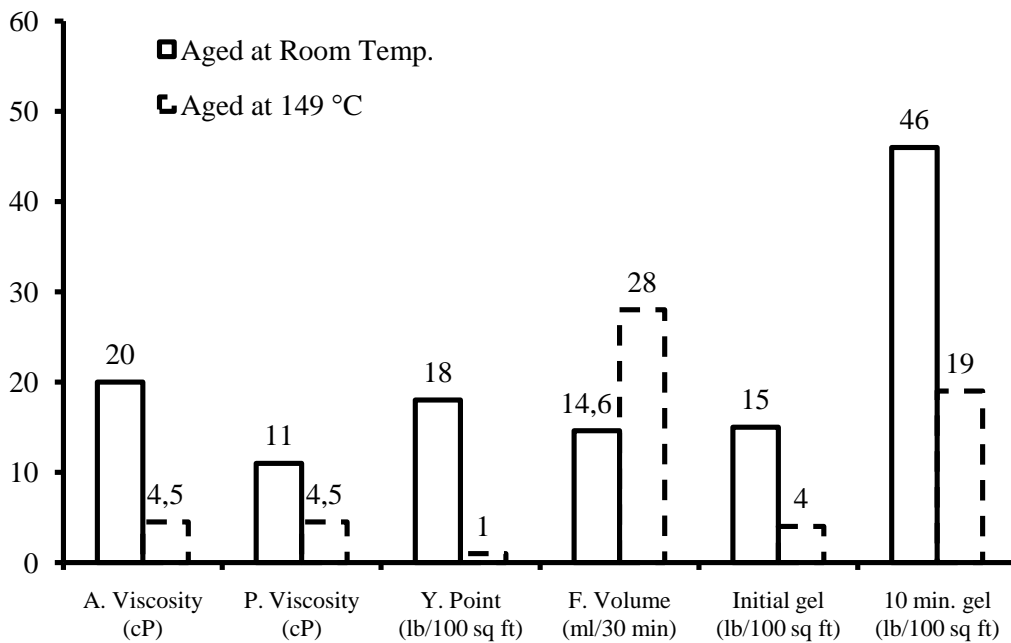


Figure 5-182 Aging of C-Ben & CMC at 149 °C



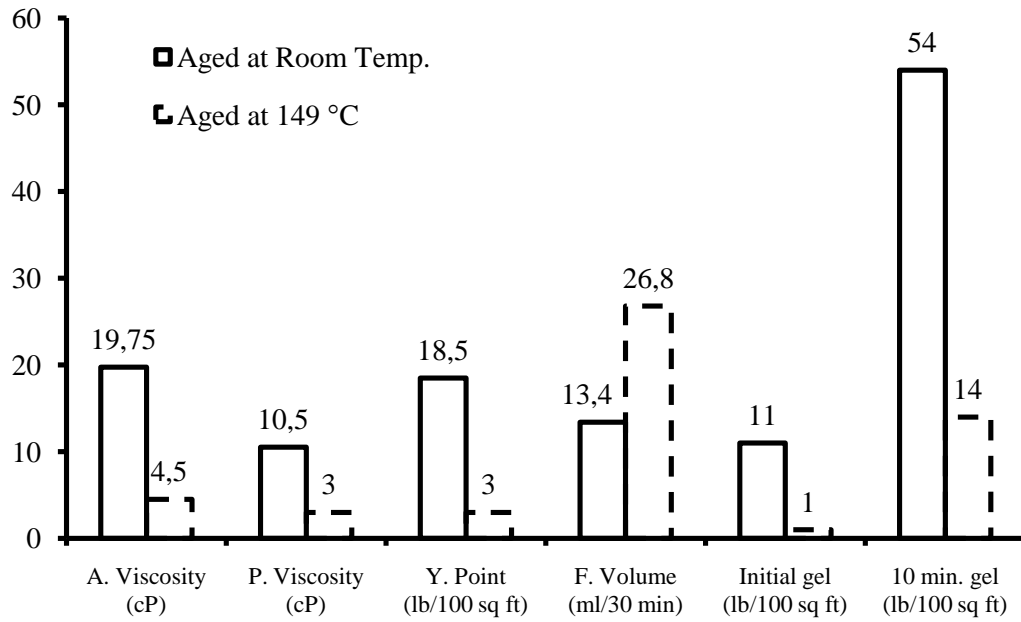


Figure 5-183 Aging of C-Ben & PAC Mud at 149 °C

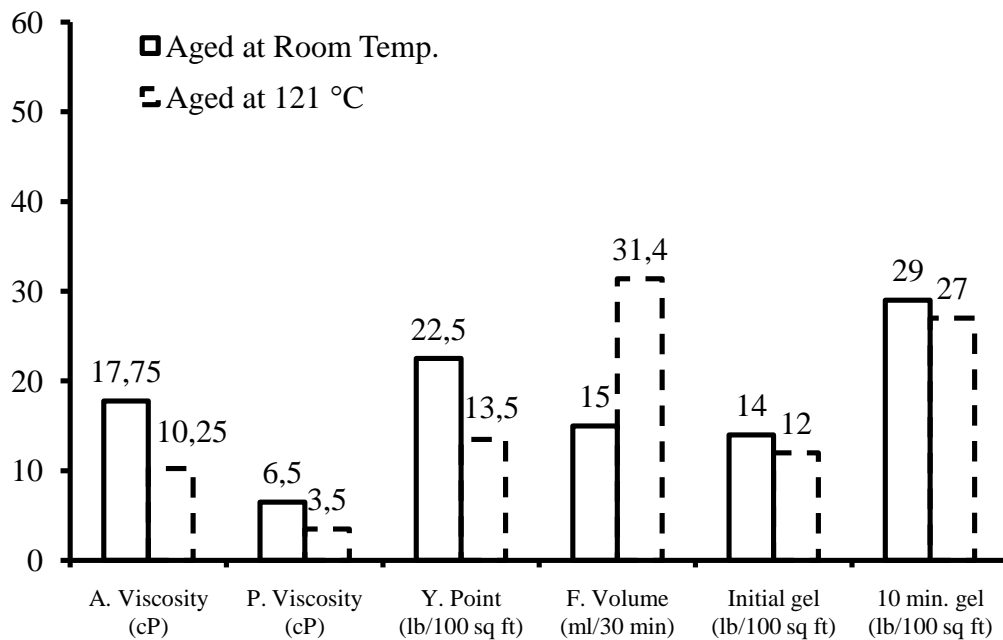


Figure 5-184 Aging of C-Ben & Xanthan Gum Mud at 121 °C

### 5.6.3 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated Bentonite and Polymers Treated Muds

The rheological and filtration loss characteristics of the following mud compositions measured at room temperature after aging at high temperature for 16 hours are given in Figures 5-185 and 5-192 (see also appendix Tables D-9 through D-16).

- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated E-Ben,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated C-Ben,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated E-Ben & 1,4% HV CMC,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated C-Ben & 0,8% HV CMC and 0,4% LV CMC mud,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated E-Ben & 1,0% PAC R mud,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated C-Ben & 0,50% PAC R and 0,30% PAC L mud,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated E-Ben & 3,0% xanthan gum,
- Mud composed of Na<sub>2</sub>CO<sub>3</sub> activated C-Ben & 0,80% xanthan gum and 2,40% dextrid mud.

The results obtained from Figure 5-185 and 5-186 show the presence of the temperature effect of the rheological properties of two muds (Na<sub>2</sub>CO<sub>3</sub> activated E-Ben and Na<sub>2</sub>CO<sub>3</sub> activated C-Ben). For one mud, while apparent viscosity, plastic viscosity and yield point increased, the gel strength decreased. For the other mud, the 10-gel strength increased from 30 to 72 lb/100 ft<sup>2</sup>.

The polymers in activated bentonite were completely degraded as shown in Figures 5-187 through 5-192. Because the filtrate loss of high temperature aging bentonite muds increased to that of activated bentonite muds aged in room temperature.

As a result, higher temperature complicated the rheological properties of mud compositions studied. As the rheological properties of bentonites treated with CMC and PAC decreased, that of bentonites treated with xanthan gum increased. A change in the apparent viscosity and gel strength may result from the different interaction between the bentonite and polymer type.

But the apparent viscosity and filtrate volume of activated bentonite did not reduce that of raw bentonite. As a result the aging of activated bentonites at high

temperature showed better rheological and filtrate properties than unactivated bentonites.

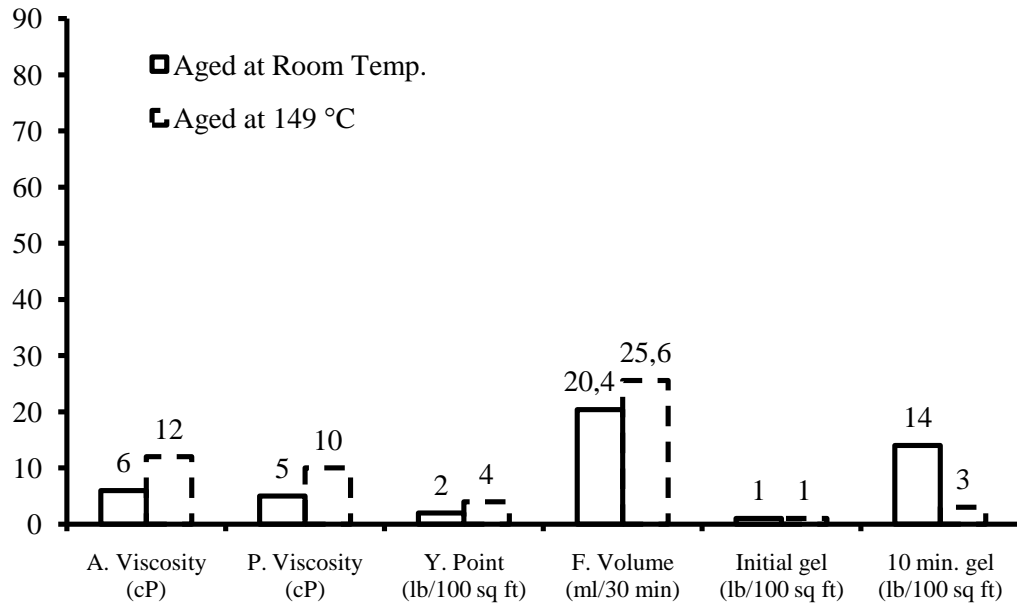


Figure 5-185 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben Mud at 149 °C

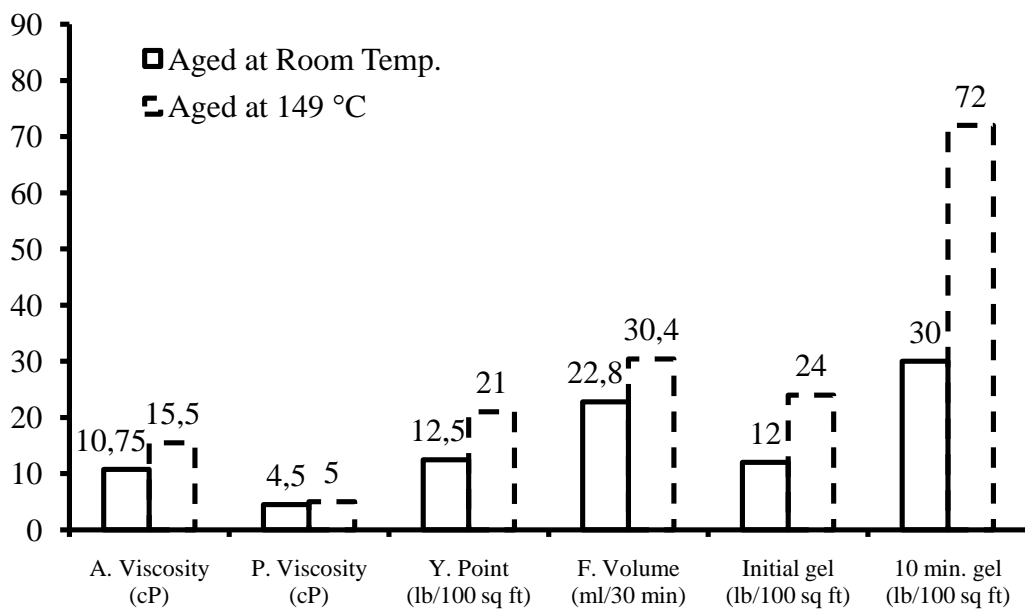


Figure 5-186 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben Mud at 149 °C

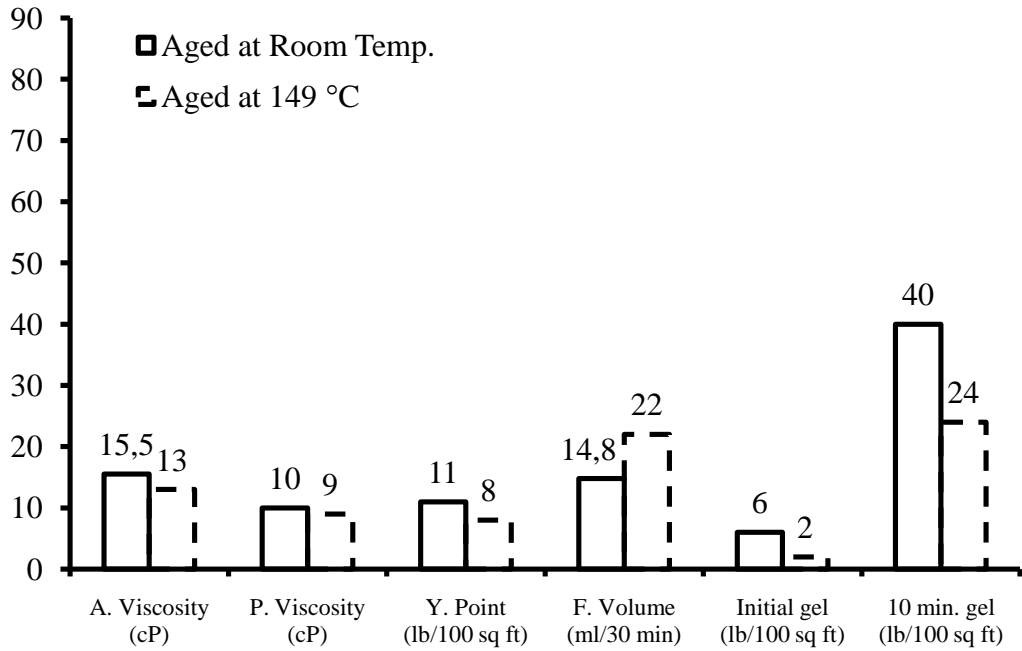


Figure 5-187 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben & CMC Mud at 149 °C

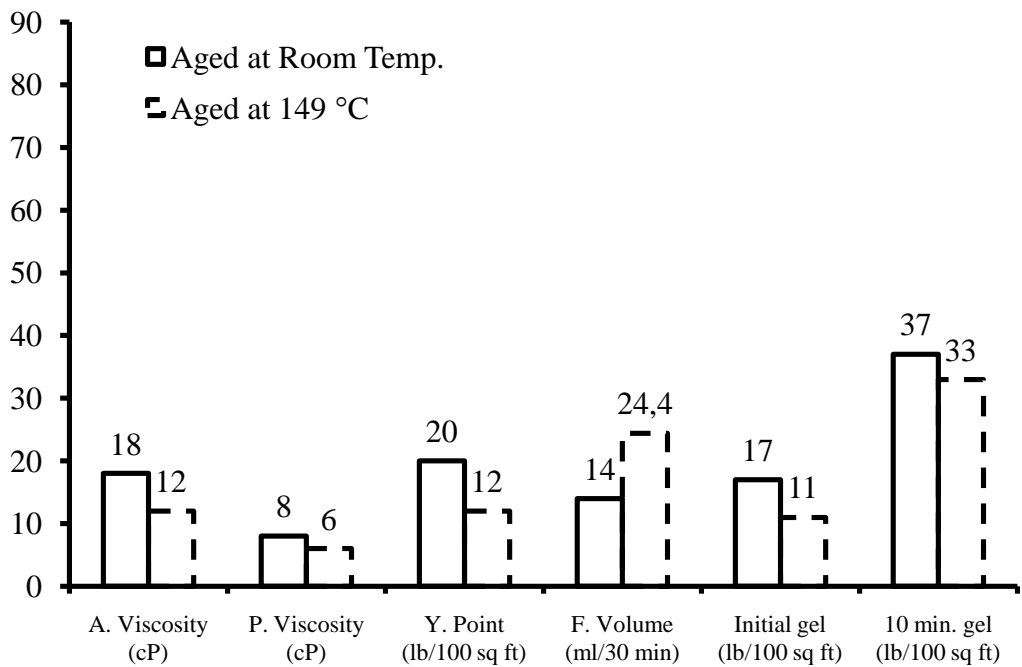


Figure 5-188 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben & CMC Mud at 149 °C

