# Biotite Influence on the Binding Properties of Bituminous Materials

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***Abstract* - The effect of biotite on rheological properties of bitumen was investigated using biotite extracted from gneiss rocks. The research methodology involves two phases. The first phase involves the separation of biotite from gneiss rock. The second phase involved tests carried out on bitumen-biotite blend to determine the effect of biotite on the rheological properties of bitumen. The biotite was ground to 75µm size and blended with bitumen AC-10 prepared in various proportions and the rheological properties determined in the laboratory. The results of this study shows that biotite improves the binding properties of bitumen. At low temperatures, the presence of biotite in bituminous mixtures in the proportion of 2 to 3.5% by weight of bitumen improves the binding properties. Conversely, at higher temperatures of about 135ºC, the biotite bitumen mix showed best result at 2%. An increase of up to 3.5% by weight of biotite resulted in a drop in the kinematic viscosity values even though there was an improvement in the binding properties over the pure AC-10 bitumen results.**

***Keywords: Biotite, Rheological Properties, Gneiss, Bitumen***

## 1.0 INTRODUCTION

Road infrastructure is a national asset and must repay its huge initial investment. Considering the importance of roads and the huge investments made in their implementation, there is the need for seasonal maintenance and possibly advancing the knowledge of the materials used in their construction.

Materials generally constitute an important structural component in many civil engineering constructions, not the least in roads [5]. It is therefore prudent to acquire the knowledge of the physical and chemical properties of the materials used their construction. A flexible pavement structure is typically composed of several layers of material. Asphalt Concrete, which is the topmost layer of the pavement is a mixture of bitumen bound materials used as pavement surfacing. They normally consist of a mixture of course aggregate, fine aggregate and filler bound with bitumen.

Bitumen is responsible for binding the mineral aggregates and is constituted by asphaltenes and maltenes. Asphaltenes correspond to the polar constituents of bitumen and can be precipitated by the addition of low molecular weight alkanes [2]. Bituminous materials consist of bitumen which is a black or dark coloured solid or viscous cementitious substances that consist mainly of high molecular weight hydrocarbons derived from the fractional distillation of petroleum or natural asphalt, has adhesive properties, and is soluble in carbon disulfide. Bitumen is a mixture of a wide variety of high-boiling point hydrocarbons with straight of branched chains, saturated rings as well as aromatics with from 1 to 6 fused rings, incorporating sulfur and a small amount of nitrogen and oxygen which can be constituted as heteroatoms in the ring structures but also as functional groups. The presence of nitrogen and oxygen in some molecules cause them to be slightly polar, but the limited amount of nitrogen and oxygen in the entire bitumen mix excludes the existence of very polar molecules. Organic materials like bitumen are kept together by strong covalent bonds between the atoms within the molecule, while the attraction between molecules is weak - dispersive, or dipole/dipole interactions.

Biotite is a large group of black mica minerals found in many rocks including granites, diorites, igneous pegmatites, schists and gneisses. Biotite exhibits the perfect single cleavage that characterizes the mica group and allows them to be easily separated into thin flexible sheets that transmit light. Its cleavage is a reflection of the mineral's crystal structure. This structure consists of aluminium silicate sheets that are weakly bound together by layers of positive ions. In biotites, these positive ions are dominantly potassium and iron with only minor magnesium. Because the chemical bonds between the aluminium silicate sheets are much weaker than those within the sheets, both of those mica minerals can be easily separated into thin sheets that are both flexible and elastic.

Aggregates are the principal materials in an asphalt pavement, therefore knowledge of aggregate properties is crucial to designing a high quality pavement. Reference [9], in their research reveal that aggregate produced from biotite-granite and granite-gneiss, because of their low porosity, and high crushing strength, possess the necessary characteristics for use in pavement construction in accordance with AASHTO, ASTM and BS standards.

Reference [12], conducted a study on the effect of physical and chemical characteristics of aggregates on bonding and recommended that methylene blue test can be used to indicate the presence of detrimental plastic fines in aggregates which may induce stripping in Hot Mix Asphalt (HMA) mixtures.

