



**FEDERAL UNIVERSITY OF TECHNOLOGY  
MINNA**

**CATALYTIC HYDROCARBON  
CONVERSIONS FOR TRANSPORTATION  
AND DOMESTIC FUELS UPGRADE**

*By*

**FOLORUNSHO ABERUAGBA**

*B.Eng'g (ABU); M.Phil (Unilag); PhD (Unilag)*

*M.N.S.Ch.E, M.N.S.E., R. Engr. (COREN)*

*Professor of Chemical Engineering*

**INAUGURAL LECTURE SERIES 53**

**10<sup>TH</sup> AUGUST, 2017**



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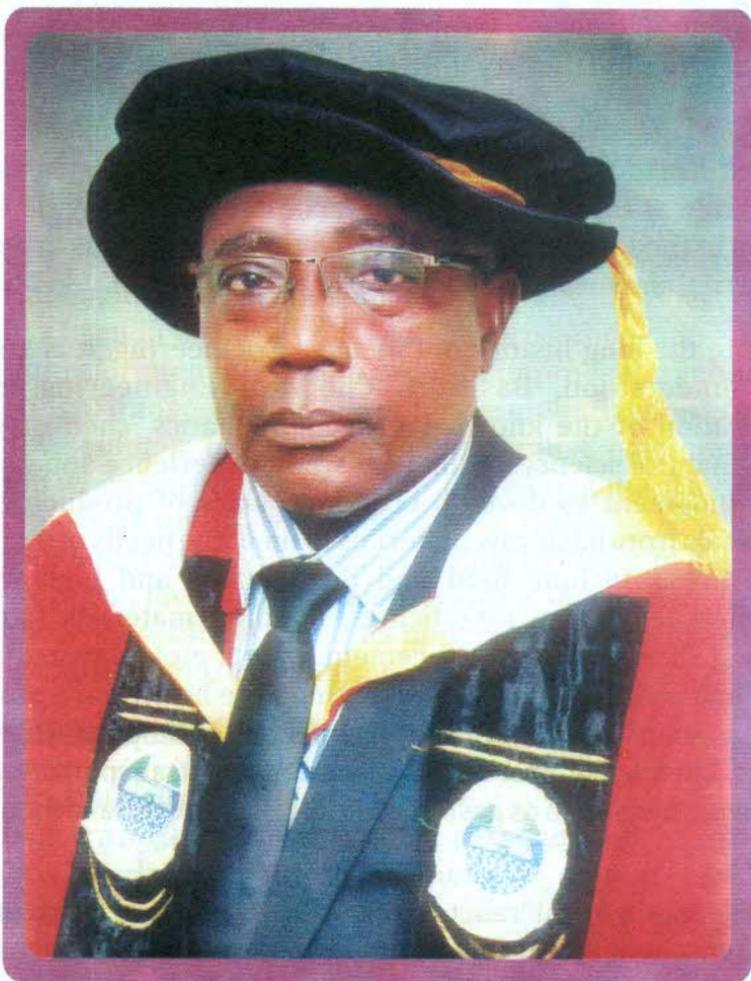
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## 1.0 INTRODUCTION

It gives me a great privilege and honour to present this Inaugural lecture, the third from the Department of Chemical Engineering and the fifty third from the University.

Chemical Engineering as a profession was first established in the United Kingdom when the first Chemical Engineering Course was given at the University of Manchester in 1887 by George E. Lewis in the form of twelve lectures covering various aspects of industrial chemical practice.

Despite the long history of Chemical Engineering, it is still not well understood. Basically, Chemical Engineering is the application of the knowledge of mathematics, chemistry and other natural sciences, gained by study, experience and practice with judgment to develop economic ways of producing bulk materials from basic raw materials to meet the needs of mankind. Such needs include food and drinks, foot and body wears, chemical fertilizers, insecticides, building materials (such as roofing sheets, metal rods, cement, paints etc), plastics, paper; dyes and writing/printing inks. Others are health care products such as soaps and detergents, disinfectants and pharmaceutical drugs, chemically processed insulators and transportation and domestic fuels such as gasoline, diesel and kerosene to mention a few.

Design of chemical reactors for the production of materials through chemical or biological transformation is an activity which uniquely differentiates a Chemical Engineer from other groups of Engineers. Most of the needs of man are met through chemical transformations, hence the importance of Chemical Engineering

An inaugural lecture can take any of the following forms (Ogunye, 1981):

- i. Concentrate on the development of the department, if the Lecturer is also the occupant of the Chair to which the leadership is attached,
- ii. Focus on the Professor's own work within the general framework of his discipline, and
- iii. Be on any general topic where the Professor considers that he has something fresh and stimulating to tell his audience.

For many years, I have been working in various aspects of the Chemical Engineering profession, but a significant aspect of the work has been in the area of catalytic hydrocarbon conversions either for reforming purpose in order to improve the antiknock properties of transportation fuel or for desulphurization in order to remove sulphur from fuel which can cause environmental pollution when burnt in engines. It is therefore natural for me to choose to speak on catalytic hydrocarbon conversions which is an essential part of the petroleum refining industry. In doing so, this lecture will focus on petroleum which is the primary source of hydrocarbons, catalysis and its importance in the petroleum refining industry and our modest contributions.

## **2.0 BACKGROUND**

### **2.1 Petroleum and its Origin**

Petroleum is a mixture of hydrocarbon gases and solids dissolved in liquids. It also contains compounds of oxygen, nitrogen, sulphur and different metals such as cobalt, nickel, potassium, calcium, sodium, silicon, copper, etc.

It is a Greek word coined from *petra* meaning rock and *oleum* meaning oil. Accordingly, petroleum means rock oil. It belongs to the family of energy resources called fossil fuel and it is a naturally occurring brown to black flammable liquid. Although exactly how it originated is not established but it is generally agreed that it is obtained from the accumulated debris of dead

plants, animals and living organisms in aquatic or marine environment over long periods of geological times and consequently organic in nature. This accumulated debris gives rise to sedimentary rocks which undergo complicated chemical transformations in the absence of air, in the presence of salt water from the sea and under high pressure. The resulting material becomes richer and richer in carbon and hydrogen, but less and less rich in oxygen and nitrogen. This new product can be liquid (petroleum) or gaseous (methane, ethane, propane, etc). The gases are formed under higher temperature conditions whereas the petroleum is formed under lower temperature conditions.

The formation of petroleum is a very slow process and is influenced by:

- i. catalytic effects of clays;
- ii. increased temperature (about 250°C);
- iii. increased pressure (50-200 atm);
- iv. biochemical activity of microorganisms.

The rocks where petroleum and gas were formed are known as the source rock. If these rocks are sealed by a layer of impermeable rock called cap rock, the petroleum accumulating within the pore spaces of the source rock is trapped to form petroleum reservoir. However, if conditions for trapping petroleum do not exist, petroleum and gas migrate under the effects of pressure and gravity, from the source rock until it is trapped in another capped (sealed) rock. The geologic structure in which petroleum reservoir has been trapped and accumulated, whether it is the source rock or the rock to which petroleum has migrated is called petroleum reservoir. Petroleum reservoirs exist in different sizes and shapes of geologic structures (Abdel-Aal et al, 2003). It is from this reservoir that petroleum is produced by drilling well(s) through which it is

pumped or forced by pressure to the surface. Typical reservoirs are shown in Figures 1-5 (Abdel-Aal *et al*, 2003).