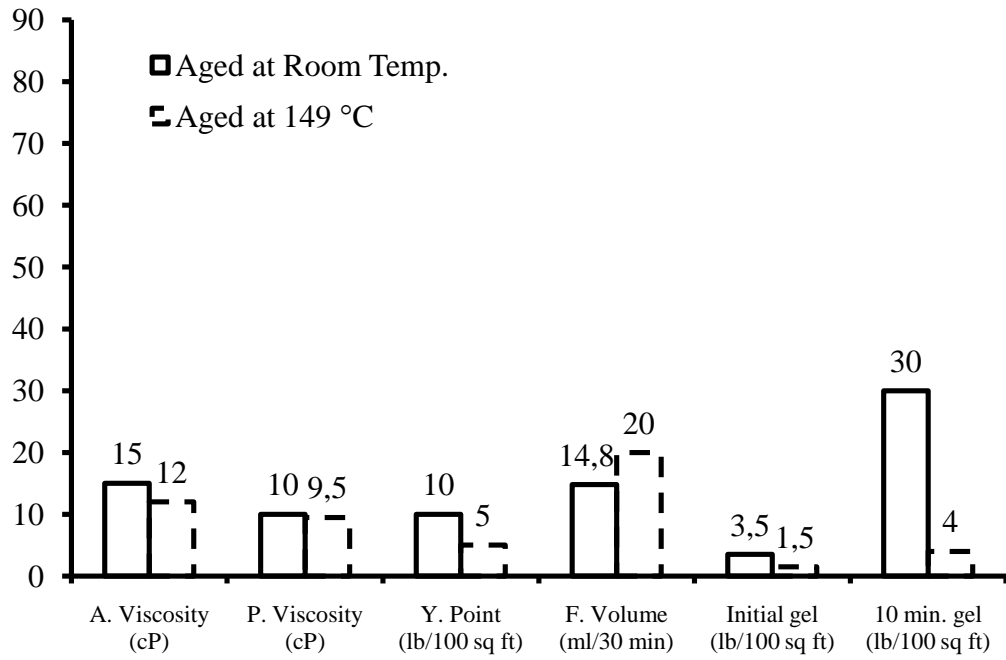


Figure 5-189 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben & PAC Mud at 149 °C

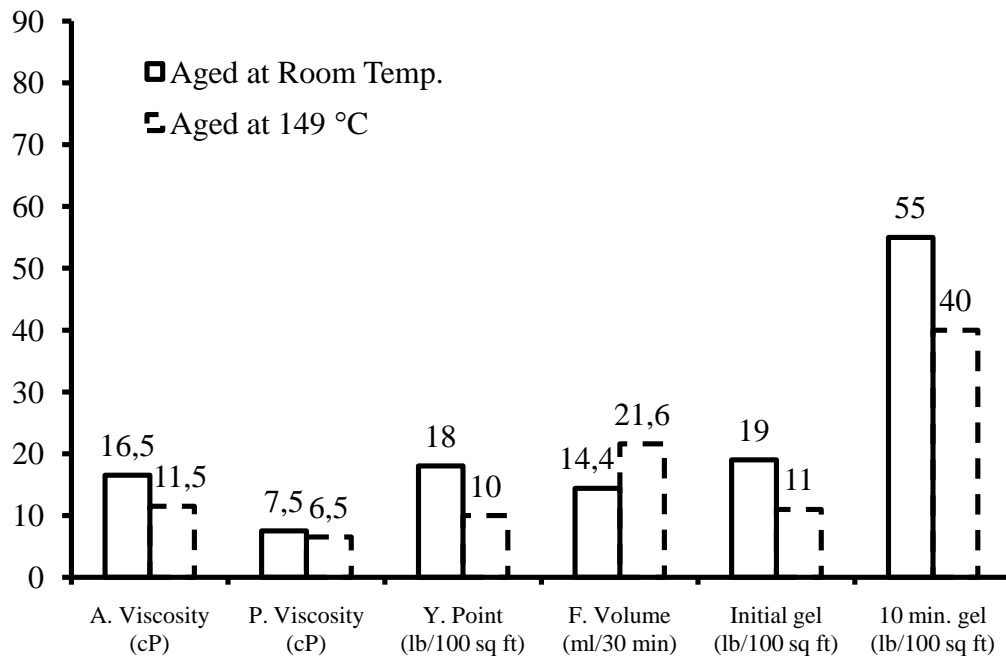


Figure 5-190 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben & PAC Mud at 149 °C

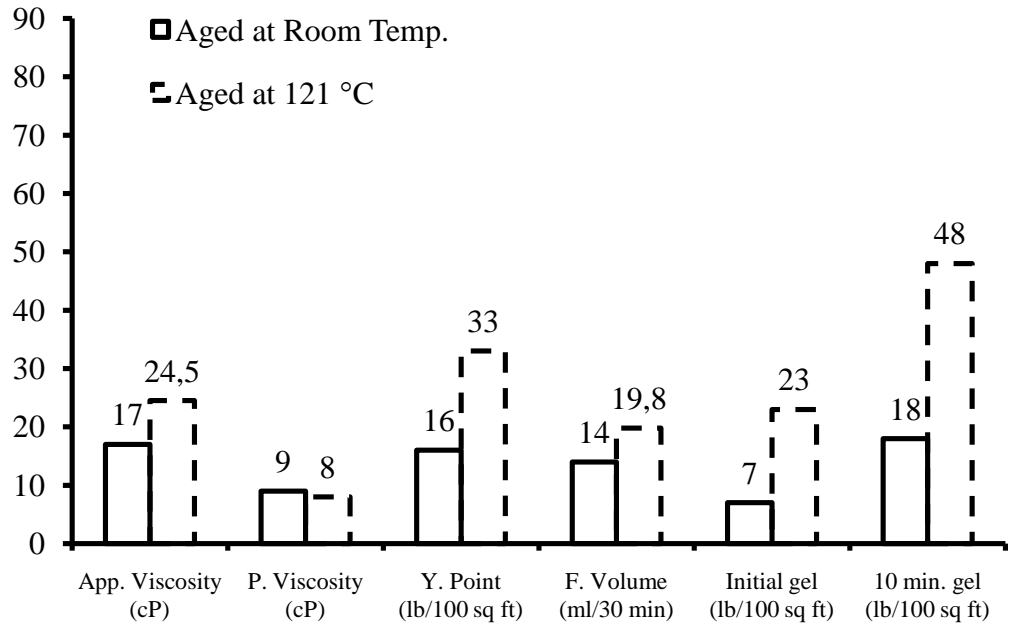


Figure 5-191 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben & Xanthan Gum Mud at 121 °C

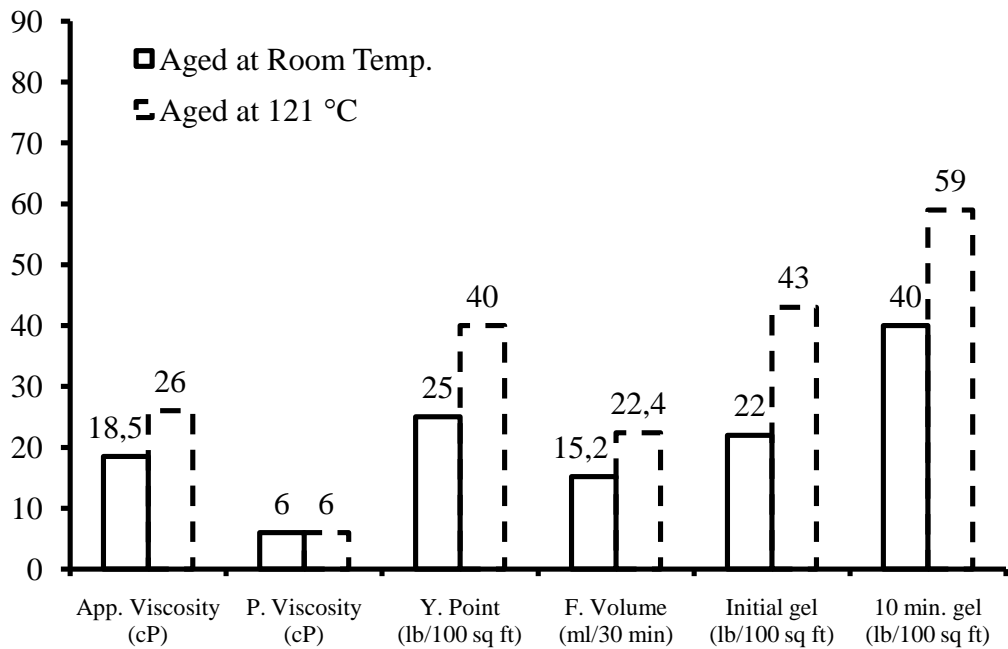


Figure 5-192 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben & Xanthan Gum Mud at 121 °C

#### 5.6.4 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated Bentonite and Polymers Treated Muds

Water-based drilling fluids containing each of the following compositions were prepared by API Standard and aged at high temperature.

- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated E-Ben,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated C-Ben,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated C-Ben & 0,8% HV CMC and 0,4% LV CMC,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated E-Ben & 2,8% HV CMC,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated E-Ben & 1,8% PAC R,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated C-Ben & 0,50% PAC R and 0,30% PAC L,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated E-Ben & 4,5% xanthan gum,
- Mud composed of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated C-Ben & 0,80% xanthan gum and 2,4% dextrans.

The rheological and filtration loss characteristics of these mud compositions measured at room temperature are given in Figures 5-193 through 5-200 (see also appendix Table D-17 through D-24).

Activated bentonite fluids demonstrated the fact that a common mud additive caused complications to the fluid rheology at high temperatures. Na<sub>2</sub>CO<sub>3</sub> activated bentonite mud exhibited viscosity and filtrate properties that are rather different from Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite mud. Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite muds showed a temperature/fluid loss relationship different from that of Na<sub>2</sub>CO<sub>3</sub> activated bentonite muds; i.e., the fluid loss of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite muds was higher than Na<sub>2</sub>CO<sub>3</sub> activated bentonite muds at high temperature (Figures 5-193 through 5-200). The main cause for these different deteriorations of Na<sub>2</sub>CO<sub>3</sub> activated bentonite and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite may be related to the reactions of CO<sub>3</sub><sup>=</sup> and borate ion in bentonite slurry at high temperature.

But after the complete degradation of polymers added in Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O activated bentonite mud, the activated bentonite showed better rheological behaviours than the same unactivated bentonites.

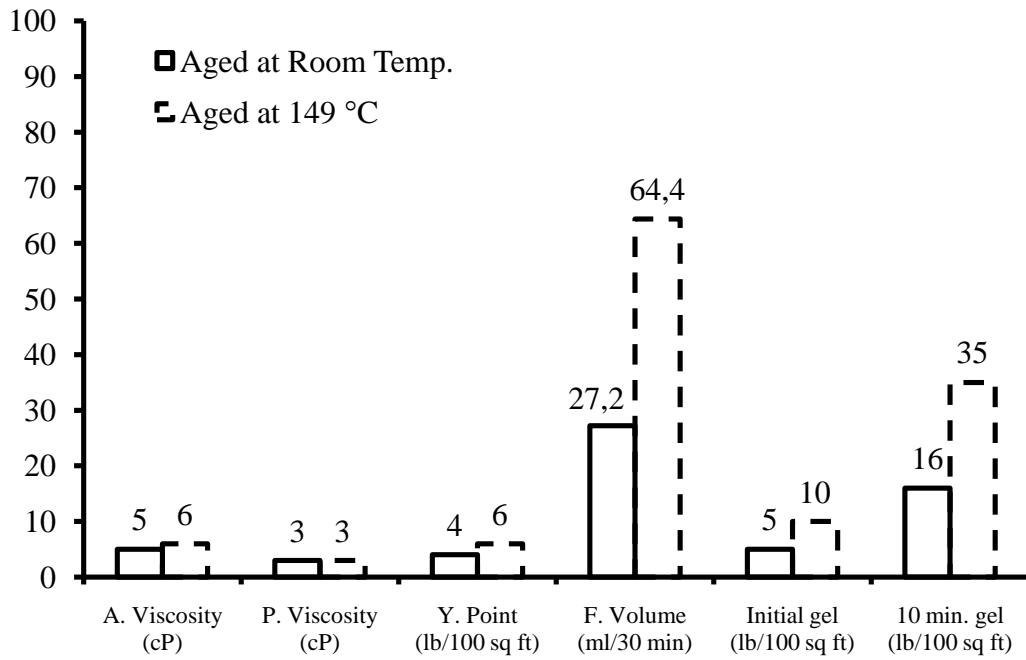


Figure 5-193 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben Mud at 149 °C

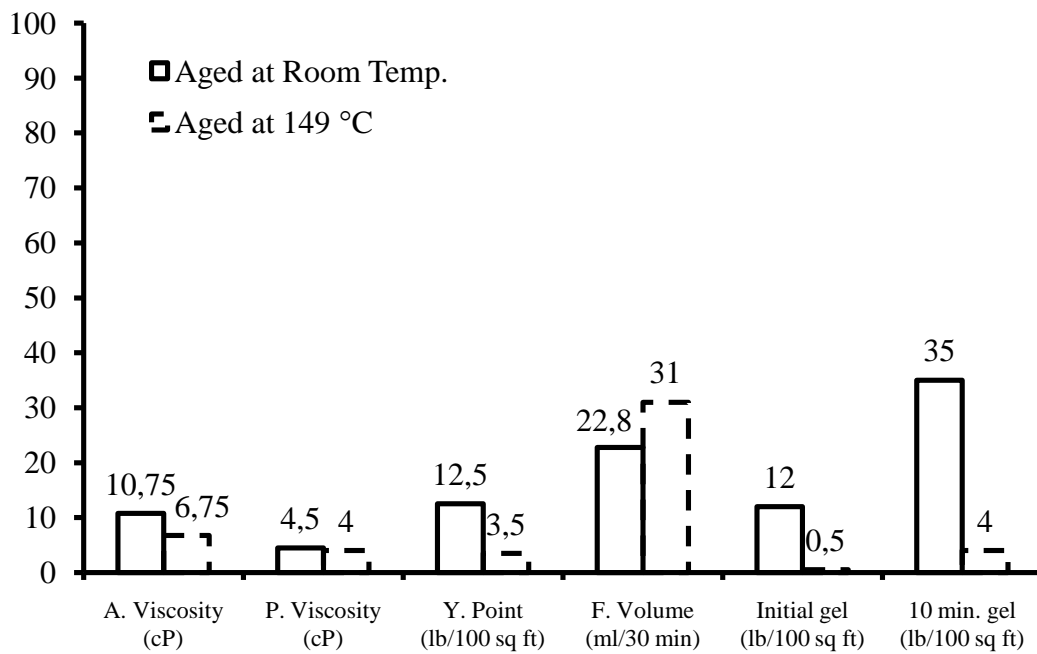


Figure 5-194 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated C-Ben Mud at 149 °C



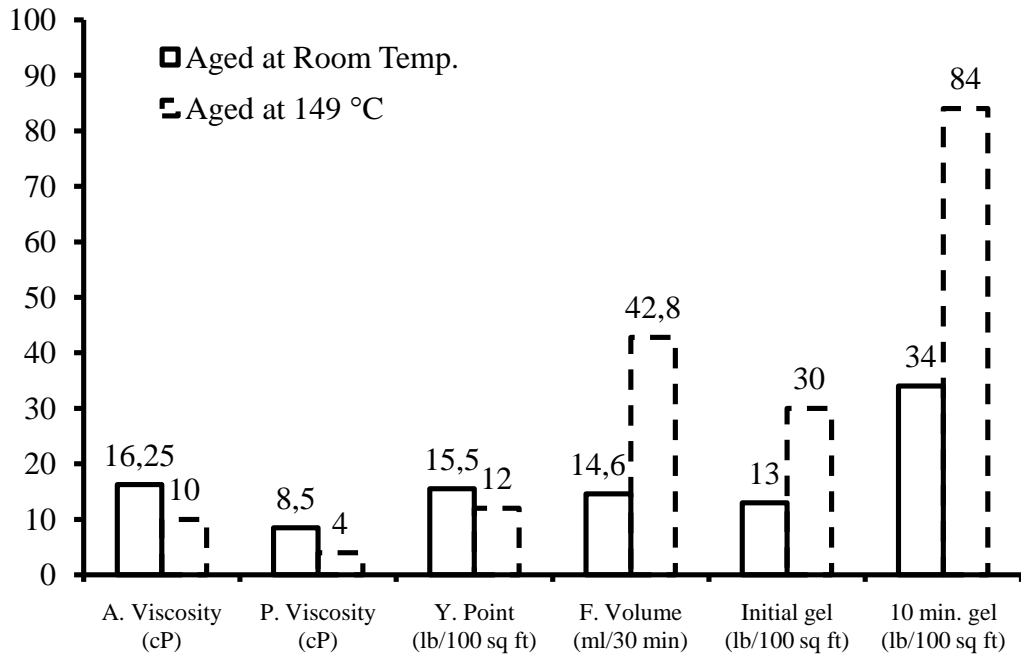


Figure 5-195 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben & CMC Mud at 149 °C