Many researchers have authenticated reliance of stripping on aggregate chemistry, mineralogy and surface morphology [4].These properties affect surface energy and chemical reactivity, and hence the positions and amounts of bonding sites (metal ions and charged species). Mineralogical composition is a natural precursor of complex processes at the aggregate surface and affects relative affinity for water and bitumen [8]. Aggregates are composed of minerals and each has a definite chemical composition and chrystalline structure. Chemical composition of aggregates determines surface chemistry and morphology of aggregates. Reference [11] showed that adsorption of bitumen onto aggregates is influenced by aggregate chemistry. Aggregates provide electrostatic and Lewis acid/acid based sites for interaction with bitumen polars ([3]; [6]).

Reference [1], conducted a research on the characterization of granite and limestone powders for use as fillers in bituminous mastics dosage and established that the adhesion action of the filler is not related to the size of the particles but instead to their form and mineralogical nature.

The mineralogical composition of aggregates are believed to have a profound influence on moisture damage susceptibility of bituminous mixtures. Reference [7], investigated the effect of aggregate mineralogical composition on moisture sensitivity of aggregate-bitumen bonds using four aggregate types and two types of bitumen and concluded that moisture absorption properties of aggregates depend strongly on certain key minerals including clay, anorthite and calcite.

Reference [10], investigated the influence of different minerals on the mechanical resistance of asphalt mixtures and concluded that the mechanical resistance of asphalt pavements is related to the chemical interactions of their constituents - bitumen and mineral aggregates. They further concluded that asphaltenes are responsible for the interaction with the surface of the rocks used as aggregates and that feldspar and biotite minerals present in these rocks are responsible for promoting such adsorptions.

Literature is replete with research on the mineralogical compositions of aggregates and their profound influence on moisture damage susceptibility of bituminous mixtures. It is however not clear as to the exact influence of biotite on the rheological properties of bitumen. This research therefore, investigates the effect of biotite on the physical and rheological properties of bitumen.

## 2.0 MATERIALS AND METHODS

The research methodology involves two phases. The first phase involves the separation of biotite from crushed gneiss rock. The second phase involved tests carried out on bitumen-biotite blend to determine the effect of biotite on the rheological properties of bitumen.

### 2.1 Determination of Mineralogical Composition of gneiss

*2.1.1Materials*

Gneiss rock samples were used for this operation. This type of rock is locally available and widely used in the construction industry in Ghana. It was sourced from a quarry site at Nsawam in Ghana.

Fig.1. Some samples of biotite in the Gneiss rock

*2.1.2 Separation of Biotite*

The rock samples were crushed and the method of winnowing was used to separate the biotite samples from the other fragments. An electric fan was placed in front of the sample and switched on to winnow out the biotite fragments from the rock particles onto a tray that was placed in front of the fan (**Fig. 2**). This separation procedure was possible because biotite fragments are lighter than the other constituent minerals.

Fig. 2. Setup for winnowing process

*2.1.3 Test on Mineralogical Composition*

In order to establish the veracity of biotite the separated samples were sent to the laboratory of the Ghana Geological Survey Department for assessment. The results of the assessment is presented under item **3.0**.

*2.2 Determination of Rheological Properties of Bitumen-biotite Mixture*

*2.2.1 Materials*

The biotite sample was ground into finer grains and sieved through 75µm sieve to obtain a definite grain size. The processed biotite sample was blended with heated AC-10 bitumen in different proportions (ranging from 0 to 3.5%) by weight of bitumen.

*2.2.2 Methods*

Laboratory tests were conducted on both conventional AC-10 bitumen and bitumen-biotite mixture in their different proportions to determine their rheological properties. The tests that were conducted include Softening Point Test, Specific Gravity Test, Absolute Viscosity Test, and Kinematic Viscosity Test.

*2.2.2.1 Absolute Viscosity Test*

The Absolute Viscosity Test was conducted using the Brookfield rotary viscometer according to ASTM D4402. The viscosity is measured at 60$^{O}$C. The mixture was poured into the viscosity can and inserted into the Viscometer. The instrument was set to the required temperature 60$^{O}$C to allow the mixture heat to that temperature. A spindle (noted spindle number 7) was then dropped into the mixture. The instrument was initially set at 2.5 rev/min for the rotation of the spindle. The digital part of the instrument shows the type of spindle (7), the torque, the speed and the temperature. When the torque goes below 10%, the speed was increased. The spindle was allowed to rotate for some time till the value of viscosity stabilizes. The various readings were recorded - torque, viscosity, and speed. The viscosity value is to be within the recommended range (800-1200) poise.

