# UNDER PEER REVIEW

### **1 Degradative Effect of I.R radiations on the Constituents of Bitumen**

### 2 Abstract

Sample of natural bitumen were taken from bitumen well in Agbabu town in 3 Odigbo Local Government of Ondo State. These samples were separately 4 irradiated with ultraviolet, infrared and X-ray radiations for a period of seven 5 hours. Part of the sample was withdrawn at interval of One, Three and Seven 6 hours. The withdrawn sample was later separated into maltene and asphaltene 7 fractions. The maltene fraction was further separated into saturated, aromatic and 8 polar fraction. The saturated and aromatic fractions were subjected to gas 9 chromatography analysis. The Saturated and aromatic profiles of the bitumen were 10 found to vary with the period of irradiation. Both the amount of saturated and 11 aromatic compounds in the bitumen decreased with the period of irradiation. Thus, 12 the radiations were found to have a degradative effect on the composition of 13 bitumen. 14

### 15 Keywords: Bitumen, radiations, Gas Chromatography, degradative effect

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#### 17 Introduction

The greatest use of bitumen is in paving and road building, particularly flexible 18 pavements. Examples of such areas of usage are in Highways Street and 19 driveways, airfields, Parking areas, service (petrol) stations and industrial floors 20 among several others [1]. Bitumen is thermoplastic as it consistency or degree of 21 hardness varies with temperature. On exposure to different radiation, bitumen 22 behaves in a different manner which will affect it quality. Bitumen is very sensitive 23 to any form of radiation which leads to a degradative effect on its constituent 24 thereby affect its usefulness for engineering purposes. [2] 25

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### **Materials and Methods**

The bitumen used for the degradation experiments were collected from one of the observatory wells in Agbabu, Ondo State, Nigeria. Agbabu is one of the major towns located in the Nigerian natural bitumen belt and the place where bitumen was first discovered in Nigeria (Adegoke, 2000) [3]. The raw natural bitumen obtained from Agbabu was purified as described by the method employed by Olabemiwo *et al* (2008) .The Chemicals used for this research are products of

- BDH Limited which includes iso octane (2,2,4-trimethylpentane) [4]
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# 36 Irradiation of Bitumen Samples

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Dry Petri dish (Pyrex) was weighed and 10g of purified natural bitumen was put on 38 it. Thin layer of the natural bitumen was formed on the petri dish. The petri dish 39 containing the purified natural bitumen was subjected to different 40 radiations(wavelength) for a period of Seven hours at interval of One, Three and 41 Seven hours respectively. Some of the irradiated sample was withdrawn into petri 42 dish at interval of One, Three and Seven hours to be analyzed. From the withdrawn 43 irradiated sample, 0.6g of it was carefully and accurately weighed into a beaker 44 and  $20 \text{cm}^3$  of iso – octane was added to precipitate out the Asphaltene component. 45 Filtration process of the solution was now carried out by making use of filter paper. 46 From the filtration process, two components were obtained which was residue and 47 filtrate. The residue is asphaltene and filtrate is maltene. The Maltene was 48 collected into a sample bottle while the asphaltene was washed about five times 49 with 20ml iso – octane. By the method employed by Olabemiwo et al (2008) using 50 Column Chromatography, maltene fraction which is the filtrate was separated into 51 saturated hydrocarbons, polycyclic aromatic hydrocarbon and polar compounds. 52

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# 54 Analysis of Gas Chromatographic

The gas chromatographic analysis used was 5890 series (Hewlet Packard) that is 55 equipped with flame ionization detector. The stationary phase used for the analysis 56 is a fused - silica capillary column coated with 0.25m film of HP-5. For 57 hydrocarbons that are saturated, about 3µL of sample was injected. The column 58 temperature started at 60°C, held for 2minutes isothermally and then increased to 59 200°C at the heating rate of 10°C/min for 20minutes. It was held at this 60 temperature for 2munutes and then increased to 320°C at the heating rate of 12°C 61 for 5minutes. The carrier gas used was nitrogen at a pressure of 30 psi. At pressure 62 of 22 and 28 psi, Hydrogen and air were introduced respectively. 300°C and 320°C 63 were used for Injector and detector temperature respectively. 64

The column temperature was held for 2minutes at about  $70^{\circ}$ C column temperature for the aromatic hydrocarbons and later increase to  $250^{\circ}$ C at heating rate of  $15^{\circ}$ C

for 20minutes. It was held at  $260^{\circ}$ C for 6 minutes isothermally and then increased

to  $320^{\circ}$ C for 6mnutes at heating rate of  $15^{\circ}$ C and it was at this temperature for

- pressure of 35psi. At a pressure of 25 and 30 psi, hydrogen and air was introduced

respectively. At temperature of 300 and  $320^{\circ}$ C was when injector and temperature

<sup>72</sup> was used respectively and the sample of injected volume is  $2\mu L$ . By making use of

73 the standards supplied by the Gas Chromatography equipment manufacturer,

- 74 Calibration curves for the standard and aromatic hydrocarbon were prepared.
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# **Result and discussion**

78 Gas Chromatography result of the saturated and aromatic fraction of irradiated

79 bitumen with infrared.