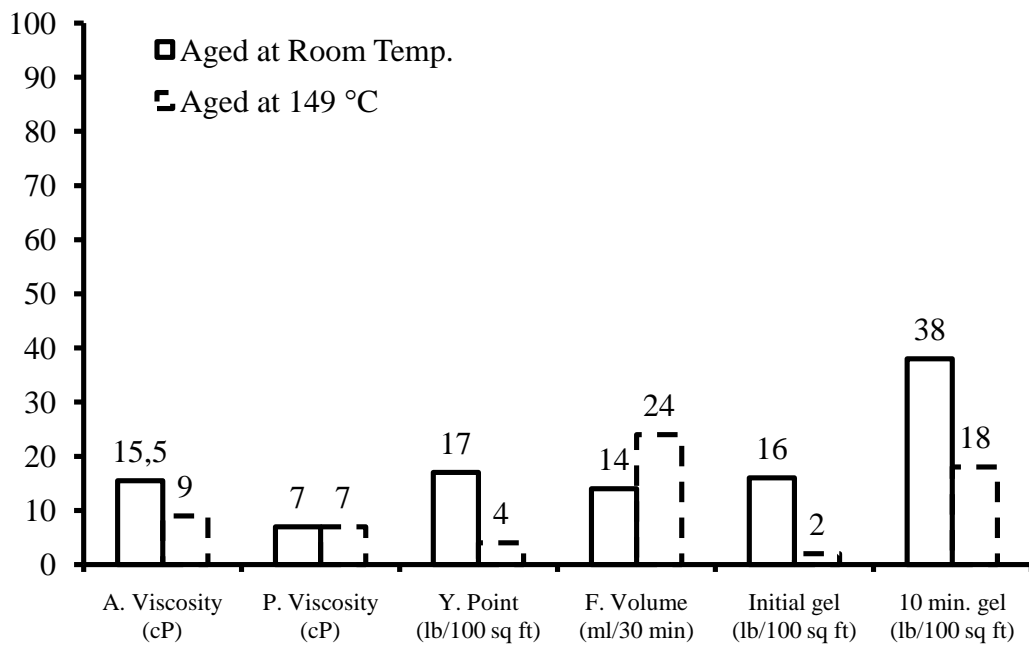


Figure 5-196 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated C-Ben & CMC Mud at 149 °C

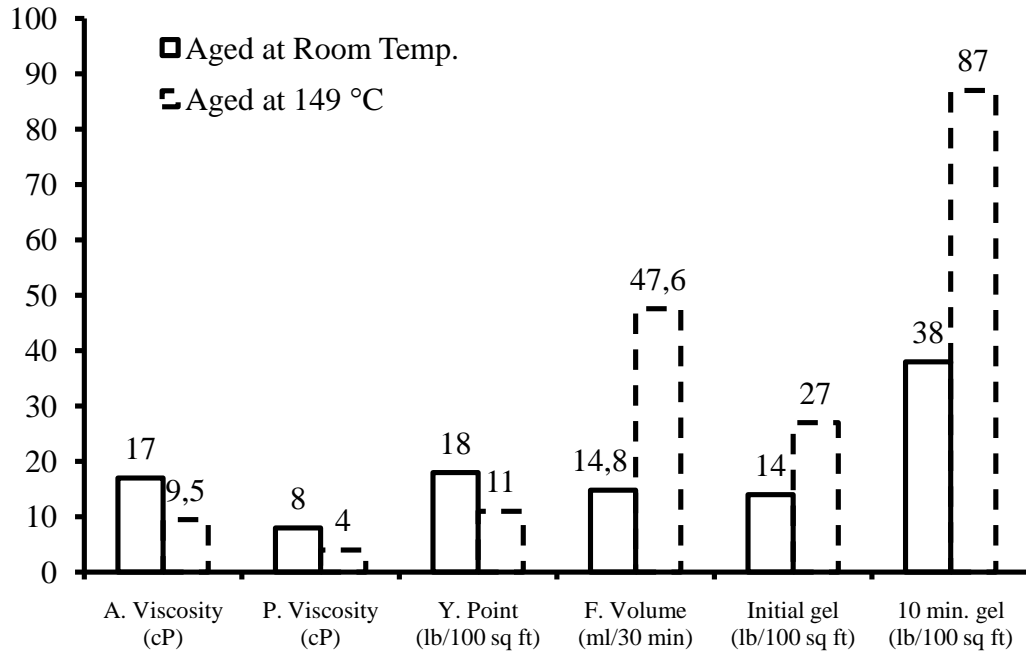


Figure 5-197 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben & PAC Mud at 149 °C

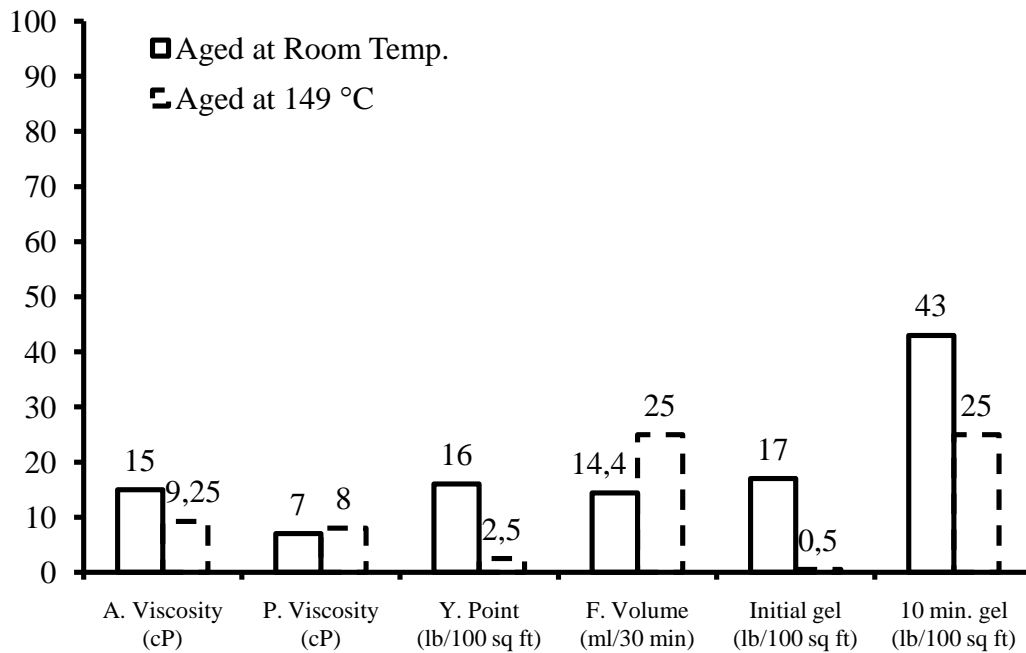


Figure 5-198 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated C-Ben & PAC Mud at 149 °C

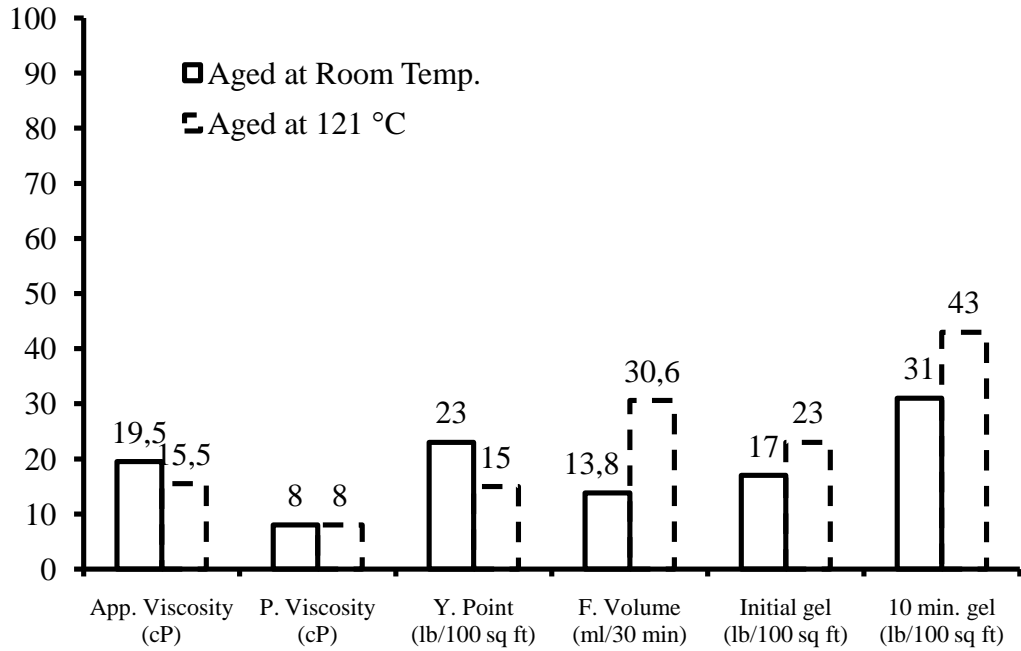


Figure 5-199 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>.5H<sub>2</sub>O Activated E-Ben & Xanthan Gum Mud at 121 °C

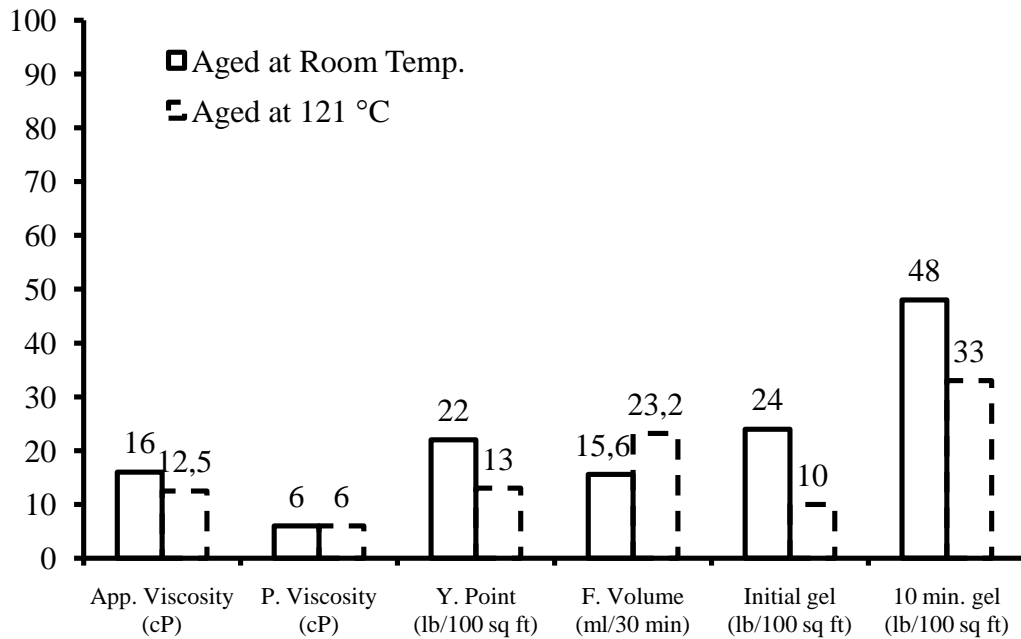


Figure 5-200 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>.5H<sub>2</sub>O Activated C-Ben & Xanthan Gum Mud at 121 °C

### 5.6.5 Aging of $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ Mixture Activated Bentonites and Polymers Treated Muds

Water-based drilling fluids containing each of the following compositions were prepared by API Standard and aged at high temperature.

- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated E-Ben,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated C-Ben,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated E-Ben & 2% HV CMC,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated C-Ben & 1,0% HV CMC and 0,7% LV CMC,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated E-Ben & 1,5% PAC R,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated C-Ben & 0,6% PAC R and 0,5% PAC L,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated E-Ben & 3,5% xanthan gum,
- Mud composed of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated C-Ben & 0,8% xanthan gum and 2,4% dextrid.

Figures 5-201 through 5-208 (see also appendix Table D-25 through D-32) show the rheological and filtration properties of these muds measured at room temperature after aging at high temperature.

Figures 5-201 through 5-208 show that higher filter losses of treated muds after aging at high temperature indicate extensive degradation of polymers. It was also observed that after subjecting  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture activated bentonite slurry to high temperature, the rheological properties changed.

As a conclusion all of the bentonite activated with inorganic salt and treated with polymer mud compositions demonstrated the fact that a common mud additive caused complications to the fluid rheology at high temperatures.

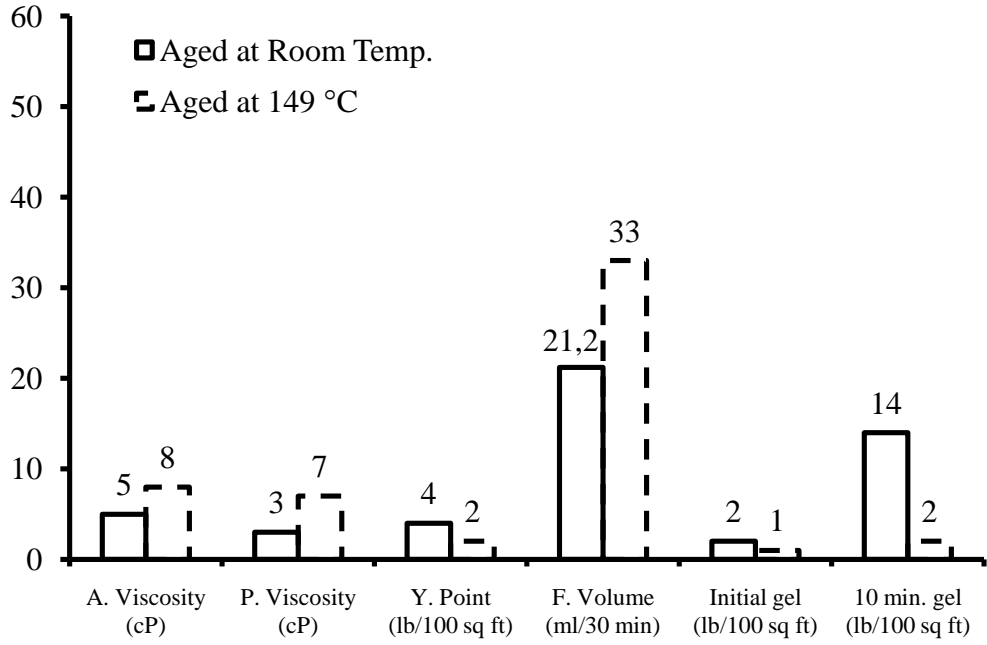


Figure 5-201 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated E-Ben Mud at 149 °C