*2.2.2.2 Kinematic Viscosity Test*

The test was conducted with the same Brookfield Viscometer. The viscosity was measured at 135$^{O}$C. The mixture was poured into viscosity can and inserted into the Viscometer. The instrument was set to the required temperature (135$^{O}$C) to allow the mixture heat to that temperature. A spindle (noted spindle number 27) was then dropped into the mixture. The instrument is initially set at 50 rev/min for the rotation of the spindle. The digital part of the instrument shows the type of spindle (27), the torque, the speed and the temperature. When the torque goes below 10%, the speed was increased. The spindle was allowed to rotate for some time till the value of viscosity stabilizes. The various readings were recorded - torque, viscosity, speed, shear stress and shear rate**.** The viscosity value is to be in the range (150 and above) poise.

*2.2.2.3 Specific Gravity Test*

The Specific gravity of bitumen is defined as the ratio of mass of given volume of bitumen of known content to the mass of equal volume of water at room temperature. A pycnometer with its stopper was weighed empty on the electronic measuring scale and the result recorded. The pycnometer with its stopper weighed again was filled with water and measured. The pycnometer with the sample was then weighed and the results recorded. The pycnometer with the sample was filled with water and weighed. The results obtained were recorded. The results were used to compute specific gravity. The specific gravity (S.G) was determined as follows:

$$S.G = \frac{C-A}{(B-A)-(D-A)}$$

Where, *A* = the weight of the empty pycnometer,
*B* = the weight of the empty pycnometer filled with water
*C* = the weight of the pycnometer with the sample,
*D* = the weight of the pycnometer with the sample and water
The Specific gravity of bitumen varies from 0.97 to 1.02.

*2.2.2.4 Softening Point Test*

Softening point is used in the classification of bitumen and is indicative of the tendency of the material to flow at elevated temperatures encountered in service. The softening point was conducted using the Ring & Ball apparatus according to ASTM D36-95. A brass ring containing test sample of bitumen is suspended in water at a given temperature. A steel ball is placed upon the bitumen sample and the liquid medium is heated at an approximately constant rate. The temperature is noted when the softened bitumen touches the metal plate which is at a specified distance below and taken as the softening point.

## 3.0 LABORATORY TEST RESULTS

The results of the test on the veracity of the biotite in the gneiss rock are summarized as follows:

| | |
|---|---|
| Mineral name: | Biotite |
| Structural formula: | K (Fe, Mg) $_3$ ($Si_3$Al) $O_{10}$ (OH, F)$_2$ |
| Crystal system: | Monoclinic |
| Class/Group: | Filosilicatos / Micas |
| Common crystal habit: | Laminar and tabulate (001), ps. hexagonal, prismatic (hk0) |
| Twinning: | (001), rarely visible |
| Cleavage: | (001) perfect |
| Color: | Brown, green, red (up to almost black) |
| Pleochroism: | Strong: Z = Y > X (yellow, green, Brown) |
| Birefringence: | 0.03-0. 07 |
| Extinction angle: | 0-9 |
| Optical type: | Binocular stereoscope microscope |
| Optical sign: | Negative |
| 2V angle: | 0-25 |
| Elongation sign: | Positive |
| Interference color: | 3rd, 4th |
| Relief: | Environment + |
| Orientation: | Z ^ to = 0-9, Y = b; PO. (010) |
| Strunz classification: | 9EC20 |

The results of other laboratory investigations are presented in Tables 1 to 4

Table 1. Absolute Viscosity Test Results

| Percentage of biotite (%) | 0 | 2.0 | 2.5 | 3.0 | 3.5 |
|---|---|---|---|---|---|
| Absolute Viscosity (Poise) | 1410 | 1740 | 1790 | 1970 | 2010 |
| Torque (%) | 14.1 | 11.0 | 11.1 | 12.3 | 12.5 |