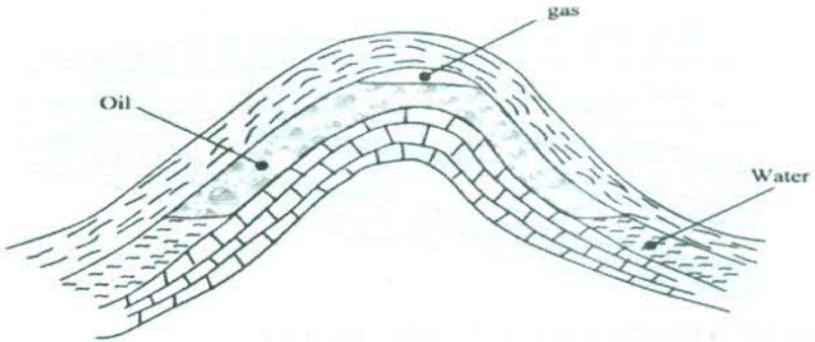


Figure 1: A reservoir formed by folding of rock layers

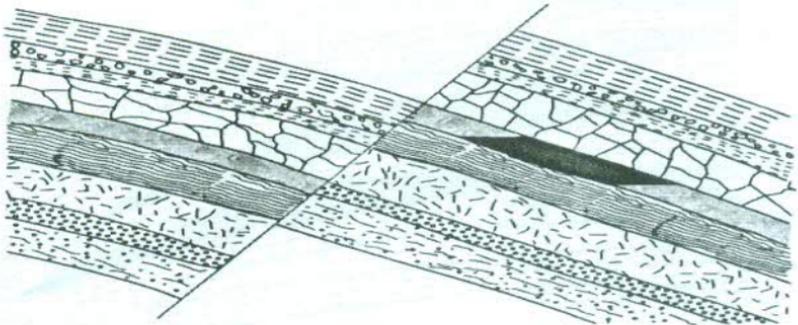


Figure 2: A cross section of a faulted reservoir

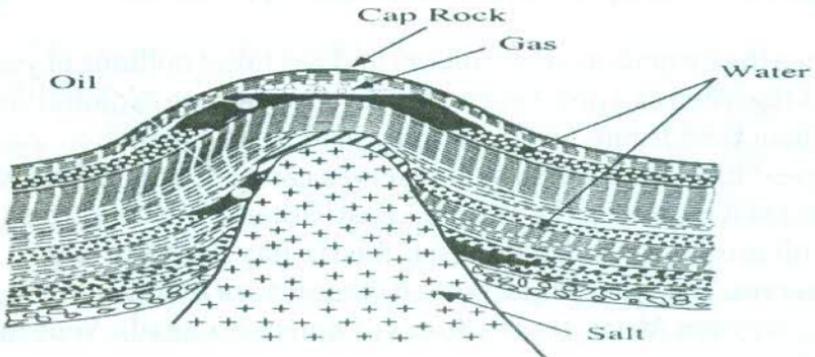


Figure 3: Section in a salt-dome structure

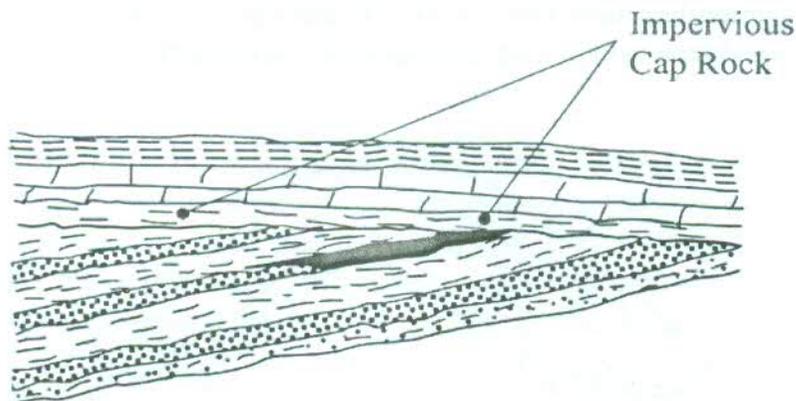


Figure 4: A reservoir formed by unconformity

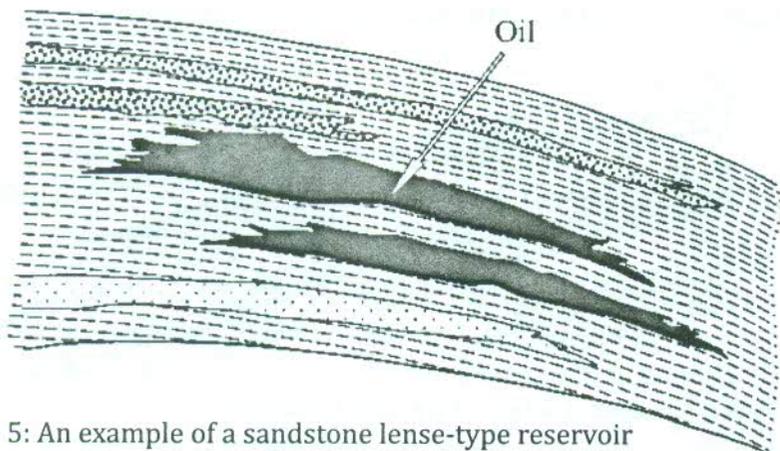


Figure 5: An example of a sandstone lense-type reservoir

Since the formation of petroleum and gas takes millions of years, it is regarded as a non-renewable resource whose exploitation in human time frame will eventually lead to its exhaustion. About three-fifths of the world's oil reserves can be found in Asia with most of it located in the Middle - East. Other Asian countries rich in oil are China and Indonesia. Russia has the next largest oil reserves. The other important oil reserves are found in Northern and Western Africa, United States of America, Canada, Venezuela and Australia.

In the United States of America, petroleum is classified into three types (Abdel-Aa *et al*, 2003):

- i. Paraffinic: Paraffinic hydrocarbons with a relatively lower percentage of aromatics and naphthenes;
- ii. Naphthenic: Cycloparaffins in a higher ratio and a higher amount of asphalt than in paraffinic petroleum;
- iii. Asphaltic: Fused aromatic compounds and asphalt in higher amount.

Other methods of classification are:

- i. Paraffinic base;
- ii. Mixed base;
- iii. Naphthenic base.

Based on this classification, a rating for the processing of petroleum is envisaged as shown in Table1 for the production of certain products and their treatments.

**Table1: Ratings for the Processing of Petroleum**

Types of oil	Lubricating oil product	Asphalt Product	Gasoline Product	Treatment of Products
Paraffinic	1	3	3	1
Mixed	2	2	2	2
Naphthenic	3	1	1	3

Rating: 1: Excellent; 2: Good; 3: Poor

## 2.2 Petroleum Production Operations

It is appropriate at this juncture to discuss the structure of the petroleum industry which consists of many parts each of which has acquired a major status and characteristics of its own. The more important of them are exploration, production, transportation, processing, marketing and distribution. The oil companies that engage in all of these activities are said to conduct integrated operations. Activities subsequent to the production of petroleum are called downstream operations and they include refining, marketing and distribution

The objective of exploration is to locate deposits of petroleum. The deposits become commercial if they occur in such quantities and circumstances that they could be produced and sold profitably under prevailing economic conditions. The volume of the oil at the market place must be sufficient to cover the initial investment costs, the continuing development and production costs and reasonable returns for the investor. The cost of exploration is usually very high and the results are not always certain.

Production follows successful exploration. By appraisal drilling and production tests, the extent and capacity of the discoveries are determined. Development wells are drilled in accordance with a predetermined production pattern. Government regulatory agencies closely monitor all production operations to ensure that they are conducted in accordance with good oil field practices. A typical oil production rig is shown in Figure 6.



Figure 6: A Typical Oil Rig.

Source:<http://en.wikipedia.org/wiki/oil-platform> (Retrieved 2<sup>nd</sup> August, 2017)

Oil production now contends with many environmental pollution problems from oil spillage and associated gas flaring. These problems engage the attention of the industry and the controlling agencies of government.

The transportation phase of the oil industry is well established in the form of pipelines and marine tankers. There are also road tankers and rail tank vehicles for petroleum products.

The refining industry is capital intensive and refineries are located either near markets or near sources of petroleum.

Product marketing is the final link in the chain of activities which began with exploration. It is the activity in which the public comes in direct contact with oil companies and their operations. The petrol filling station is the outlet for the bulk of conventional products. The oil companies may market their products directly but in many cases they operate through independent distributors known as dealers.

### **2.3 History of Oil Production**

The first modern petroleum well was dug by Colonel Edwin Drake in August 1859 near Titusville, Pennsylvania, USA (Lee, 1983). Before 1859, oil was produced from hand-dug wells in many countries including Burma, Canada, Romania and Russia.

The first attempt at prospecting for oil in Nigeria was by a German Company, the Nigeria Bitumen Corporation, which started prospecting for oil in the Araromi area of present Ondo State. Their initial efforts stopped following the outbreak of the First World War in 1914.

Oil prospecting by Shell D'Arcy (the forerunner of the present Shell Petroleum Development Company) resumed in Nigeria in 1937. This activity was again interrupted by the Second World War, but resumed in 1947. It was not until 1956 that oil in

commercial quantities was discovered at Otuabagi/Otuogadi in Oloibiri district in Ogbia LGA of Bayelsa State and the first petroleum export came from the Oloibiri field in February, 1958. Today, Nigeria ranks among the top ten petroleum producers of the World with an estimated petroleum reserve of 37.07 billion barrels (1<sup>st</sup> Jan., 2015 estimate, World fact book).

### 3.0 PETROLEUM REFINING

Petroleum has little or no direct use until it is refined into different hydrocarbon components, broken down or combined with other chemicals in a refinery to produce products which can be marketed. Petroleum refining takes place in a refinery which is a complex network of integrated unit processes for the purpose of producing a variety of products. The world's refining capacity is currently 87,913 million barrels/day whilst Nigeria which has four refineries has a combined refining capacity of 444,000 barrels per day (World Fact Book).

The history of the petroleum industry has shown that petroleum refining has evolved continuously as a result of changes in consumer demand for better and various types of products. Most of the changes in petroleum refining have been mainly in the secondary processes such as cracking, reforming, isomerization and alkylation.

From 1860 up to 1920, petroleum refining was generally limited to the production of kerosene as a cheaper and better source of light than whale oil. The advent of the automobile and its internal combustion engine in the early 20<sup>th</sup> century resulted in the demand for gasoline which was hitherto considered as nuisance. The evolution of aero planes resulted in the need for high octane jet fuel.

The size and complexity of a refinery depends on:

- (a) the nature and volume of petroleum to be processed



Petroleum refining involves:

- i) Beneficiation which is the removal of all forms of impurities such as particles, water, salts, etc.:
- ii) Distillation which is the separation of petroleum components at atmospheric pressure (760 mm Hg) or under vacuum (80 mm Hg) at 350-360°C or with the aid of steam:
- iii) Physical separation such as extraction and
- iv) Chemical modifications of the primary components to secondary or other forms as may be required.

The primary products of petroleum refining and their composition are as follows:

- i) Petroleum gas, made up of uncondensable gases (i.e. methane, ethane and occasionally some ethylene) and liquefiable or condensable gases (i.e. propane and butane). This fraction is collected at a temperature below 30°C and the hydrocarbon composition is  $C_1$  to  $C_4$ ;
- ii) Gasoline (Naphtha) containing  $C_5 - C_{12}$  hydrocarbons and is collected between 30 – 200°C;
- iii) Kerosene comprising  $C_{12}$  to  $C_{15}$  hydrocarbons and is collected between 200 – 260°C.
- iv) Gas oil (Diesel fuel) comprising  $C_{15}$  to  $C_{25}$  hydrocarbons and are collected between 250° – 350°C;
- v) Lubricating oil comprising  $C_{20} - C_{30}$  hydrocarbons and is collected under vacuum at 300°C;
- vi) Fuel oil: this is largely the residue.

The refining of petroleum does not produce some of the products in sufficient quantity hence the need for chemical transformation through cracking, alkylation, polymerization, isomerization and reforming

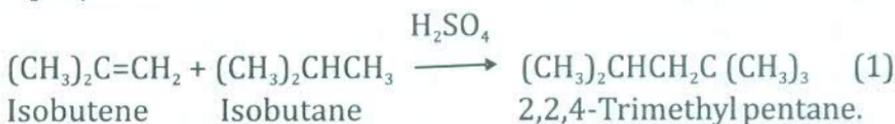
### 3.1 Cracking

Cracking ruptures C-C and C-H bonds in heavy petroleum

fractions and thus convert them to more valuable fractions. Of the various cracking processes namely: thermal cracking, hydrocracking, moving bed cracking and fluid catalytic cracking (FCC), the fluid catalytic cracking process is the most important in modern refining.

### 3.2 Alkylation

Alkylation refers to the reaction between a light olefin and a paraffin to form a highly branched chain molecules of higher octane rating. The reaction often takes place in the presence of  $H_2SO_4$  or HF



### 3.3 Isomerization

Isomerization involves the re-arrangement of n-paraffins in abundance in the C4 to C6 range into branched iso structures of much higher octane rating. Amorphous as well as zeolite catalysts are in vogue for isomerization of n-paraffins.

