Softening point and Penetration Index of bitumen from parts of Southwestern Nigeria

Olugbenga A. Ehinola, Olugbenga A. Falode and G. Jonathan

ORIGINAL SCIENTIFIC PAPER

The softening point (*SP*) and penetration index (*PI*) of bitumen from parts of southwestern Nigeria are important properties needed for better design and optimization of the upgrading process of Nigerian bitumen. This study was carried out to design and fabricate a cost effective and efficient softening point tester to classify bitumen into grades.

The Ring and ball equipment was fabricated locally according to the American Society for Testing and Materials (ASTM) and was utilized to determine the *SP* of bitumen samples collected from four locations namely: Agbabu (AB), Iluburin (IB), Loda (LB) and Ode-Omode (OB). Two disks of bitumen, cast in shouldered brass rings, were heated at a controlled rate in a liquid bath while each supports a steel ball. The *SP* was reported as the mean of the temperatures at which the two disks soften enough to allow each ball, enveloped in bitumen, to fall a distance of 25 mm. The *SP* values obtained were used to compute the various penetration indices and to predict the grade of bitumen.

The *SP* results for AB, IB, LB and OB are 30, 48, 48.10 and 38.10 °C respectively while that of *PI* results are -3.8, -1.3, -1.1, and -1.6 respectively. The bitumen grades predicted for the four locations are: temperature susceptible bitumen for AB (250/330 Grade) and conventional paving bitumen for OB, IB and LB (50/70 and 160/220 Grade). The OB, IB and LB samples are therefore suitable for road pavement, while the AB sample can be modified for industrial use.

Key words: Nigeria,softening point, bitumen, penetration index

1. INTRODUCTION

Bitumen is an oil based non-crystalline solid or viscous substance derived from petroleum that exists in the natural or as a by-product of refinery processes; has adhesive properties, and substantially soluble in carbon disulphide. It is a mixture of organic liquids that are highly viscous, black, sticky, and composed primarily of highly condensed polycyclic aromatic hydrocarbons. Bitumen is obtained by removing the lighter fractions (such as liquid petroleum gas, petrol and diesel) by distillation of blends of heavy crude oil during the refining process. The vast majority of refined bitumen is used in construction industry. Bitumen's primary use is as a constituent of products used in paving and roofing applications. Approximately 85% of all the bitumen produced worldwide is used as the binder in asphalt for roads and it is also used in other paved areas such as airport runways, car parks and footways.

Natural Bitumen forms from oil which are already generated and migrated into reservoirs and are subjected to other effects as well as normal maturation processes, these additional changes occur when the continuity of the reservoir horizons permit either up-deep or down deep gas to come in contact with the oil accumulation, one of such processes is known as water washing, which occurs when the reservoir trap is in contact with moving meteoric water. The process is simply the flushing away of the lighter hydrocarbon compound in amount proportional to their solubilities. The result is a big decrease in gasoline range hydrocarbon content and decrease in light asphaltenes and aromatics. This in turn produces an environment of heavy component and a decrease of API gravity so that heavy oil devoid of light component is the end result (bitumen).

The other process of bitumen formation is known as biodegradation, which occurs when oxygen charged meteoric water carry aerobic bacteria from the surface to the accumulation in the reservoir trap, then biodegradation occurs. These aerobic bacteria attack mainly the normal alkanes, although it can also attack the other oil components on a smaller scale. The meteoric water must have dissolved oxygen content of at least 0.8 g/litre. It must contain nutrient and be free of hydrogen sulphide (H_2S) which will poison the bacteria.

In shallow oil accumulation near the edges of the basin where the reservoir best continue to surface outcrop, a combination of water - washing and bacteria biodegradation can also convert very heavy large amount of oil into heavy tar or bitumen, an example is the deposit of bitumen in the Dahomey basin south-west Nigeria.[10] The outcrop belt of the tar sands lies within the Nigerian sector of the Benin Basin which is a marginal pull-apart10 or marginal sag basin.[9] The eastern ward limit of the Benin basin is marked by the Okiti-pupa High while the basin extends westwards into Togo and the Volta Delta (Ghana). The basin contains about 3 000 m of sediments and the stratigraphy has been described.[13,2] Omatsola and Adegoke[13] recognized three formations belonging to the Abeokuta Group. These are: Ise Formation (Neocomian -Albian) consisting essentially of continental sands, grits and siltstones, overlying the basement complex. Overlying the Ise Formation is the Afowo Formation (Turonian- Maastrichtian) consisting of coarse to medium grained sandstones with interbeds of shale, siltstones and clay. The Araromi Formation (Maastrichtian-Paleocene) conformably overlies the Afowo Formation and consists of sands, overlain by dark-grey shales with interbeds of limestones and marl.