Table 2. Kinematic Viscosity Test Results

| Percentage of biotite (%) | 0 | 2.0 | 2.5 | 3.0 | 3.5 |
|---|---|---|---|---|---|
| Kinematic Viscosity (cSt) | 240.0 | 275.0 | 270.0 | 264.5 | 257.5 |
| Torque (%) | 9.7 | 11.0 | 10.8 | 10.5 | 10.3 |
| Shear Stress (Pa) | 82.4 | 93.5 | 91.8 | 89.3 | 87.6 |
| Shear Rate ($s^{-1}$) | 34.0 | 34.0 | 34.0 | 34.0 | 34.0 |

Table 3. Softening Point Test Results

| Percentage of biotite (%) | 0 | 2.0 | 2.5 | 3.0 | 3.5 |
|---|---|---|---|---|---|
| Softening Point (°C) | 42.0 | 45.5 | 48.9 | 53.0 | 55.1 |

Table 4. Specific Gravity Test Results

| Percentage of biotite (%) | 0 | 2.0 | 2.5 | 3.0 | 3.5 |
|---|---|---|---|---|---|
| Specific Gravity (g/cm$^3$) | 0.97 | 0.99 | 1.00 | 1.02 | 1.05 |

Table 5. Standard Specifications for Viscosity-Graded Asphalt Cement for Use in Pavement Construction (ASTM D3381 - 09)

| TESTS | VISCOSITY GRADE | |
|---|---|---|
| | AC - 10 | AC - 20 |
| Penetration at 25°C, 100g, 5s, mm | 120 - 140 | 100 - 120 |
| Softening Point temperature, °C | 45 - 52 | 48 - 56 |
| Kinematic Viscosity at 135°C, min, cSt, | 150 - 250 | 200 - 300 |
| Absolute Viscosity at 60°C, P | 800 - 1200 | 1600 - 2400 |
| Specific Gravity at 25°C, g/cm$^3$ | 1.01 - 1.05 | 1.01 - 1.06 |

## 4.0 DISCUSSION

The rheological properties of the biotite-bitumen mixture were compared with that of pure bitumen AC-10 used in this experiment. On the absolute viscosity test at 60°C, it is observed that, as the percentage of the biotite additive increase from 2% to 3.5% by weight (Fig. 3), the more rapidly the biotite-bitumen mixture changes from its semi-liquid state to semi-solid state. Comparing the results of viscosity values obtained from the tests conducted in this research to the absolute viscosity standards of pure AC-20 bitumen (Table 5), it is observed that biotite-bitumen blend (with 2 to 3.5% biotite composition by weight) gives the characteristics of pure bitumen AC-20

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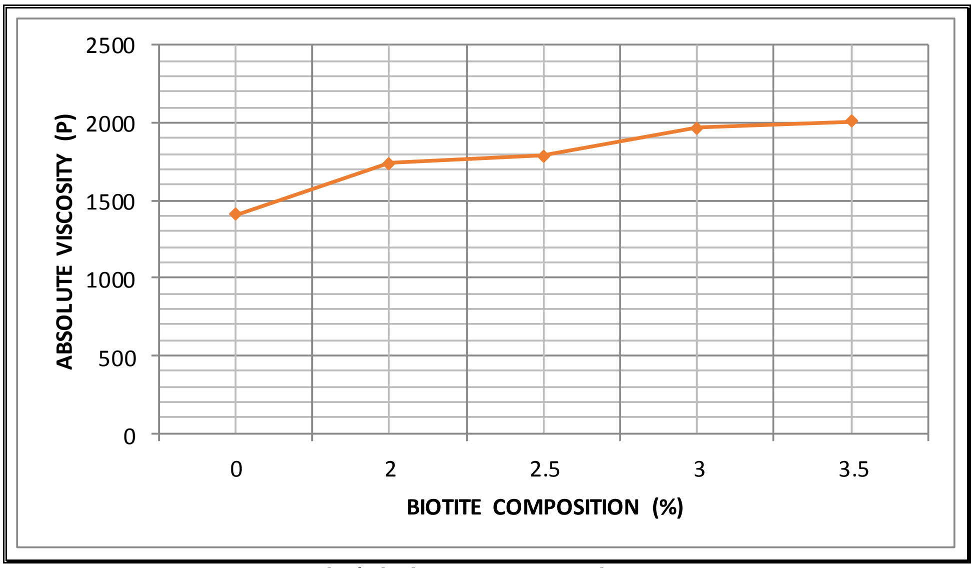