### 3.4 Catalytic Reforming

Catalytic reforming involves the re-construction of low octane hydrocarbons in the naphtha into more valuable high octane gasoline components without changing the boiling point range in the presence of a catalyst. This process results in the production of hydrogen which may make hydrotreatment and hydrocracking processes economical.

Catalytic reforming arose as a result of the phasing out of lead due to its hazardous nature and in compliance with strict environmental regulations. Usually  $\gamma-Al_2O_3$  supported Pt or Pt alloy catalysts are employed in reforming operations.

In petroleum refining, the process of reforming transforms low

octane (40 - 60) naphtha into high octane (90 - 100) gasoline (Pines H., 1981). The octane rating or number represents the ability of gasoline to resist knocking during combustion of the air-gasoline mixture in the engine cylinder. In practice, two octane ratings are measured, the Research Octane Number (RON) and the Motor Octane Number (MON) which differ in the test procedure. Research Octane Number represents the engine performance at low speed whereas MON is representative of high speed driving. By definition, the octane rating of n-heptane is zero and that of iso-octane (2,2,4-trimethylpentane) is 100. The octane rating for gasoline is defined as the volume percent of 2,2,4-trimethylpentane in blending with n-heptane that equals the knocking performance of the gasoline being tested. Some gasoline components have octane ratings exceeding 100 and have to be characterized by use of mixtures..

The major part of improvement in octane rating is due to the formation of aromatics (i.e aromatization); lesser contributions result from isomerization of straight chain paraffins to branched chain paraffins and hydrocracking of long chain hydrocarbons to lower molecular weight hydrocarbons in the gasoline boiling range. Octane ratings of typical hydrocarbons are shown in Table 2.

**Table 2: Octane rating of Pure Hydrocarbons** (Susu A. A., 1997)

Hydrocarbon	Blending Research Octane No. (Clear)	Hydrocarbon	Blending Research Octane No. (Clear)	Hydrocarbon	Blending Research Octane No. (Clear)
<b>Paraffin</b>		<b>Aromatics</b>		<b>Naphthenes</b>	
n - Butane	113	Benzene	99	Methylpentane	107
n - Pentane	63	Toluene	124	1,3 - Dimethyl - cyclopentane	96
n - Hexane	19	1,3 - Dimethyl-benzene	145	Cyclohexane	110
n - Heptane	0	Isopropyl-benzene	132	Methylcyclohexane	104
n - Octane	-19	1,3,5 Trimethyl-benzene	171	Ethylcyclohexane	43
2 - Methylhexane	41				
2,2 Dimethyl - Pentane	89				
2,3,3 Trimethyl - butane	113				

In addition to the above processes, there are processes for the upgrading of product quality such as hydrotreatment to remove impurities such as sulphur, oxygen and nitrogen containing compounds which utilize mainly molybdenum containing catalysts.

#### 4.0 CATALYSIS

Catalysts have been used by mankind for over 2000 years (Oyama and Somorjai, 1986) and the first observed uses of catalysts were in the making of wine, cheese and bread. It was found that it was always necessary to add small amounts of the previous batch to make the current batch. However, it was not until 1835 that Berzelius began to tie together observations of earlier scientists by suggesting that small amounts of a foreign source could greatly affect the course of chemical reactions. This mysterious force attributed to the substance was called catalytic. In 1894, Ostwald expanded Berzelius explanation by stating that catalysts were substances that accelerate the rate of chemical reactions without being consumed. In the over 181 years since Berzelius work, catalysts have come to play a major economic role in the world. It is estimated that the world's demand for catalyst will grow by 4.8% annually to \$20.16 billion in 2018 ([www.freedoniagroup.com](http://www.freedoniagroup.com)).

The technological advances in the production of chemicals, petro-chemicals and petroleum refining are partly due to application of catalysts. Catalysts reduce the cost of production and also make it possible for more desirable products to be produced where side reactions to undesirable products are likely to occur. The development and use of catalysts is a major part of the constant search for new ways of increasing product yield and selectivity from chemical reaction.

The application of catalyst usually changes a reaction rate by

promotion of a different molecular path (mechanism) for the reaction. This is illustrated in Figure 9.

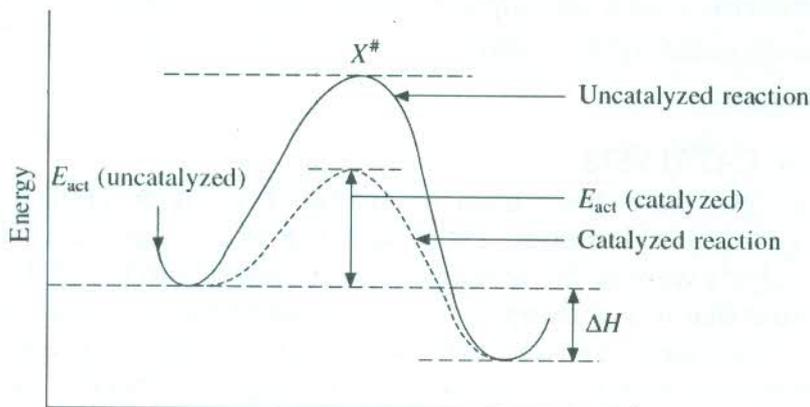


Figure 9: Reaction path for Catalyzed and Uncatalyzed reactions

Catalysts, however, have no effect on the position of equilibrium and one cannot make a reaction to proceed beyond what is dictated by thermodynamics. This is readily seen by considering a simple reversible reaction:



The standard free energy change is expressed as

$$\Delta G^\circ = -RT \ln K = -RT \ln(a_B/a_A) \quad (3)$$

Where  $a_B$  and  $a_A$  are the activities of product and reactant respectively. The presence of the solid catalyst cannot change  $\Delta G^\circ$  and hence does not change the ratio  $a_B/a_A$ .

Under actual process conditions, it is sometimes possible to improve performance over that which is calculated by thermodynamics by removing a product either chemically or physically. For example, a dehydrogenation reaction can be pushed by using oxygen to convert the free hydrogen to water. In other cases, a product may be removed by absorption or

adsorption into another phase under dynamic conditions. One can also recycle unreacted reactants.

Catalysts effective in practice range from minerals used with little or no processing and simple metal masses to substances of precisely controlled, complex composition prepared under closely controlled conditions. The latter's effectiveness in use may also require careful control of the reactor environment.

The desired catalytic action may range from the acceleration of a simple reaction that can yield only one product to a highly selective reaction that may involve complicated interactions among many intermediate species. In choosing or developing a catalyst, the difficulty of the problem may vary greatly as indicated by a suggested scheme reported by Satterfield (1981):

A. Selection among known catalysts

1. For known reactions;
2. For reactions analogous to known catalytic reactions;
3. For new reactions.

B. Search for new catalysts

1. For well known catalytic reactions;
2. For reactions analogous to those well known;
3. For reactions of new types having no analogs among well known reactions.

Solids exhibiting catalytic activity generally possess strong interatomic fields, such as that involved in ionic or metallic bonding. Covalent compounds (e.g. organics) generally are non-catalytic. A fundamental requirement is that the desired catalyst structure be stable under reaction conditions. For example, a desired metal must remain metallic and not converted to an inactive compound.

The activity of a catalyst depends mainly on its surface area, pore volume; mean pore radius, physicochemical nature of its surface

and its physical form. The particle size and the physical form of a catalyst determine the rate of intra-particle diffusion of reactant and pressure drop within reactor bed. The intra-particle diffusion limitation reduces the activity of the catalyst. It is for this reason that a powdered form of solid catalyst is used in a completely mixed or fluidized reactor in order to minimize the adverse effect of diffusion limitations. A coarse form is, however, used in a fixed bed reactor. The physicochemical properties of solid catalyst provide a lot of information on the active sites. The active site may be acidic and/or basic centers or specific sites for activation and adsorption. It may be present on the surface of the solid or generated at the presence of the reactant.

All catalysts deactivate or lose activity during use. This deactivation may be caused by:

(i). Fouling, which is a physical blockage of the catalyst surface by carbonaceous materials usually coke. This occurs simultaneously with desirable conversion reactions and it cannot be avoided. Removal of the carbonaceous material is by burning off in the presence of air (i.e. regeneration) at temperature less than  $500^{\circ}\text{C}$ . In the petroleum refining industry, platinum or platinum alloy catalyst can be used up to 10 months before regeneration for re-use;

(ii). Poisoning, which is a slow modification by chemisorptions on active sites by materials which are not easily removed. This can be avoided by the removal of impurities in the feed to the level that the catalyst can tolerate;

(iii). Sintering, which is the agglomeration of metal crystallites at high temperature. This can be avoided by operating at low enough temperatures and preventing run away temperatures during exothermic catalyst regeneration

Catalyst deactivation may be very rapid in the order of seconds or slow in the order of years. Whatever the case may be,

regeneration is required from time to time before the ultimate death of the catalyst. Omoleye and Susu (1986) have demonstrated that the mortality profile of platinum catalyst is similar to that of living organisms. They identified three stability states which are analogous to the three major stages defining human mortality, namely childhood, adulthood and old age

In the petroleum refining and chemical industry, the study of deactivation is of tremendous importance and about 50% of the time of professionals working on research and development in catalysis is devoted to the problem due to the high cost of catalysts. Platinum containing reforming catalysts are very expensive and the current price is put at \$1200.00 per ounce. The cost of a new 150,000 barrels/day refinery is about 2 billion dollars and the platinum catalyst cost is about \$576,000.00 which represents about 30% of the initial investment (Susu, 2006). The average catalyst life is 2 years before replacement and any effort to elongate its life will have a tremendous economic impact.

In order to prolong catalyst life, a quantitative and qualitative understanding of catalyst behavior especially during deactivation is necessary. As early as 1945, Voorhies succeeded in obtaining an empirical relationship between amount of coke deposited during coke deactivation in the hydroisomerization and hydrocracking of normal paraffins using mordenite bifunctional catalyst and time viz:

$$N_c = at^n$$

Where  $N_c$ =quantity of coke deposited,  $t$ =time and 'a' and 'n' are constants. Since then other deactivation models have been proposed.