The physical and chemical characteristics of the oil sands have been summarized[5,3], among others. Texturally, the sands range from fine to coarse-grained, moderately to well sorted, positively skewed and mesokurtic and represent deposits of high energy environments (e.g beaches) which have been reworked. The sand grains are sub-angular to sub-round in shape. The clay content of the sands (2-7%) is essentially kaolinite with some illite, smectite and mixed layer clays. The sands also contain some heavy minerals which include opaques, staurolite, tourmaline, zircon, rutile, garnet and andalusite some of which could be economically exploited.[5]

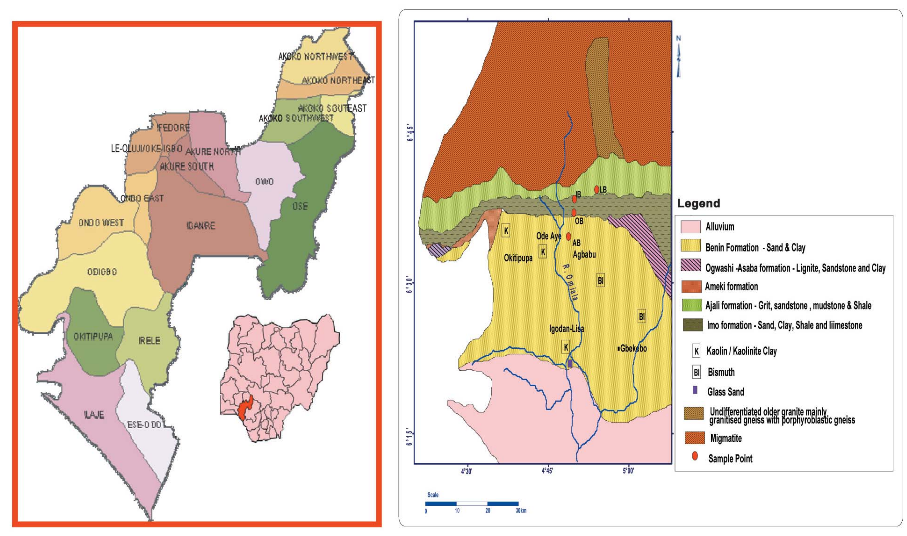

Fig. 1. Map showing the sample locations.
Sl. 1. Karta lokacija na kojima su prikupljeni uzorci

The presence of 22 major, minor and trace elements in the Nigerian tar sands with concentrations ranging from 17 to 35.5 ppm have been reported.[12] The bitumen content of the tar sands is 12-14 wt% and compares favourably with the Athabasca tar sands, except for its lower sulphur, nickel and vanadium contents. Unlike some substances (e.g. water which changes from solid to liquid at 0 °C), bituminous materials do not have a definite melting point. Instead, as the temperature rises, these materials slowly change from brittle or very thick and slow-flowing materials to softer and less viscous liquids. The kinetic parameters and temperature range for the pyrolysis of asphaltene fraction of Nigerian oil sand bitumen to release more oil-like liquid has been carried out.[14] This information is needed for better design and optimization of upgrading process of Nigerian bitumen to liquid fuel. The temperature at which bitumen softens has particular significance for materials which are to be used as pavement material, thick films, such as joint and crack fillers and roofing materials. The present study focuses on the determination of the softening point and penetration index of bitumen from southwestern Nigeria in order to ascertain their grades.

2. MATERIAL AND METHODS

The bitumen samples were collected from four study locations from Southwestern Nigeria (Fig. 1).

