

EXPERIMENTAL ANALYSIS OF SHALE FOR EVALUATING SHALE DRILLING FLUID INTERACTION IN AGBADA FORMATION

ABSTRACT

Aims: To characterize the shale samples from Agbada formation and to develop an inhibitive water-based mud for the shale types.

Study design: Experimental.

Place and Duration of Study: Department of Petroleum, University of Port Harcourt Rivers State, Agbada Formation Niger-Delta Region, between March 2011 and August 2012.

Methodology: This study includes the determination of shale properties such as mineralogy, cation exchange capacity, native moisture content and total organic carbon content of the shale samples collected from two wells in Agbada Field. It also involves the establishment of an adsorption isotherm for the shale and the modeling of these isotherms using the Guggenheim, Anderson and Deboer model.

Results: Based on the results of the dispersion and swelling tests, 3% Bentonite + 1% Potassium Chloride (KCl) were selected as the promising fluid. Well A exhibited the Type II isotherm which is common to most shale while Well B showed the Type V isotherm. Adsorption isotherm model predictions were in agreement with experimental observations. The samples from the wells exhibited dispersion after testing with deionized water. For Well A and B, 3% Bentonite + 1% Potassium Chloride (KCl) inhibits and yielded higher shale percent recovery values from dispersion tests.

Conclusion: It was observed from adsorption isotherm models that the total organic carbon content is not a significant factor in the adsorptive and dispersive behaviors exhibited by the shale samples.

Keywords: Mineralogy, Adsorption Isotherm, Dispersion Test and Swelling Test

1. INTRODUCTION

Wellbore instability in shale formations has been a significant problem in the petroleum industry for over a century. It is estimated that this problem costs the oil industry about 1 billion US dollars each year and it is also responsible for about 10 to 20% of the total drilling cost of well [1].

It is believed that the main cause of shale instability stems from unfavorable interactions between the drilling mud and shale formations [2]. Shale instability is generally caused by pore pressure changes and mechanical property alterations around the wellbore, induced by both chemical and hydraulic effects. All of these alterations are caused by water and ion movement into or out of the shale formations. Chenevert [3] showed that differences in water activity could cause an Osmotic flux of water into or out of the shale.

Models based on chemical potential and hydraulic pressure developed by Osisanya [4], Mody and Hale [5], Osisanya and Chenevert [6] and further expanded on by Van Oort *et al.* [7] have indicated the complexity of theoretical analysis of driving forces and mechanisms that govern shale stability in the borehole. Osisanya and Chenevert [6], note that if ions are added to a water-base drilling fluid; the total water activity is lowered and water movement into the shale is reduced due to osmotic effects. This effect is not long lasting because ions are not completely restricted to the wellbore fluid by semi-permeable membrane. But for oil-base mud, a very efficient membrane exists; thus, very little ion transfer occurs.

The causes of wellbore instability are often classified into either Mechanical (for example, failure of the rock around the hole because of high stresses, low rock strength or inappropriate drilling practice) or Chemical effects which arise from damaging interaction between the rock, generally shale, and the drilling fluid. These may be as a result of lack of a readily definable and irrefutable technical method to measure the interactions between drilling fluids and shale or the earlier assumption that shale's are ideal semi-permeable membranes. Depending on the shale type, water adsorption may lead to various reactions such as swelling, cuttings dispersion and increase in pore pressure creating wellbore instability to varying degrees [3]. Common failures that occur from shale instability include; Sloughing, Caving, stuck pipe and bit balling.

1.1 LITERATURE REVIEW

Several literary works have been published with the intent to clarify the uncertainties surrounding shale-fluid interaction. The study of shale fluid interactions gained momentum in the seventies after a series of publications by Chenevert revealed the importance of drilling fluid activity in the successful inhibition of shale formations. In his first paper, Chenevert [3] experimentally tested a wide range of shales expanding the little database of knowledge of shale characteristics. He determined the aqueous activity of the shales by developing adsorption isotherms relating their aqueous activities to their water contents. He discovered that all shales, including hard illitic shales thought to initially be inert, showed some reaction when immersed in water. Furthermore, at equal activities, shales with higher clay contents showed an increased weight percent of water adsorbed. O'Brien and Chenevert [8]

characterized shales with major laboratory procedures. X-Ray diffraction analyses revealed the shale composition while adsorption isotherms generated showed hydrational tendencies. Various fluids tested using the swelling and dispersion tests showed the supremacy of potassium mud in limiting swelling and instability. Osisanya [4], Osisanya and Chenevert [6] also concluded that shale is a 'leaky' membrane and no longer a semi-permeable membrane as originally proposed by Chenevert [3]. Ewy and Morton [9] assessed the efficiency of four Water Base Muds (WBMs) with varying activities to reduce pore pressure build up. Results of the study show the presence of two physical phenomena - osmotic membrane and physical plugging of the pore spaces. All mud used showed less swelling than the base case brine. In order to have the best possible results during laboratory testing, shale taken at the rig site and laboratory samples was preserved. This step is extremely important to maintain the original properties of the shale and thereby obtain results that are reliable. Poor preservation can alter the properties of the shale and cause artifacts. An incorrect preservation environment would produce poor results and wrong conclusions of gathered data.

The shale used during the laboratory testing was originally cored at a depth of 2005-2006 ft and 3462.91-3464 ft; and preserved at the rig site in polyethylene bags covered by heavy duty plastic.

2.0 MATERIAL AND METHODS

2.1 EXPERIMENTAL DETAILS

It consists of two major experiments. These are shale characterization and shale-drilling fluid interaction experiments. Characterization tests provide knowledge on the composition and properties of the shale while the shale-drilling fluid interaction tests give information on the shale's reactivity in various fluids through dispersion and swelling tests.

2.2 Experimental Procedure

The experimental studies involve the following:

1. Characterization of shale samples through mineralogy, total organic content, native moisture content, and Cation exchange capacity
2. Development of adsorption isotherms for the shale samples
3. Development and evaluation of various water-based fluid against shale samples by performing dispersion tests under atmospheric conditions and elevated temperatures of 120 °F as well as swelling tests under atmospheric conditions

2.2.1 Shale Test Sample Characterization

The shale samples were characterized using the following methods [10]:

Total organic carbon content – Low Temperature Asher

Total Organic Carbon Content (TOC) is the quantity of carbon associated with organic matter. Organic matter is formed from decaying vegetation and other metabolic activities of living organisms. Shale, being a sedimentary rock formed partly from the deposition of organic material, has organic matter present within its structure. TOC content can be determined using various methods such as high temperature combustion, photo-oxidation, and high temperature catalytic oxidation. The main principle utilized by these methods is the oxidation reaction to remove carbon. Carbolite Sheffield low temperature asher was used in this study.

Mineralogy analysis – Chemical Analysis and X-Ray Diffraction

Shale mineralogy analysis is used to identify the type and relative amounts of minerals present in shale samples. For this study, X-Ray Fluorescence analysis (XRF) and X-Ray Diffraction were used for elemental compositional analysis. X-ray fluorescence analyses were done using a very small amount of about one gram of dried solids, but 80g was submitted. An X-ray diffraction analysis requires expensive instrumentation (DX-27Min) and a knowledgeable analyst trained in the operation of the instrument and interpretation of the data. Owing to limitations of obtaining pure standards and the crystalline nature of some samples, the X-ray fluorescence data is only semi- quantitative for the mineralogical composition of the shale.

Native moisture content – Oven

Native moisture content is the amount of water present in shale as received in the laboratory after the removal of coatings for preservation. It is unique to each type of shale and the environment in which it is located. The following discussion presents the method used to obtain the native moisture content and water activity of the shales used in our study. We obtained Five (5) cubical shale samples and the initial weight of each shale sample was designated (w_i). The shale samples were dried by placing them in an oven at 200°F for 24 hours, then the weight of each dry shale sample was measured (w_d). The native moisture content of all 5 shale samples was averaged and used as the average native moisture content for the shale type.

Cation exchange capacity – Methylene blue test

The occurrence of isomorphous substitution and the subsequent adsorption of a cation in clays cause a disparity in their stability. The adsorbed cation is held loosely by the crystal structure and can readily be exchanged for another cation, thus the term cation exchange capacity (CEC). In the presence of water, the cations voluntarily undergo substitution by hydrogen or hydronium ions present. This results in a high affinity for water molecules which can cause alteration of the shale's physical properties. The methylene blue test is the

131 simplest and most common test used in the drilling industry. The test results are reported in
132 units of meq/100g (milliequivalent weights of methylene blue/100g of dry clay). The CEC of
133 the shales were determined using the methylene blue method. This method involves the
134 titration of clay suspensions using the methylene blue dye. Methylene blue is an organic dye
135 that readily displaces exchangeable cations present in the clay minerals. As it replaces these
136 cations, it is absorbed into the clay particles.

137 **2.2.2 Fluid Design and Test Matrix**

138 One of the main objectives of this study is to develop a water based mud that will minimize
139 shale dispersion and swelling for the shale samples obtained. Each shale formation requires
140 a unique drilling fluid for effective inhibition. This is achieved by providing cation exchange
141 with the clays in the shale, the K^+ or Ca^{2+} commonly replace the Sodium ion $[Na^+]$ associated
142 with the clay in the shale; creating a more stable rock that is better able to resist hydration
143 [4]. The fluid design is based on common oilfield materials. They include three salts:
144 Potassium Chloride (KCl), Calcium Chloride ($CaCl_2$) and Sodium Chloride (NaCl); as well as
145 Bentonite. The salts were used to reduce the fluids chemical potential. Water, Soda Ash,
146 PAC-L and PAC-R, Caustic Soda and Barite. Three mud systems formulated are shown in
147 Table 1 below.

148 **Table 1: Compositions of Developed Test Fluids**

S/N	Mud System	Composition
1	KCl-Bentonite Mud	3% Bentonite + 1% KCl 3% Bentonite + 0.3% KCl
2	$CaCl_2$ -Bentonite Mud	3% Bentonite + 1% $CaCl_2$ 3% Bentonite + 0.3% $CaCl_2$
3	NaCl-Bentonite Mud	3% Bentonite + 1% NaCl 3% Bentonite + 0.3% NaCl
Base Fluid = Water (350MI) + 0.20g NaOH, Density = 8.70lb/gal		

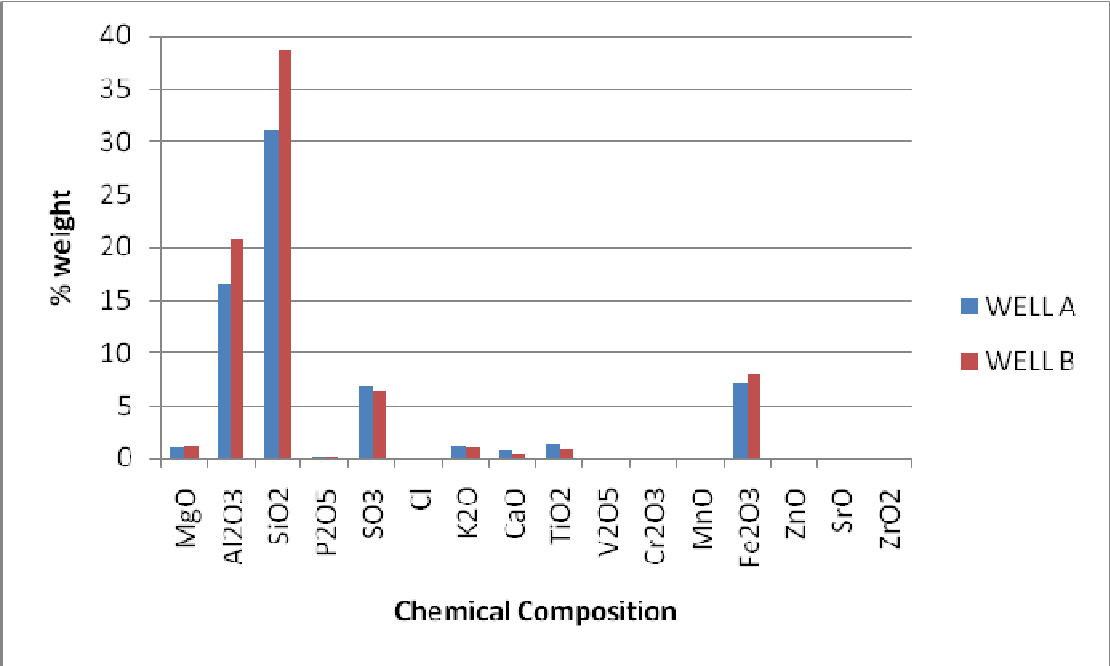
149

150 **3.0 RESULTS AND DISCUSSION**

151 **3.1 Results**

152 Mineralogical analysis indicates the relative quantities of compounds present in a shale. Clay
153 and non-clay minerals are usually present in shales. The type of clay present is an indication
154 of the degree of hydration experienced by the shale. It can also be used to estimate the
155 severity of wellbore instability issues that may arise. X-ray fluorescence (XRF)
156 characterization was performed to know the chemical compositions of the minerals that are
157 present in both Wells. The data given in Figure 1 show that the aluminum and silicon oxides
158 are present in major quantities while other minerals are present in trace amount. In both

