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The liqeufaction products of two sub-bituminous coal samples obtained from okaba and okpara mines: A comparative study

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ASTRACTS

The growing demand for alternative source of energy necessitated investigation into the structure of coal to gain insight into its chemicals components hence its utilization as a source of liquid fuel. Two sub-bimanous coal samples from Okaba and Okpara mines were investigated using soxhlet extraction. Fractionation of the extracts was done using silica – get adsorption chromatography into aliphatic, aromatic and polar fractions. Characterizations of these were carried out using infra-red spectroscopy which showed major adsorption bands associated with aliphatic and aromatic C-H groups. A preliminary conclusion from the results shows that both coal samples are made up to a low ranking.

Keywords: liquefaction, soxhlet extraction coal sub-bituminous, silica-gel, urea adduction.

INTRODUCTION

A sound economic planning and hence industrial development depends to a large extend on the resource available to a nation. This is not to say that a country can automatically develop with abundance of resources but rather an inventorized resources makes for easy planning. According to [1], the industrial revolution was largely a revolution in power availability, while [2] formulates the concept in a concrete mathematical term as follows:

$$L=\frac{R \times E \times I}{P}$$

Where L represents the standard of living of a society (measured in terms of consumption of goods and services)
R= consumption of raw materials
E= Consumption of Energy

I= consumption of ingenuity (embracing technology, political, socio-economic and management components).
P= Population of the society

In Nigeria, for instance. More people own cars and privates plants today compared to the early 1930's and 1940's. As a result of this, there has been an increase in the use and demand of energy in terms power generation and transportation, which has so far been supplied by stock fossils like crude oil and to a lesser extent natural gas.

There is a worldwide growing concern on the rate of depletion, and final exhaustion of these fossils. The life span of this resource in the world which at present is the main source of energy and petrochemical by-products is now measured in few decades [3]. In events of depletion of the crude reserves, the ugly scenario that could be predicated by the energy crises will be better imagined than experienced. The fact that oil has a "turbulence factor" in contemporary international affairs has lead to a boast in coal research in the technologically advanced countries in the west geared toward reducing the dependence on the O.P.E.C nations [4].

In the light of this, it is high time our research efforts should be focused on other alternative sources of energy such as biomass, tar sand, solar energy and of course coal. Coal is the most abundant fossils fuel in the world with proven reserve of about 698 billion tones [5]. This represents about 80% of the world's non-renemable energy reserves [6]. Nigeria is endowed with a large reserve of these recourses: coal and lignite- 359 and 216 million tones respectively. The coal belt spans a considerable part of the country, twelve out of the former thirty states [7]. Preliminary works have showed that Nigerian coals have high volatile matter content, about 40% [8]. This point to their high potential as a source of energy and feed stock for the country's chemical and allied industries in the future [9].

Coal deposits were found from the remains of vast forests of trees, shrubs and plants, which flourished in the hot humid climates about 250 to 400 millions years ago on earth. This flora died and was partially rottened, buried and consolidated under sediments deposited by encroaching seas. It has a very complex chemical structure containing compounds made up of carbons, hydrogen, oxygen, nitrogen and sulphur [10]. The use of coal as an energy substitute for crude oil and natural gas cannot be over emphasized. It is used as fuel for electric power generation, industrial heating, steam generation, driving of locomotive engines, smokeless briquettes from coal can be used for domestic cooking. The further use of coke and fuel gas also contributes to the coal consumption for heat. Also, the growth of synthetic fuels from coal will provide substantial quantities of by-products including elemental sulpur, fertilizer, ammonia or its salt and a range of liquid products in the chemical industry [4]. For efficient utilization of coal, its basic structure and the nature if its reactions have to be well understood [11].

The objective of this study, is it extract and analyze fractions obtained from solvent extraction of okaba coal using I.R. spectrometry from these instrument could give an insight into the possible constituents of the coal sample, thus enabling a rational utilization of this potent resource as fuel and chemical feed stock in our chemical industries.

MATERIALS AND METHODS

Sample preparation

Coal samples form both, Okaba and Okpara mines were obtained from the National Metallurgical Research and Development Council (NMRDC), Jos, Plateau State. The coal samples were dried, crushed and ground to powder, then sieved with a 200 mesh (75µm) B.S Test sieve to increase the surface area and allow for swelling. 60g of each sample was extracted in a Soxhlet extractor at 80°C for hours using a mixture of methanal and benzene in the ratio of 1:3 (v/v). [3]. Each extract was concentrated using a rotator evaporator and stored. This was repeated for both samples.