These include:

1. Agbabu (AB), sample was collected from a drilled well with metal casing.
2. Ode-Omode Mile 2 (OB), Sample was collected from a drilled well with metal casing.
3. Ilubirin (IB), sample was collected from tar sand.
4. Loda (LB), sample was collected from tar sand.

3. FABRICATION OF APPRATUS

The fabrication of the softening point apparatus was carried out according to the American Society for Testing and Materials (ASTM). The material used for the fabrication was brass and the specification of the apparatus are stated in Figure 2.

1. Ring holder

Brass plate of 89 mm (length) by 30 mm (breadth) by 25 mm (thickness).

(a) Marking out-: This involve using the following tools

(i) Scriber: used to make lines.

(ii) Steel Tool: Used for measurement.

(iii) Centre Punch: Used to make mark on the measured line

(b) Drill-:1st drill--5.6 mm diameter drill was used to make five holes out of the length.

2nd drill-19 mm diameter drill was used to expand the holes.

(c) Bench-:This involved the use of a hacksaw and file to shape out the edges into dimension.

2. Ball centering guide

Brass rod of 23mm by 9mm was used to fabricate the ball centering guides; this was achieved with the aid of a LATHE MACHINE. The ball centering guides were drilled and machined to the required size.

(I) Tap / Die, Die / Wrench, Tap / wrench, Die / Stud.

(II) Milling machine/dividing head-:This was used to make holes on the centering guide and also to make sure the holes were equal to each other.

(III) Cutting Tools used-: Knife edge, Drill and Boring Tool were used.

3. Shouldered rings

A brass rod of 23 mm by 9 mm was used to fabricate the shouldered rings, the following processes were used to achieve this:

(I) The rod was turned to the required size.

(II) The rod was then stepped turned.

(III) It went through a drilling process.

(IV) Then it went through boring and step boring.

4. Bottom plate

This was drilled the same way as the ring holder.

5. Screws

2BA Screw and 2BA Tap were used.

4. EXPERIMENTAL PROCEDURES

Specimens were prepared exactly as specified in ASTM D36-95 in precisely dimensioned brass rings and maintained at a temperature of not less than 10 °C below the expected softening point (*SP*) for at least 30 minutes before the test. The preparation involved heating the bitumen samples until it boiled and was able to be poured (pouring temperature). Some of the samples were filtered with a sieve to remove impurities while pouring; the samples were then allowed to set for 30 minutes. The rings and assembly and two ball bearings, were then placed in a liquid bath filled to a depth of 600±3 mm and the whole system maintained at a temperature of 5±1 °C for 15 minutes (Freshly boiled distilled water was used for the bitumen softening point).

A 9.5 mm steel ball bearing (weighing 3.50±0.05 g) was centered on each specimen and heat was then applied to the beaker so as to raise the temperature by 5±0.5 °C per minute. The temperature at which each bitumen specimen touches the base plate was recorded to the nearest 0.2 °C. The pouring temperature and the room temperature were recorded.

5. PENETRATION INDEX (*PI*) COMPUTATION

The penetration index represents a quantitative measure of the response of bitumen to variation in temperature. Knowing the penetration index of particular bitumen, it is possible to predict its behavior in an application. Therefore, asphalt binders with high penetration numbers (called "soft") are used for cold climates while asphalt binders with low penetration numbers (called "hard") are used for warm climates. All bitumen display thermoplastic properties i.e. they become softer when heated and harden when cooled. Several equations exist that define the way that the viscosity (or consistency) changes with temperature. One of the best known is that developed by Pfeiffer and Van Doormaal which states that:

If the logarithm of penetration, *P*, is plotted against temperature, *T*, a straight line is obtained such that:

