Optimization of Biodiesel Production: Transesterification of Edible and Non-Edible Vegetable Oils

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ABSTRACT

In this study, both edible (palm oil) and non-edible oils (Jathropha and Neem oil) were used to optimize the biodiesel production process variables like catalyst concentration, amount of alcohol required for reaction, reaction time and reaction temperature. Biodiesel production from different edible and non-edible vegetable oils was compared in order to optimize the biodiesel production process. A two-step and single-step transesteri? cation process was used to produce biodiesel from high free fatty acid (FFA) non-edible oils and edible vegetable oils, respectively. This process gives yields of about 90–92% for J. curcas, 82–84% for neem, and 90–95% for palm oil using potassium hydroxide (KOH) as a catalyst.

Keywords: *Biodiesel, Transesterification, Optimization, Jatropha, Neem, Groundnut oil.*

1.0 INTRODUCTION

The increased use of diesel fuel resulted in depletion of its fossil reserves. This triggers for many initiatives to search for alternate fuel, which can supplement or replace such fossil fuel. In recent years, research has been directed to explore plant-based fuels and plant oils and fats as fuels have bright future (Martini and Shell, 1998). The most common that is being developed and used at present is biodiesel, which is fatty acid methyl esters of seed oils and fats and have already been found suitable for use as fuel in diesel engine. Presently the world's energy needs are met through non-renewable resources such as petrochemicals, natural gas and coal. Since the demand and cost of petroleum based fuel is growing rapidly, and if the present pattern of consumption continues, these resources will be depleted in few years. Hence, efforts are being made to explore for alternative source of energy. An alternative fuel must be technically feasible, economically competitive, environmentally acceptable and readily available (Srivastara and Prasad, 2000) Biodiesel is found to be environmentally safe, non-toxic and biodegradable (Harrington, 1986).

The raw materials being exploited commercially by the developed countries constitute the edible fatty oils derived from rapeseed, soya bean, palm, sun? ower, coconut, linseed, etc. (Korbitz, 1999). Use of such edible oil to produce biodiesel in Nigeria is not feasible in view of a big gap in demand and supply of such oils in the country. Increased pressure to augment production of edible oil has also put limitation on the use of these oils for production of biodiesel. Under such conditions, those crops that produce non-edible oil in appreciable quantities can be grown in large scale in non-cropped marginal lands and wastelands only considered for biodiesel production (Mohibbe and Waris, 2005).

Fatty acid methyl esters derived from renewable sources such as vegetable oils has gained importance as an alternative fuel for diesel engines. The edible oils such as soyabean oil in USA, rapeseed oil in Europe and palm oil in countries with tropical climate such as Malaysia are being used for the production of biodiesel to fuel their compression ignition engines (Knothe, 2002). In Nigerian context, the use of edible oils for engine fuel is not feasible; however, there are several non-edible oilseed species such as Jatropha (*Jatropha curcas*) and Neem (*Azadirachta indica*), which could be utilized as a source for production of oil.

Extensive work has been done on the transesteri? cation of non-edible and edible vegetable oils; however, no signi? cant work has been done on the optimization of Jathropha

curcas, neem and bleached palm oil. An optimization study on biodiesel production for the edible (bleached palm) and non-edible (Jathropha curcas and neem) vegetable oils were done in detail with one-step alkali transesteri?cation process and two-step acid esteri?cation process, respectively with fuel property analysis of these oils.

2.0 MATERIALS AND METHODS

Edible (Bleached palm oil) and non-edible (Jatropha and neem oil) oils were used in this experiment. These oils were extracted at NARICT, Zaria processing plant. All the oils were ?rst ?ltered using muslin cloth mainly to remove the dirt and other inert materials from the oil and then placed in a ?ask. A round bottom ?ask was used as laboratory scale reactor for the experimental studies in this work, and a hot plate with magnetic stirrer arrangement was used for heating the mixture in the ?ask. For both edible and non-edible oils, the mixture was stirred at the same speed of 450 rpm for all test runs.

Under agitation, the raw oil was heated up to nearer to the boiling point to remove the water contaminant present in the oil. After that oil is allowed to cool down under room temperature, and the treated oil alone was taken for biodiesel production purpose. Again, under agitation, the above treated oil was heated up to a desired temperature on a hot plate. A ?xed amount of freshly prepared sodium hydroxide–methanol solution was added into the oils, taking this moment as the starting time of the reaction. When the reaction reached the preset reaction time, heating and stirring were stopped. The products of reaction were allowed to settle. During settling two distinct liquid phases were formed: crude ester phase at the top and glycerol phase at the bottom. The crude ester phase separated from the bottom glycerol phase was then washed by cold water several times until the washed water became clear. The excess methanol and water in ester phase were then removed by heating > 100^0C.