Agrawa *et al* (1980) stated that if deactivation is due to the reduction in the number of active sites, then the kinetic behavior should remain unchanged with deactivation and the reduction in

the rate constant will then be attributed to the fractional reduction in the number of active sites. However, if deactivation is due to electronic factors, the value of the kinetic parameters would be expected to change on deactivation and this will provide a new insight into the deactivation mechanism. Dumez and Froment (1976), Omoleye and Susu (1989) have developed models which relate activity to coke content of the catalyst.

Mr. Vice-Chancellor sir, I shall now present our modest contributions in catalytic hydrocarbon conversion research. These studies are relevant in the gathering of reactivity and rate data useful in the design of chemical reactors for transportation and domestic fuels upgrade.

## **5.0 HYDROCARBON CONVERSION RESEARCH**

### **5.1 Catalytic Reforming and Deactivation Studies**

My first effort at hydrocarbon conversion research started at the University of Lagos in 1991 where I had the opportunity of working for my PhD in the area of catalytic reforming under the supervision of a world acclaimed authority in catalysis in person of Professor Alfred A. Susu who is also a past winner of both Nigerian National Order of Merit Award and NLG prize.

A variety of catalysts are active for reforming but reforming processes using platinum or platinum alloy catalysts have the highest current application and are usually bi-functional in nature.

Platinum is one of the rarest precious metals and its largest reserves are found in South Africa and in the former Soviet Union. There is no known platinum deposit in Nigeria at the moment and search could begin with the identification of ultramafic layered bodies.

The reactions occurring in catalytic reforming are very complex and the understanding of the complex kinetics has been made possible through the investigation of the catalytic reforming of pure hydrocarbons present in the naphtha.

The focus of our research in catalytic reforming has been in studying reaction mechanisms of hydrocarbon reactions, improving selectivity and of catalyst deactivation using commercial platinum and platinum-rhenium catalysts. The mechanisms of reactions are important to the Chemical Engineer whose responsibility is the design of chemical reactors in which these reactions take place.

Our studies cover a variety of hydrocarbon reactions on commercial Pt/Al<sub>2</sub>O<sub>3</sub> and Pt-Re/Al<sub>2</sub>O<sub>3</sub> catalysts. Both catalysts were identical except for the amount of Re in the bimetallic catalyst (0.3% Pt-0.3% Re) and the sulphur content (0.3% in Pt/Al<sub>2</sub>O<sub>3</sub> and 0.005% in Pt-Re/Al<sub>2</sub>O<sub>3</sub>). Surface area, chlorine content and pore volume of both catalysts were 180m<sup>2</sup>/g, 1%, and 0.5ml/g, respectively.

These studies were carried out in a pulse microcatalytic reactor whose set up is illustrated in Figure 10. A pulse microcatalytic reactor offers the following advantages:-

- i) Kinetic data are obtained in relatively shorter time than that of a flow reactor;
- ii) It is cost effective because it reduces the reactant cost to the barest minimum;
- iii) It allows for a comprehensive study of initial rates, poisoning effects, catalyst deactivation and other important relaxation phenomena which often give an insight into the reaction mechanism on the surface.

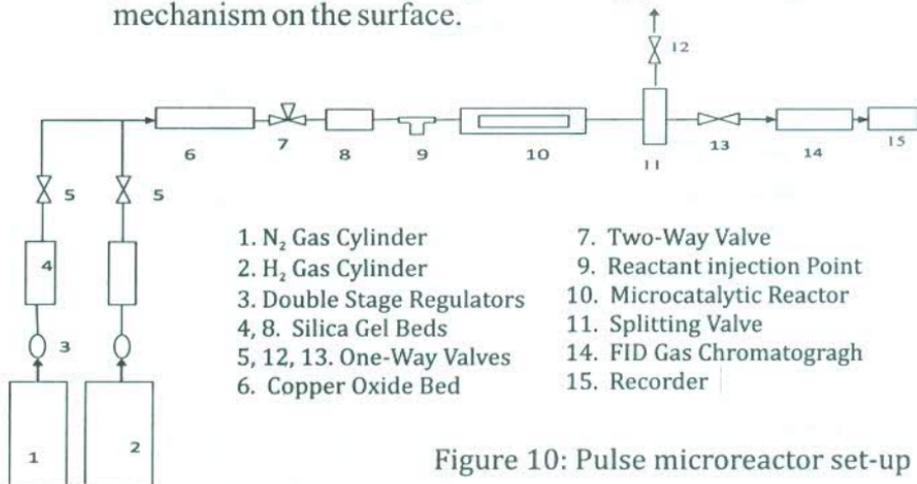


Figure 10: Pulse microreactor set-up

### 5.1.1 Nitrogen influence on reforming

Aberuagba and Susu,1999a first reported nitrogen influence on aromatic selectivity during n-heptane reforming on fresh and deactivating platinum/alumina and platinum - rhenium/alumina catalysts in which an unusual role of nitrogen in imparting aromatic selectivity on a fresh platinum-rhenium catalyst which is ordinarily a strong cracking catalyst was observed. The fresh Pt-Re/ $\text{Al}_2\text{O}_3$  catalyst has no intrinsic aromatization activity; the cracking selectivity was unity in  $\text{H}_2$ . Other researchers have previously reported that the superior activity of Pt-Re is imparted by sulphur which is usually present in the reforming feed (Biloen et al, 1980).

The monometallic Pt/ $\text{Al}_2\text{O}_3$  did not show this effect. Additionally, results in  $\text{N}_2$ -  $\text{H}_2$  mixtures showed that this effect only occurs up to a threshold of 50%  $\text{N}_2$  and at  $\text{N}_2$  levels below this value, aromatization is completely suppressed and the catalyst reverts back to its cracking activity. This is illustrated in Figures 11-14 for a typical W/F value of 1.5 mg-min/mL and pressure of  $4.0\text{kg/cm}^2$

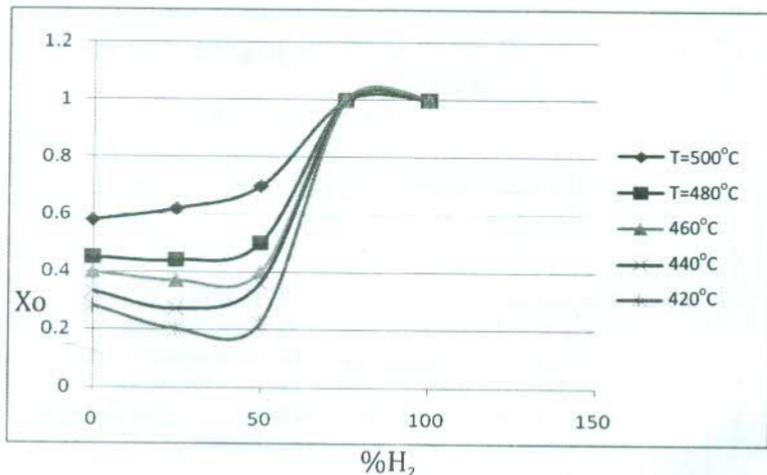


Figure 11: Effect of  $\text{H}_2$  and  $\text{N}_2$  on total conversion on n-heptane reforming on Pt-Re/Alumina catalyst at varying temperatures.

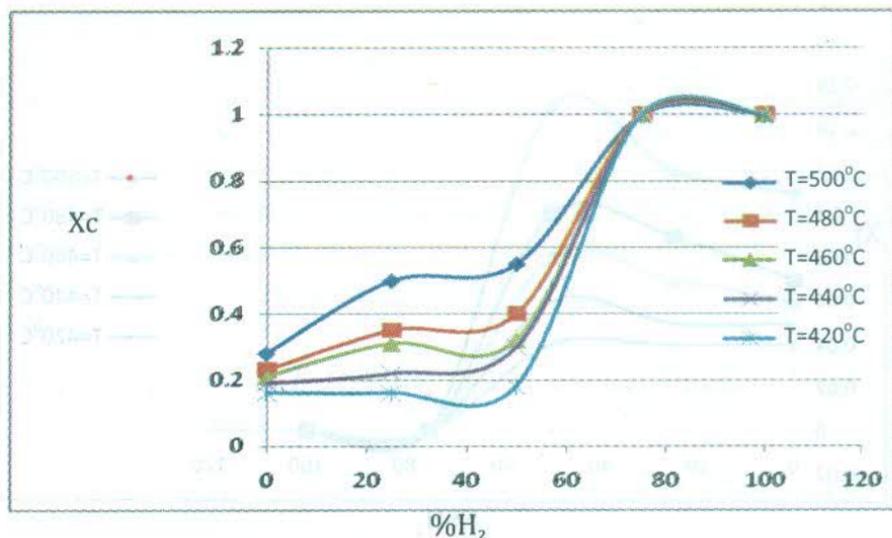


Figure 12: Effect of H<sub>2</sub> and N<sub>2</sub> mixture on cracked products production during n-Heptane reforming on Pt-Re/Alumina catalyst at varying temperatures

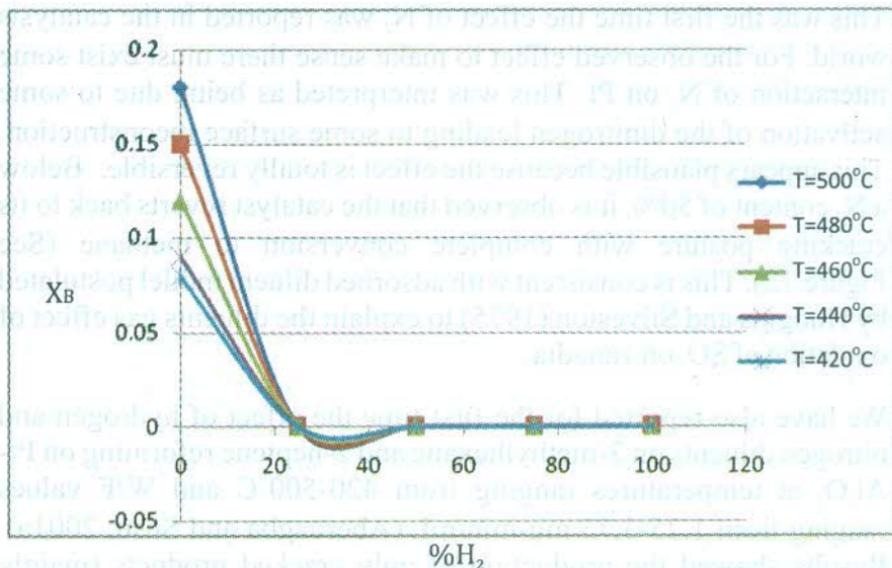


Figure 13: Effect of H<sub>2</sub> and N<sub>2</sub> mixtures on benzene formation during n-Heptane reforming on Pt-Re/Alumina catalyst at varying temperatures.