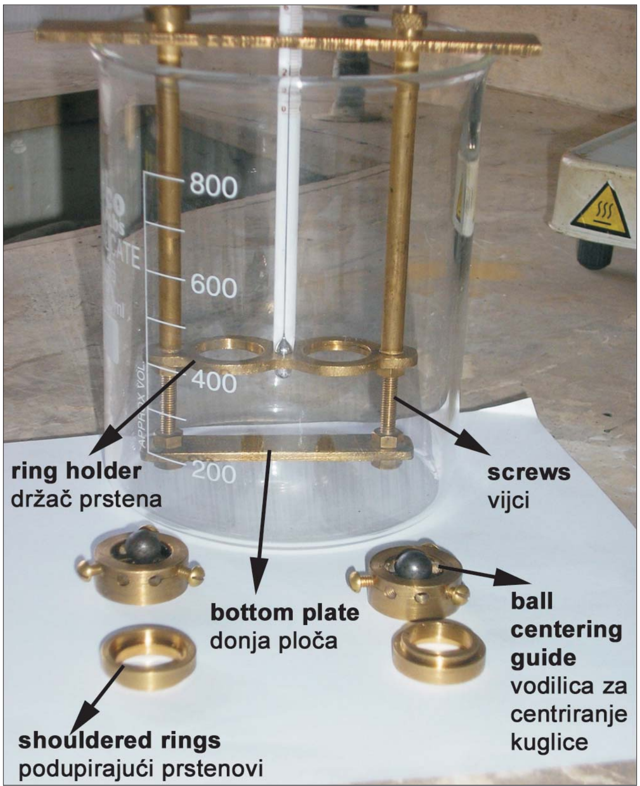

Fig. 2. Fabricated Ring and ball Apparatus (ASTM D36-95 specification)

Sl. 2. Izrađeni uređaj s prstenom i kuglicom (ASTM D36-95 opis)

$$\log P = AT + K \qquad (1)$$

where

A = The temperature susceptibility

P = Penetration at temperature T

K = Constant

The value of *A* varies from 0.001 5 to 0.06 showing that there may be considerable difference in temperature response. Pfeiffer and Van Doormaal developed an equation for the temperature response that assumes a value of about zero for road bitumen. For this reason they defined the penetration index (*PI*) as:

$$\frac{20 - PI}{10 + PI} = 50A \qquad (2)$$

or explicitly,

$$PI = \frac{20(1 - 25A)}{1 + 50A} \qquad (3)$$

The value of *PI* ranges from around -3 for high temperature susceptible bitumens to around +7 for highly blown low temperature susceptible (high *PI*) bitumen. The *PI* is an unequivocal function of *A* and hence it may be used for the same purpose.

The values of *A* and *PI* can be derived from penetration measurements at two temperatures, T_1 and T_2 using the equation:

Table 1. Results of the softening point from the study area

Sample Source	Softening Point Temperature (°C)	Pouring Temperature (°C)	Room Temperature (°C)	Bath Liquid	Time (min.)
AGBABU (AB)	30	104	30	Water	1
ILUBIRIN (IB)	48	110	30	Water	8
ODE-OMODE (OB)	38.10	102	30	Water	4
LODA (LB)	48.10	110	30	Water	8

Table 2. Results of penetration and sulphur content from the study area

Locations	Penetration (decimillimetre)	Sulphur content (weight %)	Cobalt/Nickel ratio (weight %)
Ilubirin (IB)	60	0.45	0.040
Loda (LB)	65	0.52	0.106
Ode-Omode (OB)	170	0.42	0.046
Agbabu (AB)	330	0.64	0.392

Table 3. The predictive Grade of Bitumen

Bitumen location	Grade	Penetration index	Best possible use
Agbabu (AB)	250/330	-3.8	Temperature susceptible bitumen
Ode-Omode Mile 2(OB)	160/220	-1.6	Conventional paving bitumen.
Loda (LB)	50/70	-1.1	Conventional paving bitumen.
Ilubirin (IB)	50/70	-1.3	Conventional paving bitumen.

$$A = \frac{\log pen\ at\ T_1 - \log pen\ at\ T_2}{T_1 - T_2} \quad (4)$$

Pfeiffer and Van Doormaal (year) found that most bitumen had a penetration of about 800 dmm (deci-millimetre) at the ASTM softening point temperature. Replacing T_2 in the above equation by the ASTM softening point temperature and the penetration at T_2 by 800 they obtained equation (5):

$$A = \frac{\log pen\ T_1 - \log 800}{T_1 - \text{ASTM softening point}} \quad (5)$$

Substituting equation (5) in equation 3 and assuming a penetration test temperature of 25 °C gives:

$$PI = \frac{1952 - 500 \log pen - 20 \text{ softening point}}{50 \log pen - \text{softening point} - 120} \quad (6)$$

Equations (3) and (5) were applied in this study to calculate first for *A* (temperature susceptibility of bitumen) and *PI* (penetration index). These were calculated from the measured softening point temperatures and penetrations.