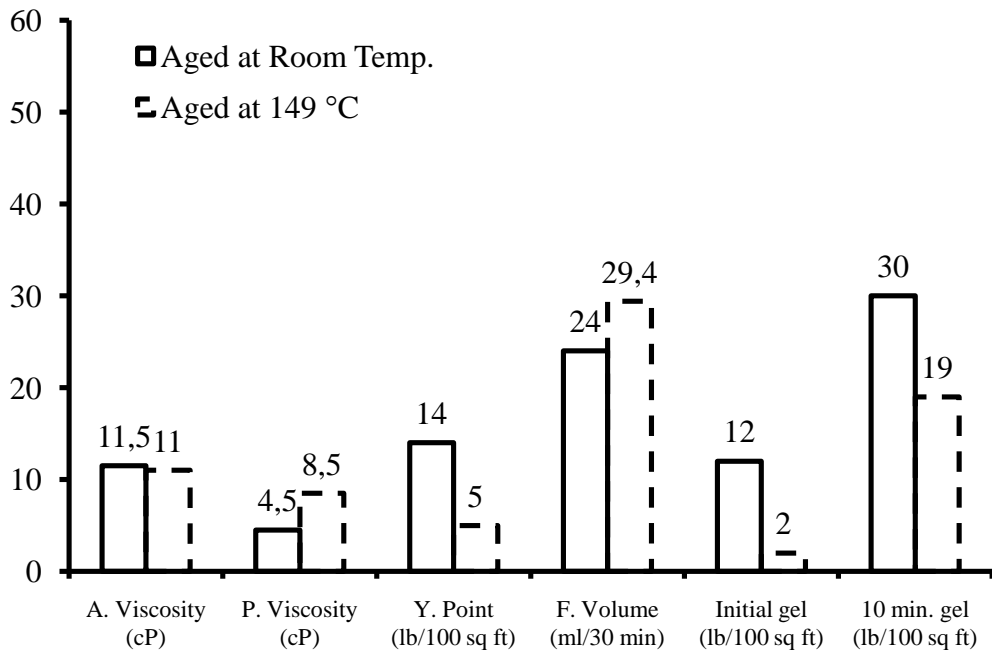


Figure 5-202 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated C-Ben Mud at 149 °C

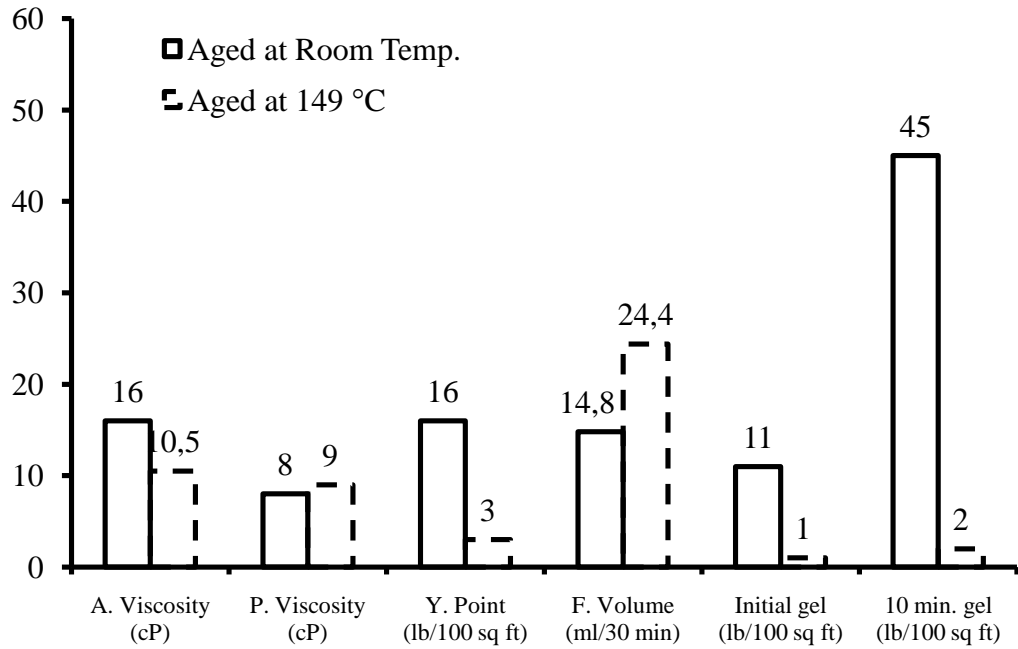


Figure 5-203 Aging of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated E-Ben & CMC Mud at 149 °C

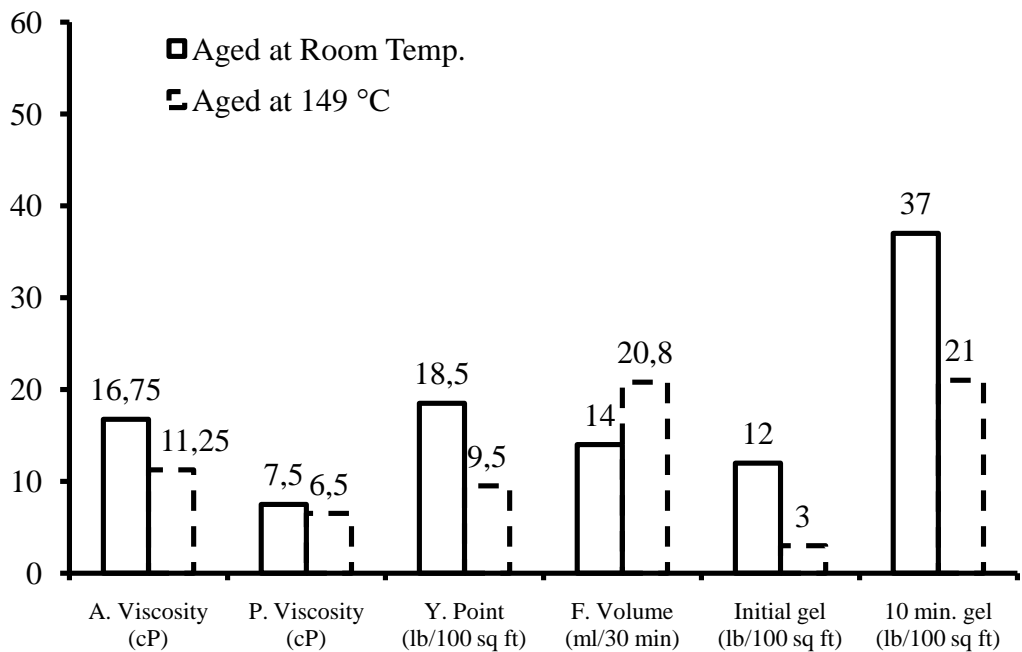


Figure 5-204 Aging of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated C-Ben & CMC Mud at 149 °C

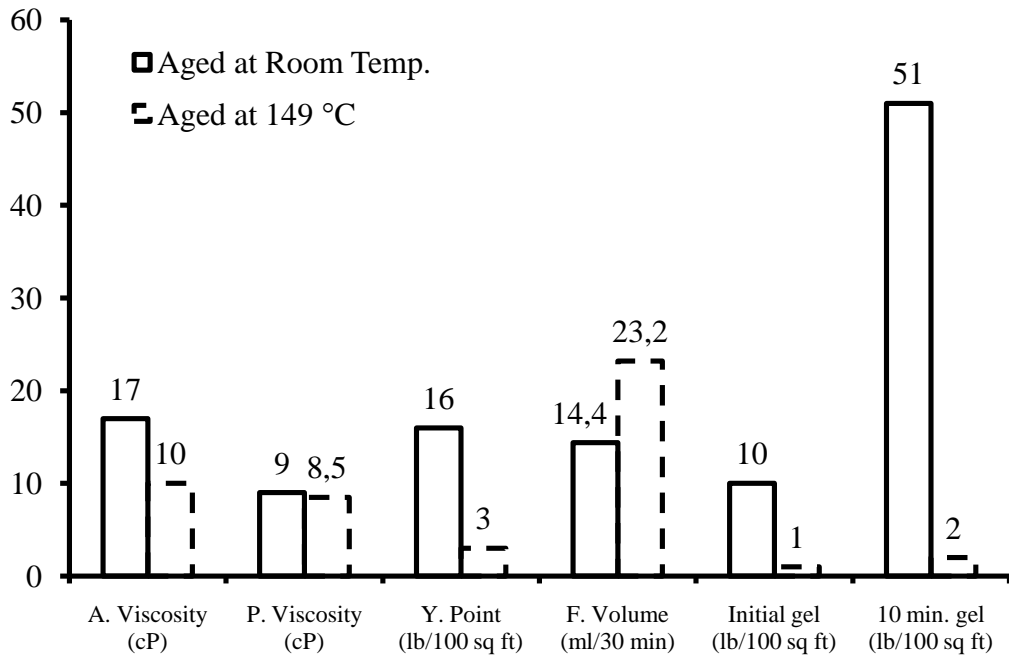


Figure 5-205 Aging of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated E-Ben & PAC Mud at 149 °C

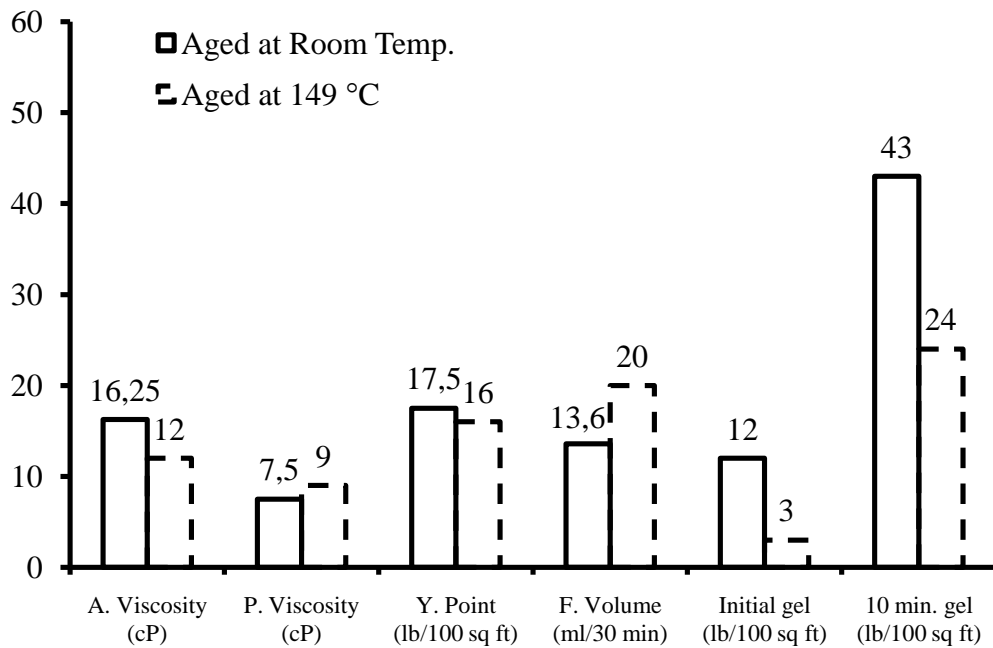


Figure 5-206 Aging of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated C-Ben & PAC Mud at 149 °C

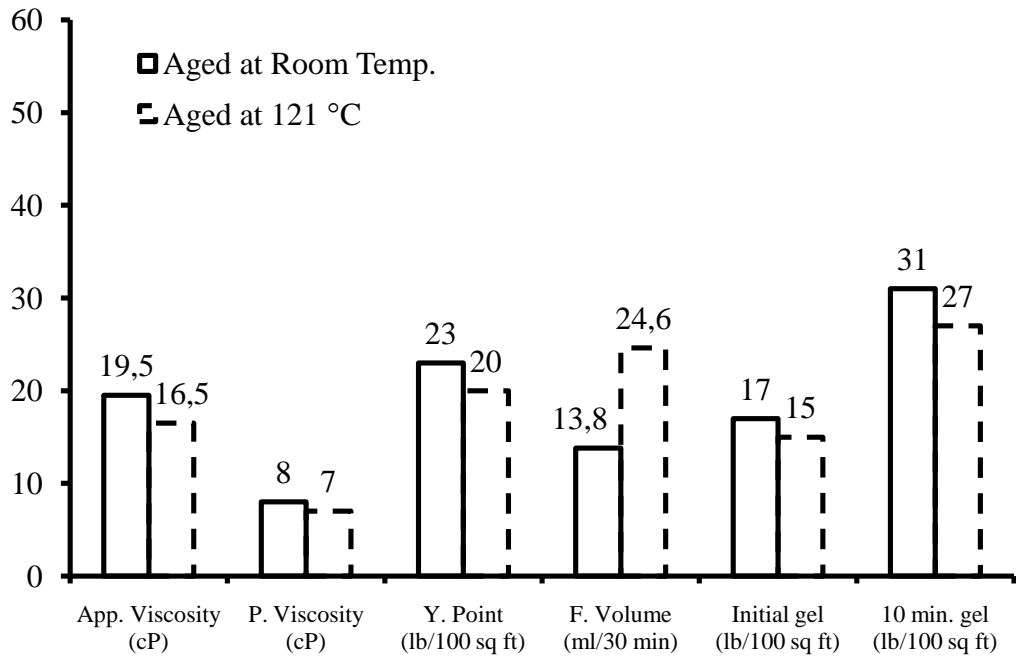


Figure 5-207 Aging of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated E-Ben & Xanthan Gum Mud at 121 °C

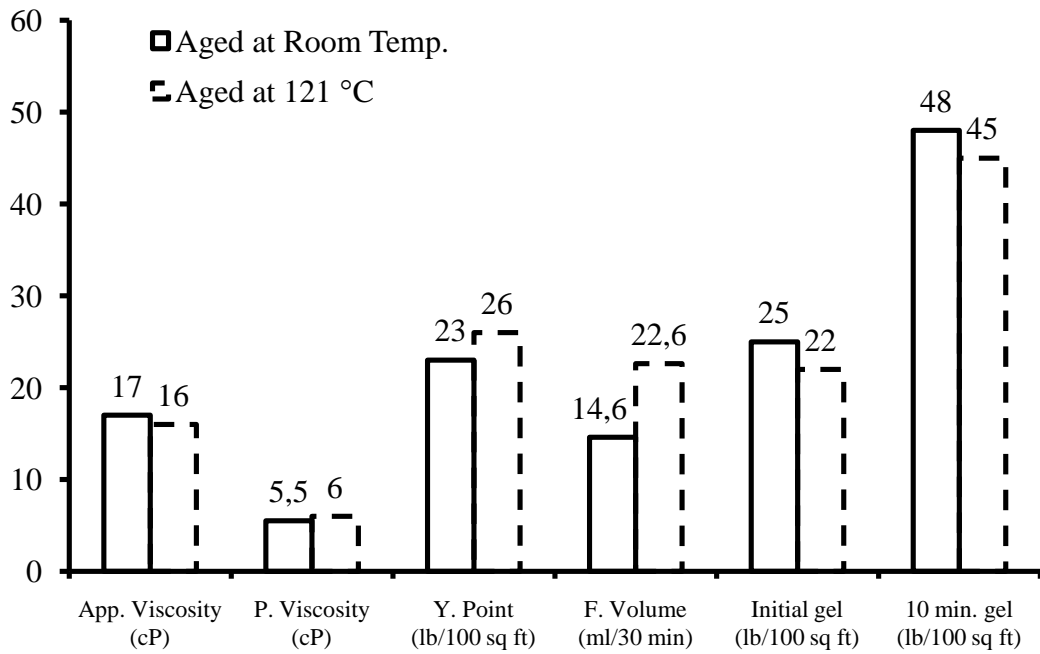


Figure 5-208 Aging of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated C-Ben & Xanthan Gum Mud at 121 °C



## CHAPTER 6

### CONCLUSIONS AND RECOMMENDATIONS

Based on the data and discussions presented in this study, the following conclusions can be drawn:

- 1- Rheological and filtrate properties of bentonites (E-Ben and C-Ben) were found to improve upon activation with  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ .
- 2- Slightly larger dosages were required for sodium Na-borate activation (3,78%  $\text{Na}_2\text{O}$  equivalent for E-Ben and 0,76%  $\text{Na}_2\text{O}$  equivalent for C-Ben) than Na-carbonate activation (2,90%  $\text{Na}_2\text{O}$  equivalent for E-Ben and 0,58%  $\text{Na}_2\text{O}$  equivalent for C-Ben).
- 3- Neither E-Ben nor C-Ben met the API requirements when activated with only  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  and the mixture of the two; polymer addition was deemed to be necessary.
- 4- The two bentonites tested were found to meet the drilling mud requirement after treating with polymers, namely CMC, PAC and xanthan gum.
- 5- In terms of aging at 36 °C for a 21-day period, it was determined that aging has no adverse effect on the rheological and filtrate properties of bentonite treated with CMC and PAC.
- 6- The rheological and filtrate properties of bentonites treated with xanthan gum were found to deteriorate upon aging. However E-Ben activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  and the mixture of  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  mixture showed a stable rheological properties. Presence of Na-borate in the mud appears to alleviate this adverse effect.
- 7- The deterioration of polymers present in the mud at high temperature (149 °C for CMC and PAC and 121 °C for xanthan gum) demonstrated that the rheological and filtrate properties of mud changed differently depending on the type of bentonites and activators.
- 8- Future studies for the effect of borates on drilling muds should focus on Na-bentonite to understand the mechanism involved in the behaviour of the Na-

borate added muds. The following studies could be undertaken to reach this end.