159 shale samples there exist concentration of Magnesium and Calcium in oxide fraction, which
160 could be connected with character of their matrix and their origin. Magnesium could create
161 mixed hydroxide minerals with other cations like Zinc, Manganese, Aluminum and iron,
162 which influence mobility and bioaccumulation. The content of Calcium in the shale samples
163 could be connected with a presence of clay and high content of organic matter.
164 The chemical composition of Well A is rich in the following oxides: SiO₂, Al₂O₃, SO₃ and
165 Fe₂O₃ while shale B proved to be rich in SiO₂, Al₂O₃, SO₃ and Fe₂O₃.



166 **Figure 1:** Shale Samples % weight and Chemical Composition
167
168
169

170 **3.2 Discussion of Results**

171 The mineralogy analysis for both Well A and B samples are presented in Table 2. The Well
172 A (Usan) cored at a depth of 2005 ft – 2006 ft, is made up of 20% Quartz and 52% Clay. It
173 contains other non-clay minerals such as feldspar and carbonates in minimal amounts. The
174 bulk of the clay content consists of illite and mixed clays with small amount of smectite. The
175 existence of smectite indicates the probability of some swelling and dispersion in aqueous
176 solution. Well B cored at a depth of 3462.91 ft – 3464 ft, is composed of 22% Quartz and
177 51% Clay. It also contains negligible amount of non-clay minerals such as feldspar and
178 carbonates. Zero smectite levels indicate low swelling tendencies.

179
180
181

182

Table 2: Shale Sample Mineralogy using X-Ray Diffraction

Analysis	Well A	Well B
Depth (ft)	2005 - 2006	3462.91 - 3464
Clay Content (Wt %)		
Illite	14.90	17.10
Kaolinite	10.10	6.40
Smectite	2.90	0
Halloysite	4.70	7.60
Mixed layer	19.30	20.10
Total Clay Content	51.90	51.20
Non-Clay Mineral Content (Wt %)		
Quartz	20.20	21.90
Carbonates	8.05	10.10
Feldspar	8.80	10.80
Apatite	4.60	5.30
Dolomite	6.40	1

183

184 The native moisture content obtained for Well A was 5.04% while that of Well B was 1.79%.
185 Well A contains more moisture than Well B and is expected to have a higher isotherm curve
186 and native activity. Moisture content as we know from literature is a function of various
187 factors with the most important being clay mineral composition and preservation techniques.
188 High moisture contents indicate the presence of expandable clays with the ability to store
189 moisture by adsorption. Total organic carbon analysis provides a generic test of the overall
190 organic carbon content of the shale. Furthermore, unpreserved shales lose their in-situ
191 moisture when brought to the surface because of the change in environmental conditions.
192 The total organic content for Well A was 10.02% while that of Well B was 10.62% both
193 shales have relative organic contents. Table 3 shows the test results for Native moisture

194 contents (NMC), Cation Exchange Capacity (CEC) and Total organic carbon contents (TOC)
195 for well A and B.

196

197 **Table 3: Native Moisture Content, Cation Exchange Capacity (CEC) and Total Organic**
198 **Content Values**

Parameter	Well A	Well B
Native Moisture Content (% weight)	5.04	1.79
Total Organic Content (%)	10.02	10.62
Cation Exchange Capacity (CEC) (Meq/100g of Solid)	4.20	3.15

199

200 The cation exchange capacity (CEC) of shales is a measure of the intensity of the negative
201 charge environment between clay platelets and hence co-ions electrical exclusion property
202 of shales. High cation exchange capacity indicates strong electrical repulsion of anions;
203 therefore, the cation exchange capacity should influence the shale membrane efficiency [11].
204 The higher the CEC, the more reactive the shale. **Cation Exchange Capacity test** show that
205 Well A has CEC value of 4.20 Meq/100g of solid while Well B has CEC value of 3.15
206 Meq/100g of solid. The CEC values of both shales are below moderately reactive shales and
207 greater than non-reactive sand stone and limestone. A low CEC value in the range of 1 to 10
208 Meq/100g can still be problematic if the small amount of clays present swell and cause the
209 shale to break apart. **Table 3 shows the result for the cation exchange capacity (CEC) of the**
210 **shales.** Both shales showed a large amount of interchangeable cation C^{2+} , which gives these
211 shale samples a low reactivity profile.

212 **3.3 Adsorption Isotherms and Modeling**

213 Adsorption isotherms provide information on a material's water content at certain equilibrium
214 conditions. The behavior of a shale sample under these conditions can directly be related to
215 its hydration and swelling potential. The isotherm can also give information on the
216 expandable clay content of the shale. Two pieces of information are needed from the
217 adsorption isotherm in order to make these conclusions. First, the shape of the adsorption
218 isotherm, for compositionally and structurally similar clays; the shape of the adsorption
219 isotherm should approximately be similar to one of the already established isotherms.

Second, the amount of moisture adsorbed that is expressed on the y-axis of the curve shows which shale is more likely to hydrate [10]. The adsorption isotherm for Well A sample is shown in figure 2. The shape is similar to the Type II Adsorption Isotherm. Type II Adsorption isotherm shows large deviation from Langmuir model of adsorption. Type II isotherms do not exhibit a saturation limit. This type of isotherm indicates an indefinite multi-layer formation after completion of the monolayer and is found in adsorbents with a wide distribution of pore size. The intermediate flat region in the isotherm corresponds to monolayer formation, following which adsorption occurs in successive layers.

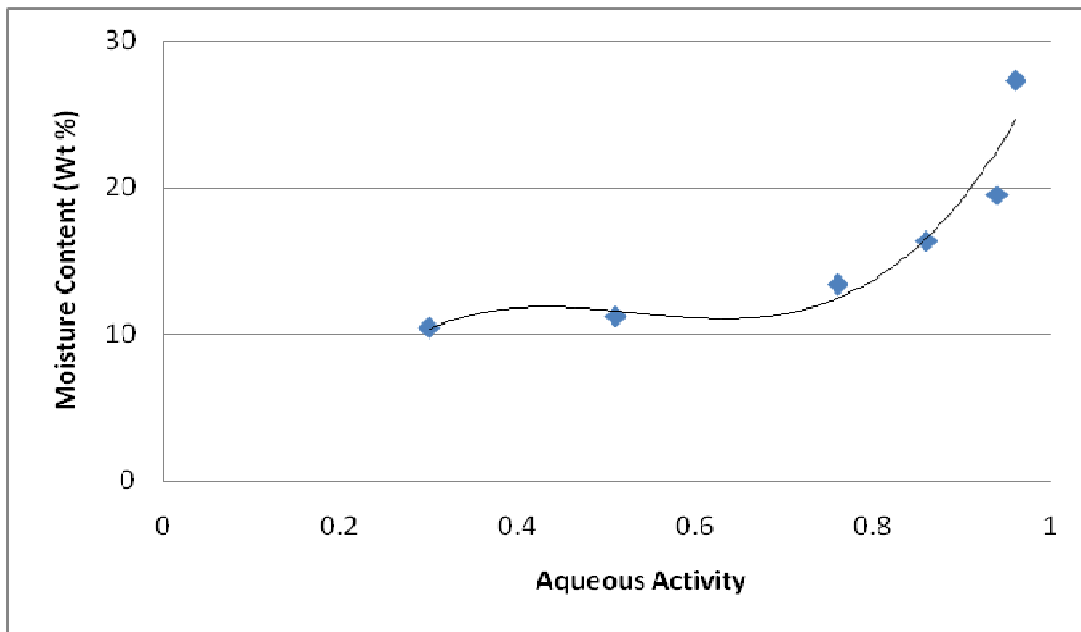


Figure 2: Adsorption Isotherm for Well A

Several authors have reported isotherms with similar Type II shape [3, 4]. An example is the adsorption of water vapour on carbon black at 30°C. Using the native moisture content obtained previously as 5.04% and the adsorption isotherm, we can obtain the equilibrium activity of approximately 0.26.

Figure 3 shows the adsorption isotherm for Well B sample. The shape observed is characteristic of the Type V adsorption isotherm.

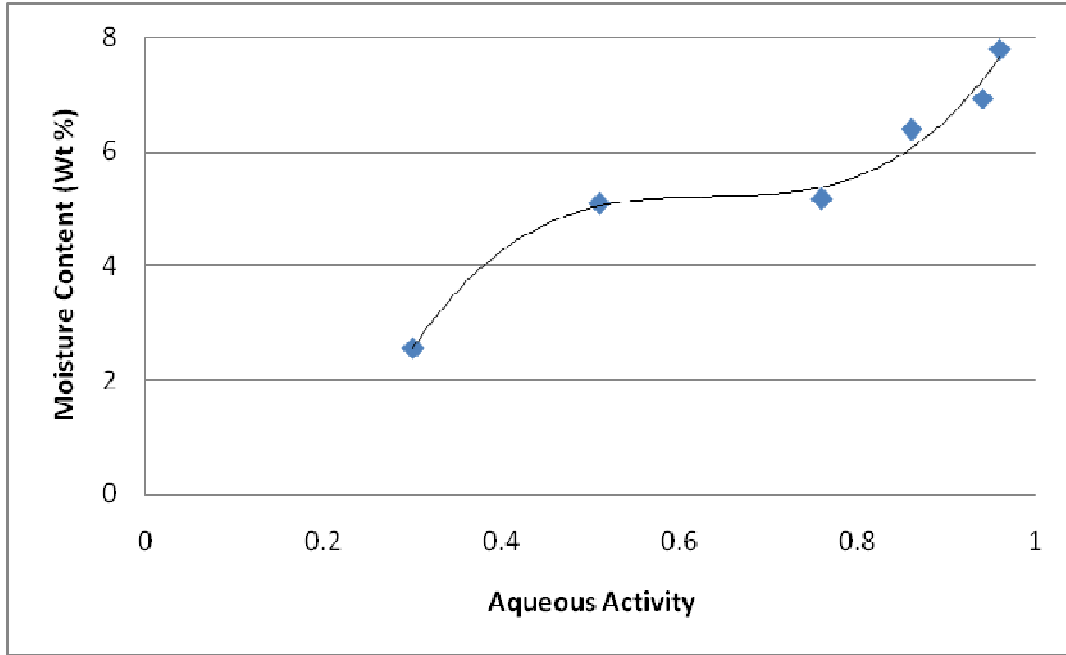


Figure 3: Adsorption Isotherm for Well B

Type V adsorption isotherm shows phenomenon of capillary condensation of gas. The saturation level reaches at pressure below the saturation vapour pressure; this can be explained on the basis of possibility of gases getting condensed in the tiny capillary pores of adsorbent at pressure below the saturation pressure of the gas. Example of Type V adsorption isotherm is adsorption of water vapour at 100°C on Charcoal. An equilibrium activity of approximately 0.20 was obtained from the native moisture content of 1.79% and the adsorption isotherm.