Fig. 3. A graph of Absolute Viscosity against biotite composition

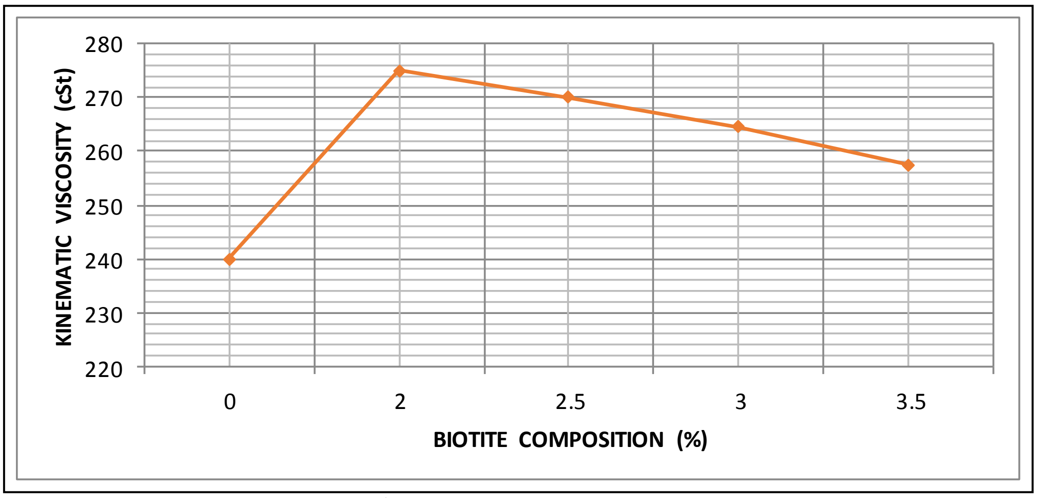


Fig. 4. A graph of Kinematic Viscosity against biotite composition

Analyzing the results obtained in Fig. 4, it is noted that, the addition of 2% by weight of biotite to the bitumen shows a sharp increase in the kinematic viscosity of the mixture from 240.0 poise for pure bitumen AC-10 to 275.0 poise at 135$^{o}$C. A further increase in the percentage of biotite from 2 to 3.5% by weight of bitumen shows a decrease in the kinematic viscosity of bitumen, but remains within the recommended limit of 150-250 cSt. The torque and shear stress also show similar trends.

Reference [1], in their research explained that the biotite separated from rocks with electronegative characteristics adsorbed the molecules of the bitumen by chemisorption. He further stated that biotite particles react with acids in the bitumen binder to promote improvement in the binder properties. The mineralogical nature and chemical composition of biotite is the main factor determining the degree of modification of the bituminous binder at low temperatures.

The biotite, like other aggregate minerals, reacts with the bitumen to influence the mechanical resistance of asphalt pavements. Reference [10], observed that the biotite minerals, apart from feldspar, are responsible for promoting adsorption as asphaltenes in bitumen react with aggregate minerals.

Interpreting the behavioural trends of the results in Fig. 5, the softening point showed a general increase with the addition of biotite to the bitumen. It is necessary to emphasize here that, according to the softening point standard (ASTM D3381 - 09), the result obtained for the pure bitumen used in this research reaches value below the minimum recommended value of 45$^{o}$C, (Table 5) - that is 3$^{o}$C below the minimum limit specified. However, the addition of 2% biotite by weight has a notable influence with an increase of 3.5$^{o}$C in the softening point value from 42.0 $^{o}$C to 45.5 $^{o}$C. A further increase in the percentage composition of biotite additive from 2 to 3.5% shows increase in the softening point of the mixture. The increase in the softening point will minimize bleeding problems that characterize high tropical climates.