6. RESULTS AND DISCUSSION

The *SP* and *PI* results for the bitumen samples are presented in tables 1 and 2 respectively. The *SP* results ranged from 30 to 48.10 °C and the *PI* ranged from 60 to 330 mm (Figs. and 4). The sulphur contents are between 0.42 and 0.64 wt% (Table 2). Based on the *SP* and *PI* results, IL, OB and LB samples meet the requirement for conventional pavement bitumen. Sample AB could be categorized as temperature susceptible bitumen.Three of the four samples collected can be applied successfully on road construction in the tropics, but this can only be achieved after they must have been upgraded. In fact, the result of the sulphur content shows that all the samples have low sulphur. However, for bitumen to be used as road pavement material, its sulphur content should be between 4-6%. Sulphur usually reduces the high temperature viscosity of bitumen; improving its workability when hot and its deformation to resistance when cold, thereby making it compacting.

The bitumen grade is summarized in Table 3. Agbabu (AB) sample flows at room temperature and are usually best applied for pavement in temperate regions of the

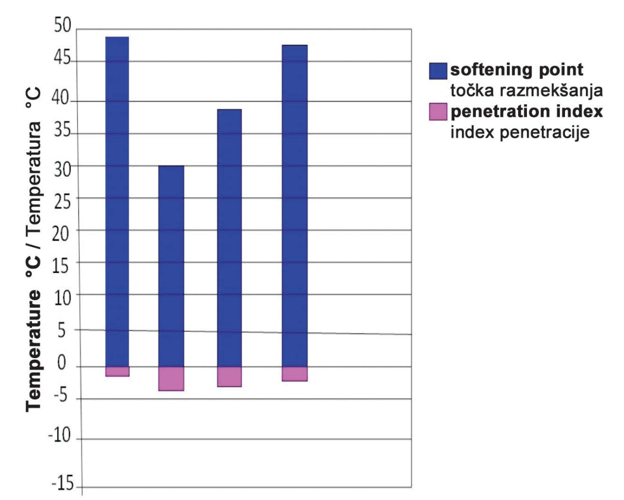

Fig 3. Bar chart showing softening point and penetration index plot of AB, OB, LB and IB.
Sl. 3. Položeni stupčani grafikon s točkama razmekšanja i indeksom penetracije za AB, OB, LB i IB

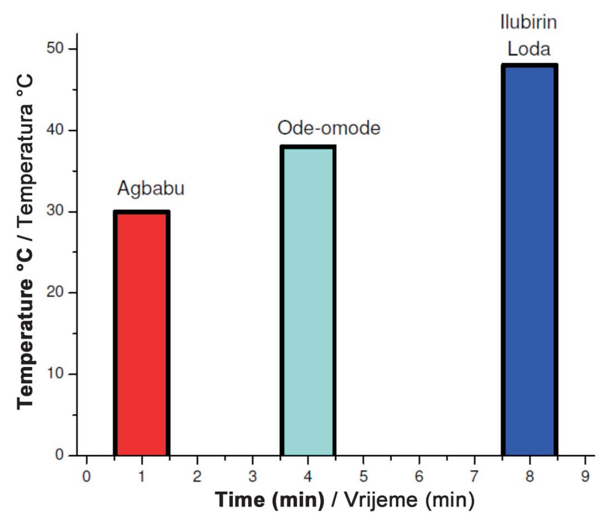

Fig 4. A bar chart of softening point (degrees) against time in minutes

Sl. 4. Položeni stupčani grafikon s točkama razmekšanja (stupnjeva) nasuprot vremena u min.

world but not in tropic regions like Nigeria. Low grade bitumen are also applied in industrial processes, alternatively these bitumen reserves can be cracked to get other petroleum products.[14]

The penetration of bituminous material is the most widely used method of measuring the consistency of a bituminous material at a given temperature. It is a means of classification rather than a measure of quality (The engineering term consistency is an empirical measure of the resistance offered by a fluid to continuous deformation when it is subjected to shearing stress). The consistency is a function of the chemical constituents of bitumen, viz. the relative proportions of asphaltenes (high molecular weight, responsible for strength and stiffness), resins (responsible for adhesion and ductility) and oils (low molecular weight, responsible for viscosity and fluidity). The type and amount of these constituents are determined by the source petroleum and the method of processing at the refinery.