It can be deduced that Well A has higher adsorptive potential than Well B and a conclusion that more adsorption took place in Well A than in Well B can be made; therefore more expandable clays are present.

The Guggenheim, Anderson and DeBoer (GAB) model being the most commonly accepted model was used to fit the isotherms of Well A and B. The model has two advantages over the BET model. It fits a wider range of activity values between 0-0.99. Other shale adsorption isotherms developed by Osisanya [4] for Mancos, Wellington and Pierre shales were also fitted. The GAB model equation is expressed as follows:

$$\frac{m}{m_0} = \frac{CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)} \quad 1$$

Input Values

m = moisture content of the material on a dry basis (Kg/Kg % dry matter)

258 a_w = water activity

$$C = C_o \exp\left(\frac{\Delta h_c}{RT}\right) \quad 2$$

$$K = K_o \exp\left(\frac{\Delta h_k}{RT}\right) \quad 3$$

$$\Delta h_k = \Delta h_{s, multi} - \Delta h_{vap}$$

$$\Delta h_c = \Delta h_{s, mono} - \Delta h_{vap}$$

263 C and K are constants associated with the energies of interaction between the first and the
264 distant adsorbed molecules at the individual adsorption sites. M_o is the monolayer value, that
265 is, moisture content corresponding to saturation of all primary sites by one molecule of
266 water. The constant K permits the model to be applicable to higher water activity (at
267 multilayer moisture region). All parameters used in GAB equation have physical meaning
268 associated with them. Where Δh_c is the specific bonding enthalpy of water monolayer (J/kg),
269 Δh_k is the mean specific bonding enthalpy of the water multilayer (J/kg), R is the universal
270 gas constant (J/kg/K), T is the absolute temperature (K), $\Delta h_{s, mono}$ is the specific sorption
271 enthalpy of water monolayer (J/kg), Δh_{vap} is the specific vaporization enthalpy of water
272 (J/kg), $\Delta h_{s, multi}$ is the mean specific sorption enthalpy of the water multilayer (J/kg), C_o and
273 K_o are adjustable parameters accounting for temperature effect.

274 The most important parameter in the model is the monolayer moisture content M_o . Reactive
275 clay minerals such as smectite have a higher number of active interlayers and increased
276 isomorphic substitution than less reactive clays. Shales with higher reactive clays will
277 therefore have more area for monolayer coverage by water molecules before multiple layers
278 begin to form. Thus, the M_o of reactive shale should be higher than that of less reactive
279 shale. In our study, Well A and B have M_o values of 6.728 g/g (dry basis) and 2.999 g/g (dry
280 basis). Thus, Well A is more reactive than Well B.

281 Microsoft Office Excel solver was used to fit the curves to the model equation. It was
282 programmed to use a nonlinear regression-least squares method for curve fittings. The
283 square of the correlation coefficient (R^2) for the fit should range between 0.9 and 1.00 to
284 show a good fit for the model.

285 Figure 4 and 5 show the adsorption isotherms obtained from experimental data and how it
286 fits to the GAB model. The figure shows the position of each salt used in the adsorption test
287 when the shale samples from both Well A and B were placed in the desiccators. The static
288 gravimetric method, also known as the isopiestic method was used to develop the shale
289 adsorption isotherms. In this method, weight measurements were taken of shale samples
290 under varying relative humidity conditions at constant temperature and pressure. The

relative humidity environments were created using saturated salt solutions in desiccators. The moisture adsorbed physically is the difference between the water content as expressed in initial weight and the water content at equilibrium known as the final weight of the sample. For this study, tests were carried out at ambient conditions. Table 4 gives the M_o of each shale from the model along with the value of the CEC previously obtained.

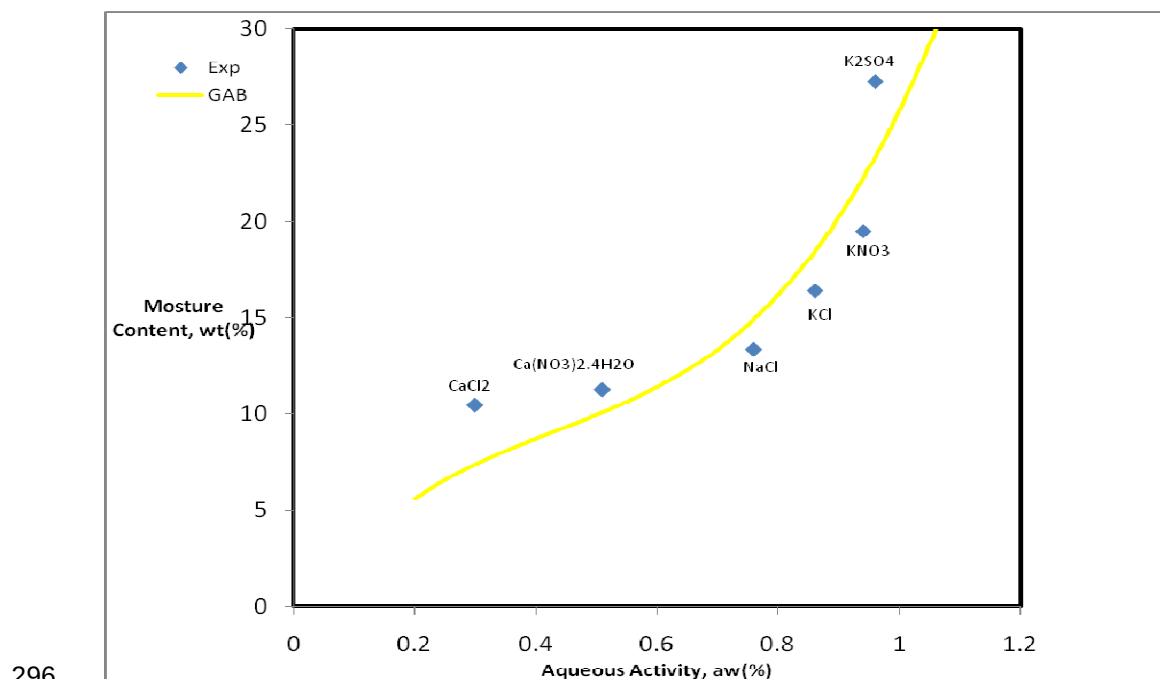


Figure 4: Adsorption Isotherm of Well A fitted to the GAB model

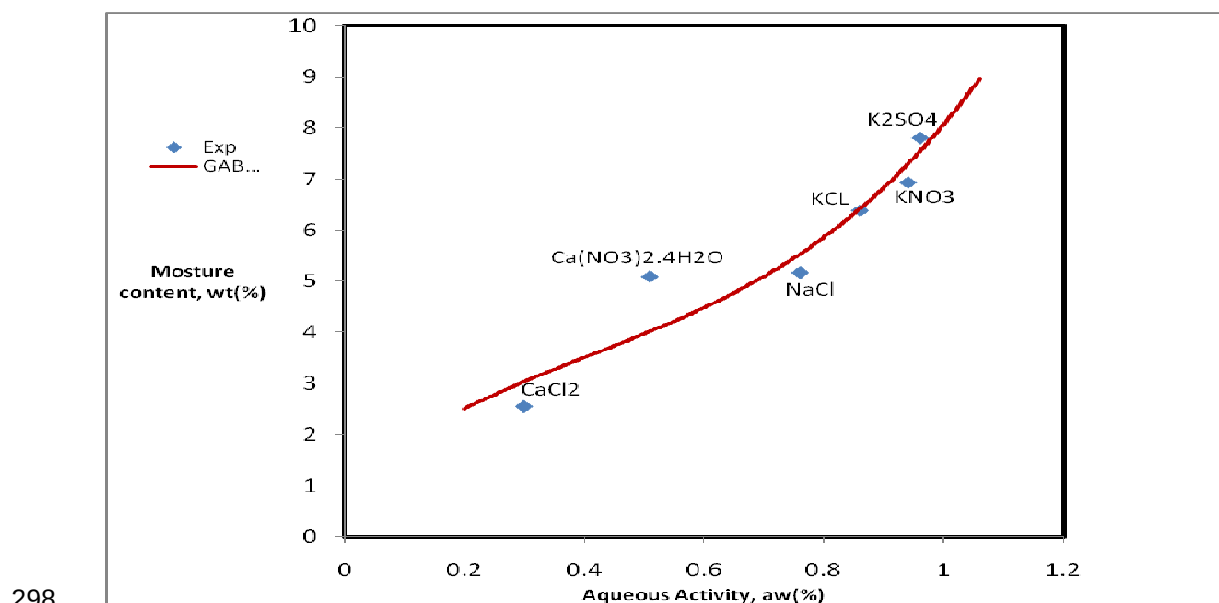


Figure 5: Adsorption Isotherm of Well B fitted to the GAB model

300 **Table 4: Cation Exchange Capacities and Monolayer Content for the Shale**

	Well A	Well B
CEC (Meq/100g of solid)	4.20	3.15
M _o (g/g dry basis)	6.728	2.999

301

302 From Table 4, the CEC and M_o values appear to exhibit a trend for both shale samples.
 303 Higher CEC value were observed with higher monolayer moisture value.

304

305 **3.4 Shale-Drilling Fluid Interaction**

306 The shale dispersion test (rolling test) is a common procedure that is used to measure the
 307 interactions between drilling fluids and shales. The shale rolling test depend on the moisture
 308 content of the shale, the shale composition, the viscosity of the test fluid, the rotation speed
 309 of the rollers, and the test temperature. The rheological behavior of the test fluid has the
 310 strongest influence on test results. The rolling test is a useful technique to measure the
 311 interactions of electrolytes with shale granules in partially formulated drilling fluids. This test
 312 is used to design fluids and screen the effectiveness of inhibitor additives to maintain the
 313 integrity of the cuttings and minimize the interaction of fluids with the shale sections during
 314 the drilling and completion operations. It gives an indication of how the cuttings might survive
 315 in the drilling fluid as they travel up the annulus and it does provide some measure of the
 316 ability of the drilling fluid to inhibit shale. Dispersion tests were carried out using various
 317 drilling fluids for Shale samples in Well A and B at 120°F. The dispersion test involves
 318 exposing a weighed quantity of sized shale pieces (2-4 Sieve Opening Millimeters) to a
 319 formulated fluid in a conventional roller-oven cell. The test provides long-term exposure of
 320 the shale to the fluid under mild agitation. Under such conditions, dispersion of the shale
 321 into the fluid will occur depending on the tendency of the shale to disperse and the inhibitive
 322 properties of the fluid. Shale dispersion is a process by which shale cuttings disintegrate into
 323 smaller sizes. It is a function of mechanical factors such as shear and chemical factors such
 324 as hydration. The fluid and shale are rolled together in a roller oven for 16 hours at 120°F.
 325 After cooling to room temperature, the fluid is poured over a 0.023 mesh size and the
 326 retained shale pieces are recovered, washed, weighed, and dried overnight at 200°F.
 327 Afterwards, the sample is re-weighed to determine the percent recovery. Fluid rheological
 328 properties were also measured using a six speed Fann 35 viscometer before and after
 329 dispersion tests.

330 Percent recovery, a measure of shale recovered after dispersion tests were calculated.
 331 Table .5 shows the percent recoveries of Shale in Well A and B for the bentonitic fluids.

Maximum recovery was obtained at 120°F using a fluid composition of 3% Bentonite + 1% KCl followed by 3% Bentonite + 1% CaCl₂ for both Well A and B.