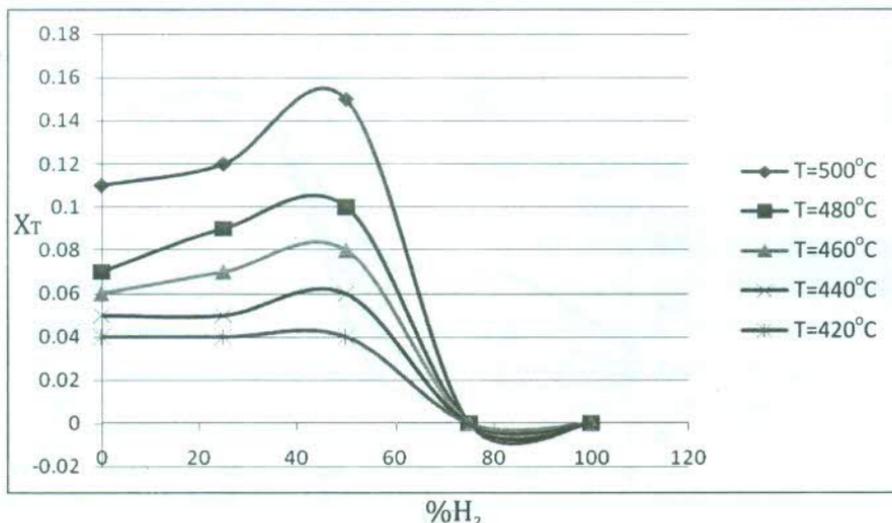
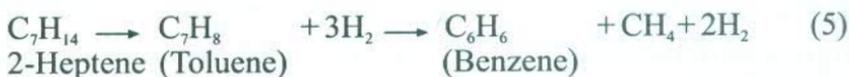
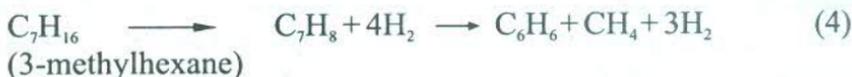


Figure 14: Effect of H<sub>2</sub> and N<sub>2</sub> mixtures on Toluene formation during n-Heptane reforming on Pt-Re/Alumina catalyst at varying temperatures

This was the first time the effect of N<sub>2</sub> was reported in the catalysis world. For the observed effect to make sense there must exist some interaction of N<sub>2</sub> on Pt. This was interpreted as being due to some activation of the dinitrogen leading to some surface reconstruction. This appears plausible because the effect is totally reversible. Below a N<sub>2</sub> content of 50%, it is observed that the catalyst reverts back to its cracking posture with complete conversion to methane (See Figure. 12). This is consistent with adsorbed diluent model postulated by Hudgins and Silveston (1975) to explain the diluents gas effect of oxidation of SO<sub>2</sub> on vanadia.

We have also reported for the first time the effect of hydrogen and nitrogen diluents on 3-methylhexane and 2-heptene reforming on Pt-Al<sub>2</sub>O<sub>3</sub> at temperatures ranging from 420-500°C and W/F values ranging from 1.25-3.75 mg-min/mL (Aberuagba and Susu, 2001a). Results showed the production of only cracked products (mainly methane) from deep fragmentation of 3-methylhexane and 2-heptene in hydrogen which sharply contrasts with the reaction in nitrogen

where intrinsic aromaticity was imparted to the catalyst with the production of methane, benzene and toluene. In hydrogen-nitrogen mixtures, 50% and 75% H<sub>2</sub> were required to reduce this aromatization activity of the catalyst for 3-methylhexane and 2-heptene respectively to zero. Furthermore, the reforming of 3-methylhexane and 2-heptene yields the same amounts of methane and benzene due to the demethylation of toluene and the fact that 2-heptene was more reactive than 3-methylhexane. The reaction networks of both reactants conversion in N<sub>2</sub> are:



The amount of adsorbed hydrogen has been reported to govern the type of chemisorbed specie on the catalyst surface: It also determines selectivity (Paal, 1980). In the absence of H<sub>2</sub> in the feed, dehydrogenation species are formed. These species are strongly adsorbed on the catalyst surface and may yield high molecular weight polymers (coke). The presence of N<sub>2</sub> probably allows the freezing of the reactive intermediates for facilitation of subsequent ring closure to form toluene. Paal (1980) have also reported the formation of aromatics (benzene) from n-hexane in helium on platinum black in a pulse reactor. This was attributed to a more deeply dehydrogenated surface intermediate leading to benzene, a process assisted by H<sub>2</sub> depleted surface. In pure H<sub>2</sub> carrier gas, the formation of deeply dehydrogenated specie was probably suppressed leading to fragmentation to cracked products and eventually to methane. It has been known that supported industrial platinum catalyst may produce more or less fragments in addition to useful products (such as aromatics and isomers, etc). Fragments may be simple or occur in multiple steps with the former process resulting in two fragments from one molecule, and the latter yielding extensive breakdown to

methane (Pines, 1981) A preliminary kinetic analysis showed a first order kinetics for both reactants with activation energies of 202.0 kJ/mol in  $H_2$  and 107.0 kJ/mol in  $N_2$  for 3-methylhexane and 124.7 kJ/mol in  $H_2$  and 102.6 kJ/mol in  $N_2$  for 2-heptene. However, this preliminary analysis would suggest that whatever the complexity of the reaction equations obtained with the more exhaustive models, it may be reduced to first order reactions with suitable assumptions especially with regard to the magnitudes of the equilibrium constants for the reactants and products. The similarity in product types and comparable activation energies in  $N_2$  suggest that the reforming of both reactants proceeds through similar pathways in  $N_2$ . However, the differences observed in  $H_2$  carrier gas suggest significant differences in the reaction in  $H_2$ . A comparative reaction study of methylcyclohexane and 3-methylhexane in  $H_2$  and  $N_2$  on  $Pt-Al_2O_3$  catalysts has also been reported (Aberuagba and Susu, 2002).

A significant contribution of this work, like that of the preceding one, is the impartment of aromatic selectivity to the  $Pt/Al_2O_3$  catalyst when the carrier gas was  $N_2$ . A similar behavior was found for n-heptane aromatization over  $Pt-Re/Al_2O_3$  (Aberuagba and Susu, 1999) showing that the observed effect is not limited to the bimetallic catalyst. Susu et al (1980), Hudgins *et al* (1975) and Paal (1980) had also reported on the diluent gas effect on cyclohexane dehydrogenation on  $Pt-Re/Al_2O_3$  catalyst,  $SO_2$  oxidation on vanadia catalyst and n-hexane conversion over Pt black, respectively. The explanation for the diluent effect has not been satisfactorily made. Susu *et al* (1980) observed a diluent gas effect with a difference in activation energies between hydrogen and inert ( $N_2$ , He, Ar) which was attributed to a change in mechanism on the coked catalyst surface. Hudgins *et al* (1975) selected the adsorbed diluent model as the most likely explanation for the observed diluent effect during the oxidation of  $SO_2$  on vanadia.

### 5.1.2. The role of added carbon on reforming

Apart from the above works on the unusual role of nitrogen in imparting aromatic selectivity during n-heptane, 3-methylhexane and 2-heptene reforming, our work (Aberuagba and Susu, 1999b) on the role of added carbon on the behaviour of Pt/Al<sub>2</sub>O<sub>3</sub> and Pt - Re/Al<sub>2</sub>O<sub>3</sub> is worthy of mention. This work, for the first time, showed that C1 carbon incorporated catalyst is more selective for aromatization reaction and also leads to higher stability. Furthermore, the carbon incorporated surface was found to be different from surface covered by carbonaceous deposit from a reacting hydrocarbon

In a gas consisting of 75% H<sub>2</sub> in N<sub>2</sub> at a total pressure of 4.0 kg/cm<sup>2</sup>, W/F value of 1.5mg-min/mL and temperature ranging from 420-500°C, the products on Pt/ Al<sub>2</sub>O<sub>3</sub> were hydrogenolysis products (C<sub>1</sub>-C<sub>6</sub>), toluene and benzene from demethylation of toluene whereas only methane from deep fragmentation of n-heptane were produced on Pt-Re/Al<sub>2</sub>O<sub>3</sub>. However on successful carbon incorporation by the injection of optimum 1μL carbon tetrachloride at 500°C, no effect was found with Pt-Al<sub>2</sub>O<sub>3</sub>, but the product distribution was markedly affected on Pt-Re/Al<sub>2</sub>O<sub>3</sub> where a dramatic increase in toluene selectivity at the expense of methane production was observed. In order to confirm that this dramatic effect was due to carbon and not chlorine in the carbon tetrachloride (CCl<sub>4</sub>), a similar injection of hydrochloric acid (HCl) was applied to both catalysts at the same conditions. This chlorine addition neither affected the total conversion nor the product distribution. A comparison between carbon incorporated on the Pt-Re catalyst at 500°C from separate feeds of CCl<sub>4</sub> and CH<sub>4</sub> is shown in Table 3. The comparable amount of carbon atoms deposition on the catalyst surface, the catalyst activity, hydrogenolysis reduction and concomitant toluene production were quite similar proving that the observed effect is due to carbon alone.

