



Chemical, Morphological Characterizations of Ririwai Biotite and Determination of Yield Point of its Weighting Agent Application in Drilling Mud

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Abstract

Chemical, morphological characterizations and drilling mud yield point impact of Ririwai biotite have been investigated and reported in this work. Local Ririwai biotite mined in Doguwa Local Government Area of Kano State was used as a weighting agent in drilling mud formulation. Scanning Electron Microscopy (SEM) characterization, X-ray Diffraction (XRD) and Electron Dispersion X-ray (EDX) analysis of the Ririwai biotite were carried out. Water-based drilling mud was prepared using commercial bentonite according to the API 13A Standard. Effect of gradual addition of Ririwai weighting agent; 0 – 100 wt%, on the yield point of the formulated drilling mud was studied. XRD analysis showed that the dominant mineral phase in the material was biotite. Morphological analysis carried out showed that the Ririwai biotite had a sheet-like morphology while the commercial bentonite had clumpy morphology. The estimated average particle sizes were 60 and 25 μm for the Ririwai biotite and commercial bentonite, respectively. EDX analysis showed that silica-alumina ratio of the Ririwai biotite was 9.3 while that of the commercial bentonite was 1.58. The optimum yield point of the formulated drilling mud was 2.0 lb / 100 ft^2 corresponding to formulation having 0 – 30 wt% weighting agent composition. The specific gravity of Ririwai biotite was determined as 2.4.

Keywords: Ririwai biotite, SEM, EDX, drilling mud, yield point, morphology

1. INTRODUCTION

Drilling mud is a suspension fluid that is indispensable in well drilling. Drilling mud performs key roles in petroleum production [1], such as cooling and lubricating drilling tools, carrying and suspending cuttings, cleaning wellbore, minimizing fluid loss across permeable formation, and maintain the stability of the wellbore and formation [2–5]. The main functions of drilling fluids include providing hydrostatic pressure to prevent formation fluids from entering into the well bore, keeping the drill bit cool and clean during drilling. The drilling fluid used for a particular job is selected to avoid formation damage and to limit corrosion [6]. Water-based bentonite drilling fluid is widely used in the petroleum industry because of its cost effectiveness and being advantageous environmentally [7, 8]. Water based drilling mud can be prepared using bentonitic clay precursor and other additives.

Bentonite is a clay mineral belonging to the montmorillonite mineral family. Montmorillonite structural architecture consists of an octahedral

alumina layer sandwiched between two tetrahedral silica sheets. They are regarded as 1:2 category of clay [9]. Natural bentonites are either monovalent-cation based or divalent-cation based montmorillonites. The monovalent cation associated with natural bentonite mineral is majorly Na^+ while the associated divalent cation is majorly Ca^{2+} . Natural bentonites can also exist with mixed interlayer cations; $\text{Ca}^{2+}/\text{Na}^+$ [10]. Biotite is a Fe^{2+} -rich phyllosilicate, it is a major constituent of rock and granite [11]. Its chemical formula can be represented as $\text{K}(\text{Mg},\text{Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{OH},\text{F})_2$. The presence of weighting agent in drilling fluid stabilizes hydrostatic pressure in the borehole thereby minimizing fluid loss as a result of deposition of thick filter cake on the wall of the well, it also increases the penetration rate of the borehole [12]. The aim of this work is to determine the morphological and chemical characterization of Ririwai biotite and investigate its impact on the yield point of drilling mud when it is applied as weighting agent.

2. MATERIALS AND METHODS

2.1. Materials

The materials used include raw biotite mined from Ririwai Village, Tudun Wada Local Government Area of Kano State, Nigeria, commercial

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bentonite and distilled water. Apparatuses used include viscometer (Maker: Fann Model 35A), electronic compact scale (Maker: Kerro BL10001), mud stirrer, digital stop watch, beakers and measuring cylinder, spatula and Mixing bowl.