334

335 **Table 5: Well A Percent Recoveries for Bentonitic Fluids**

Fluid Composition	% Recovery	
	Well A	Well B
Base Fluid	47.40	50.40
3% Bentonite + 0.3% KCl	69.89	70.40
3% Bentonite + 1% KCl	79.67	77.20
3% Bentonite + 0.3% CaCl ₂	70.56	66.80
3% Bentonite + 1% CaCl ₂	77.15	71.80
3% Bentonite + 0.3% NaCl	66.34	65.80
3% Bentonite + 1% NaCl	76.83	70.80

336

337 The results are in agreement with the literature, that K⁺ and Ca²⁺ ions are added to the
 338 water-base mud to inhibit the clay from dispersing, to stop it from breaking up when attacked
 339 by aqueous solution. These ions commonly replace the sodium ion (Na⁺) associated with the
 340 clay in the shale, creating a more stable rock that is able to resist hydration.

341 The results show that the mud with high concentration inhibit the shale samples more from
 342 dispersion and this account for higher value in Percentage Recovery of these muds. The
 343 dispersion is a function of changes in structure of the shale matrix and in the bound and
 344 crystalline water caused by the hydration of the cores.

345 Further analysis of the dispersion tests involved the measurement of the fluid rheology
 346 before and after the tests. Values of shear stress (τ) and shear rate (γ) were calculated using
 347 the following equations:

348 For shear stress,

$$349 \quad \tau = 0.01066 \times \theta_i \times N$$

4

350

351 Where,

352 τ = shear stress (lb_f / ft²)

353 = dial reading at ith rpm

354 N = spring factor = 1

355 For shear rate,

356
$$\gamma = 1.703 \times \text{RPM}$$

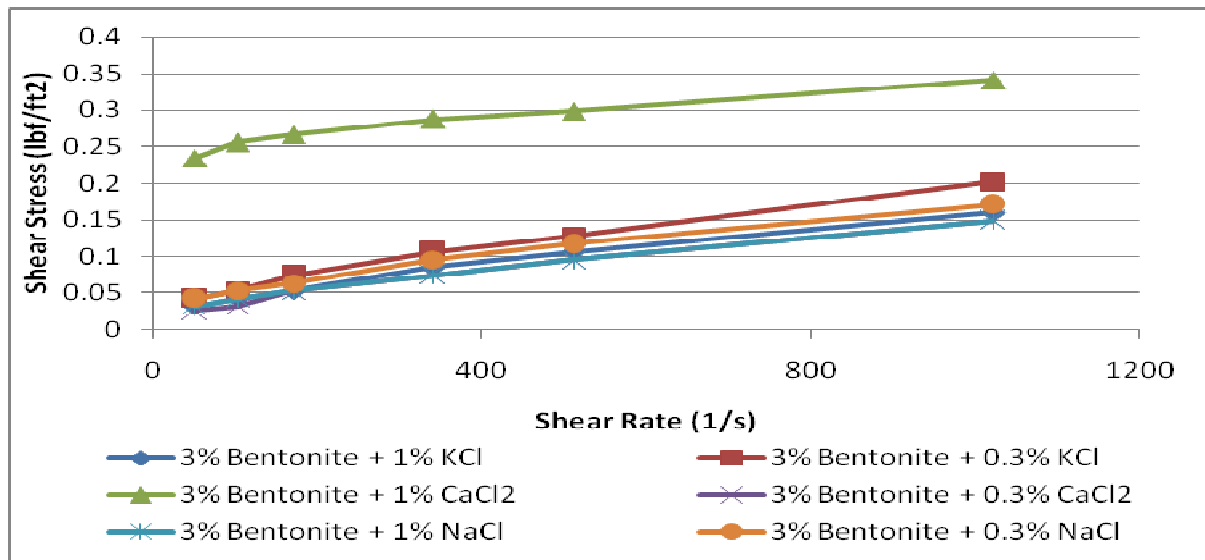
5

357 Where,

358 γ = shear rate (1 / s)

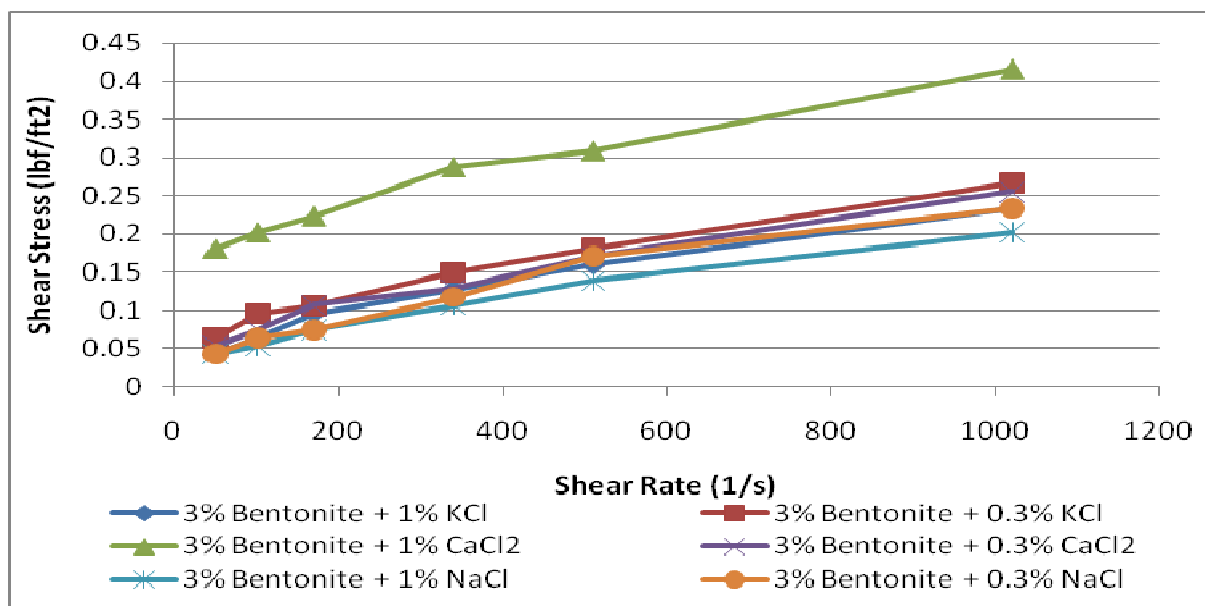
359 RPM = viscometer rotational speed

360 A rheogram which is a plot of shear stress versus shear rate was developed for the fluids
361 before and after dispersion tests. Figure 6 shows the plots for the bentonitic fluids before
362 dispersion test at 120°F while Figures 7 and 8 shows the plots for the bentonitic fluids after
363 dispersion test with Shales from Well A and B respectively at 120°F.



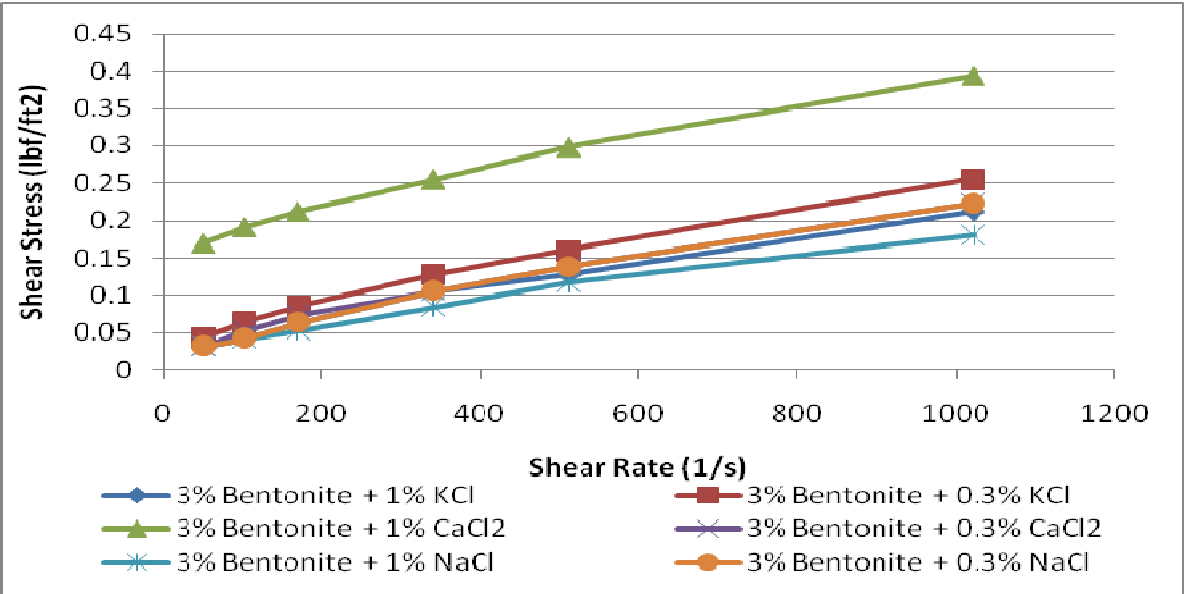
364

365 **Figure 6:** Rheogram for Bentonitic Fluids before Dispersion Test at 120°F



366

367 **Figure 7:** Rheogram for Bentonitic Fluids after Dispersion Tests with Well A
 368



369
 370 **Figure 8:** Rheogram for Bentonitic Fluids after Dispersion Tests with Well B
 371

372 The plastic viscosity (PV) and yield point (YP) were calculated before and after dispersion
 373 using the following equations:

374
 375
$$\mu_p = \theta_{600} - \theta_{300} \quad 1$$

376
 377
$$\tau_o = \theta_{300} - \mu_p \quad 2$$

378
 379 Where,
 380 μ_p = plastic viscosity (cp)
 381 τ_o = yield stress ($\text{lb}_f / 100\text{ft}^2$)
 382 θ_{600} = dial reading at 600 rpm
 383 θ_{300} = dial reading at 300 rpm

384 Table 6 show the PV and Table 7 show the YP values obtained for the bentonitic fluids.

385
 386
 387
 388
 389

390 **Table 6: Plastic Viscosity Values for Bentonitic Fluids before and after hot rolling with**
 391 **Shales in Well A and Well B**
 392

Fluid Composition	Well A Plastic Viscosity (centipoise, cP) At 120°F		Well A Plastic Viscosity (centipoise, cP) At 120°F	
	Before	After	Before	After
3% Bentonite + 1% KCl	5	7	5	8
3% Bentonite + 0.3% KCl	7	8	7	9
3% Bentonite + 1% CaCl ₂	4	10	4	9
3% Bentonite + 0.3% CaCl ₂	5	8	5	8
3% Bentonite + 1% NaCl	5	6	5	6
3% Bentonite + 0.3% NaCl	5	6	5	8

393
 394
 395 **Table 7: Yield Point Values for Bentonitic Fluids before and after hot rolling with**
 396 **Shales in Well A and Well B**

Fluid Composition	Well A Yield Point (lbs/100ft ²) At 120°F		Well B Yield Point (lbs/100ft ²) At 120°F	
	Before	After	Before	After
3% Bentonite + 1% KCl	5	8	5	4
3% Bentonite + 0.3% KCl	5	9	5	6
3% Bentonite + 1% CaCl ₂	24	19	24	19
3% Bentonite + 0.3% CaCl ₂	4	8	4	5
3% Bentonite + 1% NaCl	4	7	4	5
3% Bentonite + 0.3% NaCl	6	10	6	5

397

398 A comparison of PV values before and after hot-rolling the shale samples showed distinct
399 outcome with the salt/bentonite fluids. The salt/bentonite fluids showed an increase in PV
400 when hot-rolled at 120°F; which indicates shale dispersion.