- The influence of borates on rheological properties of drilling muds made of Na-bentonite, only.
- The effects of borates on rheological properties of drilling muds under high temperature and pressure.
- The effects of borates on rheological properties of drilling muds in the presence of polymer (susceptible to bacterial degradation).

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APPENDIX A

(RHEOLOGICAL DATA FOR INORGANIC SALT ACTIVATION)

Table A-1 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Swelling of E-Ben

	<b>Na<sub>2</sub>CO<sub>3</sub> addition level; %Na<sub>2</sub>O equivalent</b>								
<b>%Na<sub>2</sub>O</b>	<b>0</b>	<b>0,58</b>	<b>1,16</b>	<b>1,74</b>	<b>2,32</b>	<b>2,90</b>	<b>3,48</b>	<b>4,06</b>	<b>4,64</b>
<b>Swelling (mL)</b>	8,5	11	18	22	34	40	29	24	23

Table A-2 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Swelling of C-Ben

	<b>Na<sub>2</sub>CO<sub>3</sub> addition level; %Na<sub>2</sub>O equivalent</b>								
<b>%Na<sub>2</sub>O</b>	<b>0</b>	<b>0,29</b>	<b>0,44</b>	<b>0,58</b>	<b>0,73</b>	<b>0,87</b>	<b>1,16</b>	<b>1,74</b>	
<b>Swelling (mL)</b>	29	33,5	36	41	39	38,5	36	28	

Table A-3 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Swelling of E-Ben

	<b>Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O addition level; %Na<sub>2</sub>O equivalent</b>								
<b>%Na<sub>2</sub>O</b>	<b>0</b>	<b>0,76</b>	<b>1,52</b>	<b>2,29</b>	<b>3,05</b>	<b>3,82</b>	<b>4,58</b>	<b>5,35</b>	
<b>Swelling (mL)</b>	8,5	11	14	17	22,5	36	35	35	

Table A-4 Effect of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Dissolved at 55 °C on the Swelling of E-Ben

	<b><math>\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}</math> addition level; %<math>\text{Na}_2\text{O}</math> equivalent</b>							
<b>%<math>\text{Na}_2\text{O}</math></b>	<b>0</b>	<b>3,02</b>	<b>3,40</b>	<b>3,78</b>	<b>4,15</b>	<b>4,53</b>	<b>4,90</b>	<b>5,66</b>
<b>Swelling (mL)</b>	8,5	22,5	27	34	30	29,5	29	27

Table A-5 Effect of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  Dissolved at Room Temperature on the Swelling of C-Ben

	<b><math>\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}</math> addition level; %<math>\text{Na}_2\text{O}</math> equivalent</b>						
<b>%<math>\text{Na}_2\text{O}</math></b>	<b>0</b>	<b>0,38</b>	<b>0,57</b>	<b>0,76</b>	<b>0,95</b>	<b>1,14</b>	<b>1,52</b>
<b>Swelling (mL)</b>	29	31	32,5	37,5	33,5	33	33

Table A-6 Effect of  $\text{Na}_2\text{CO}_3$  Dissolved at Room Temperature on the Rheology and Filtrate Properties of E-Ben

<b>Properties</b>	<b><math>\text{Na}_2\text{CO}_3</math> addition level; %<math>\text{Na}_2\text{O}</math> equivalent</b>								
	<b>0</b>	<b>0,58</b>	<b>1,16</b>	<b>1,74</b>	<b>2,32</b>	<b>2,90</b>	<b>3,48</b>	<b>4,06</b>	<b>4,64</b>
<b>AV</b>	1,5	2	2,5	3	4,5	6	3,25	2,75	2,75
<b>PV</b>	1	1,5	2	2,5	4	5	3	2,5	2,5
<b>YP</b>	1	1	1	1	1	2	0,5	0,5	0,5
<b>YP/PV</b>	1	0,4	0,5	0,4	0,25	0,4	0,17	0,2	0,2
<b>0-10 gel</b>	0,5-1	0,5-2	0,5-3	1-5	1-6	1-14	1-4	1-3	0,5-2
<b>pH</b>	7,5	8,5	9,0	9,3	9,7	10,0	10,1	10,3	10,3
<b>Filtrate V.</b>	111	53	30	24	23,4	20,4	26,8	27,6	29,6

Table A-7 Effect of Na<sub>2</sub>CO<sub>3</sub> Dissolved at Room Temperature on the Rheology and Filtrate Properties of C-Ben

Properties	Na <sub>2</sub> CO <sub>3</sub> addition level; %Na <sub>2</sub> O equivalent							
	0	0,29	0,44	0,58	0,73	0,87	1,16	1,74
<b>AV</b>	5,75	7,25	8	10,75	9,5	8,5	6,5	3
<b>PV</b>	3,5	4	4	4,5	4	3,5	4	2,5
<b>YP</b>	4,5	6,5	8	12,5	11	10	5	1
<b>YP/PV</b>	1,28	1,65	2	2,77	2,75	2,85	1,25	0,4
<b>0-10 Gel</b>	5-9	8-18	9-25	12-30	9-26	9-25	5-19	4-8
<b>pH</b>	8,5	8,8	9,1	9,4	9,3	9,5	9,7	10,0
<b>Filtrate V.</b>	30	24,8	23,8	22,8	23,2	23,8	26,0	28

Table A-8 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Rheology and Filtrate Properties of E-Ben

Properties	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O addition level; %Na <sub>2</sub> O equivalent							
	0	0,76	1,52	2,29	3,05	3,82	4,58	5,35
<b>AV</b>	1,5	2	2,5	3,5	4,75	6,75	6	5,5
<b>PV</b>	1	1	1,5	2,5	3,5	4,5	4	4
<b>YP</b>	1	2	2	2	2,5	4,5	4	3,5
<b>YP/PV</b>	1	2	0,75	0,8	0,71	1	1	0,875
<b>0-10 gel</b>	0,5-1	0,5-1	1-2,5	2-3	7-18	13-42	7-35	5-30
<b>pH</b>	7,5	9	9,2	9,2	9,2	9,3	9,3	9,3
<b>Filtrate V.</b>	108	90	83	72	34,4	22	23,8	24,8

Table A-9 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at 55 °C on the Rheology and Filtrate Properties of E-Ben

Properties	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O addition level; %Na <sub>2</sub> O equivalent							
	0	3,02	3,40	3,78	4,15	4,53	4,90	5,66
AV	1,5	3,75	4	4,75	4,25	4	4	3,75
PV	1	3	3	3	3	3	3	3
YP	1	1,5	2	3,5	2,5	2	2	1,5
YP/PV	1	0,5	0,66	1,66	0,83	0,66	0,66	0,5
0-10 Gel	0,5-1	4-8	4-11	5-15	4-15	3-12	2-11	1-10
pH	7,5	9,2	9,2	9,2	9,2	9,3	9,3	9,3
Filtrate V.	111	46,4	34	27	26,8	28,2	28,6	29,0

Table A-10 Effect of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Dissolved at Room Temperature on the Rheology and Filtrate Properties of C-Ben

Properties	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O addition level; %Na <sub>2</sub> O equivalent						
	0	0,38	0,57	0,76	0,95	1,14	1,52
AV	5,75	6,25	7	10	7,5	7	6,5
PV	3,5	3,5	3,5	4	3,5	3	3
YP	4,5	5,5	7	12	8	8	7
YP/PV	1,28	1,57	2,66	3	2,28	2,66	2,33
0-10 Gel	5-9	6-13	8-22	11-25	10-23	9-23	8-21
pH	8,2	8,7	9	9,1	9,2	9,2	9,2
Filtrate V.	30	29,2	28,2	25	25,4	26,2	26,4

Table A-11 Effect of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Dissolved at Room Temperature on the Rheology and Filtrate Properties of E-Ben

Properties	$\text{Na}_2\text{CO}_3/\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ addition level; % $\text{Na}_2\text{O}$ equivalent				
	0/3,82	0,73/2,86	1,45/1,91	2,18/0,95	2,90/0
<b>AV</b>	6,75	7	6	5	6
<b>PV</b>	4,5	4	3	3	5
<b>YP</b>	4,5	6	6	4	2
<b>YP/PV</b>	1,0	1,5	2,0	1,3	0,4
<b>0-10 gel</b>	13-42	14-48	11-38	2-14	1-14
<b>pH</b>	9,3	9,4	9,5	9,7	10,0
<b>Filtrate Volume</b>	22	21,8	21	21,2	20,4

Table A-12 Effect of  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Dissolved at Room Temperature on the Rheology and Filtrate Properties of C-Ben

Properties	$\text{Na}_2\text{CO}_3/\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ addition level; % $\text{Na}_2\text{O}$ equivalent				
	0/0,76	0,14/0,57	0,29/0,38	0,44/0,19	0,58/0
<b>AV</b>	10	9,5	11,5	10	10,75
<b>PV</b>	4	4	4,5	4	4,5
<b>YP</b>	12	11	14	12	12,5
<b>YP/PV</b>	3,0	2,8	3,1	3	2,8
<b>0-10 gel</b>	11-25	11-31	12-30	11-27	12-35
<b>pH</b>	9,1	9,2	9,2	9,2	9,2
<b>Filtrate Volume</b>	25	25,6	24	23,4	22,8

## APPENDIX B

### (RHEOLOGICAL DATA FOR THE EFFECT OF POLYMERS)

Table B-1 Effect of HV CMC on the Rheology and Filtrate Properties of E-Ben

<b>Properties</b>	<b>HV CMC (%)</b>			
	<b>0</b>	<b>2,5</b>	<b>5</b>	<b>7,5</b>
<b>AV</b>	1,5	5,5	11,25	15,5
<b>PV</b>	1	4,5	8,5	11,5
<b>YP</b>	1	2	5,5	8
<b>YP/PV</b>	1	0,4	0,6	0,7
<b>0-10 Gel</b>	0,5-1	0,5-1	1-2	1-2
<b>pH</b>	7,5	7,5	7,5	7,5
<b>Filtrate Volume</b>	111	26	16	14

Table B-2 Effect of PAC R on the Rheology and Filtrate Properties of E-Ben

<b>Properties</b>	<b>PAC R (%)</b>			
	<b>0</b>	<b>1,25</b>	<b>2,5</b>	<b>3,75</b>
<b>AV</b>	1,5	5,5	11,25	16
<b>PV</b>	1	4,5	8	10,5
<b>YP</b>	1	2	6,5	11
<b>YP/PV</b>	1	0,4	0,8	1
<b>0-10 gel</b>	0,5-1	0,5-1	1-1,5	1,5-2
<b>pH</b>	7,5	7,5	7,5	7,5
<b>Filtrate Volume</b>	111	24,8	16,4	15

Table B-3 Effect of Xanthan Gum on the Rheology and Filtrate Properties of E-Ben

<b>Properties</b>	<b>Xanthan Gum (%)</b>			
	<b>0</b>	<b>2,5</b>	<b>5</b>	<b>7,5</b>
<b>AV</b>	1,5	7,75	13,25	19,5
<b>PV</b>	1	5	7	8
<b>YP</b>	1	5,5	12,5	23
<b>YP/PV</b>	1	1,1	1,8	2,9
<b>0-10 gel</b>	0,5-1	3-3	7-9	11-16
<b>pH</b>	7,5	7,5	7,5	7,5
<b>Filtrate Volume</b>	111	21,6	16	13,6

Table B-4 Rheological and Filtrate Properties of Polymers Used in the Treatment of E-Ben

<b>Properties</b>	<b>7,5% HV CMC</b>	<b>3,75% PAC R</b>	<b>7,5% Xant. Gum</b>
<b>AV</b>	12,75	17	23,5
<b>PV</b>	10,5	10	13,5
<b>YP</b>	4,5	14	21
<b>YP/PV</b>	0,4	1,4	1,6
<b>0-10 gel</b>	1-2	1,5-2	11-11
<b>pH</b>	6,5	6,5	6,0
<b>Filtrate Volume</b>	28,0	High	23,2

Table B-5 Effect of HV CMC on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

<b>Properties</b>	<b>HV CMC (%)</b>						
	<b>0</b>	<b>0,2</b>	<b>0,4</b>	<b>0,6</b>	<b>0,8</b>	<b>1</b>	<b>1,2</b>
<b>AV</b>	6	8	9,25	10,25	12	13,25	15,5
<b>PV</b>	5	5,5	6,5	7	7,5	8	9
<b>YP</b>	2	5	5,5	7	9	10,5	13
<b>YP/PV</b>	0,4	0,9	0,8	1	1,2	1,3	1,4
<b>0-10 gel</b>	1-14	2-23	2-26	3-31	4-33	5-36	6-40
<b>pH</b>	10,0	10,0	10,0	10,0	10,0	10,0	10,0
<b>Filtrate V.</b>	20,4	19,2	18,4	17,2	16,2	15,8	14,8



Table B-6 Effect of PAC R on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

	<b>PAC R (%)</b>					
<b>Properties</b>	<b>0</b>	<b>0,2</b>	<b>0,4</b>	<b>0,6</b>	<b>0,8</b>	<b>1</b>
<b>AV</b>	6	8,5	10,5	12	13,25	15
<b>PV</b>	5	6	7	8	8,5	9
<b>YP</b>	2	5	7	8	9,5	12
<b>YP/PV</b>	0,4	0,8	1	1	1,1	1,3
<b>0-10 gel</b>	1-14	2-20	3-25	3-26	3-28	4-30
<b>pH</b>	10,0	10,0	10,0	10,0	10,0	10,0
<b>Filtrate Volume</b>	20,4	17,2	16,2	15,4	15,2	14,8

Table B-7 Effect of Xanthan Gum on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

	<b>Xanthan Gum (%)</b>			
<b>Properties</b>	<b>0</b>	<b>1</b>	<b>2</b>	<b>3</b>
<b>AV</b>	6	9,5	13,25	17
<b>PV</b>	5	6	7	9
<b>YP</b>	2	7	12,5	16
<b>YP/PV</b>	0,4	1,2	1,8	1,8
<b>0-10 gel</b>	1-14	3-15	5-16	7-18
<b>pH</b>	10,0	10,0	10,0	10,0
<b>Filtrate Volume</b>	20,4	17,2	15,6	14

Table B-8 Rheological and Filtrate Properties of Polymers Used in the Treatment of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

Properties	1,2% HV CMC	1,0% PAC R	3,0% Xant. Gum
AV	3,5	6	9,75
PV	3	4	6
YP	1	4	7,5
YP/PV	0,33	1	1,3
0-10 gel	½-1	½-1	3-3
pH	6,5	6,5	6,0
Filtrate Volume	70,8	High	32

Table B-9 Effect of HV CMC on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O

Properties	HV CMC (%)							
	0	0,4	0,8	1,2	1,6	2	2,4	2,8
AV	5	6	7,5	8,5	10	11,75	14	16,25
PV	3	3,5	4,5	5	6	6,5	8	8,5
YP	4	5	6	7	8	10,5	12	15,5
YP/PV	1,3	1,4	1,3	1,4	1,3	1,6	1,5	1,8
0-10 gel	5-16	5-20	6-22	7-24	8-28	9-31	10-32	13-34
pH	9,2	9,2	9,2	9,2	9,2	9,2	9,2	9,2
Filtrate V.	27,2	24,4	20,4	18,6	17	16,4	15,6	14,6

Table B-10 Effect of PAC R on the Rheology and Filtrate Properties of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

	<b>PAC R (%)</b>					
<b>Properties</b>	<b>0</b>	<b>0,4</b>	<b>0,8</b>	<b>1,2</b>	<b>1,6</b>	<b>1,8</b>
<b>AV</b>	5	7	9,5	13,25	15,5	17
<b>PV</b>	3	4	5	6,5	7,5	8
<b>YP</b>	4	6	9	13,5	16	18
<b>YP/PV</b>	1,3	1,5	1,8	2,0	2,1	2,3
<b>0-10 gel</b>	5-16	7-24	9-30	12-34	13-37	14-38
<b>pH</b>	9,2	9,2	9,2	9,2	9,2	9,2
<b>Filtrate V.</b>	27,2	20	16	15,6	15,2	14,8

Table B-11 Effect of Xanthan Gum on the Rheology and Filtrate Properties of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

	<b>Xanthan Gum (%)</b>			
<b>Properties</b>	<b>0</b>	<b>2</b>	<b>4</b>	<b>4,5</b>
<b>AV</b>	5	12,5	18,5	19,5
<b>PV</b>	3	6	7,5	8
<b>YP</b>	4	13	22	23
<b>YP/PV</b>	1,3	2,2	2,9	2,9
<b>0-10 gel</b>	5-16	13-23	17-29	18-31
<b>pH</b>	9,2	9,2	9,2	9,2
<b>Filtrate Volume</b>	27,2	19,2	15,6	13,8