2.2. Mud Preparation

The drilling mud was prepared according to the API 13A standard. For every preparation 6% of drilling mud solid was applied in distilled water and was thoroughly mixed for about 30 min until a homogenous mixture was obtained. For 0% weighting agent formulation, the drilling mud sample was prepared by dissolving 30g of commercial bentonite in 500 ml of distilled water. The mixture was thoroughly mixed for about 30 min using a Hamilton Beach commercial mixer until the drilling mud becomes completely homogeneous. For a 10% weighting agent formulation, the above procedure was repeated but 10% of the initial bentonite (3g) was substituted with Biotite. Therefore, the formation was 27g bentonite plus 3g biotite dissolved in 500 ml distilled. Similarly for 20% – 100% weighting agent formulations, the above procedure was repeated with biotite introduced as weighting agent at various weight percent ranging from 20% – 100% at interval of 10%.

2.3. Yield Point Determination

The mud sample prepared was poured into viscometer cup to the scribed mark. The viscometer cup was adjusted until the sleeve of the viscometer was into the mud unto the level indicated in the spindle. The viscometer knob was adjusted to obtain 300 and 600 rpm dial readings. The dial reading of the various mud formulations at 600 rpm was denoted as A. The dial reading of the various mud formulations at 300 rpm was denoted as B. Therefore, the yield point of the various mud formulations (in lb/100 ft²) was determined by Eq. (1).

$$\text{Yield point} = 2B - A \quad (1)$$

2.4. Specific Gravity

Specific gravity of the weighting agent was measured using density bottle. The specific gravity was determined using Eq. (2).

$$\text{Specific gravity} = \frac{(W_2 - W_1)}{(W_4 - W_1) - (W_3 - W_2)} \quad (2)$$

where, W_1 is weight of empty bottle, W_2 is weight of bottle + clay, W_3 is weight of bottle + clay + water and W_4 is weight of bottle + full water.

2.5. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX)

SEM and EDX analyses were carried out using PhenomWorld Scanning Electron Microscope; Prox, manufactured by Eindhoven the Netherlands. The SEM machine was allowed to stabilize at about vacuum of 9×10^{-5} torr. The solid was pretreated by pulverization and spurting. Few

sample of the solid, about 20 mg was mounted on the stub of a spurting machine to coat the solid surface with about 40 nm of gold. Then the spurted sample was placed in the SEM sample holder. Using the computer interface, magnification setting of 500× was made and images at different selected areas were collected. Similarly, at other magnification settings of 1000× and 1500× images at different selected areas were collected. Also using the computer interface EDX data of the sample was collected at about 1000 counts/s.

2.6. X-ray Diffraction

XRD patterns were recorded from Bragg's angle (2θ) of 5° to 705°; Rigaku Miniflex600, operated at continuous scanning, scanning speed of 55°/min, generator settings of 10 mA and 40 kV, and CuK α node material.

3. RESULTS AND DISCUSSION

Figure 1 shows the viscometer dial readings at 300 and 600 rpm against weight percent of biotite weighting agent. For the viscometer spindle rotation of 600 rpm, it could be observed that a continuous trend of decrease in dial reading was observed with increase in weight percent of the weighting agent from 0 – 70 wt%. Between weighting agent concentration of 70 – 90 wt% the dial reading was relatively constant at 4 lb/100 ft². At weighting agent concentration of 100 wt%, the dial reading further dropped to 4 lb/100 ft². For the viscometer spindle rotation of 300 rpm, it could be observed that similar to the 600 rpm reading a continuous trend of decrease in dial reading was observed with increase in weight percent of the weighting agent from 0 – 60 wt%. Between 60 – 70 wt% weighting agent concentration the dial reading was constant at 2 lb/100 ft². Further from 70 wt% the dial reading decreased to 1 lb/100 ft² at 80 wt% and remained constant between 80 – 100 wt%.