РАН	AMOUNT (g/mg)				
	RAW BT	IRO 1 SAT	IRO 3 SAT	IRO 7 SAT	
C <sub>11</sub>	304.33	371.745	227.238	194.829	
C <sub>12</sub>	3.7772	2.5146	1.5918	2.3541	
C <sub>13</sub>	37.3414	25.7805	56.4245	24.6311	
C <sub>14</sub>	4.4330	2.99145	1.7349	3.1931	
C <sub>15</sub>	45.1838	32.5694	19.69	27.9708	
C <sub>16</sub>	4.1187	2.7674	1.6251	2.6861	
C <sub>17</sub>	6.6893	5.0672	2.8083	4.3397	
C <sub>18</sub>	4.0167	3.3583	1.9249	2.7063	
C <sub>19</sub>	3.9041	2.9776	1.7255	2.5150	
C <sub>20</sub>	8.9376	9.1302	5.0815	6.2955	
C <sub>21</sub>	3.9808	6.6905	3.5853	4.0090	
C <sub>22</sub>	11.4659	8.1386	6.0837	6.2387	
C <sub>23</sub>	2.2465	2.2687	1.2912	1.6358	
C <sub>24</sub>	115.834	40.9511	40.6723	50.120	
C <sub>25</sub>	5.9590	6.3874	4.9352	5.5857	
C <sub>26</sub>	39.1534	25.049	16.561	17.788	
C <sub>27</sub>	10.5277	13.1155	8.0830	9.3792	
C <sub>28</sub>	7.3891	17.6333	4.0959	4.9437	
C <sub>29</sub>	1.0401	0.8327	0.7083	0.7928	
$C_{30}$	1.8928 × 10 <sup>-5</sup>	-	-	9.16933 * 10 <sup>-5</sup>	
TOTAL PAH'S	620.4383	579.9684	405.8604	372.0145	

80 Table 1: Aliphatic Hydrocarbon Profile of Bitumen Irradiated with Infra – red Radiation

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### 82 Table 2: Polycyclic Aromatic Hydrocarbon Profile of bitumen irradiated with infra – red

РАН	AMOUNT (g/mg)				
	RAW BT	IRO 1 SAT	IRO 3 SAT	IRO 7 SAT	
Napthalene	0.7122	0.1160	0.12459	0.11831	
Acenapthylene	0.0000	0.0000	0.0000	0.0000	
Acephathene	0.0083	0.01394	0.005290	0.00575	
Fluorine	0.1980	0.08268	0.06521	0.026631	
Phenathrene	0.12949	0.07757	0.062855	0.02038	
Anthracene	0.02884	0.02988	0.010598	0.00679	
Fluoranthene	0.02289	0.012464	0.006254	0.00255	
Pyrene	0.01795	0.03190	0.01187	0.006787	
Benzo(a)	-	0.01750	0.0085474	0.0031555	
anthracene					
Chrysene	-	0.01833	0.07822	0.002763	
Benzo (b)	0.02399	0.003922	-	7.858 * 10 <sup>-4</sup>	
fluoranthene					
Benzo (k)	0.10900	-	-	0.0010659	
fluoranthene					
Benzo (b)	0.0000	-	-	0.0000	
pyrene					
Indeno (1,2,3	0.08438	-	-	-	
- cd)					
Dibenzo (a,h)	-	-	-	-	
anthracene					
Benzo (g,h,i)	0.0246	-	-	-	
TOTAL	1.3596	0.4041	0.3734	0.1949	
PAH'S					

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# **Bitumen samples irradiated with Infra – red radiations**

Saturated Fractions: The total amount in g/kg of the aliphatic hydrocarbons was found to decrease as the period of exposure of the bitumen to ultraviolet radiation increases. This can be contributed to cracking and recombination of product. The irradiation of bitumen brought about the cracking of some higher molecular mass

92 hydrocarbons to lower molecular mass radicals.[5]

Aromatic Fractions: The total amount in g/kg of the polycyclic aromatic 93 hydrocarbons was found to decrease as the period of exposure of the bitumen to 94 infra-red radiation increases. Benzo (a) anthracenes and chrysene which were 95 absent in the control sample appeared after irradiation with infra-red radiations. 96 Indeno (1, 2, 3-cd) pyrene and Benzo (g, h, i) perylene which were present in the 97 after irradiation with sample disappeared infra-red control radiations. 98 Acenaphthylene, Benzo (a) pyrene and Dibenzo (a, h) anthracene were absent in 99 the control and irradiated sample. Benzo (b) fluoranthene which was present after 100 one hour of irradiation disappeared after three hours of irradiation. Benzo(k) 101 fluoroanthene which was present in the control sample disappeared after three 102 hours of irradiation and later reappeared in minimal amounts of seven hours if 103 irradiation. 104

### Conclusion

The Gas Chromatogram result for Aromatic fraction of bitumen irradiated with infrared radiation respectively shows that the total number of polycyclic hydrocarbons decreases as the time of irradiation is increases. This reduces the quality of bitumen used for construction purposes. Therefore, bitumen to be used for construction or any other purposes should not be exposed for radiation. Hence, Gas chromatography analysis shown that bitumen consists of Sixteen Polycyclic hydrocarbon and these are responsible for the quality of bitumen.

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126	Appendix
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128	Definition of abbreviation
129	PAH: Polycyclic Aromatic Hydrocarbon
130	RAW BT: Raw Bitumen
131	IRO SAT 1: Saturated fraction of bitumen irradiated with infrared for one
132	hour
133	IRO SAT 3: Saturated fraction of bitumen irradiated with infrared for three
134	hours
135	IRO SAT 7: Saturated fraction of bitumen irradiated with infrared for Seven
136	hours
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