401 Yield point for the salt/bentonite fluids showed a decrease in YP after hot-rolling at 120°F.
402 YP is a function of electrostatic forces between fluid particles in motion [12]. For Bingham
403 fluids, it is also the shear stress required to initiate flow in fluids. From YP results, the
404 dispersion tests reduced the attractive force between the solid particles significantly. In this
405 study, rheological parameters were determined assuming Bingham plastic fluid model.

406 The percent recoveries shown can only be used as a comparison between the fluids
407 represented in this study.

408 **3.5 Swelling Test Result**

409 Salt additives {Potassium Chloride (KCl), Sodium Chloride (NaCl), Calcium Chloride (CaCl₂)}
410 when used in the drilling fluid have proved to be beneficial in stabilizing shale formations.
411 They also lower the water activity of the drilling fluid and thus generate osmotic potentials.

412 Linear Swelling tests were carried out on Shale samples from Well A and B using Water
413 Base-mud and deionized water. Table 8 shows the summary of the linear swelling test
414 results. Both shale samples swell in deionized water after immersion for 24 hours. They also
415 showed reactivity when placed in all the test fluids. Maximum linear swell for this shale was
416 obtained in the deionized water. The maximum reading on the digital Veneer caliper
417 experienced by Shale in Well A was 0.0510 inches and for Shale in Well B was 0.0457
418 inches. This gave an 8.15% and 7.30% linear swell respectively. A sample calculation is
419 shown below.

420

421

422

423

424

425

426

427

428

429

430

431

432

433

434 **Table 8: Linear Swelling Results**

Drilling Fluid Composition	Maximum Percent Linear Swelling (%)		Qualitative Description	
	Shale A	Shale B	Shale A	Shale B
De-ionized Water	8.15	7.30	Disintegration	Disintegration
Base Fluid	6.54	5.12	Partial Disintegration	Partial Disintegration
3% Bentonite + 1% KCl	1.30	0.20	Intact, soft	Intact, firm
3% Bentonite + 0.3% KCl	3.33	1.56	Intact, soft	Intact, soft
3% Bentonite + 1% CaCl ₂	0.59	2.80	Intact, firm	Intact, soft
3% Bentonite + 0.3% CaCl ₂	2.02	3.26	Intact, soft	Intact, soft
3% Bentonite + 1% NaCl	2.74	2.35	Intact, soft	Intact, soft
3% Bentonite + 0.3% NaCl	1.70	3.91	Intact, soft	Intact, soft

435

436 *Sample calculation for Well A*

437 L = 0.626 in.

438 ΔL = 0.0510 in.

439

440
$$\epsilon = \frac{\Delta L}{L} \times 100$$

441

442
$$\epsilon = \frac{0.0510 \text{ in}}{0.626 \text{ in}} \times 100 = 8.15\%$$

443

444 Where,

445 L = length of shale slab (in)

446 ΔL = change in shale length (in)

447 ε = percent linear swell (%)

448

449 For Well A, the maximum readings on the digital Veneer Caliper for 3% Bentonite + 0.3%

450 KCl, 3% Bentonite + 0.3% CaCl₂ and 3% Bentonite + 1% NaCl were 0.0208 inches, 0.0127

451 inches and 0.0171 inches respectively. The percent linear swellings calculated from these
452 values are 3.33%, 2.02% and 2.74%.

453 For Well B, the maximum readings on the digital Veneer Caliper for 3% Bentonite + 0.3%
454 KCl, 3% Bentonite + 0.3% CaCl_2 and 3% Bentonite + 0.3% NaCl were 0.0098 inches,
455 0.0204 inches and 0.0245 inches respectively. The percent linear swellings calculated from
456 these values are 1.56%, 3.26% and 3.91%. All of these are lower than the value obtained for
457 the de-ionized water. That is, they all provided inhibition to some extent. The 3% Bentonite +
458 1% CaCl_2 and 3% Bentonite + 1% KCl fluids superseded all other fluids as minimum swelling
459 was observed using this fluids for Well A and B shale samples respectively; while the 3%
460 Bentonite + 0.3% KCl and 3% Bentonite + 0.3% NaCl fluids gave the highest linear swelling
461 for Shale samples in Well A and B respectively.

462 The results obtained from swelling tests are similar to those from the dispersion tests. The
463 3% Bentonite + CaCl_2 and 3% Bentonite + KCl fluids provided maximum inhibition for both
464 tests. Though, swelling and dispersion are different phenomena. The magnitude of inhibition
465 observed in the swelling test was higher than in the dispersion tests. Therefore, shale
466 swelling is more sensitive to fluid selection than dispersion.

467 This research provides information on the characterization of shale from Agbada formation
468 and the development of inhibitive water-based drilling fluid.

469 We observed from chemical and mineralogy analyses that the type and relative amounts of
470 mineral present in a shale sample may differ from one oil field to another. These minerals
471 contained in each shale formation should be considered during well planning so as to select
472 the best water-based drilling fluid that will inhibit the shale formation without any wellbore
473 instability problems.

474

475

476

477

478

479

480

481

482

483

484

485

486

487 **Table 9: Applications of Potassium Systems for Drilling Shale with Water-Base Fluid**

Shale Type	Dispersion- Limiting Ability of KCl	Ability of KCl to provide Sufficient Hydration Reduction	Is the use of KCl suggested?
Soft, Fairly high Dispersion, Fairly high montmorillonite, High in interlayered Clays.	Good	Good	Yes
Medium-hard, Moderate Dispersion, Chlorite, High in illite.	Excellent	Very Good	Yes

488

489 **Table 10: Applications of Calcium Systems for Drilling Shale with Water-Base Fluid**

Shale Type	Dispersion- Limiting Ability of CaCl ₂	Ability of CaCl ₂ to provide Sufficient Hydration Reduction	Is the use of CaCl ₂ suggested?
Soft, Fairly high Dispersion, Fairly high montmorillonite, High in interlayered Clays.	Excellent	Good	Yes
Medium-hard, Moderate Dispersion, Chlorite, High in illite.	Good	Fair	Depends on Shale Type

490

491

492

493

494

495

496 **Table 11: Applications of Sodium Systems for Drilling Shale with Water-Base Fluid**

Shale Type	Dispersion- Limiting Ability of NaCl	Ability of NaCl to provide Sufficient Hydration Reduction	Is the use of NaCl suggested?
Soft, Fairly high Dispersion, Fairly high montmorillonite, High in interlayered Clays.	Fair	Fair	Depends on Shale Type
Medium-hard, Moderate Dispersion, Chlorite, High in illite.	Fair	Fair	Depends on Shale Type

497

498

499 **4. CONCLUSION**

500

501 Based on the experimental results obtained from this study, the following conclusions were
502 made.

503

1. Adsorption isotherms modeled for the shale samples from both wells using GAB model indicate the dependence of shale adsorptive properties on cation exchange capacity (CEC) alone, since their total organic carbon content is approximately the same.

504

505

506

507

2. Shale from Agbada formation that exhibit the same characteristics with the sample from Well A will be drilled using Base fluid (water + 3% Bentonite) + KCl salt, because of the Montmorillonite clay present. Shale from this same formation that exhibit the same characteristics with the sample from Well B will be drilled using Base fluid (water + 3% Bentonite) + KCl salt.

508

509

510

511

512

3. This study has shown that when drilling shale formations, the actual, not higher salt content in drilling fluids will reduce wellbore instability and improve drilling performance. The rheology is more affected by the concentration of the additives in this study.

513

514

515

516

4. Water-based fluids require a much lower activity than the shale to control water transport; from the dispersion test it was observed that the shale samples dispersed more in low concentrations of salt.

517

518

520

521

522

523

524

525

526

527

528

529

530

531

532

533

534

535

536

537

538

539

540

541

542

543

544

545

546

547

548

549

550

551

552

- 1 Chenevert ME. Shale alteration by water adsorption. Journal of Petroleum Technology. Paper presented at SPE/ISRM 78162, Rock Mechanics Conference, Texas 2002.
- 2 Oort EV. New flat-rheology Synthetic-Based mud for improved deep water drilling. Paper SPE 90987, presented at the SPE annual Technical conference and exhibition, Houston, Texas, USA, September 26-29, 2003.
- 3 Chenevert ME. Shale Control with Balanced Activity Oil Muds, JPT Trans AIME, 1970: **249**, pp 1309-1316.
- 4 Osisanya SO. Experimental Studies of Wellbore Stability in Shale Formations, Ph.D Dissertation. The University of Texas at Austin, 1991.
- 5 Mody FK, Hale, A.H. A borehole stability model to couple the mechanics and chemistry of drilling fluid shale interaction. SPE/IADC 25728, SPE/IADC Drilling conference, Amsterdam, February 23-25, 1993.
- 6 Chenevert ME, Osisanya SO. Shale swelling at elevated temperature and pressure. Presented at the 33rd Symposium in Rock Mechanics, Santa Fe, New Mexico, June 8-10, 1996.
- 7 Oort VE, Hale AH, Mody FK, Roy S. Critical parameters in modeling the chemical aspects of Borehole stability in shales and in designing improved water-based shale drilling fluids. SPE 28309, paper presented at the SPE annual conference, New Orleans, September 26-28, 1996.
- 8 O'Brien DE, Chenevert ME. Stabilizing sensitive shales with inhibited Potassium based drilling fluids. Journal of Petroleum Technology, 1973, September, 255.
- 9 Ewy R, Morton E. Wellbore stability performance of water base mud additives. Journal of petroleum Technology, 2008.
- 10 Kehinde Adesoye. Shale characterization for evaluating shale-drilling fluid interaction. University of Oklahoma, MSc. Thesis, 2009.
- 11 Keijzer THJS, Kleingeld PJ, Loch JPG. Chemical osmosis in compacted clayey material and the prediction of water transport. Engineering Geology, 1999: **53**, 151-159.
- 12 Roscoe Moss Company. Handbook of ground water development. New York, John Wiley and Sons, 439p, 1990.

- 553 13 O'Brien DE, Chenevert ME. Shale control with balanced activity oil continuous
554 muds. Journal of Petroleum Technology, 1973.
- 555 14 Al-Awad NJ, Smart BGD. Characterization of shale-drilling fluid interaction
556 mechanisms related to wellbore instability, J. King Saud Univ., 1996: **8**, Eng.
557 Sci. (2), pp 187-215.
- 558 15 Osisanya SO. Practical Approach to solving wellbore instability problems. 50th
559 anniversary of the SPE Distinguished Lecturer program.
- 560 16 Yu M, Chenevert ME, Sharma MM. Chemical-mechanical wellbore instability
561 model in shales. Journal of Petroleum Science & Technology, 2002.
- 562 17 Bourgoyne AT, Millheim KK, Chenevert ME, Young FS. Applied drilling
563 engineering. 2nd ed. Society of petroleum Engineers, Callister, W.D. 1991.