Figure 2 shows the mud yield point against weight percent of biotite weighting agent. It could be observed that the yield point of the drilling mud was constant at 2.0 lb/100 ft² for weighting agent composition of 0 – 30 wt%. Between 30 – 50 wt% of weighting agent the yield point of the mud decreased steadily until it was 0.0 lb/100 ft² at 50 wt% and above. It can be deduced that the rheological property of the mud was plastic for weighting agent composition of 0 – 30 wt%. Above 30 wt% the mud lost its plasticity and gradually became brittle. At weighting agent composition of 50 wt% and above the mud was completely brittle. The region between 30 – 50 wt% could be seen as the transitional region between plastic and brittle behavior of the formulated mud. A drilling fluid formulation using weighting agent composition higher than 50 wt% will result into a poor petroleum production as a result of substantial pressure loss in the reservoir production line occasioned by presence of several local fracture fronts in the drilling mud. The brittle mud will further

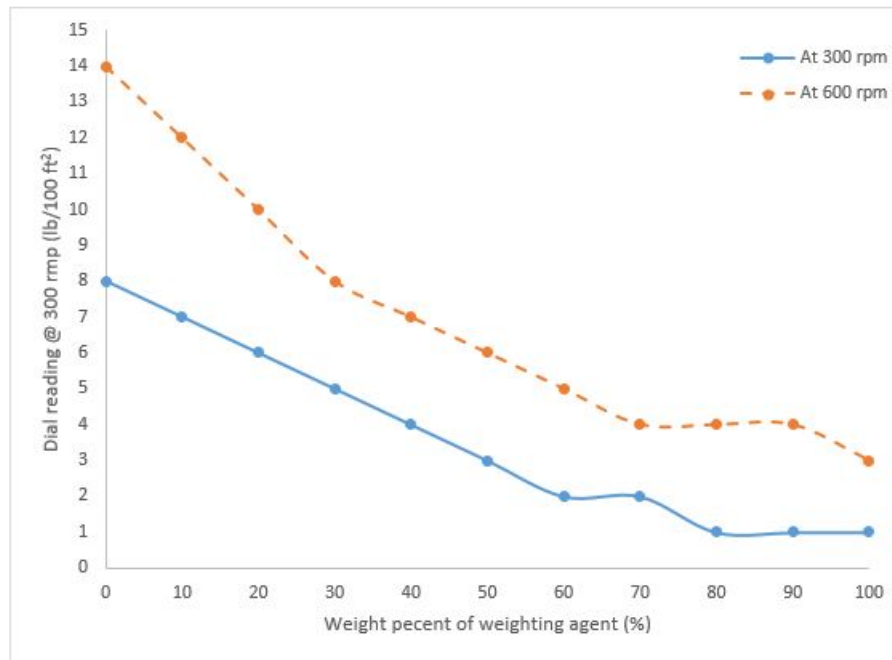


Figure 1: Viscometer dial reading at 300 and 600 rpm against weight percent of weighting agent.

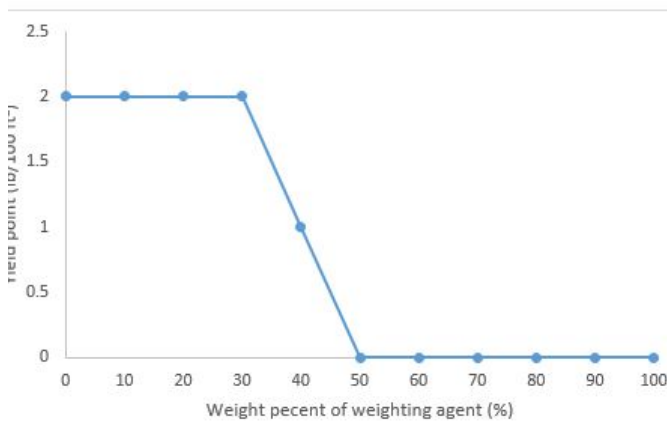


Figure 2: Mud yield point against weight percent of weighting agent .

reduce petroleum production by its abrasive impact on the walls of the production well.