**Table 3: Comparison between carbons incorporated from  $\text{CCl}_4$  and  $\text{CH}_4$  on Pt-Re catalyst at  $500^\circ\text{C}$  and carrier gas flow rate of  $100\text{mL}/\text{min}$**

	<b><math>\text{CCl}_4</math> incorporated on Pt-Re</b>	<b><math>\text{CH}_4</math> incorporated on Pt-Re</b>
Total conversion( $X_o$ )	0.955	1.00
Methane mol. frac.( $X_c$ )	0.64	0.67
Toluene mol. frac.( $X_t$ )	0.32	0.33
Vol of carbon source retained	0.25 $\mu\text{L}$	0.22 mL
Single carbon atoms retained	$5 \times 10^{18}$	$4.11 \times 10^{18}$

The observed effect may be attributable to the stabilization of carbon atoms incorporated onto the surface matrix of the catalyst. That the carbon incorporation did not affect the monometallic catalyst is probably due to the fact that the optimum distribution of Pt ensembles needed for aromatization is only created by the deposit of amorphous deposit since Pt-C bonds are relatively weak. The aromatization efficiency of the Pt-Re catalyst has been widely reported as being enhanced by the addition of sulphur to form inactive Re-S or with the addition of other inactive additives like tin or lead (Sachtler, 1984; Ribeiro *et al*, 1994). In a similar fashion, the incorporation of single carbon atoms should facilitate the formation of strong Re-C bonds which then divides the Pt-Re surface into smaller Pt ensembles favouring aromatization.

Furthermore, this new site configuration in the bimetallic catalyst surface excludes sites for demethylation and further cracking of toluene to benzene and hydrogenolysis products. The carbon incorporated bimetallic catalyst was also found to be stable as a result of increased dispersion of the metal on the catalyst surface as less deleterious coke deposition is expected on a more dispersed surface.

### 5.1.3. Identification of reaction intermediates

In the formulation of the sequence of steps for the elucidation of the reaction mechanisms of a particular reaction, it is very helpful to identify in separate experiments the species that are likely reactive intermediates. This has become necessary because of the rather expensive method of isotope labeling for the identification of reaction intermediates.

We have shown interest in charting the reaction pathways for n-heptane reaction in nitrogen and hydrogen atmospheres. In doing this, we have located some hydrocarbons which are likely candidates as reactive intermediates for n-heptane reforming namely: 2-heptene representing a likely dehydrogenated intermediate, 3-methylhexane representing the formation of an isomer prior to further reaction and methylcyclohexane representing the aromatization reaction which is needed for toluene production. In analyzing the suitability of the hydrocarbon species as likely reactive intermediates for n-heptane reaction, selectivity is a good indicator for characterizing the complex n-heptane reaction. In this regard studies on comparative aromatic selectivity during reforming of n-heptane, heptene-2, 3-methylhexane and methylcyclohexane on Pt/Al<sub>2</sub>O<sub>3</sub> were carried out (Aberuagba *et al*, 2001b). It was found that aromatization products manifested under N<sub>2</sub> atmospheres for all hydrocarbon reactants investigated. However, under H<sub>2</sub> atmosphere, aromatization was only observed for n-heptane at all temperatures (420–500°C) investigated – and for methylcyclohexane at temperature below 460°C. In contrast only cracked products were observed for heptene-2 and 3-methylhexane at all temperatures investigated. What are the implications of these results on the prediction of reactive intermediates for n-heptane reaction?

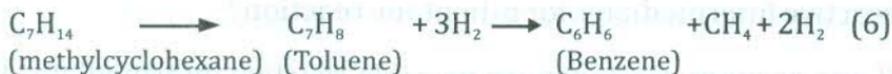
If one or more reactants are possible reactive intermediates for another reactant, there will exist a unique rate determining step

that will provide a unique product profile and rate of reaction for a given set of process conditions. Applying this principle to 2-heptene, 3-methylhexane and methylcyclohexane, we note that for any of these reactants to qualify as a reactive intermediate for n-heptane reaction, it must possess the same rate determining step yielding the same product profile. The fact that the product profiles for n-heptane are different from those for heptene-2 and 3-methylhexane means that it is unlikely that these hydrocarbons are intermediates in the reforming of n-heptane. The likely rate determining step for n-heptane is a dehydrocyclization step. Since methylcyclohexane occurs downstream of dehydrocyclization step, it can have a different product profile and still qualify as an intermediate for the n-heptane reaction. In addition, the fact that aromatization occurred in H<sub>2</sub> for n-heptane whilst only cracking occurred for heptene-2 and 3-methylhexane corroborated this conclusion.

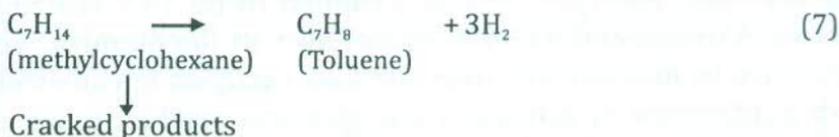
This was the first time a hypothesis of this nature was proposed in the identification of reaction intermediates.

#### 5.1.4 Kinetic analysis of methycyclohexane

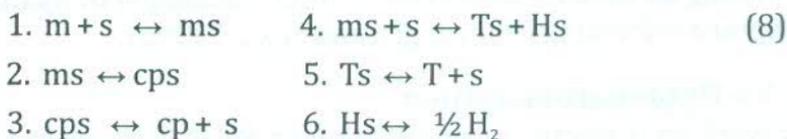
The identification of possible reaction intermediates enables us to carry out comprehensive kinetic analysis of various hydrocarbons. This involves determination of reaction mechanisms, rate controlling steps, the rate equation and the determination of kinetic parameters through appropriate mathematical techniques. In this regard, we have reported a comprehensive kinetic analysis on methylcyclohexane aromatization over Pt-Re/Al<sub>2</sub>O<sub>3</sub> and Pt/Al<sub>2</sub>O<sub>3</sub> catalyst (Aberuagba and Susu, 2004). Under nitrogen atmosphere, methylcyclohexane reforming proceeded by the following reaction network:



However in hydrogen there are no demethylation sites and the reforming reaction proceeded by the following reaction network:



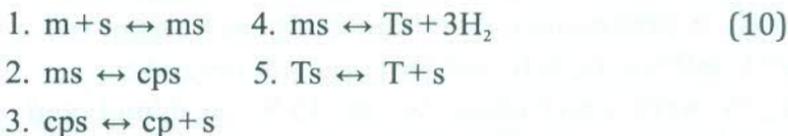
The study showed that based on the reaction network in hydrogen, over Pt-Re/ Al<sub>2</sub>O<sub>3</sub> catalyst, the following mechanism applied with desorption of toluene as the rate determining step:



The rate expression was determined to be:

$$-r = \frac{2.75 \times 10^{-7} m - 0.472 [T]}{1 + 5.25 \times 10^{-4} [m] + 5.25 \times 10^{-4} [CP]} \quad (9)$$

However, over Pt/Al<sub>2</sub>O<sub>3</sub>, the following mechanism applies with desorption of toluene similarly rate controlling:



The rate expression was determined to be:

$$-r = \frac{4.97 \times 10^{-6} [m] - 0.06 [T]}{1 + 0.0559 [m] + 0.174 [CP]} \quad (11)$$

Where: m=methylcyclohexane; s=vacant site; cp=cracked products; T=toluene; ms=adsorbed methylcyclohexane;

cps=adsorbed cracked products; Ts=adsorbed toluene; Hs=adsorbed hydrogen.

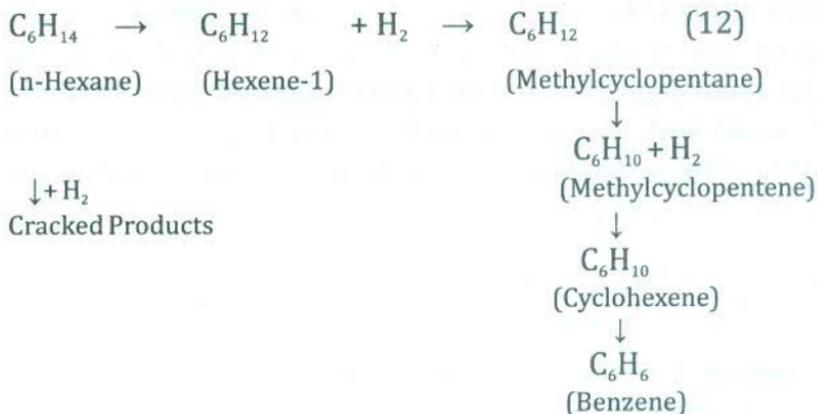
The activation energies were determined to be 19.7 kcal/mol over Pt/Alumina and 34.94 kcal/mol over Pt-Re/Alumina. The difference in mechanisms over the two catalysts is consistent with a difference in activation energies and product types on both catalysts as only toluene was formed on Pt/Al<sub>2</sub>O<sub>3</sub> whereas toluene and cracked products were formed on Pt-Re/Al<sub>2</sub>O<sub>3</sub> at the temperature of 350°C considered here. The cracking accompanying aromatization on Pt-Re/Al<sub>2</sub>O<sub>3</sub> in H<sub>2</sub> is not surprising as we have observed its high cracking activity during n-heptane reforming (Aberuagba and Susu, 1999a).

### 5.1.5 n-Hexane aromaization

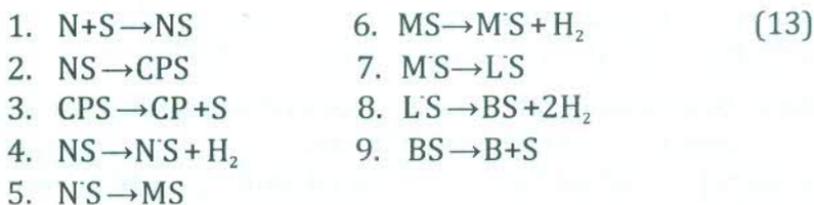
Our work on n-hexane aromatization on Pt-Al<sub>2</sub>O<sub>3</sub> catalyst under H<sub>2</sub> and He atmospheres (Agbajelola and Aberuagba, 2007) revealed that, while cracked products and benzene were produced in He, cracked products, methylcyclopentane and benzene were however produced in H<sub>2</sub>. The difference in product types in both carrier gases showed that n-hexane aromatization proceed through different mechanisms. Reactions in H<sub>2</sub> and He mixtures revealed that the yield of methylcyclopentane increases with % H<sub>2</sub> while benzene appears to pass through a maximum at 50% H<sub>2</sub>. A preliminary kinetic investigation suggested a first order kinetics in both H<sub>2</sub> and He atmospheres and the activation energies were determined to be 15.92 kcal/mol and 19.5 kcal/mol respectively. The difference in activation energies in both carrier gases clearly confirmed the difference in the mechanism for n-hexane aromatization in both carrier gases. A similar study over a bimetallic Pt-Re/Al<sub>2</sub>O<sub>3</sub> catalyst (Agbajelola and Aberuagba, 2009a) showed that in H<sub>2</sub> only, cracked products were formed whereas in He, cracked products, methylcyclopentane and benzene were formed. However,

introduction of more than 25% He in H<sub>2</sub> imparted aromatic selectivity on the catalyst with the production of cracked products, methylcyclopentane and benzene. This was the first time a diluent gas effect was reported for n-hexane aromatization on Pt-Re/Al<sub>2</sub>O<sub>3</sub>.