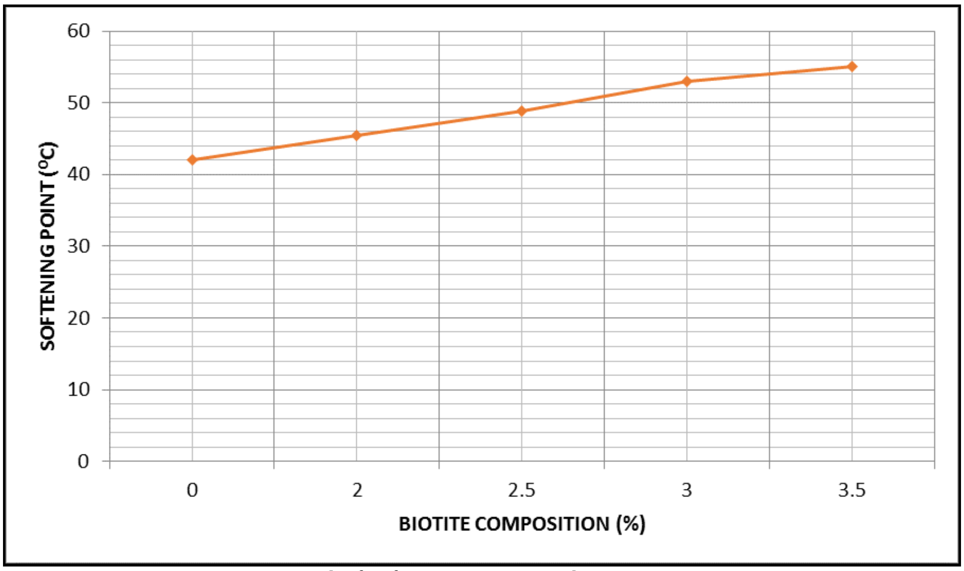


Fig. 5. Graph of Softening Point against biotite composition

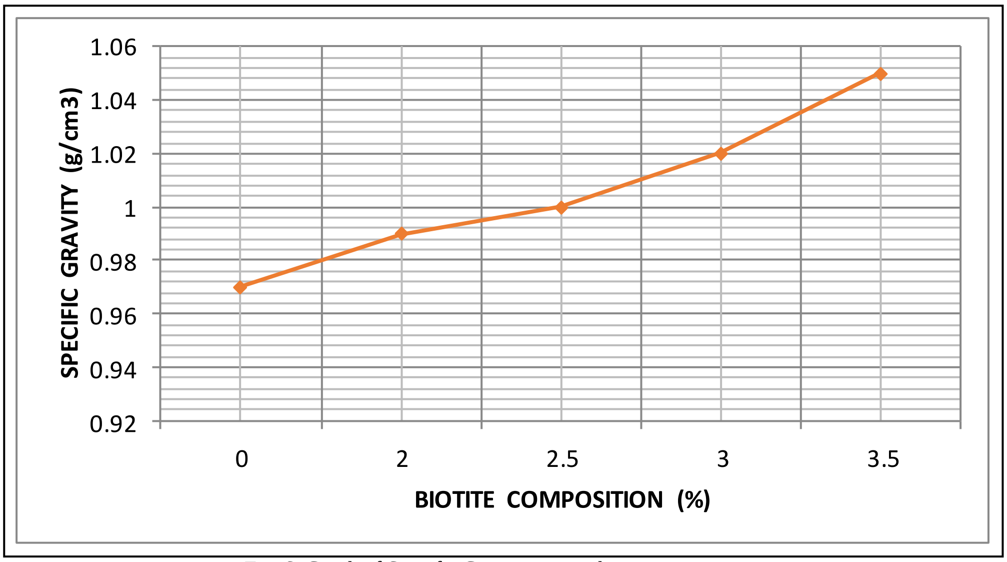


Fig. 6. Graph of Specific Gravity against biotite composition

The specific gravity test results indicate slight increase in values as the percentage composition by weight of biotite increases from 2% to 3.5% (Fig. 6). This is as a result of higher specific gravity of biotite of 3.1 g/cm$^3$ as compared to 1.0 g/cm$^3$ for bitumen.

## 5.0 CONCLUSIONS

The results of this study shows that at low temperatures, the presence of biotite in bituminous mixtures in the proportion of 2 to 3.5% by weight of bitumen improves the viscosities and softening point values and, therefore, the binding properties. At higher temperatures of about 135°C, the best result is achieved at 2% by weight of biotite. The improvement in the softening point values will minimize bleeding problems associated with flexible pavements in high tropical climates.

The results of this research indicates that artificial sand with high amounts of biotite is good for improving the binding properties of bituminous mixtures when used as fine aggregate component or filler that can withstand adverse tropical climates.

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