Table B-12 Rheological and Filtrate Properties of Polymers Used in the Treatment of E-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O

<b>Properties</b>	<b>2,8% HV CMC</b>	<b>1,8% PAC R</b>	<b>4,5% Xant. Gum</b>
<b>AV</b>	5,5	10	12,5
<b>PV</b>	4,5	5,5	8
<b>YP</b>	2	9	9
<b>YP/PV</b>	0,44	1,6	1,1
<b>0-10 gel</b>	½-1	½-1	7-7
<b>pH</b>	6,5	6,5	6,0
<b>Filtrate Volume</b>	51,6	High	30,4

Table B-13 Effect of HV CMC on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

<b>Properties</b>	<b>HV CMC (%)</b>				
	<b>0</b>	<b>0,5</b>	<b>1</b>	<b>1,5</b>	<b>2</b>
<b>AV</b>	5	7,5	10,5	13	16
<b>PV</b>	3	5	7	8	9
<b>YP</b>	4	5	7	10	14
<b>YP/PV</b>	1,3	1,0	1,0	1,3	1,5
<b>0-10 gel</b>	2-14	4-23	6-34	10-40	11-45
<b>pH</b>	9,4	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	21,2	18,4	17,2	16,2	14,8

Table B-14 Effect of PAC R on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

	<b>PAC R (%)</b>			
<b>Properties</b>	<b>0</b>	<b>0,5</b>	<b>1</b>	<b>1,5</b>
<b>AV</b>	5	9,5	13,5	17
<b>PV</b>	3	5,5	7,5	9
<b>YP</b>	4	8	12	16
<b>YP/PV</b>	1,3	1,5	1,6	1,8
<b>0-10 gel</b>	2-14	4-33	6-45	10-51
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	21,2	18	15,6	14,4

Table B-15 Effect of Xanthan Gum on the Rheology and Filtrate Properties of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

	<b>Xanthan Gum (%)</b>			
<b>Properties</b>	<b>0</b>	<b>1,5</b>	<b>3</b>	<b>3,5</b>
<b>AV</b>	5	11	16,5	19
<b>PV</b>	3	6	8	9
<b>YP</b>	4	10	17	20
<b>YP/PV</b>	1,3	1,7	2,1	2,2
<b>0-10 gel</b>	2-14	6-21	11-26	12-29
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	21,2	17,6	15,2	14,4

Table B-16 Rheological and Filtrate Properties of Polymers Used in the Treatment of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O mixture

<b>Properties</b>	<b>2,0% HV CMC</b>	<b>1,5% PAC R</b>	<b>3,5% Xant. Gum</b>
<b>AV</b>	4,5	8,75	10
<b>PV</b>	3,5	5,5	6,5
<b>YP</b>	2	6,5	7
<b>YP/PV</b>	0,57	1,2	1,1
<b>0-10 gel</b>	½-1	1-1	4-4
<b>pH</b>	6,5	6,5	6,0
<b>Filtrate Volume</b>	62	High	31,2

Table B-17 Effect of HV CMC on the Rheology and Filtrate Properties of C-Ben

<b>Properties</b>	<b>HV CMC (%)</b>			
	<b>0</b>	<b>1</b>	<b>2</b>	<b>3</b>
<b>AV</b>	5,75	9,5	13,5	20
<b>PV</b>	3,5	6,5	9	11
<b>YP</b>	4,5	6	9	18
<b>YP/PV</b>	1,3	0,9	1	1,6
<b>0-10 gel</b>	5-9	7-26	10-36	15-46
<b>pH</b>	8,5	8,5	8,5	8,5
<b>Filtrate Volume</b>	30	19,2	15,6	14,0

Table B-18 Effect of PAC R on the Rheology and Filtrate Properties of C-Ben

<b>Properties</b>	<b>PAC R (%)</b>				
	<b>0</b>	<b>0,5</b>	<b>1</b>	<b>1,5</b>	<b>2</b>
<b>AV</b>	5,75	9	12,5	15,5	19,75
<b>PV</b>	3,5	6,5	8	9	10,5
<b>YP</b>	4,5	5	9	13	18,5
<b>YP/PV</b>	1,3	0,8	1,1	1,4	1,8
<b>0-10 gel</b>	5-9	6-30	7-35	8-41	11-44
<b>pH</b>	8,5	8,5	8,5	8,5	8,5
<b>Filtrate Volume</b>	30	21	17	15,6	13,4

Table B-19 Effect of Xanthan Gum on the Rheology and Filtrate Properties of C-Ben

<b>Properties</b>	<b>Xanthan Gum (%)</b>			
	<b>0</b>	<b>1</b>	<b>2</b>	<b>2,5</b>
<b>AV</b>	5,75	9	14,5	17,75
<b>PV</b>	3,5	6	6,5	6,5
<b>YP</b>	4,5	6	18,5	22,5
<b>YP/PV</b>	1,3	1	2,9	3,5
<b>0-10 gel</b>	5-9	8-14	12-17	14-20
<b>pH</b>	8,5	8,5	8,5	8,5
<b>Filtrate Volume</b>	30	21,2	15,2	14

Table B-20 Rheological and Filtrate Properties of Polymers Used in the Treatment of C-Ben

<b>Properties</b>	<b>3,0% HV CMC</b>	<b>2,0% PAC R</b>	<b>2,5% Xant. Gum</b>
<b>AV</b>	5,75	9,5	7,75
<b>PV</b>	4,5	6	5,5
<b>YP</b>	2,5	7	4,5
<b>YP/PV</b>	0,5	1,2	0,8
<b>0-10 gel</b>	½-1	½-1	3-3
<b>pH</b>	6,5	6,5	6,0
<b>Filtrate Volume</b>	48	High	35

Table B-21 Effect of HV CMC on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

<b>Properties</b>	<b>HV CMC (%)</b>			
	<b>0</b>	<b>0,2</b>	<b>0,4</b>	<b>0,6</b>
<b>AV</b>	10,75	14,5	18,75	23,5
<b>PV</b>	4,5	5	5,5	7
<b>YP</b>	12,5	19	26,5	33
<b>YP/PV</b>	2,7	3,8	4,8	4,7
<b>0-10 gel</b>	12-30	13-21	14-18	14-18
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	22,8	20,6	17,6	16,8



Table B-22 Effect of PAC R on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

	<b>PAC R (%)</b>			
<b>Properties</b>	<b>0</b>	<b>0,1</b>	<b>0,2</b>	<b>0,3</b>
<b>AV</b>	10,75	16,25	19,5	24
<b>PV</b>	4,5	5	5,5	6
<b>YP</b>	12,5	22,5	28,5	36
<b>YP/PV</b>	2,7	4,5	5,2	6
<b>0-10 gel</b>	12-30	13-18	13-18	15-18
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	22,8	20	17,6	17,2

Table B-23 Effect of Xanthan Gum on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

	<b>Xanthan Gum (%)</b>			
<b>Properties</b>	<b>0</b>	<b>0,2</b>	<b>0,4</b>	<b>0,6</b>
<b>AV</b>	10,75	12	14	15,5
<b>PV</b>	4,5	4,5	5	5,5
<b>YP</b>	12,5	15,5	18	20
<b>YP/PV</b>	2,7	3,4	3,6	3,6
<b>0-10 gel</b>	12-30	16-35	21-40	24-44
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	22,8	21,2	20,4	19,6

Table B-24 Effect of HV CMC and LV CMC on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

<b>HV CMC (%)</b>	<b>1,25</b>	<b>0,80</b>	<b>0,70</b>	<b>0,60</b>
<b>LV CMC (%)</b>	<b>0,75</b>	<b>0,40</b>	<b>0,35</b>	<b>0,30</b>
<b>AV</b>	18	18	18,5	18,5
<b>PV</b>	9	8	8	8
<b>YP</b>	18	20	21	21
<b>YP/PV</b>	2	2,5	2,6	2,6
<b>0-10 gel</b>	16-52	17-37	16-28	16-28
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	12,4	14	14,4	16

Table B-25 Effect of PAC R and PAC L on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

<b>PAC R (%)</b>	<b>0,70</b>	<b>0,50</b>	<b>0,40</b>	<b>0,40</b>
<b>PAC L (%)</b>	<b>0,85</b>	<b>0,30</b>	<b>0,30</b>	<b>0,20</b>
<b>AV</b>	16,75	16,5	16	19,5
<b>PV</b>	10,5	7,5	6,5	9
<b>YP</b>	12,5	18	19	21
<b>YP/PV</b>	1,2	2,4	2,9	2,3
<b>0-10 gel</b>	14-48	19-55	15-41	18-30
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	12,4	14,0	14,8	16

Table B-26 Effect of Xanthan Gum and Dextrid on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

<b>Xant. Gum (%)</b>	<b>0,4</b>	<b>0,6</b>	<b>0,8</b>	<b>0,8</b>
<b>Dextrid (%)</b>	<b>1,6</b>	<b>2,0</b>	<b>2,2</b>	<b>2,4</b>
<b>AV</b>	14	15,25	18	18,5
<b>PV</b>	5	5	5,5	6
<b>YP</b>	18	20,5	25	25
<b>YP/PV</b>	3,6	4,1	4,5	4,1
<b>0-10 gel</b>	19-31	20-34	24-42	22-40
<b>pH</b>	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	16,6	16,4	15,6	15,2

Table B-27 Rheological and Filtrate Properties of Polymers Used in the Treatment of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>

<b>Properties</b>	0,80% HV CMC 0,40% LV CMC	0,50% PAC R 0,30% PAC L	0,8% Xant. Gum 2,4% Dextrid
<b>AV</b>	2,5	4	2,75
<b>PV</b>	2	3	2,5
<b>YP</b>	1	2	0,5
<b>YP/PV</b>	0,5	0,66	0,2
<b>0-10 gel</b>	½-1	½-1	½-½
<b>pH</b>	6,5	6,5	6,0
<b>Filtrate Volume</b>	High	High	24

Table B-28 Effect of HV CMC on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

<b>Properties</b>	<b>HV CMC (%)</b>			
	<b>0</b>	<b>0,2</b>	<b>0,4</b>	<b>0,6</b>
<b>AV</b>	10	12,5	14,5	17,5
<b>PV</b>	4	4,5	5	6
<b>YP</b>	12	16	19	23
<b>YP/PV</b>	3	3,5	3,8	3,8
<b>0-10 gel</b>	11-25	14-20	14-20	14-20
<b>pH</b>	9,1	9,1	9,1	9,1
<b>Filtrate Volume</b>	25	21	19,2	17,4

Table B-29 Effect of PAC R on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

<b>Properties</b>	<b>PAC R (%)</b>			
	<b>0</b>	<b>0,1</b>	<b>0,2</b>	<b>0,3</b>
<b>AV</b>	10	13	16	20,5
<b>PV</b>	4	5	5	5
<b>YP</b>	12	16	23	31
<b>YP/PV</b>	3,0	3,2	4,6	6,2
<b>0-10 gel</b>	11-25	13-18	13-17	13-16
<b>pH</b>	9,1	9,1	9,1	9,1
<b>Filtrate Volume</b>	25	22	19,6	17,2

Table B-30 Effect of Xanthan Gum on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

<b>Properties</b>	<b>Xanthan Gum (%)</b>			
	<b>0</b>	<b>0,3</b>	<b>0,6</b>	<b>0,9</b>
<b>AV</b>	10	12	14	16
<b>PV</b>	4	4	4	4,5
<b>YP</b>	12	16	20	23
<b>YP/PV</b>	3	4	5	5,1
<b>0-10 gel</b>	11-25	18-35	24-47	27-52
<b>pH</b>	9,1	9,1	9,1	9,1
<b>Filtrate Volume</b>	25	21,4	20	18,8

Table B-31 Effect of HV CMC and LV CMC on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$

<b>HV CMC (%)</b>	<i>1,30</i>	<i>1,00</i>	<i>0,80</i>	<i>0,70</i>
<b>LV CMC (%)</b>	<i>0,50</i>	<i>0,40</i>	<i>0,40</i>	<i>0,30</i>
<b>AV</b>	16,5	15	15,5	15
<b>PV</b>	8	7	7	7
<b>YP</b>	17	16	17	16
<b>YP/PV</b>	2,12	2,25	2,4	2,29
<b>0-10 gel</b>	15-47	17-43	16-38	14-34
<b>pH</b>	9,1	9,1	9,1	9,1
<b>Filtrate Volume</b>	14	13,6	15	15,6

Table B-32 Effect of PAC R and PAC L on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O

<b>PAC R (%)</b>	<b>0,85</b>	<b>0,60</b>	<b>0,50</b>	<b>0,45</b>
<b>PAC L (%)</b>	<b>0,60</b>	<b>0,30</b>	<b>0,30</b>	<b>0,25</b>
<b>AV</b>	17	16	15	15,5
<b>PV</b>	9	8	7	7
<b>YP</b>	16	16	16	17
<b>YP/PV</b>	1,8	2	2,3	2,4
<b>0-10 gel</b>	17-62	17-48	17-45	17-42
<b>pH</b>	9,1	9,1	9,1	9,1
<b>Filtrate Volume</b>	12,2	14	14,4	16

Table B-33 Effect of Xanthan Gum and Dextrid on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O

<b>Xant. Gum (%)</b>	<b>0,4</b>	<b>0,6</b>	<b>0,8</b>	<b>0,8</b>
<b>Dextrid (%)</b>	<b>1,6</b>	<b>2,0</b>	<b>2,2</b>	<b>2,4</b>
<b>AV</b>	13	15	15,75	16
<b>PV</b>	5	5,5	5,5	6
<b>YP</b>	16	19	20,5	22
<b>YP/PV</b>	3,2	3,5	3,7	3,7
<b>0-10 gel</b>	20-41	22-46	21-47	24-48
<b>pH</b>	9,1	9,1	9,1	9,1
<b>Filtrate Volume</b>	16,8	16,6	16,0	15,6

Table B-34 Effect of HV CMC on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

Properties	HV CMC (%)			
	0	0,2	0,4	0,6
AV	11,5	13,75	15,5	18
PV	4,5	5	5	5,5
YP	14	17,5	21	25
YP/PV	3,1	3,5	4,2	4,5
0-10 gel	12-30	12-28	13-29	13-30
pH	9,2	9,2	9,2	9,2
Filtrate Volume	24	20,6	18,8	17,2

Table B-35 Effect of PAC R on the Rheology and Filtrate Properties of C-Ben Activated with  $\text{Na}_2\text{CO}_3\text{-Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture

Properties	PAC R (%)			
	0	0,1	0,2	0,3
AV	11,5	14,5	16	18
PV	4,5	5,5	5,5	6
YP	14	18,5	21	24
YP/PV	3,1	3,4	3,8	4
0-10 gel	12-30	13-29	13-30	13-30
pH	9,2	9,2	9,2	9,2
Filtrate Volume	24	21,8	19,4	16,8

Table B-36 Effect of Xanthan Gum on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

Properties	Xanthan Gum (%)			
	0	0,3	0,6	0,9
AV	11,5	12,5	14	16
PV	4,5	4,5	5	5,5
YP	14	16	18	21
YP/PV	3,1	3,6	3,6	3,8
0-10 gel	12-30	16-35	19-39	22-42
pH	9,2	9,2	9,2	9,2
Filtrate Volume	24	21,8	20,6	19,2

Table B-37 Effect of HV CMC and LV CMC on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

HV CMC (%)	0,7	0,8	0,9	1,0
LV CMC (%)	0,5	0,5	0,6	0,7
AV	15	16,5	16,5	16,75
PV	6	7	6,5	7,5
YP	18	19	20	18,5
YP/PV	3	2,7	3	2,5
0-10 gel	12-30	12-36	12-38	12-37
pH	9,2	9,2	9,2	9,2
Filtrate Volume	16,2	16	15	14



Table B-38 Effect of PAC R on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

<b>PAC R (%)</b>	<b>0,50</b>	<b>0,50</b>	<b>0,60</b>	<b>0,70</b>
<b>PAC L (%)</b>	<b>0,30</b>	<b>0,40</b>	<b>0,50</b>	<b>0,50</b>
<b>AV</b>	16,5	15,5	16,25	17
<b>PV</b>	7	7	7,5	8
<b>YP</b>	19	17	17,5	18
<b>YP/PV</b>	2,7	2,4	2,3	2,3
<b>0-10 gel</b>	12-43	12-46	12-48	13-56
<b>pH</b>	9,2	9,2	9,2	9,2
<b>Filtrate Volume</b>	16	15,2	13,6	13