7. CONCLUSION

The ring and ball apparatus has shown that the bitumen samples from the study area are not the same but of two grades. Samples OB, IL, LB are Conventional pavement grades and Sample AB is temperature susceptible grade. The particular kind of grade applied in the construction of road goes a long way to determine the durability or failure of the road. The low sulphur content would have definitely affected the *SP* and *PI* of the bitumen and study on the sulphurisation of Nigeria bitumen would possibly help to improve the grade.

References

1. ASTM (1978): "Standard Tests Method for Resistance to Plastic Flow of Bituminous Mixtures using Marshall Apparatus." Annual Book of ASTM Standards, Part 15, pp.425 -433.
2. Bilman, H.G. 1992. "Offshore Stratigraphy and Paleontology of the Dahomey (Benin) Embayment, West Africa". NAPE Bull. 7(2):121-130.
3. Ekweozor, C. M. (1990): "Geochemistry of Oil Sands of Southwestern Nigeria." Proceedings, First Workshop on Nigerian Tar Sands. The Nigerian Mining and Geoscience Society, Ago-Iwoye, Aho, B.D. and Enu, E. I. - Editors, pp. 81-88.
4. Ekweozor, C. M. and Nwachukwu, J. I. (1989): "The Origin of Tar Sands of South-Western Nigeria," Nigerian Association of Petroleum Exploration, Bulletin 4, pp. 82 - 94.
5. Enu, E.I. 1985. "Textural Characteristic of the Nigerian Tar Sands". Sedimentary Geology. 44:65-81.
6. John Read and David Whiteoak(2003): The Shell Bitumen Book, PP.135-138
7. John Read and David Whiteoak(1995): The Shell Bitumen Industrial Book, PP 44-64
8. Kennedy, T. W. and Hudson, W. R. (1968) "Application of the Indirect Tensile to Stabilized Materials." Highway Research Record No.235, Highway Research Board, pp.36 -48.
9. Kingston, D.R., Dishroon, C.P., and Williams, P.A. 1983. "Global Basin Classification System AAPG. Bull. 67:2175-2193.
10. Klemme, H.D. 1975. "Geothermal Gradients, Heatflow and Hydrocarbon Recovery". In: A.G. Fischer and S. Judson (eds). Petroleum and Global Tectonics. Princeton University Press: Princeton, NJ. 251-304.
11. Monismith, C. I. (l.961): "Asphalt Paving Mixtures - Properties, Design and Performance," The Institute of Transportation and Traffic Engineering, University of California, Berkeley, Los Angeles, U. S. A.
12. Oluwole. A. F. (1985): "Chemical Composition of Bitumen Extracted from Nigerian Tar Sands", Proceedings, Third International Conference on Heavy Crudes and Tar Sands, California, U.S.A., pp. 373- 379.
13. Omatsola, M.A. and Adegoke, O.S. 1981. "Tectonic Evolution and Cretaceous Stratigraphy of the Dahomey Basin". J. Min. Geol. 18(1): 130-137.
14. Sonibare, O.O., Egashira, R and Ehinola, O.A., 2003.Pyrolysis study of Nigeria bitumen and its fractions. Nafta, Oil and Gas Journal, vol. 54, no. 3, p.105-110.

❖

Authors:

Olugbenga A. Ehinola, Energy and Environmental Research Group, Department of Geology, University of Ibadan, Ibadan, 200284, Oyo State, Nigeria, ehinola01@yahoo.com

Olugbenga A. Falode, Energy and Environmental Research Group, Department of Petroleum Engineering, University of Ibadan, Ibadan, 200284, Oyo State, Nigeria

George Jonathan, Energy and Environmental Research Group, Department of Geology, University of Ibadan, Ibadan, 200284, Oyo State, Nigeria, ehinola01@yahoo.com