Using Eq. (2), the specific gravity of the commercial bentonite was determined as 1.03 while that of the weighting agent was 2.40. The local material used could be considered as a promising weighting agent as its specific gravity was 2.3 times the specific gravity of the commercial bentonite used. However, the specific gravity of the local material was relatively lower than the specific gravity of barite, a common weighting agent used for drilling muds which is reported to have average specific gravity of 4.4 [13, 14].

Figure 3 shows XRD patterns of the weighting agent used. It could be observed that the predominant mineral present in the material was biotite. The biotite phases were identified at 2θ values of

9° , 24° , 26.8° and 36.7° [15]. Other minor phases present in the weighting agent were quartz and microcline which phases are indicated by Q and M, respectively [13].

Figures 4a – 4c show SEM images of commercial bentonite used for the water based drilling mud, at various magnifications. Figures 4a and 4b are images at $500\times$ and $1000\times$ magnification, respectively. Although, Fig. 4b shows clearer particle distribution of the bentonite than Fig. 4a, both images show relatively even distribution of micro-particles of the bentonite having irregular sizes and shape. Figure 4c shows a micrograph of the bentonite at magnification of $1500\times$. The micrograph is clearer as it reveals clearer details of the particles. It could be observed that the particles have clumpy morphology and irregular crystal shapes. The average particle size was estimated as $25\ \mu\text{m}$.

Figures 5 show SEM images of Ririwai biotite used as weighting agent in the prepared drilling mud, at various magnifications. Figures 5a and 5b are images at $500\times$ and $1000\times$ magnification, respectively. Although, Fig. 5b shows clearer particle distribution of the bentonite than Fig. 5a, both images show uneven distribution of particles of the biotite having irregular sizes. Figure 5c shows a micrograph of the biotite at magnification of $1500\times$. The micrograph is clearer as it reveals clearer details of the particles. It could be observed that the particles have sheet-like morphology and hexagonal crystal shape with some degree of crystal shape irregularity possibly due to high degree of crystal defects. The average particle size was estimated as $60\ \mu\text{m}$.

Table 1 presents the Energy dispersive X-ray (EDX) elemental analysis of Ririwai biotite and

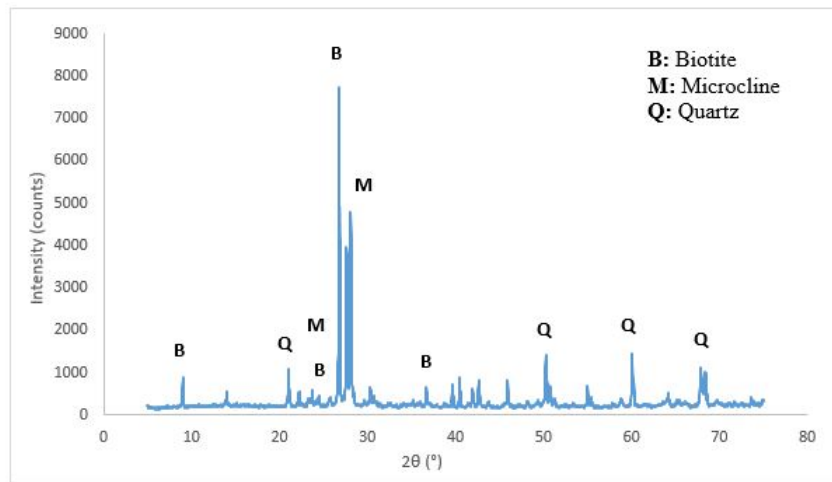


Figure 3: XRD patterns of Ririwai biotite used as weighting agent.