564 **DEFINITIONS, ACRONYMS, ABBREVIATIONS**

565	a	= activity
566	a_w	= water activity
567	BET	= Brunauer, Emmett and Teller
568	C	= constant
569	C	= Guggenheim constant
570	°C	= degree Celsius
571	CEC	= Cation Exchange Capacity, Meq/100g Solid
572	°F	= degree Fahrenheit
573	GAB	= Guggenheim, Anderson, and DeBoer
574	Δh_c	= specific bonding enthalpy of water monolayer, J/Kg
575	Δh_k	= mean specific bonding enthalpy of the water multilayer, J/Kg
576	$\Delta h_{s, mono}$	= specific sorption enthalpy of water monolayer, J/Kg
577	Δh_{vap}	= specific vaporization enthalpy of water monolayer, J/Kg
578	$\Delta h_{s, multi}$	= mean specific sorption enthalpy of water multilayer, J/g
579	K	= sorption energies if multiple layers of molecule
580	K	= temperature dependent constant
581	K_o	= adjustable parameter accounting for temperature effect
582	ΔL	= change in shale length, in
583	L	= original length of shale sample, in
584	MC	= moisture content, %
585	Meq/100g	= milliequivalent weights of methylene blue per 100g of solid
586	N	= spring factor
587	NMC	= native Moisture Content, %

588	OBM	= oil-Based Mud
589	P	= pressure
590	P_o	= gas saturation pressure
591	P/P_o	= relative vapor pressure
592	P_{π}	= swelling pressure
593	P_{ads}	= adsorptive Pressure
594	PHPA	= partially hydrolyzed polyacrylamide
595	R	= universal gas constant, J/Kg/K
596	T	= temperature, K
597	TOC	= Total Organic Content, %
598	W_i	= initial weight, g
599	W_f	= final weight, g
600	W_c	= weight of carbon removed by ashing, g
601	W_s	= weight of shale before experimental procedure, g
602	W_u	= weight of undispersed shale, g
603	W_w	= weight of water removed by drying, g
604	W_g	= weight of water gained by the shale sample, g
605	WBM	= Water-Based Mud
606	XRD	= X-Ray Diffraction Analysis

607

608 **Greek Symbols**

609	θ	= fraction of available active sites
610	θ_{300}	= dial reading at 300 rpm
611	θ_{600}	= dial reading at 600 rpm
612	ϵ	= percent linear swell, %
613	τ	= shear stress, lbf/ft ²
614	τ_o	= yield stress, lbf/100ft ²
615	μ_p	= plastic viscosity, cp
616	γ	= shear rate, 1/s

617

618

619

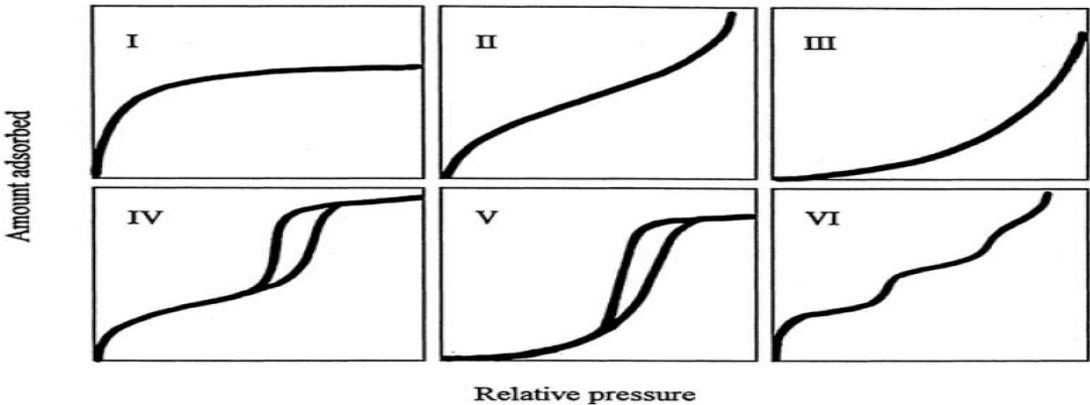
620

621

APPENDIX A: DATA SPREAD SHEETS

IUPAC CLASSIFICATION OF ADSORPTION ISOTHERMS FOR GAS-SOLID EQUILIBRIA

(AFTER DONOHUE 1998)



622

623

624

Table A.1: Adsorption Isotherms

	WELL A			WELL B		
Water	Weight _{Initial}	Weight _{Final}	%	Weight _{Initial}	Weight _{Final}	%
Activity	(g)	(g)	Difference	(g)	(g)	Difference
0	0.00	0.00	0.00	0.00	0.00	0.00
0.30	23.36	27.05	10.44	23.96	24.57	2.55
0.51	23.42	26.05	11.23	23.30	24.74	5.09
0.76	23.23	25.40	13.37	23.23	24.43	5.17
0.86	23.96	27.88	16.36	23.45	24.50	6.38
0.94	23.10	28.02	17.51	23.07	25.50	6.93
0.96	23.98	29.70	27.6	23.91	25.54	7.80

625

626

Table A.2: Total Organic Content

Shale	Crucible	Crucible +	Sample	Crucible +	Sample	%
Sample	(g)	Sample Before	Before	Sample After	After	TOC
		(g)	(g)	(g)	(g)	
Well A	25.346	32.754	7.480	32.012	6.666	10.02
Well B	26.670	34.155	7.480	33.360	6.690	10.62

627 **Table A.3: Adsorption Isotherm Modeling for Well A and B**

628

Shale A			Shale B		
$m_o =$	6.7284		$m_o =$	2.9987	
$C =$	19.9324		$C =$	18.7147	
$K =$	0.7486		$K =$	0.6410	
Aqueous	Experimental		Aqueous	Experimental	
Activity	Data	Model Data	Activity	Data	Model Data
0.3	10.44	7.3962	0.3	2.55	3.0322
0.51	11.23	10.0663	0.51	5.09	4.0136
0.76	13.37	15.0383	0.76	5.17	5.5359
0.86	16.36	18.3807	0.86	6.38	6.4040
0.94	19.51	22.2396	0.94	6.93	7.2879
0.96	27.26	23.4574	0.96	7.8	7.5443

629
630
631
632
633
634
635
636
637
638
639
640
641
642
643
644
645
646
647
648
649
650
651
652

Table A.4: Well A Regression Statistics Summary Output

Regression Statistics					
Multiple R	0.972494562				
R Square	0.945745674				
Adjusted R Square	0.932182092				
Standard Error	1.688980572				
Observations	6				
ANOVA					
	df	SS	MS	F	Significance F
Regression	1	198.9066425	198.9066425	69.72683923	0.001124419
Residual	4	11.41062149	2.852655371		
Total	5	210.3172639			

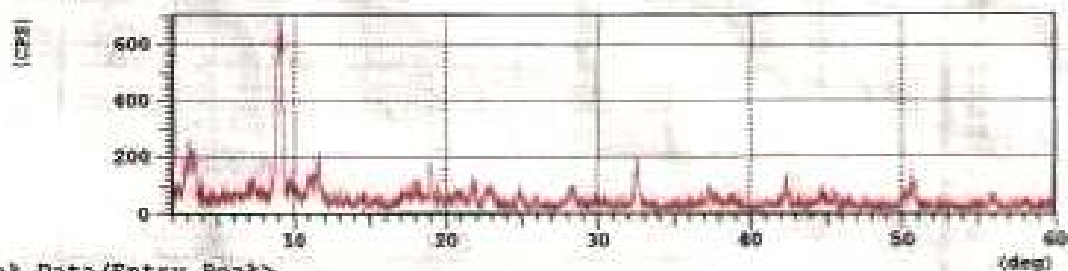
Table A.5: Well B Regression Statistics Summary Output

Regression Statistics					
Multiple R	0.98853539				
R Square	0.97720222				
Adjusted R Square	0.97150277				
Standard Error	0.30556391				
Observations	6				
ANOVA					
	df	SS	MS	F	Significance F
Regression	1	16.00869531	16.00869531	171.45566	0.000196402
Residual	4	0.373477209	0.093369302		
Total	5	16.38217252			

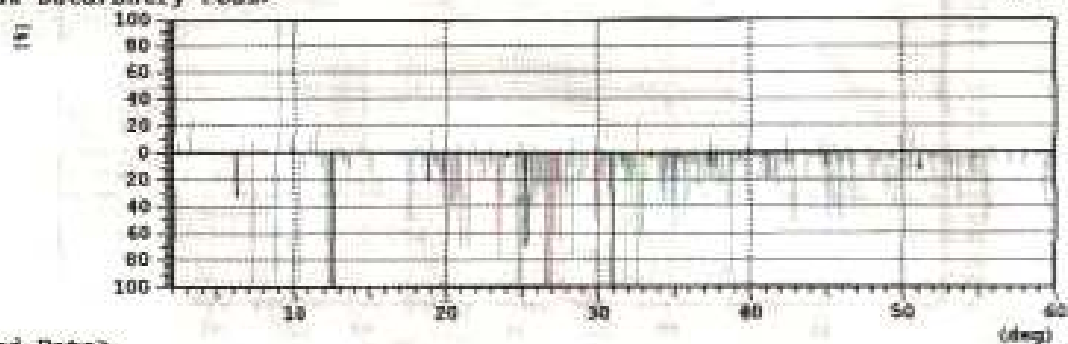
APPENDIX B – X-RAY DIFFRACTION RESULTS

DATA NAME: WELL A
 File Name: Sample A
 SAMPLE NAME: AGBADA SHALE
 Comment : DARK-SHALE
 Date & Time : 04-05-12 08:58:04