Table B-39 Effect of Xanthan Gum and Dextrid on the Rheology and Filtrate Properties of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

<b>Xant. Gum (%)</b>	<b>0,4</b>	<b>0,6</b>	<b>0,8</b>	<b>0,8</b>
<b>Dextrid (%)</b>	<b>1,6</b>	<b>2,0</b>	<b>2,2</b>	<b>2,4</b>
<b>AV</b>	13	15	16,75	18
<b>PV</b>	5	5,5	5,5	6
<b>YP</b>	16	19	22,5	24
<b>YP/PV</b>	3,2	3,5	4,1	4
<b>0-10 gel</b>	18-40	20-45	24-48	25-48
<b>pH</b>	9,2	9,2	9,2	9,2
<b>Filtrate Volume</b>	16,0	15,2	14,8	14,6

Table B-40 Rheological and Filtrate Properties of Polymers Used in the Treatment of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture

<b>Properties</b>	1,00% HV CMC 0,70% LV CMC	0,60% PAC R 0,50% PAC L	0,8% Xant. Gum 2,4% Dextrid
<b>AV</b>	3	4,75	2,75
<b>PV</b>	2,5	3	2,5
<b>YP</b>	1	3,5	0,5
<b>YP/PV</b>	0,4	1,2	0,2
<b>0-10 gel</b>	½-1	½-1	½-½
<b>pH</b>	6,5	6,5	6,0
<b>Filtrate Volume</b>	92	High	24

APPENDIX C

(RHEOLOGICAL DATA FOR AGING AT 36 °C)

Table C-1 Aging of API Bentonite

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	15,25	15,50	15,25	15,25	15,50
<b>PV</b>	10	10	10	10	10
<b>YP</b>	10,5	11	10,5	10,5	11
<b>YP/PV</b>	1,1	1,1	1,1	1,1	1,1
<b>0-10 gel</b>	12-28	10-28	8-26	8-29	10-26
<b>pH</b>	9,0	9,0	9,0	9,0	9,0
<b>Filtrate Volume</b>	11,2	11,6	11,4	11,2	11,2

Table C-2 Aging of Reşadiye Bentonite

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	19,5	19,5	19,5	20	20,25
<b>PV</b>	15,5	15	15,5	16	16
<b>YP</b>	8	9	8	8	8,5
<b>YP/PV</b>	0,5	0,6	0,5	0,5	0,5
<b>0-10 gel</b>	1-2	1-2	1-2	1-2	1-2
<b>pH</b>	9,7	9,7	9,7	9,7	9,7
<b>Filtrate Volume</b>	12,4	12,4	12,6	12,8	12,4

Table C-3 Aging of 7,5% HV CMC

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	11,25	11,5	11,5	12	12,5
<b>PV</b>	9,5	9,5	9,5	9,5	10
<b>YP</b>	3,5	4	4	5	5
<b>YP/PV</b>	0,4	0,4	0,4	0,5	0,5
<b>0-10 gel</b>	1/2-1	1/2-1	1/2-1	1/2-1	1/2-1
<b>pH</b>	6,5	6,5	6,5	6,5	6,5
<b>Filtrate Volume</b>	28,4	28,6	28,6	29	30

Table C-4 Aging of 3,75% PAC R

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	17,25	17,5	18	18,25	18
<b>PV</b>	10	10	11	11	11
<b>YP</b>	14,5	15	14	14,5	14
<b>YP/PV</b>	1,5	1,5	1,3	1,3	1,3
<b>0-10 gel</b>	1,5-2	1,5-2	1,5-2	1,5-2	1,5-2
<b>pH</b>	6,5	6,5	6,5	6,5	6,5
<b>Filtrate Volume</b>	High	High	High	High	High

Table C-5 Aging of 7,5% Xanthan Gum

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	19,5	15	12	8,5	2
<b>PV</b>	10,5	5,5	5	5	1
<b>YP</b>	18	19	15	9	2
<b>YP/PV</b>	1,7	3,5	3	1,8	2
<b>0-10 gel</b>	11-11	11-11	7-7	3-3	½-½
<b>pH</b>	6,0	5,5	5,0	4,8	4,7
<b>Filtrate Volume</b>	24,2	High	High	High	High

Table C-6 Aging of 3% Xanthan Gum

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	8	6,5	5,5	5,5	3
<b>PV</b>	4,5	4,5	4	4	2
<b>YP</b>	7	5	3	3	2
<b>YP/PV</b>	1,6	1,1	0,75	0,75	1
<b>0-10 gel</b>	3-3	2-3	½-1	½-1	0-0
<b>pH</b>	6,0	5,3	5,1	4,7	4,6
<b>Filtrate Volume</b>	33	High	High	High	High

Table C-7 Aging of 4,5% Xanthan Gum

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	11	9	8	7	2
<b>PV</b>	6,5	4	4	4	1
<b>YP</b>	9	10	8	6	2
<b>YP/PV</b>	1,4	2,5	2	1,5	2
<b>0-10 gel</b>	6-6	2-3	2-3	2-2	0-0
<b>pH</b>	6,0	5,4	5,0	4,8	4,7
<b>Filtrate Volume</b>	30,6	High	High	High	High

Table C-8 Aging of 3,5% Xanthan Gum

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	8	7	6	5,5	3
PV	5	3,5	4	4	2
YP	6	7	4	3	2
YP/PV	1,2	2	1	0,75	1
0-10 gel	4-4	2-3	2-3	1-1	0-0
pH	6,0	5,3	4,9	4,7	4,6
Filtrate Volume	31,4	High	High	High	High

Table C-9 Aging of 2,5% Xanthan Gum

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	6	5,75	3	2,75	2
PV	5	4,5	1,5	2	2,5
YP	2	2,5	3	1,5	1
YP/PV	0,4	0,6	2	0,75	0,4
0-10 gel	2-2	2-2	½-½	½-½	½-½
pH	6,0	5,4	5,0	4,7	4,7
Filtrate Volume	36	High	High	High	High

Table C-10 Aging of 0,8% Xanthan Gum and 2,4% Dextrans

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	2,5	2,25	1,25	1	1
<b>PV</b>	2	2	1,5	1	1
<b>YP</b>	1	0,5	0,5	0	0
<b>YP/PV</b>	0,5	0,25	0,33	0	0
<b>0-10 gel</b>	0-1/2	0-0	0-0	0-0	0-0
<b>pH</b>	6,0	5,4	5,0	4,8	4,7
<b>Filtrate Volume</b>	26	High	High	High	High

Table C-11 Aging of E-Ben & HV CMC Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	15,25	16	16,25	16	15,5
<b>PV</b>	12	12	12	12	12
<b>YP</b>	6,5	8	8,5	8	7
<b>YP/PV</b>	0,5	0,7	0,7	0,7	0,6
<b>0-10 gel</b>	1,5-2	1,5-2	1,5-2	1-1,5	1-1,5
<b>pH</b>	7,5	7,5	7,5	7,5	7,5
<b>Filtrate Volume</b>	14,2	14,2	14,4	14,4	14,8



Table C-12 Aging of C-Ben & HV CMC Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	19	22	21	20	20,5
<b>PV</b>	11,5	12	11	11,5	11
<b>YP</b>	15	20	20	18	19
<b>YP/PV</b>	1,3	1,7	1,8	1,5	1,7
<b>0-10 gel</b>	13-46	15-47	14-45	16-47	16-47
<b>pH</b>	8,2	8,2	8,2	8,2	8,2
<b>Filtrate Volume</b>	14,2	14,4	14,6	14,4	14,6

Table C-13 Aging of E-Ben & PAC R Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	16	15	15,5	14,75	14,25
<b>PV</b>	10,5	10,5	10,5	10	9,5
<b>YP</b>	11	9	10	9,5	9,5
<b>YP/PV</b>	1	0,9	1	0,8	1
<b>0-10 gel</b>	1,5-2	1,5-2	1,5-2	1,5-2	1-1,5
<b>pH</b>	7,5	7,5	7,5	7,5	7,5
<b>Filtrate Volume</b>	15	15,4	15,6	15,8	15,8

Table C-14 Aging of C-Ben & PAC R Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	19,5	21,5	20,25	20,75	21,25
<b>PV</b>	11	12	11	11,5	11,5
<b>YP</b>	17	19	17,50	18,5	19,5
<b>YP/PV</b>	1,5	1,6	1,6	1,6	1,7
<b>0-10 gel</b>	10-50	13-52	13-53	14-53	14-54
<b>pH</b>	8,2	8,2	8,2	8,2	8,2
<b>Filtrate Volume</b>	13,6	13,6	13,8	13,8	13,6

Table C-15 Aging of E-Ben & Xanthan Gum Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	20,5	17,25	13,75	12	9,5
<b>PV</b>	9,5	7,5	7,5	7	6
<b>YP</b>	21,5	19,5	12,5	10	7
<b>YP/PV</b>	2,3	2,6	1,7	1,4	1,2
<b>0-10 gel</b>	12-16	9-12	7-9	5-7	3-4
<b>pH</b>	7,5	7,0	6,4	6,0	5,8
<b>Filtrate Volume</b>	13,8	14,4	15,6	16,8	17,6

Table C-16 Aging of C-Ben & Xanthan Gum Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	17,25	20	15	12	9,5
<b>PV</b>	5,5	7,5	5,5	4	3
<b>YP</b>	23,5	25	19	16	13
<b>YP/PV</b>	4,3	3,3	3,5	4	4,3
<b>0-10 gel</b>	15-19	16-23	14-20	15-26	12-24
<b>pH</b>	8,2	8,0	7,8	7,5	7,2
<b>Filtrate Volume</b>	14,2	14,8	19,6	25,2	32

Table C-17 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & HV CMC Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	17,5	18,75	19,25	19	19
<b>PV</b>	10	10	10,5	10	10
<b>YP</b>	15	17,5	17,5	18	18
<b>YP/PV</b>	1,5	1,7	1,6	1,8	1,8
<b>0-10 gel</b>	11-48	11-49	8-53	9-52	10-48
<b>pH</b>	10,0	10,0	10,0	10,0	10,0
<b>Filtrate Volume</b>	14,8	15	15,4	15,6	15,4

Table C-18 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & HV CMC and LV CMC Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	18	20	19,25	20,25	19,5
PV	8	9	8,5	9	8,5
YP	20	22	22,5	22,5	22
YP/PV	2,5	2,4	2,6	2,5	2,6
0-10 gel	20-48	20-50	23-49	24-49	23-51
pH	9,4	9,4	9,4	9,4	9,4
Filtrate Volume	14,2	14,4	14,6	14,6	14,4

Table C-19 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & PAC R Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	14,75	16	17	17,5	17,5
PV	9	9,5	10	10	10
YP	11,5	13	14	15	15
YP/PV	1,3	1,4	1,4	1,5	1,5
0-10 gel	3-30	4-33	5-36	5-38	4-37
pH	10,0	10,0	10,0	10,0	10,0
Filtrate Volume	15	15,2	15,6	15,4	15,6

Table C-20 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & PAC R and PAC L Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	17	18,5	18,5	18	18,5
<b>PV</b>	8	8,5	8,5	8	8,5
<b>YP</b>	18	20	20	20	20
<b>YP/PV</b>	2,3	2,4	2,4	2,5	2,4
<b>0-10 gel</b>	22-55	25-58	23-57	22-56	19-55
<b>pH</b>	9,4	9,4	9,4	9,4	9,4
<b>Filtrate Volume</b>	14,2	14,4	14,4	14,6	14,4

Table C-21 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & Xanthan Gum Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	17,5	19,75	25	25	25
<b>PV</b>	9,5	9,5	11	11	10,5
<b>YP</b>	16	20,5	28	28	29
<b>YP/PV</b>	1,7	2,1	2,6	2,6	2,8
<b>0-10 gel</b>	9-15	10-18	15-25	14-27	15-27
<b>pH</b>	9,8	9,6	9,3	9,2	9,1
<b>Filtrate Volume</b>	14,8	14,8	15	15,2	15

Table C-22 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub> & Xanthan Gum and Dextrid Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	17,75	30	25,5	28	27,5
<b>PV</b>	6	4	2	2	2
<b>YP</b>	23,5	52	47	52	51
<b>YP/PV</b>	3,9	13	23,5	26	25,5
<b>0-10 gel</b>	22-48	45-66	38-61	40-60	39-63
<b>pH</b>	9,2	9,0	8,6	8,5	8,1
<b>Filtrate Volume</b>	16,0	19,2	26,4	29,2	33

Table C-23 Aging of E-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O & HV CMC Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	16,75	16	16,5	15,5	15
<b>PV</b>	7,5	8	8	7	8
<b>YP</b>	18,5	16	17	17	16
<b>YP/PV</b>	2,5	2	2,1	2,4	2,0
<b>0-10 gel</b>	16-40	15-39	18-43	18-42	19-39
<b>pH</b>	9,2	9,2	9,2	9,2	9,2
<b>Filtrate Volume</b>	15,4	15,6	15,8	16,0	16,2

Table C-24 Aging of C-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O & HV CMC and LV CMC Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	15,25	15,5	15,5	16	16,25
PV	6,5	6,5	6,5	7	7
YP	18,5	17	17	18	18,5
YP/PV	2,8	2,6	2,6	2,6	2,6
0-10 gel	17-43	18-44	18	16-44	18-47
pH	9,1	9,1	9,1	9,1	9,1
Filtrate Volume	15,2	15,4	15,4	15,6	15,6

Table C-25 Aging of E-Ben Activated with Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O & PAC R Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	17,25	16	15,5	15,25	15
PV	7,5	7	7,5	7,5	8
YP	19,5	18	16	15,5	14
YP/PV	2,6	2,6	2,1	2,1	1,8
0-10 gel	20-43	19-44	18-42	17-39	15-38
pH	9,2	9,2	9,2	9,2	9,2
Filtrate Volume	15,6	15,8	16	16	16,2

Table C-26 Aging of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  & PAC R and PAC L Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	15,25	15,75	14,5	15	15
PV	7	7	6,5	6,5	6,5
YP	16,5	17,5	16	17	17
YP/PV	2,4	2,5	2,5	2,6	2,6
0-10 gel	16-50	17-52	20-50	19-53	17-54
pH	9,1	9,1	9,1	9,1	9,1
Filtrate Volume	14,6	14,4	14,8	14,6	14,6

Table C-27 Aging of E-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  & Xanthan Gum Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	21,5	22	22	22,5	24,75
PV	8,5	9	9	9	9,5
YP	26	26	26	27	26,5
YP/PV	3	2,9	2,9	3	2,9
0-10 gel	17-30	16-24	16-22	16-19	14-19
pH	9,2	9,2	9,1	9,1	9,0
Filtrate Volume	14,8	15	15,2	15,2	15,2



Table C-28 Aging of C-Ben Activated with  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  & Xanthan Gum and Dextrid Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	16,75	17,75	24,75	24,5	25
PV	4,5	4,5	4	2	1,5
YP	24,5	26,5	41,5	45	47
YP/PV	6,1	5,9	10,4	22,5	20,8
0-10 gel	30-62	28-49	42-50	41-52	39-46
pH	9,0	8,8	8,5	8,3	7,9
Filtrate Volume	15,6	16,8	17,6	22	27,2

Table C-29 Aging of E-Ben Activated with  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture & HV CMC Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	16	15,5	15,5	15,5	15
PV	9	9	8,5	9	8,5
YP	14	13	14	13	13
YP/PV	1,6	1,4	1,6	1,4	1,5
0-10 gel	9-46	10-48	11-49	12-48	11-45
pH	9,4	9,4	9,4	9,4	9,4
Filtrate Volume	15	15,2	15,4	15,2	15,4

Table C-30 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & HV CMC and LV CMC Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	15,5	17,25	16,5	17	16,75
PV	7	8	7	7,5	7
YP	17	18,5	19	19	19
YP/PV	2,4	2,3	2,7	2,5	2,5
0-10 gel	19-57	19-59	20	25-62	12-37
pH	9,2	9,2	9,2	9,2	9,2
Filtrate Volume	14,2	14,2	14,4	14,6	14,2

Table C-31 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & PAC R Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	17,5	17	16,75	17	16,5
PV	9,5	9,5	9	9	9
YP	16	15	15	16	15
YP/PV	1,7	1,6	1,7	1,8	1,7
0-10 gel	8-49	9-50	7-53	9-52	10-51
pH	9,4	9,4	9,4	9,4	9,4
Filtrate Volume	14,4	14,8	14,6	14,8	15