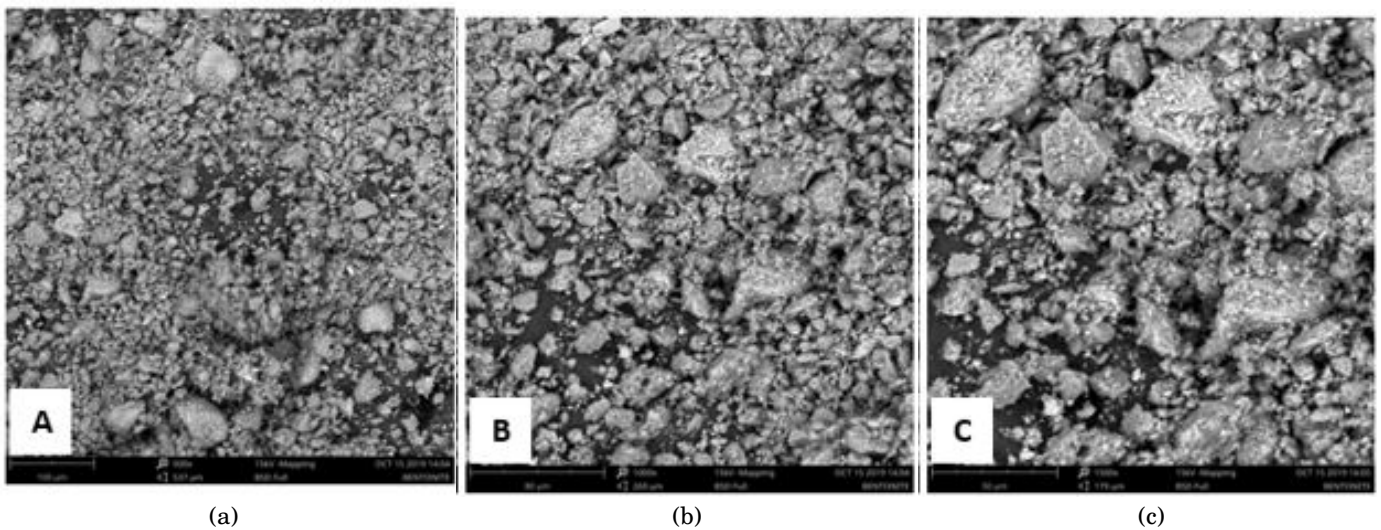


Figure 4: SEM images of commercial bentonite at magnifications of (a) 500x, (b) 1000x and (c) 1500x .