A comprehensive kinetic analysis of n-hexane aromatization on Pt/Al<sub>2</sub>O<sub>3</sub> in H<sub>2</sub> had also been reported (Agbajelola and Aberuagba, 2009b) based on the following reaction network:



Mechanistic reaction studies showed that mechanism involving unadsorbed hydrogen viz:



described the reaction and the rate determining step was determined to be the conversion of adsorbed hexene-1 to adsorbed methylcyclopentane with the following reaction rate;

$$-r = (k_f C_N - k_r C_B) / (1 + K_N C_N + K_{CP} C_{CP} + K_B C_B) \quad (14)$$

Where: N = n-Hexane; NS= adsorbed n-Hexane; CPS= adsorbed cracked products; CP=cracked products; N<sup>\*</sup>=Hexene-1; M=Methylcyclopentane; M<sup>\*</sup>=Methycyclopentene; MS=adsorbed Methylcyclopentane; M<sup>\*</sup>S = adsorbed Methylcyclopentene; L = Cyclohexene; B=Benzene; BS=adsorbed Benzene;  $k_p$ ,  $k_r$  =rate constants;  $K_N$ ,  $K_{CP}$ ,  $K_B$  =equilibrium constants.

### 5.1.6 Deactivation model for Pt/Al<sub>2</sub>O<sub>3</sub> catalyst

Folorunsho Aberuagba (1998) tested various models for the deactivation of Pt/Al<sub>2</sub>O<sub>3</sub> in 60% hydrogen with nitrogen diluents, a compromise mixture between a sharp deactivation under nitrogen and a negligible deactivation under hydrogen. Of all the various deactivation models tested to predict the deactivation of Pt/ Al<sub>2</sub>O<sub>3</sub>, only a first order reaction rate with first order deactivation rate viz:

$$\ln \ln \left( \frac{1}{1-X} \right) = \ln k_2 - (i-1)k_d \tau$$

Where:

X = conversion,  $k_1$  = constant,  $k_d$  = deactivation,  $i$  = pulse number,  
 $\tau$  = pulse residence time

was found to adequately describe the decay of Pt/ Al<sub>2</sub>O<sub>3</sub> during n-heptane reforming. The parameters obtained are tabulated in Table 4. The activation energies at various flows for the deactivation of platinum catalyst is shown in Table 5.

**Table 4: Deactivation rate constants for first order rate of reaction and first order deactivation rate.**

Temp(°C)	60 ml/min.		100 ml/min.		140 ml/min.	
	$k_d$	$k_2$	$k_d$	$k_2$	$k_d$	$k_2$
440	0.030	0.24	0.019	0.37	0.015	0.07
460	0.026	1.38	0.019	1.82	0.017	0.50
480	0.032	3.06	0.027	4.95	0.022	1.92
500	0.032	3.06	0.027	4.95	0.022	1.92

**Table 5: Activation energies at varying flow rates of the carrier gas**

Flow ml/min	$\Delta E$ (Kcal/g mol)
60	2.48
100	7.19
140	7.50

The low values of the activation energies obtained 2.48–7.50 K<sub>cal</sub>/gmol suggest that the decay reaction is only limited by thermodynamic effects rather than by kinetic effects. Ayo and Susu (1988) have also established that the deactivation of a bimetallic Pt/Al<sub>2</sub>O<sub>3</sub> catalyst during cyclohexane under helium atmosphere is unactivated.

## 5.2. Hydrocarbon Desulphurization Studies

In 1999, I obtained a Third World Academy of Sciences Fellowship for post-doctoral research tenable at the Indian Institute of Petroleum, Dehradun. This Institute is a world acclaimed center for research in all aspects of petroleum processing. In the catalyst laboratory of the Institute, I had the opportunity of collaborating with eminent scientists who are acclaimed authorities in the field of catalysis like Drs. Gudimella Murlidhar and Lakshmi Datt Sharma. With the assistance of others, we jointly investigated the use of mixed oxides as support for hydrotreatment catalysts. Hydrotreatment is undertaken for the removal of sulphur compounds. Fuels containing sulphur compounds are not only poisonous to catalyst used in reforming process and automobile catalytic converter but also such fuels on combustion produce sulphur oxide, thus causing serious environmental pollution leading to global warming and several human health concerns such as cardiovascular diseases, cancer, creation of asthmatic symptoms and other respiratory diseases.

Sulphided Co and Ni promoted molybdenum (Mo) or tungsten (W) metals supported on gamma alumina ( $\gamma\text{-Al}_2\text{O}_3$ ) are currently in use for hydrotreatment. To reduce or eliminate environmental pollution, the sulphur content in the fuel needs to be reduced to a lower level (<50 ppm). To meet the new challenges of environmental regulations and to process heavier feed stocks economically, highly active and selective catalysts are needed.

One of the strategies that is used in the search for a better catalyst is taking advantage of preparation methods to develop a support which result in better interaction with the metal component leading to desired activity and selectivity. Support plays an important role in controlling the specific characteristics of a catalyst. The textural and chemical properties of a support can be controlled by the method of preparation and by using different combinations of mixed oxides.

### **5.2.1 Preparation and characterization of zirconia/alumina and magnesia/alumina mixed oxides**

Zirconia based mixed oxides have been found to be thermally stable and consist of the desired textural and structural characteristics more suitable for hydrodesulphurization catalyst. Magnesia based mixed oxides have not been widely studied. Consequently, we have used the method of precipitation by urea hydrolysis for the preparation of  $\text{MgO}/\text{Al}_2\text{O}_3$  mixed oxide (Aberuagba *et al* 2002) and  $\text{ZrO}_2\text{-Al}_2\text{O}_3$  mixed oxides (Aberuagba *et al* 2003). This method is cheap and permits the formation of a precipitate at a nearly ideal rate which is rarely attained by conventional methods. These mixed oxides were also characterized by determining their BET surface areas, micro pore surface areas, total pore volume, absorption-desorption isotherms, the x-ray diffractogram, surface acidity and catalytic functionality. A key finding in these studies is that incorporation of 50% magnesia or zirconium oxide into alumina yielded mixed oxide of the highest surface area. Also beyond 50% magnesia or

zirconium oxide incorporation into alumina, renders the mixed oxide more amorphous. The catalytic functionality for cumene cracking and total acidity measurements indicates that while the incorporation of magnesia into alumina decreases its acidity since it is itself basic, the incorporation of zirconium oxide into alumina increases its acidity due to a synergistic effect.

### 5.2.2 Preparation and characterization mixed oxides supported molybdenum catalysts.

We also studied the characteristics of mixed oxides supported molybdenum hydrotreatment catalyst (prepared by incipient wetness procedure) by determining their BET surface area, x-ray diffraction, temperature programmed reduction, oxygen chemisorptions and catalytic functionalities for hydrodesulphurization, hydrogenation and hydrocracking (Manoj Kumar *et al*, 2004; Aberuagba *et al*, 2004). These studies showed an optimum metal loading of 8% on both  $ZrO_2/Al_2O_3$  and  $Mg/Al_2O_3$  mixed oxides. Typical results of the effect of support on the catalytic functionalities are shown in Tables 6 and 7.

**Table 6: The Catalytic activities of MgO supported Mo catalysts**

Catalyst	R <sub>HDS</sub> (mol/hg)	R <sub>HYD</sub> (mol/hg)	R <sub>HYC</sub> (mol/h.g)
8 wt% Mo/MgO	0.0526	0.0156	0.02365
8 wt% Mo/Al <sub>2</sub> O <sub>3</sub>	0.02356	0.0364	0.03234
8 wt% Mo/MgO- Al <sub>2</sub> O <sub>3</sub>	0.01178	0.0305	0.03625

**Table 7: The Catalytic activities of ZrO<sub>2</sub> supported Mo catalysts**

Catalyst	R <sub>HDS</sub> (mol/hg)	R <sub>HYD</sub> (mol/hg)	R <sub>HYC</sub> (mol/h.g)
8 wt% Mo/ZrO <sub>2</sub>	0.0240	0.01323	0.03023
8 wt% Mo/Al <sub>2</sub> O <sub>3</sub>	0.0235	0.0304	0.0324
8 wt% Mo/ZrO <sub>2</sub> - Al <sub>2</sub> O <sub>3</sub>	0.0330	0.0308	0.0429

Results in Tables 6 and 7 clearly demonstrate that the  $ZrO_2-Al_2O_3$  supported catalyst is far superior to the alumina supported and  $MgO/Al_2O_3$  supported catalysts for hydrotreatment. The incorporation of Co as promoter enhanced the catalytic functionalities as shown in Tables 8 and 9.

**Table 8: Promotional effect of Co on 8% Mo/MgO- $Al_2O_3$  catalyst**

% Co	$R_{HDS}(\text{mol/hg})$	$R_{HYD}(\text{mol/hg})$	$R_{HYC}(\text{mol/h.g})$
0	0.01178	0.0305	0.03625
1	0.0423	0.0359	0.03635
3	0.0585	0.0473	0.0376
5	0.0488	0.0375	0.03287

**Table 9: Promotional effect of Co on 8% Mo/ $ZrO_2-Al_2O_3$  catalyst**

% Co	$R_{HDS}(\text{mol/hg})$	$R_{HYD}(\text{mol/hg})$	$R_{HYC}(\text{mol/h.g})$
0	0.03303	0.0308	0.0429
1	0.0355	0.03398	0.0431
3	0.0406	0.0394	0.05588
5	0.0231	0.0382	0.05311

Tables 8 and 9 show an optimum loading of 3% Co for the highest catalytic activities for hydrodesulphurization, hydrogenation and hydrocracking.