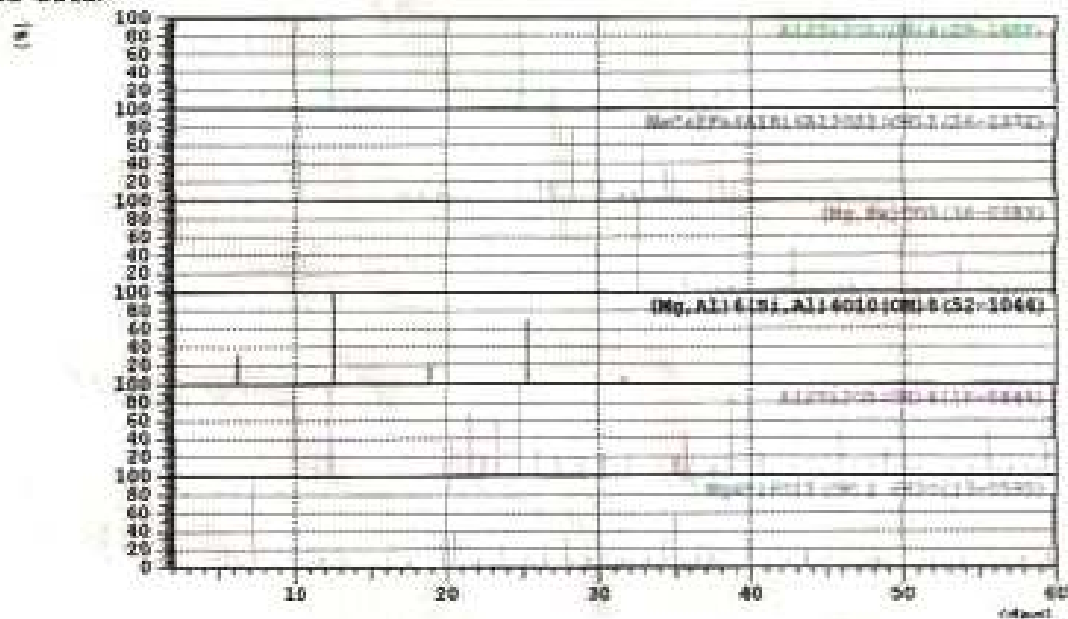
<Raw Data>

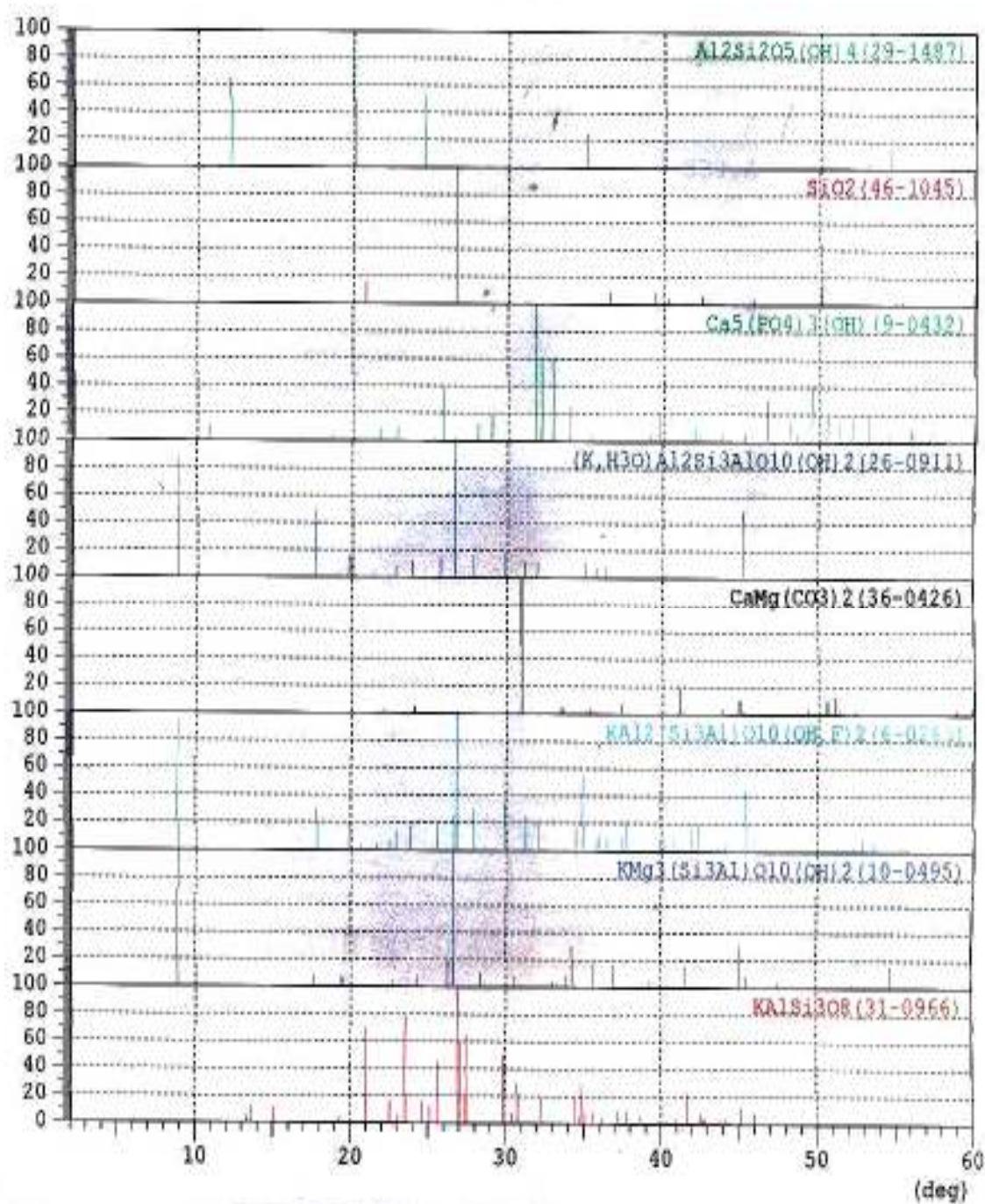


<Peak Data/Entry Peak>



<Card Data>





Data; Well A

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (Å)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	7	9.1795	9.62627	100	0.50570	96	2398
2	2	3.5255	25.04150	26	0.71670	25	920
3	26	32.6153	2.74328	23	0.35000	22	419

Peak Data List

peak no.	2Theta (deg)	d (Å)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.8030	31.49436	7	0.44000	7	170
2	3.5255	25.04150	26	0.71670	25	920
3	4.2242	20.90097	3	0.04000	3	20
4	6.4415	13.71053	3	0.03000	3	10
5	7.4677	11.82859	8	0.44000	8	236
6	8.0791	10.93476	4	0.12000	4	36
7	9.1795	9.62627	100	0.50570	96	2398
8	9.9400	8.89142	7	0.36000	7	126
9	10.1583	8.70083	5	0.20000	5	73
10	11.2111	7.88601	11	0.24000	11	145
11	11.6684	7.57795	17	0.56000	16	431
12	12.6435	6.99563	3	0.04000	3	12
13	13.1165	6.74440	4	0.23000	4	87
14	14.6859	6.02700	5	0.14000	5	73
15	17.4891	5.06678	5	0.12000	5	75
16	18.2577	4.85518	6	0.30000	6	155
17	19.0989	4.64319	16	0.26670	15	218
18	21.0299	4.22101	5	0.42000	5	171
19	21.9376	4.04836	11	0.28000	11	181
20	22.9003	3.88031	9	0.59000	9	300
21	24.9611	3.56442	8	0.30000	8	146
22	26.0092	3.42311	4	0.28000	4	68
23	28.3356	3.14713	10	0.42000	10	259
24	29.9034	2.98560	4	0.16000	4	98
25	30.3928	2.93863	3	0.06000	3	35
26	32.6153	2.74328	23	0.35000	22	419
27	35.1378	2.55191	3	0.04000	3	20
28	37.3858	2.40347	7	0.26000	7	138
29	37.8055	2.37774	3	0.14000	3	44
30	38.8547	2.31592	4	0.24000	4	72
31	41.0931	2.19479	3	0.12000	3	34
32	42.4422	2.12809	13	0.30000	12	217
33	44.7009	2.02566	7	0.26000	7	142
34	45.5854	1.98839	6	0.29000	6	115
35	46.5499	1.94942	4	0.20000	4	68
36	47.7193	1.90434	4	0.18000	4	69
37	50.2181	1.81528	9	0.22000	9	110
38	50.6829	1.79972	15	0.37000	14	276
39	55.9358	1.64251	5	0.22000	5	89
40	58.1450	1.58525	4	0.24000	4	82

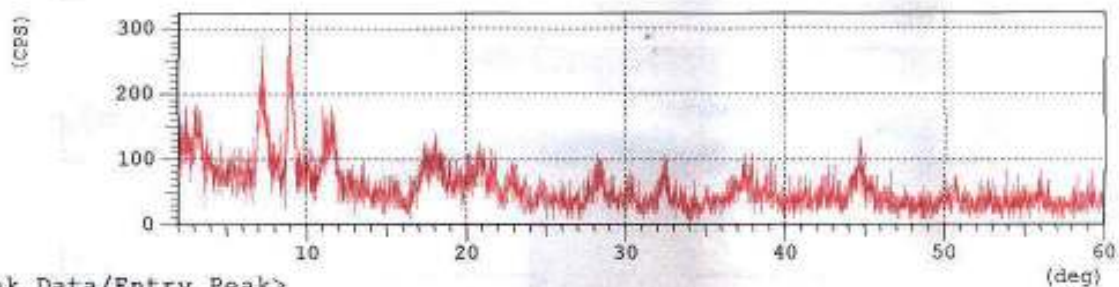
Group Name : Well A
 Data Name : SAMPLE-A
 File Name : SAMPLE-A.PSE
 Sample Name : AGBADASHALE
 Comment : DARK-SHALE

<Entry Card>

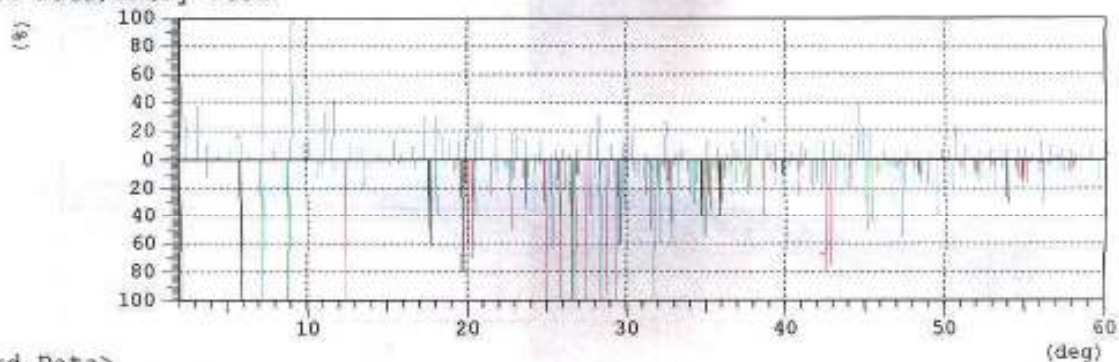
No.	Card	Chemical Formula	S	L	d	I	B
		Chemical Name (Mineral Name)		Dx	WT%	S.G.	
1	29-1488	Al ₂ Si ₂ O ₅ (OH) ₄	0.142	0.714(10/21)	0.590	-----	0.422
		Aluminum Silicate Hydroxide (Kaolinite-1\	-----			-----	
2	26-1372	NaCa ₂ Fe ₄ Al ₈ Si ₆ Al ₂ O ₂₂ (OH) ₂	0.164	0.667(10/15)	0.605	-----	0.403
		Sodium Calcium Iron Aluminum Silicate Hydr	-----			-----	
3	36-0383	(Mg,Fe)CO ₃	0.273	0.625(5/14)	0.632	-----	0.395
		Iron Magnesium Carbonate (Magnesite, ferr	-----			-----	
4	52-1044	(Mg,Al) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	0.139	0.625(5/12)	0.597	-----	0.373
		Magnesium Aluminum Silicate Hydroxide (Ch	-----			-----	
5	10-0446	Al ₂ Si ₂ O ₅ (OH) ₄	0.170	0.560(21/39)	0.592	-----	0.336
		Aluminum Silicate Hydroxide (Dickite-2\IT	-----			-----	
6	13-0595	Mg ₄ Si ₆ O ₁₅ (OH) ₂ ·6H ₂ O	0.301	0.583(21/39)	0.574	-----	0.335
		Magnesium Silicate Hydroxide Hydrate (Sep	-----			-----	
7	29-1487	Al ₂ Si ₂ O ₅ (OH) ₄	0.099	0.667(4/ 7)	0.488	-----	0.325
		Aluminum Silicate Hydroxide (Halloysite-7	-----			-----	
8	46-1045	SiO ₂	0.625	0.385(5/42)	0.811	-----	0.312
		Silicon Oxide (Quartz, syn)	-----			-----	
9	9-0432	Ca ₅ (PO ₄) ₃ (OH)	0.484	0.514(10/42)	0.563	-----	0.290
		Calcium Phosphate Hydroxide (Hydroxylapat	-----			-----	
10	26-0911	(K,H ₃ O)Al ₂ Si ₃ AlO ₁₀ (OH) ₂	0.504	0.471(6/18)	0.607	-----	0.286
		Potassium Aluminum Silicate Hydroxide (Il	-----			-----	
11	36-0426	CaMg(CO ₃) ₂	0.627	0.412(7/41)	0.640	-----	0.263
		Calcium Magnesium Carbonate (Dolomite)	-----			-----	
12	6-0263	KAl ₂ (Si ₃ Al)O ₁₀ (OH,F) ₂	0.461	0.405(17/42)	0.643	-----	0.260
		Potassium Aluminum Silicate Hydroxide (Mu	-----			-----	
13	10-0495	KMg ₃ (Si ₃ Al)O ₁₀ (OH) ₂	0.741	0.542(13/26)	0.466	-----	0.253
		Potassium Magnesium Aluminum Silicate Hydr	-----			-----	
14	31-0966	KAlSi ₃ O ₈	0.333	0.405(17/42)	0.614	-----	0.248
		Potassium Aluminum Silicate (Orthoclase)	-----			-----	

```
Data Name      : WELL B
File Name     : SAMPLE-B.PKR
Sample Name    : AGBADA SHALE
Comment       : DARKISH-SHALE
Date & Time    : 04-04-12 14:03:39
```