Fractionation

180g of silica-gel (60-120mesh size) was activated in an oven at 160°C for 24 hours and cooled at room temperature in a dessicator. Slurry of the gel was made with n-hexane and carefully packed into a glass column of dimension 37x2.5cm internal diameter 3g of the extract was dissolved in 3cm^3 of benzene and poured on top of the column. The elution commenced first with 150cm^3 of n-benzene, followed by 150cm^3 of methanol [12]. This fractionation process was repeated on the other sample. The elutes were again concentrated and kept for further analysis

Urea abduction

20g of urea were ground in a mortar to powder, and the n-hexane eluate was mixed with the urea in a 6:1 (vol/wt) ratio and stirred at 1,00rpm in an ice cold bath and latter allowed to stand for 10mintues. The mixtures was washed with ice benzene and left over-night. A white solid adducts was formed and then filtered. The adduct was dried under room temperature and was decomposed with hot water at 80^{0}C. The oily layer on top of the water was separated using a separating funnel and kept inside a dessicator to remove any moisture.

Instrumentation (Infra-red Analysis)

Extract characterization was carried out using a Perkins Elmer 1310 infra-red spectrophotometer under the following conditions. Expansion- 0.5; Scan time – 3mintues; Drive time- 3 minutes; cell path-KBR and Reference- polystyrene. Spectrums were obtained (not presented here and the interpretation were made (Table 2)

RESULTS AND DISCUSSION

Solvent extraction: the amount of crude extract recovered is summarized in Table 1: below

Table 1: result of the crude extracts obtained during solvent extraction of coal samples

Sample	1st batch weight loss(g)	2nd batch weight	Total Weight	Percentage weight loss (%)
Okaba	2.61	2.35	4.96	4.31
Okpara	2.23	2.42	4.65	3.88

I.R Analysis:

The result of these analysis is summarized in table 2 below:

Table 2 spectral characteristic of extracts from Okaba and Okpara coal

Sample	Band, cm^{-1} 1st Batch	Assignment (vibration Characteristics)
OKABA EXTRACTS		
N-Hexane:	3100	Aromatic C-H, Ali CH_3, CH_2, CH
	1600	Aromatic C=C
Benzene Eluate	3020	Aromatic C-H
	2900-2800	Aliphatic C-H
	≈1600	C=O or C=C conjugated alkeness
Methanol	3400	OH and NH
	1300-1200	C=O from carbonyl Compounds
	≈1050	In-plane PH from primary alcohols
OKPARA EXTRACT:		
n-hexane Eluate:	3100	Aromatic C-H
	1650	Aromatic ring C=C
Benzene Eluate	3100	Aromatic C-H
	≈1450	Skeletal aromatic C=C
	750	H on substituted aromatic ring
Methanol Eluate	3400-3200	Oh from alcohols and phenols
	3300	H-bonded alc Phenol,
	1500-1300	NO_2 from nitro compound

The frequencies with which motions sensitive to detection by infra-red spectroscopy can be directly correlated with the structure of hydrocarbons and the identification of aromatic fragments [13]

The peaks identified are 3100-3000 cm^{-1}, indicating aromatic C-H 2900-2880cm^{-1} also indicate C-H group from aliphatic chains. Also, 1650-1600cm^{-1}, indicating aromatic C=C bonds; 1500-1300cm^{-1} indicating NO_2 from nitro compounds and 760cm^{-1} indicative of hydrogen atoms on substituted benzene ring [14]. These bands indicate the presence of aromatic and aliphatic fragments in both coal samples. These observations have also been made by [15].

Also prominent are the peaks 3400-3200cm^{-1}, 1300-1000cm^{-1}, indicative of polar compounds which are found in waxes and resins in colas[16]. [17] Compared the intensities of out-of-plane (OOP) C-H bond vibration with protion NMR of pyridine and CS_2 soluble coal extracts. They reported that, for both the extract and coal, the aromatic region increase with increasing carbon content suggesting that coal contain large materials which are aromatic and contain large polynuclear condensed ring systems. This is consistent with the result obtained.

CONCLUSION

The results from this instrument gave a consistent insight of the coal compounds. The I.R. spectra clearly showed major absorptions associated with aliphatic and aromatic C-H groups. In the liquefaction process (Soxhlet extraction), the aliphatic chains are liable as such fragmentation of the structure occurred at those points, Coal samples are of a low rank. Aromatization is a prove of strong grapheme layers which is a function of high energy vale of the two coal samples.

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