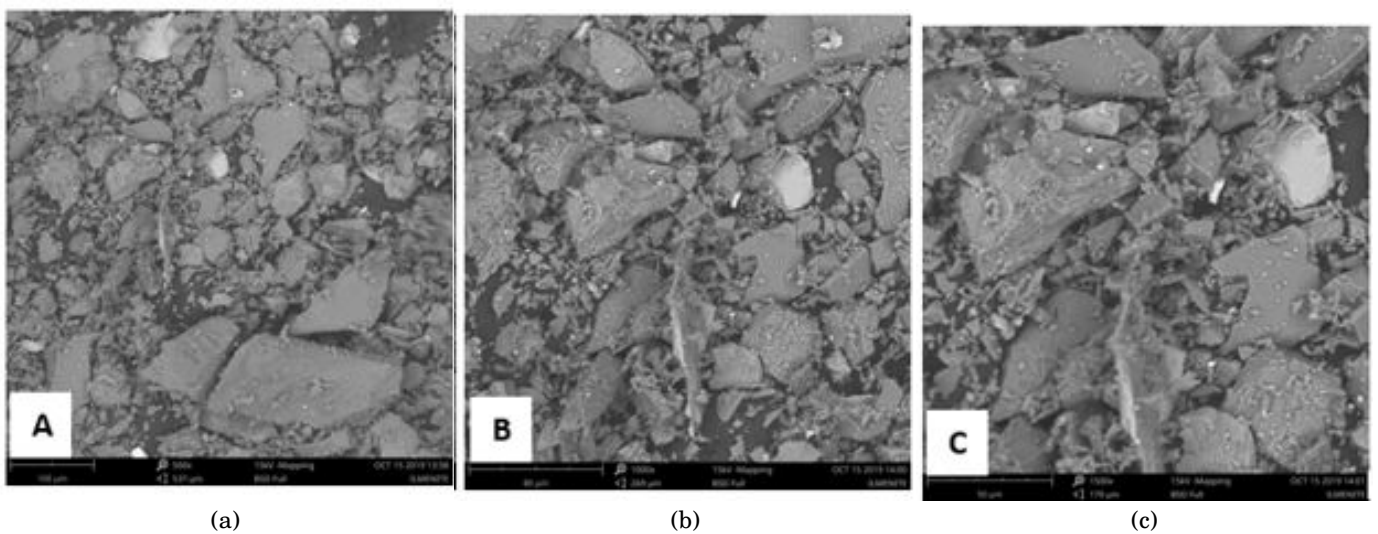


Figure 5: SEM images of Ririwai biotite at magnifications of (a) 500× (b) 1000× (c) 1500×.

Table 1: Energy dispersive X-ray (EDX) elemental analysis of Ririwai biotite and commercial bentonite.

Elemental composition	Composition (wt%)	
	Bentonite	Biotite
Si	39.37	69.22
Al	24.87	7.47
Fe	7.39	6.45
Ca	4.32	0.91
Ba	4.24	*ND
Ti	3.77	0.51
Nb	3.40	1.89
Ag	3.08	2.72
Mn	2.64	0.39
K	1.92	8.42
V	1.75	0.37
Cr	1.72	*ND
Mg	0.89	0.27
Na	0.63	1.36
TOTAL	99.99	99.98

*ND: Not determined

the commercial bentonite used in the formation of drilling mud. The two materials are aluminosilicate minerals. Most of the constituent metals are in their oxide forms as it is known of all aluminosilicate minerals [16]. The dominant composition of the bentonite was silica which was present up to 39.37 wt% followed by alumina which was 24.87 wt%. Therefore, the silica-alumina ratio of the bentonite was 1.58. The low silica alumina ratio of the bentonite indicates the dominating presence of montmorillonite mineral in the material. The elemental analysis of the commercial bentonite used in this study indicated that the bentonite contains both monovalent and divalent cations. The calcium oxide content was 4.32 wt% while the sodium oxide was 0.63 wt%. The bentonite was a mixed interlayer cation based, but largely calcium based, meaning that the Ca^{2+} is the exchangeable interlayer cation in the 1:2 structural architecture of the montmorillonite mineral constituting the bentonite. The calcium cation dominance was seven times multiple of the sodium cation presence. The bentonite was rich in iron, barium, titanium, niobium, silver and manganese to the tune of 7.39, 4.24, 3.77, 3.40, 3.08 and 2.64 wt%, respectively. These are likely to be majorly contributed by associated mixed mineral phases in the bentonite or chemical additives added to the processed bentonite to improve its rheological characteristic to make it suitable for drilling fluid application. In particular, the barium oxide of 4.24 wt% was likely due to presence of barite additive. Vanadium and chromium were present at 1.75 and 1.72 wt%, respectively, they are likely due to presence of associated impurities. The potassium and magnesium compositions of 1.92 and 0.89 wt% were likely due to salt additives for enhancement of the rheology of the drilling mud.

The dominant chemical constituent of the biotite was silica having 69.22 wt% composition. The alumina composition was 7.47 wt%. The