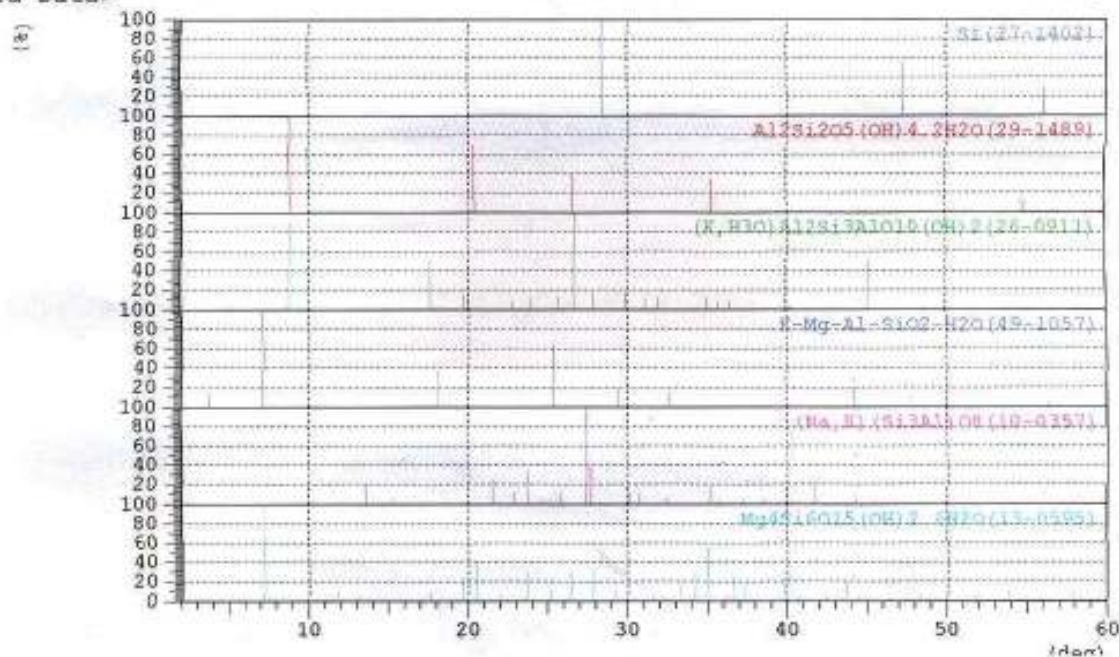
<Raw Data>

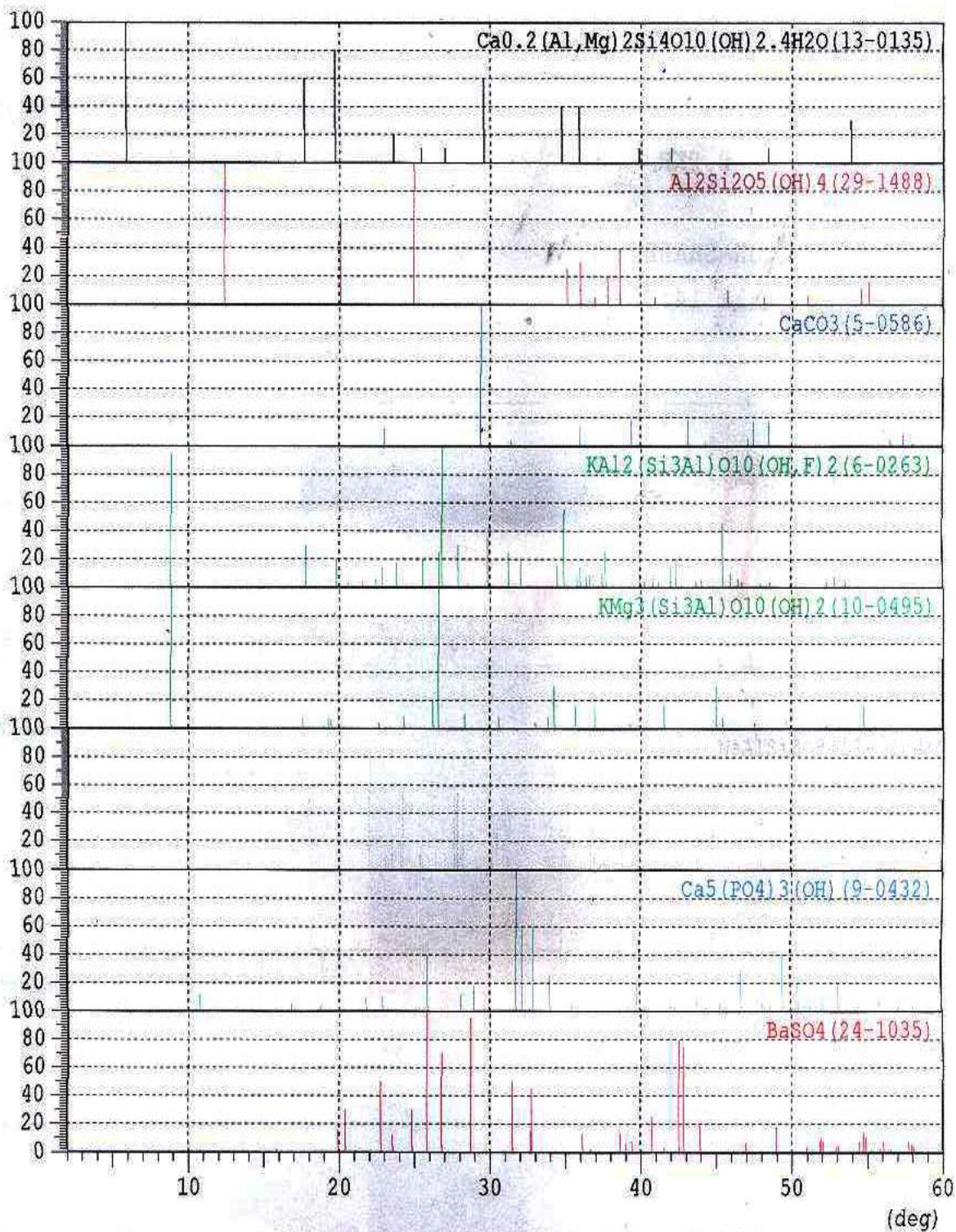


<Peak Data/Entry Peak>



<Card Data>





DATA ; WELL B

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (Å)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	11	9.1783	9.62753	100	0.43670	30	631
2	8	7.4234	11.89908	80	0.51000	24	681
3	15	11.7712	7.51200	43	0.40670	13	276

Peak Data List

peak no.	2Theta (deg)	d (Å)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.8115	31.39916	27	0.50000	8	217
2	3.4966	25.24841	37	0.52000	11	297
3	4.1108	21.47728	10	0.04000	3	18
4	4.7961	18.40989	3	0.00000	1	0
5	5.4523	16.19559	7	0.08000	2	9
6	6.0896	14.50201	13	0.15000	4	33
7	6.5443	13.49539	3	0.00000	1	0
8	7.4234	11.89908	80	0.51000	24	681
9	8.0791	10.93476	7	0.00000	2	0
10	8.4347	10.47454	13	0.16000	4	58
11	9.1783	9.62753	100	0.43670	30	631
12	9.9202	8.90912	10	0.04000	3	13
13	10.3220	8.56320	13	0.19000	4	37
14	11.2508	7.85827	33	0.32000	10	186
15	11.7712	7.51200	43	0.40670	13	276
16	12.9422	6.83484	10	0.04000	3	14
17	13.3007	6.65141	7	0.04000	2	9
18	13.6643	6.47522	10	0.05000	3	22
19	14.6560	6.03923	3	0.00000	1	0
20	15.5833	5.68189	13	0.06000	4	29
21	15.9923	5.53748	3	0.00000	1	0
22	16.7905	5.27599	10	0.08000	3	25
23	17.4991	5.06391	30	0.34000	9	247
24	18.1779	4.87632	30	0.30000	9	233
25	18.6072	4.76477	17	0.00000	5	0
26	19.0657	4.65120	13	0.12000	4	41
27	19.6040	4.52468	13	0.04000	4	25
28	20.1025	4.41359	20	0.16000	6	119
29	20.7008	4.28736	23	0.08000	7	55
30	21.0199	4.22299	27	0.44000	8	199
31	21.8678	4.06113	17	0.14000	5	65
32	22.9053	3.87947	20	0.22000	6	98
33	23.2246	3.82685	17	0.10000	5	47
34	23.7735	3.73972	13	0.20000	4	46
35	24.3722	3.64919	3	0.00000	1	0
36	24.7615	3.59270	13	0.14000	4	73
37	25.3903	3.50513	3	0.00000	1	0
38	25.7098	3.46229	7	0.08000	2	12
39	26.0991	3.41152	7	0.06000	2	18
40	26.5284	3.35728	3	0.00000	1	0
41	26.9577	3.30478	7	0.14000	2	19
42	27.8064	3.20581	17	0.12000	5	55
43	28.3206	3.14876	30	0.31000	9	246
44	29.1544	3.06058	10	0.10000	3	45
45	29.8335	2.99244	10	0.06000	3	20

Data Name : WELL B
 File Name : SAMPLE-B.PKR
 Sample Name : AGBADA SHALE
 Comment : DARKISH-SHALE

<Entry Card>

No.	Card	Chemical Formula	S	L	d	I	R
		Chemical Name (Mineral Name)	Dx	WT%	S.G.		
1	27-1402	Si	0.270	1.000(3/11)	0.569	0.728	0.414
		Silicon (Silicon, syn)	-----		-----		
2	29-1489	Al ₂ Si ₂ O ₅ (OH) ₄ ·2H ₂ O	0.564	1.000(5/ 7)	0.619	0.540	0.334
		Aluminum Silicate Hydroxide Hydrate (Hall	-----		-----		
3	26-0911	(K,H ₃ O)Al ₂ Si ₃ AlO ₁₀ (OH) ₂	0.567	0.882(15/18)	0.757	0.481	0.321
		Potassium Aluminum Silicate Hydroxide (Il	-----		-----		
4	49-1057	K-Mg-Al-SiO ₂ -H ₂ O	0.629	0.909(10/22)	0.690	0.498	0.313
		Potassium Iron Magnesium Aluminum Silicate	-----		-----		
5	10-0357	(Na,K)(Si ₃ Al) ₈	0.632	0.941(16/17)	0.716	0.460	0.310
		Potassium Sodium Aluminum Silicate (Sanid	-----		-----		
6	13-0595	Mg ₄ Si ₆ O ₁₅ (OH) ₂ ·6H ₂ O	0.564	0.917(33/39)	0.709	0.451	0.293
		Magnesium Silicate Hydroxide Hydrate (Sep	-----		-----		
7	13-0135	Ca _{0.2} (Al,Mg) ₂ Si ₄ O ₁₀ (OH) ₂ ·4H ₂ O	0.289	0.923(12/17)	0.596	0.530	0.292
		Calcium Magnesium Aluminum Silicate Hydrox	-----		-----		
8	29-1488	Al ₂ Si ₂ O ₅ (OH) ₄	0.326	1.000(14/21)	0.619	0.470	0.291
		Aluminum Silicate Hydroxide (Kaolinite-1\	-----		-----		
9	5-0586	CaCO ₃	0.398	0.833(10/42)	0.743	0.402	0.249
		Calcium Carbonate (Calcite, syn)	-----		-----		
10	6-0263	KAl ₂ (Si ₃ Al) ₈ O ₁₀ (OH,F) ₂	0.583	0.833(35/42)	0.693	0.430	0.248
		Potassium Aluminum Silicate Hydroxide (Mu	-----		-----		
11	10-0495	KMg ₃ (Si ₃ Al) ₈ O ₁₀ (OH) ₂	0.603	0.833(20/26)	0.719	0.386	0.231
		Potassium Magnesium Aluminum Silicate Hydr	-----		-----		
12	19-1184	NaAlSi ₃ O ₈	0.402	0.667(28/42)	0.773	0.423	0.218
		Sodium Aluminum Silicate (Albite, ordered	-----		-----		
13	9-0432	Ca ₅ (PO ₄) ₃ (OH)	0.500	0.886(31/42)	0.619	0.393	0.215
		Calcium Phosphate Hydroxide (Hydroxylapat	-----		-----		
14	24-1035	BaSO ₄	0.341	0.829(34/42)	0.731	0.355	0.215
		Barium Sulfate (Barite, syn)	-----		-----		

663
 664
 665