From Mossbauer emission spectroscopy (MES), Co in sulphided  $Mo/Al_2O_3$  catalyst have been reported to be present in several forms, the ones easily identifiable being Co-Mo-S,  $Co_9S_8$  and  $Co-Al_2O_3$  (Topsoe and Clausen, 1984). These various forms, however, depend on catalyst preparation method and sulphidation. Of all the forms of Co structure, Co-Mo-S structure was reported to be responsible for the promotional effect on catalyst activity for

HDS and the activity was reported to be proportional to the amount of Co present. Co-Mo-S structures are not restricted to Co-Mo-Al<sub>2</sub>O<sub>3</sub> catalysts. Such structures have been observed in carbon supported Co-Mo catalyst (Brysse *et al*, 1982). A similar structure could be attributed to the promotional effect observed on the mixed oxide supported catalyst reported here.

### 5.2.3 Adsorptive desulphurization

Due to high cost of hydrogen used for hydrotreatment, much effort are being devoted to developing techniques that can reduce or remove refractory sulphur compounds by oxidation (Guoxian Y. *et al* 2007) , extraction(Jiang et al 2008), adsorption (Zhao *et al* 2006) and biodesulphurization (Gomez *et al* 2006). However, adsorption is the most preferred alternative to achieve ultra clean fuels. Adsorption is a mass transfer process wherein molecules in a free phase become bound to a surface by intermolecular forces. We have undertaken a comparative analysis of adsorptive desulphurization of crude oil by activated manganese dioxide and activated zinc oxide. The results indicated that activated manganese dioxide has a higher adsorption capacity than activated zinc oxide. Useful kinetic /adsorption data were also reported (Adeyi and Aberuagba, 2012).

### 5.2.4 Kerosene deodourization

We have also used acid treated local clays from Kagara in Niger State for kerosene deodourization (Aberuagba, 1997). The kerosene produced in most refineries are not suitable for direct use as a solvent because of the presence of aromatics and sulphur which causes objectionable odour and is responsible for smokiness (i.e. formation of soot) when burnt in internal combustion engines. Deodourized kerosene can be used as a cheaper substitute for volatile solvents like ethanol which is used as a propellant or as base oil in the cosmetic and allied industries. Of the three types of clays, namely, laterite red kaolin, light grey

kaolin and dark grey kaolin, the light grey kaolin showed the greatest promise removing 46% of the aromatics and sulphur in a single stage. In order to facilitate the commercial production of deodourized kerosene using this clay, suitable design adsorption data were obtained. For example, a Freundlich adsorption isotherm described the adsorption process with an order of adsorption of 0.225 and an adsorption equilibrium constant value of 0.387. Apart from the use of clays for kerosene deodorization, we have attempted the use of activated carbons from agricultural wastes namely goat bones, saw dust and groundnut shells for the same purpose (Kagher, 2008). Results showed that these materials are suitable for kerosene deodorization and the Freundlich isotherm also fits the adsorption process, the best being goat bones activated carbons with an order of adsorption value of 0.9 and adsorption equilibrium constant of 0.7783.

## CONCLUSIONS

1. The reforming products of n-heptane, heptene-2, 3-methylhexane, methycyclohexane and n-hexane on Pt/Al<sub>2</sub>O<sub>3</sub> and Pt-Re/Al<sub>2</sub>O<sub>3</sub> were determined to be either cracked products (methane) or benzene or toluene or a combination of all or two of them while that of n-hexane are cracked products and benzene or methylcyclopentane or a combination of all depending on the catalyst type and carrier gas. Of these products, the aromatics (benzene and toluene) and naphthene (methylcyclopentane) serve as octane number boosters in transportation fuel (gasoline) and thus enhance its antiknock properties. A preliminary kinetic analysis of the reforming processes suggested a first order reaction rate, but a comprehensive analysis suggested a more complex reaction rate equation for each process.
2. Our studies had also shown how selectivity for the

production of the aforementioned aromatics could be significantly improved through nitrogen gas or helium gas diluent effect or through single carbon incorporation on the catalyst surface. These beneficial effects were first reported by us.

3. We have shown how reaction intermediates can be identified in order to facilitate the determination of reaction mechanisms which is crucial in kinetic analysis for the evaluation of important reactor design parameters. This procedure was first reported by us as far as we know and the method is much cheaper than the conventional isotope labelling method.
4. We have shown the superior activity of  $\text{ZrO}_2\text{-Al}_2\text{O}_3$  (in the ratio 1:1) supported Mo hydrotreatment catalyst for sulphur removal in fuels compared to mostly used  $\text{Al}_2\text{O}_3$  supported Mo catalysts. The beneficial effect of incorporation of 3% cobalt ions on activity was also shown.
5. Due to high hydrogen costs, we have demonstrated the efficacy of adsorptive desulphurization using activated manganese dioxide and activated zinc oxide for sulphur removal. The efficacy of local kaolin clays from Kagara was also demonstrated. Useful process design parameters were also obtained

## 7.0 RECOMMENDATIONS

1. A country like Nigeria despite its huge energy resources still has challenges of development arising from inadequate infrastructure, slow and ineffective legislative system, monumental corruption, net importer of food and industrial products, poor maintenance culture, regulatory constraints, near absence of the spirit of nationalism and security risks limiting new investment in oil and natural gas production. The

challenges of development call for a more aggressive reorientation of Nigerians and prudent management of all resources from petroleum (which provides 95% of foreign exchange earnings) to ensure the development of other sectors of the economy, particularly agriculture and solid minerals which used to be the main stay of Nigeria's economy before the discovery of petroleum.

2. The vast arable land in Nigeria (which is under cultivated) is suitable for the cultivation of crops and the large quantity of agricultural waste there from can be used for the production of bioethanol which is the current fuel of interest for automobiles on account of its high octane number, of biodegradability and because it burns cleanly with oxygen and hence reduces green house gas emissions. Many countries have embraced the use of bioethanol wholly or blended it with petrol to power their automobiles and they include countries like, Brazil, United States of America, Indonesia, the Philippines, Guatemala, Costa Rica, South Africa, etc. Recently, France indicated its plan to ban the use of gasoline and petroleum diesel by 2040. It has been proved that gasoline blended with up to 30% bioethanol can be used in automobiles without engine modification. The use of 100% bioethanol would, however, require engine modification. Apart from the use of bioethanol as fuel, it can also be catalytically converted to ethylene which is an important feedstock in the petrochemical industry for various categories of plastic materials. Our efforts in bioethanol production from agricultural wastes and process design for same are well documented.
3. Nigeria can also produce biodiesel and biolubricants from vegetable oils to replace petroleum diesel and lubricants for which there are many documented works including ours in the literature.

4. Nigeria is also endowed with a lot of solid minerals from virtually all geo-political zones which can be processed to useful end products including catalysts. The adjoining sea bed is yet to be exploited also for precious metals suitable for jewelry and as sources of catalysts. Very little is being done in the area of catalyst synthesis. Hence our Universities and Research Institutes need to collaborate in this direction as the cost of catalyst is a significant factor in the cost of petroleum and chemical products. The setting up of a center of excellence for catalyst development may not be out of place.

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Mr. Vice-Chancellor sir, I hope you understood me. I am done!

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## **BIOGRAPHY OF ENGR. PROF. FOLORUNSHO ABERUAGBA**

**E**ngr. Prof. Folorunsho Aberuagba was born in Minna, Niger State on Wednesday, 22nd February, 1956 to late Dr. Gabriel Adeoye Aberuagba and Hajia Aishatu Mohammed.

He attended Baptist Transfer School, Minna for his first school leaving certificate from 1963-1969. In 1970, he proceeded to Baptist High School, Shaki in present Oyo State where he started his secondary education. In 1971, he transferred to Kufena College (Formerly St. Paul's College), Wusasa, Zaria where he completed his secondary education in 1974 with Division One in the West African School Certificate Examination.

In 1974, he proceeded to the School of Basic Studies, Ahmadu Bello University, Zaria and was absorbed into the Chemical Engineering programme of the same University in 1975. He latter graduated with the B.Eng (Chemical Engineering) degree in 1979 in the second class upper division.

He was posted to Oyo state for National service between 1979 and 1980 and did his primary assignment at Lafia Canning Factory, Ibadan where he served as a Process Overseer.

After his National service, he joined the services of the Federal Polytechnic, Bida and later proceeded to the University of Lagos, Lagos in 1983 and obtained the M.Phil (Chemical Engineering) degree in 1985. In the same year he joined the services of the then Mona Juice Ltd, Minna as Production manager.

As a result of his desire to make a career in Academics, he joined the services of the Federal University of Technology, Minna as a Lecturer 2 in 1987. In 1991 he proceeded to the University of Lagos, Lagos for a PhD (Chemical Engineering) degree programme and graduated in 1995.

He has been involved in teaching, research, and community service since joining the university and has held many academic

and administrative responsibilities. He became a Professor of Chemical Engineering in 2005 and has over 62 publications in both local and international journals to his credit.

He has worked in various aspects of the Chemical Engineering Profession but his major work has been in the area of catalyst preparation, characterization and functionally studies, catalytic conversion of hydrocarbons for petroleum products upgrade and for petrochemical feedstock production, renewable energy studies with particular interest in biofuels and enzyme synthesis and characterization.

He has supervised many undergraduate and postgraduate research projects at both the M.Eng. and PhD. levels and has served as External Examiner for same in many Universities. He has also assessed many Journal and Professorship publications for publishers and Universities respectively. Furthermore he has served as resource person for both NUC and COREN for accreditation purposes.

He is a past recipient of Federal Government of Nigeria Scholarship for both undergraduate and postgraduate studies and also a recipient of a Fellowship of the Third World Academy of Sciences for postdoctoral research at the Indian Institute of Petroleum, Dehradun.

He is a registered Engineer with the Council for the Regulation of Engineering in Nigeria (COREN) and member of both the Nigerian Society of Engineers and Nigeria Society of Chemical Engineers.

He is listed by International Biographical Center as one of 2000 outstanding Intellectuals of the 21st century, Cambridge Book man of the year 2005 and one of leading Educators of the world, 2005. He is also listed by Marquis Who's Who in Science and Engineering.

He is happily married with a wife and three (3) children.