Table C-32 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & PAC R and PAC L Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	16,25	17,50	16,5	16,5	17
PV	7,5	8	7,5	7	7,5
YP	17,5	18,5	18	19	19
YP/PV	2,3	2,3	2,4	2,7	2,5
0-10 gel	14-57	15-60	15-59	14-58	12-48
pH	9,2	9,2	9,2	9,2	9,2
Filtrate Volume	13,8	14	14,2	14,2	14

Table C-33 Aging of E-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & Xanthan Gum Mud

Properties	Aging Time (Days)				
	1	4	8	15	21
AV	17,5	17	18,25	17,5	17,25
PV	8	7	8,5	7	8
YP	19	20	19,5	21	18
YP/PV	2,4	2,85	2,3	3	2,3
0-10 gel	12-24	11-22	10-19	11-19	8-16
pH	9,4	9,4	9,3	9,3	9,2
Filtrate Volume	14,4	14,4	14,8	14,4	14,6

Table C-34 Aging of C-Ben Activated with Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture & Xanthan Gum and Dextrid Mud

<b>Properties</b>	<b>Aging Time (Days)</b>				
	<b>1</b>	<b>4</b>	<b>8</b>	<b>15</b>	<b>21</b>
<b>AV</b>	18,5	19	25	23,5	26
<b>PV</b>	7	6	2	2	2
<b>YP</b>	23	26	46	43	48
<b>YP/PV</b>	3,3	4,3	23	21,5	24
<b>0-10 gel</b>	29-65	29-51	42-50	40-45	42-49
<b>pH</b>	9,0	8,8	8,5	8,3	8,2
<b>Filtrate Volume</b>	15,0	15,2	27,2	28,4	29,2

## APPENDIX D

### (RHEOLOGICAL DATA FOR AGING AT HIGH TEMPERATURE)

Table D-1 Aging of API Bentonite at 149 °C

<b>Properties</b>	<b>API Bentonite at Room Temp.</b>	<b>API Bentonite at 149 °C</b>
<b>AV</b>	15,5	25
<b>PV</b>	10	13
<b>YP</b>	10,5	24
<b>YP/PV</b>	1,0	1,8
<b>0-10 Gel</b>	13-30	30-59
<b>pH</b>	9,0	9,0
<b>Filtrate Volume</b>	12,4	14,8

Table D-2 Aging of Reşadiye Bentonite at 149 °C

<b>Properties</b>	Reşadiye Bentonite at Room Temp.	Reşadiye Bentonite at 149 °C
<b>AV</b>	18,5	27,5
<b>PV</b>	15	21
<b>YP</b>	7	13
<b>YP/PV</b>	0,5	0,6
<b>0-10 Gel</b>	2-3	3-4
<b>pH</b>	9,7	9,7
<b>Filtrate Volume</b>	13,4	15

Table D-3 Aging of E-Ben & CMC Mud at 149 °C

<b>Properties</b>	Unactivated E-Ben & CMC mud at room temp.	Unactivated E-Ben & CMC mud at 149 °C
<b>AV</b>	17,5	1,5
<b>PV</b>	13,5	1,5
<b>YP</b>	8	0
<b>YP/PV</b>	0,6	0
<b>0-10 Gel</b>	1-2	0-0
<b>pH</b>	7,5	7,5
<b>Filtrate Volume</b>	14	120

Table D-4 Aging of E-Ben & PAC Mud at 149 °C

<b>Properties</b>	Unactivated E-Ben & PAC mud at room temp.	Unactivated E-Ben & PAC mud at 149 °C
<b>AV</b>	16	1,5
<b>PV</b>	10,5	1,5
<b>YP</b>	11	0
<b>YP/PV</b>	1	0
<b>0-10 Gel</b>	1,5-2	0-0
<b>pH</b>	7,5	7,5
<b>Filtrate Volume</b>	15	120

Table D-5 Aging of E-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Unactivated E-Ben & Xanthan Gum mud at room temp.	Unactivated E-Ben & Xanthan Gum mud at 121 °C
<b>AV</b>	19,5	2,5
<b>PV</b>	8	2
<b>YP</b>	23	1
<b>YP/PV</b>	2,9	0,5
<b>0-10 Gel</b>	11-16	1-1
<b>pH</b>	7,5	7,0
<b>Filtrate Volume</b>	13,6	130

Table D-6 Aging of C-Ben & CMC at 149 °C

<b>Properties</b>	Unactivated C-Ben & CMC mud at room temp.	Unactivated C-Ben & CMC mud at 149 °C
<b>AV</b>	20	4,5
<b>PV</b>	11	4,5
<b>YP</b>	18	1
<b>YP/PV</b>	1,6	0,2
<b>0-10 Gel</b>	15-46	4-19
<b>pH</b>	8,5	8,5
<b>Filtrate Volume</b>	14,6	28

Table D-7 Aging of C-Ben & PAC Mud at 149 °C

<b>Properties</b>	Unactivated C-Ben & PAC mud at room temp.	Unactivated C-Ben & PAC mud at 149 °C
<b>AV</b>	19,75	4,5
<b>PV</b>	10,5	3
<b>YP</b>	18,5	3
<b>YP/PV</b>	1,8	1
<b>0-10 Gel</b>	11-54	1-14
<b>pH</b>	8,5	8,5
<b>Filtrate Volume</b>	13,4	26,8



Table D-8 Aging of C-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Unactivated C-Ben & Xanthan Gum mud at room temp.	Unactivated C-Ben & Xanthan Gum mud at 121 °C
<b>AV</b>	17,75	10,25
<b>PV</b>	6,5	3,5
<b>YP</b>	22,5	13,5
<b>YP/PV</b>	3,5	3,9
<b>0-10 Gel</b>	14-29	12-27
<b>pH</b>	8,5	8,0
<b>Filtrate Volume</b>	15	31,4

Table D-9 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben at 149 °C
<b>AV</b>	6	12
<b>PV</b>	5	10
<b>YP</b>	2	4
<b>YP/PV</b>	0,4	0,4
<b>0-10 Gel</b>	1-14	1-3
<b>pH</b>	10,0	9,7
<b>Filtrate Volume</b>	20,4	25,6

Table D-10 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben at 149 °C
<b>AV</b>	10,75	15,5
<b>PV</b>	4,5	5
<b>YP</b>	12,5	21
<b>YP/PV</b>	2,77	0,4
<b>0-10 Gel</b>	12-30	24-72
<b>pH</b>	9,4	9,4
<b>Filtrate Volume</b>	22,8	30,4

Table D-11 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben & CMC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben & CMC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben & CMC mud at 149 °C
<b>AV</b>	15,5	13
<b>PV</b>	10	9
<b>YP</b>	11	8
<b>YP/PV</b>	1,1	0,9
<b>0-10 Gel</b>	6-40	2-24
<b>pH</b>	10,0	9,5
<b>Filtrate Volume</b>	14,8	22

Table D-12 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben & CMC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben & CMC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben & CMC mud at 149 °C
<b>AV</b>	18	12
<b>PV</b>	8	6
<b>YP</b>	20	12
<b>YP/PV</b>	2,5	2
<b>0-10 Gel</b>	17-37	11-33
<b>pH</b>	9,4	9,2
<b>Filtrate Volume</b>	14	24,4

Table D-13 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben & PAC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben & PAC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben & PAC mud at 149 °C
<b>AV</b>	15	12
<b>PV</b>	10	9,5
<b>YP</b>	10	5
<b>YP/PV</b>	1	0,5
<b>0-10 Gel</b>	3,5-30	1,5-4
<b>pH</b>	10,0	9,8
<b>Filtrate Volume</b>	14,8	20

Table D-14 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben & PAC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben & PAC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben & PAC mud at 149 °C
<b>AV</b>	16,5	11,5
<b>PV</b>	7,5	6,5
<b>YP</b>	18	10
<b>YP/PV</b>	2,4	1,5
<b>0-10 Gel</b>	19-55	11-40
<b>pH</b>	9,4	9,2
<b>Filtrate Volume</b>	14,4	21,6

Table D-15 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated E-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben & Xanthan Gum mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated E-Ben & Xanthan Gum mud at 121 °C
<b>AV</b>	17	24,5
<b>PV</b>	9	8
<b>YP</b>	16	33
<b>YP/PV</b>	1,8	4,1
<b>0-10 Gel</b>	7-18	23-48
<b>pH</b>	10,0	9,5
<b>Filtrate Volume</b>	14	19,8

Table D-16 Aging of Na<sub>2</sub>CO<sub>3</sub> Activated C-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben & Xanthan Gum mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> activated C-Ben & Xanthan Gum mud at 121 °C
<b>AV</b>	18,5	26
<b>PV</b>	6	6
<b>YP</b>	25	40
<b>YP/PV</b>	4,1	6,6
<b>0-10 Gel</b>	22-40	43-59
<b>pH</b>	9,4	9,1
<b>Filtrate Volume</b>	15,2	22,4

Table D-17 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben at 149 °C
<b>AV</b>	5	6
<b>PV</b>	3	3
<b>YP</b>	4	6
<b>YP/PV</b>	1,33	2
<b>0-10 Gel</b>	5-16	10-35
<b>pH</b>	9,2	9,2
<b>Filtrate Volume</b>	27,2	64,4

Table D-18 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated C-Ben Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated C-Ben at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated C-Ben at 149 °C
<b>AV</b>	10,75	6,75
<b>PV</b>	4,5	4
<b>YP</b>	12,5	3,5
<b>YP/PV</b>	2,77	0,9
<b>0-10 Gel</b>	12-35	½-4
<b>pH</b>	9,1	9,0
<b>Filtrate Volume</b>	22,8	31

Table D-19 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben & CMC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben & CMC mud at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben & CMC mud at 149 °C
<b>AV</b>	16,25	10
<b>PV</b>	8,5	4
<b>YP</b>	15,5	12
<b>YP/PV</b>	1,82	2
<b>0-10 Gel</b>	13-34	30-84
<b>pH</b>	9,2	9,2
<b>Filtrate Volume</b>	14,6	42,8

Table D-20 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated C-Ben & CMC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated C-Ben & CMC mud at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated C-Ben & CMC mud at 149 °C
<b>AV</b>	15,5	9
<b>PV</b>	7	7
<b>YP</b>	17	4
<b>YP/PV</b>	2,4	0,6
<b>0-10 Gel</b>	16-38	2-18
<b>pH</b>	9,1	9,1
<b>Filtrate Volume</b>	14	24

Table D-21 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben & PAC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben & PAC mud at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben & PAC mud at 149 °C
<b>AV</b>	17	9,5
<b>PV</b>	8	4
<b>YP</b>	18	11
<b>YP/PV</b>	2,25	2,75
<b>0-10 Gel</b>	14-38	27-87
<b>pH</b>	9,2	9,2
<b>Filtrate Volume</b>	14,8	47,6

Table D-22 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated C-Ben & PAC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated C-Ben & PAC mud at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated C-Ben & PAC mud at 149 °C
<b>AV</b>	15	9,25
<b>PV</b>	7	8
<b>YP</b>	16	2,5
<b>YP/PV</b>	2,3	0,3
<b>0-10 Gel</b>	17-43	½-8
<b>pH</b>	9,1	9,1
<b>Filtrate Volume</b>	14,4	25

Table D-23 Aging of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Activated E-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben & Xanthan Gum mud at room temp.	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O activated E-Ben & Xanthan Gum mud at 121 °C
<b>AV</b>	19,5	15,5
<b>PV</b>	8	8
<b>YP</b>	23	15
<b>YP/PV</b>	2,9	1,9
<b>0-10 Gel</b>	17-31	23-43
<b>pH</b>	9,2	9,2
<b>Filtrate Volume</b>	13,8	30,6



Table D-24 Aging of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Activated C-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	$\text{Na}_2\text{CO}_3$ activated C-Ben & Xanthan Gum mud at room temp.	$\text{Na}_2\text{CO}_3$ activated C-Ben & Xanthan Gum mud at 121 °C
<b>AV</b>	16	12,5
<b>PV</b>	6	6
<b>YP</b>	22	13
<b>YP/PV</b>	3,7	2,2
<b>0-10 Gel</b>	24-48	10-33
<b>pH</b>	9,1	9,1
<b>Filtrate Volume</b>	15,6	23,2

Table D-25 Aging of  $\text{Na}_2\text{CO}_3$ - $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$  Mixture Activated E-Ben Mud at 149 °C

<b>Properties</b>	$\text{Na}_2\text{CO}_3$ and $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ mixture activated E-Ben mud at room temp.	$\text{Na}_2\text{CO}_3$ and $\text{Na}_2\text{B}_4\text{O}_7\cdot 5\text{H}_2\text{O}$ mixture activated E-Ben mud at 149 °C
<b>AV</b>	5	8
<b>PV</b>	3	7
<b>YP</b>	4	2
<b>YP/PV</b>	1,3	0,2
<b>0-10 Gel</b>	2-14	1-2
<b>pH</b>	9,4	9,4
<b>Filtrate Volume</b>	21,2	33

Table D-26 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated C-Ben at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben mud at 149 °C
<b>AV</b>	11,5	11
<b>PV</b>	4,5	8,5
<b>YP</b>	14	5
<b>YP/PV</b>	3,1	0,6
<b>0-10 Gel</b>	12-30	2-19
<b>pH</b>	9,2	9,2
<b>Filtrate Volume</b>	24	29,4

Table D-27 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated E-Ben & CMC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated E-Ben & CMC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated E-Ben & CMC mud at 149 °C
<b>AV</b>	16	10,5
<b>PV</b>	8	9
<b>YP</b>	16	3
<b>YP/PV</b>	2	0,3
<b>0-10 Gel</b>	11-45	1-2
<b>pH</b>	9,4	9,4
<b>Filtrate Volume</b>	14,8	24,4

Table D-28 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated C-Ben & CMC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben & CMC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben & CMC mud at 149 °C
<b>AV</b>	16,75	11,25
<b>PV</b>	7,5	6,5
<b>YP</b>	18,5	9,5
<b>YP/PV</b>	2,5	1,5
<b>0-10 Gel</b>	12-37	3-21
<b>pH</b>	9,2	9,2
<b>Filtrate Volume</b>	14	20,8

Table D-29 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated E-Ben & PAC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated E-Ben & PAC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated E-Ben & PAC mud at 149 °C
<b>AV</b>	17	10
<b>PV</b>	9	8,5
<b>YP</b>	16	3
<b>YP/PV</b>	1,8	0,4
<b>0-10 Gel</b>	10-51	1-2
<b>pH</b>	9,4	9,4
<b>Filtrate Volume</b>	14,4	23,2

Table D-30 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated C-Ben & PAC Mud at 149 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben & PAC mud at room temp.	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben & PAC mud at 149 °C
<b>AV</b>	16,25	12
<b>PV</b>	7,5	9
<b>YP</b>	17,5	16
<b>YP/PV</b>	2,3	1,8
<b>0-10 Gel</b>	12-43	3-24
<b>pH</b>	9,1	9,1
<b>Filtrate Volume</b>	13,6	20

Table D-31 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated E-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated E-Ben & Xant. Gum mud at room temp	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated E-Ben & Xant. Gum mud at 121 °C
<b>AV</b>	19,5	16,5
<b>PV</b>	8	7
<b>YP</b>	23	20
<b>YP/PV</b>	2,9	2,9
<b>0-10 Gel</b>	17-31	15-27
<b>pH</b>	9,4	9,4
<b>Filtrate Volume</b>	13,8	24,6

Table D-32 Aging of Na<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·5H<sub>2</sub>O Mixture Activated C-Ben & Xanthan Gum Mud at 121 °C

<b>Properties</b>	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben & Xant. Gum mud at room temp	Na <sub>2</sub> CO <sub>3</sub> and Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·5H <sub>2</sub> O mixture activated C-Ben & Xant. Gum mud at 121 °C
<b>AV</b>	17	16
<b>PV</b>	5,5	6
<b>YP</b>	23	26
<b>YP/PV</b>	4,2	4,3
<b>0-10 Gel</b>	25-48	22-45
<b>pH</b>	9,1	9,1
<b>Filtrate Volume</b>	14,6	22,6

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### PUBLICATIONS

1. Toka B., Atalay U., *Flotation Behaviour of Galena and Pyrite*, Proceedings of the 6<sup>th</sup> International Mineral Processing Symposium, Kusadası-Turkey, 1996.
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### HOBBIES

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