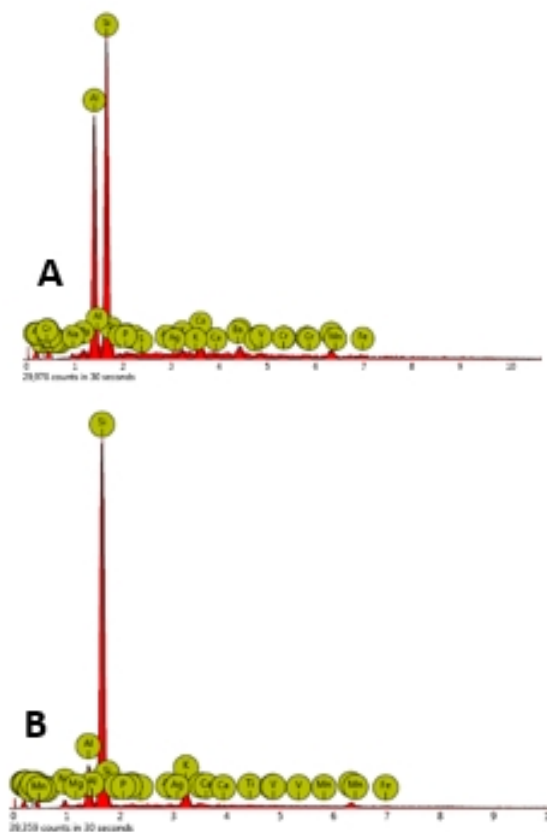


Figure 6: EDX elemental distribution mapping of (a) commercial bentonite (b) biotite .

silica-alumina ratio was very high, up to 9.3, making the mineral highly silicious. The high silica content was likely due to the presence of substantial quartz as shown in the X-ray diffractogram in Fig. 3, as quartz is simply a crystalline silica. The potassium oxide, magnesium oxide and iron oxide compositions of 8.42, 0.27 and 6.45 wt%, respectively are part of the structural framework of the biotite mineral. Other compositions such as calcium, titanium, niobium, silver, manganese, vanadium and sodium analyzed as 0.91, 0.51, 1.89, 2.72, 0.39, 0.37 and 1.36 wt%, respectively were due to impurity associated with the biotite mineral, these are likely due to the microcline mineral identified as associated impurity phase in the X-ray diffractogram in Fig. 3.

Fig 6 shows the elemental distribution mapping of the energy dispersive X-ray (EDX) for; (a) the commercial bentonite used and; (b) Ririwai biotite. The distribution mapping for the commercial bentonite was collected at 1000 counts/s while that of the biotite was collected at 1312 counts/s. It could be observed that the dominant peak in both was due to Si followed by Al. This indicates that both minerals are aluminosilicate minerals as already established earlier.

4. CONCLUSION

Mineralogical analysis of the local material used showed that the dominant mineral phase in

the local weighting material was biotite. Morphological analysis of the Ririwai biotite has shown that the mineral possesses a sheet-like morphology with hexagonal crystal shape and an estimated average particle size of 60 μm . SEM analysis of the commercial bentonite revealed that the mineral possesses clumpy morphology with irregular crystal shapes and an estimated average particle size of 25 μm . The chemical compositional analysis of the Ririwai biotite indicated that the material is a highly siliceous aluminosilicate mineral having a silica-alumina ratio of 9.3. Chemical compositional analysis of the commercial bentonite indicated that the material is an aluminosilicate mineral having a silica-alumina ratio of 1.58. The bentonite was a mixed interlayer cation-based possessing Na^+ monovalent exchangeable interlayer cation and Ca^{2+} divalent exchangeable interlayer cation. The divalent cation was predominant as the calcium oxide content was 4.32 wt% while the sodium oxide was 0.63 wt%. The yield point of the formulated drilling mud using Ririwai biotite weighting agent had yield point of 2.0 lb/100 ft² for formulation having 0 – 30 wt% weighting agent composition. Above 30 wt% the yield point decreased steadily until it was 0.0 lb/100 ft² at 50 wt% weighting agent composition. The Mud formulated was plastic at weighting agent concentration (C) of $0 \leq C \leq 30$ wt% and this region corresponds to a yield point value of 2.0 lb/100 ft². At $30 \leq C < 50$ wt% the mud displayed necking property with continuous drop in the yield point value. At $C \geq 50$ wt% the mud becomes brittle having yield point value of 0.0 lb/100 ft². The specific gravity of Ririwai biotite was 2.4 which was about 55% the average specific gravity of barite, a common weighting agent for drilling mud [13, 14]. On the basis of its relative lower specific gravity, Ririwai biotite is not likely to serve as an excellent drilling mud weighting agent when compared to the common weighting agents like barite. Nonetheless, Ririwai biotite can be considered as a fair weighing agent on the basis of its moderate specific gravity which is 2.3 times the specific gravity of the commercial bentonite used.

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