After that, weight of the ester was taken for product yield calculation. The reaction was investigated step by step. The optimal value of each parameter involved in the process was determined while the rest of the parameters were kept constant. After each optimal value was attained, this value was adopted for the optimization of the next parameter. The parameters such as alcohol to oil molar ratio, catalyst amount, reaction temperature, and reaction time were analyzed.

(a) Two-step esterification procedure for non-edible oils

In acid esterification, oil was poured into the flask and heated to about 55^0C. Then 20%(v/w) of methanol was added and stirred at low stirring speed for 30 min. followed by 0.5% (v/v) of sulfuric acid. The reaction mixture was then poured into a separation funnel to remove excess alcohol, sulfuric acid and impurities. The treated oil having acid value less than < 1. 0.25 mg KOH/g was used for the alkali transesterification reaction. It has been reported that transesterification would not occur if FFA content in the oil was above 2% (Canakci and VanGerpen, 1992). The J. curcas oil has an initial acid value of 8mg KOH/g and neem oil has an acid value of 12 mg KOH/g. The experimental set-up for alkali catalyzed transesterification was the same as that was used for of acid catalyzed pretreatment. 1% of KOH was dissolved in 20 %(w/w) of methanol and half of that was poured into the flask containing an unheated mixture from acid esterification step and stirred for 10 min. After 10 min, the mixture was heated and stirred continuously to about 60^0C, and then the remaining methoxide was added to it. The reaction was continued for next 1 hr.

(b) One-step alkali transesterification procedure for edible oils

In alkali transesterification, treated oil from pretreatment procedure was allowed to cool at normal temperature. Meanwhile, 1 % of KOH was dissolved in 20% (w/w) methanol and half of it was poured into the unheated oil and stirred for 10 min. The mixture was heated to temperature of 55^0C in the flask. Then added remaining mixture of methanol and catalyst to it and reaction was continued for 1h at 450rpm. After the alkali transesterification reaction was

completed in edible and non-edible oils, the reactant mixture was allowed to be separated into two layers. The bottom layer having a brownish red color and containing the impurities and crude glycerol was drawn off. The esters along with the catalyst remained in the upper layer were then separated from the reactant mixtures. The transesterified product (biodiesel) after separation was first distilled to remove the unreacted methanol and then washed 2–3 times with hot water to remove the dissolved glycerol in the biodiesel phase.

(c) Effect of catalyst concentration.

The effect of sodium hydroxide concentration on the transesterification of the edible and non-edible oils was investigated with its concentration varying from 0.25 to 1.25 wt. % (based on the weight of raw oil). The operation conditions during the whole reaction process were fixed at the optimal level: reaction temperature of 55^{o}C reaction time of 90 min. and 180 and 210 ml of methanol for edible and non-edible oils, respectively.

(d) Effect of reaction time

The reaction time of the transesterification reaction conducted at 55^{0}C was optimized with the highest achievable mixing degree, an excess amount of alcohol (220 ml per liter of oil) and optimal sodium hydroxide concentration of 1.25 wt. % for all the oils. The transesterification time vary from 25 to 105min.

(e) Effects of methanol amount.

The effect of methanol amount on yield of the transesterification experiments was conducted with different ratio of methanol to oil in the range of 3:1 to 15:1. The optimized catalyst concentration time as obtained in the above sections were adopted.

(f) Effect of reaction temperature.

To study the effect of reaction temperature on methyl esters' formation, the transesterification reaction was carried out under the optimal conditions obtained in the previous sections. The experiments were conducted at temperature ranging from 20 to 80^{o}C interval.

3.0 RESULTS AND DISCUSSION

Experimental results showed changes in ester yield content with varied catalyst concentration. As the sodium hydroxide concentration increased the conversion of triglyceride as well as the ester content also increased. Insufficient amount of sodium hydroxide resulted in incomplete conversion of triglycerides into the esters as indicated from its lower ester content. The ester content reached an optimal value when the sodium hydroxide concentration reached 1wt. % and further increase in catalyst concentration in all the cases, ester production amount decreased as shown in Fig. 1. Large amount of soap was observed in excess amount of sodium hydroxide added experiments. This is because addition of excess alkaline catalyst caused more triglycerides' participation in the saponification reaction with sodium hydroxide, resulting in the production of more amount of soap and reduction of the ester yield.

The changes in product composition with reaction time during the transesterification of the oils and the distribution of various components in the reaction system can be clearly seen. When the reaction time reached 65 min., no triglyceride was left in the product mixture, indicating complete conversion. In this experiment, glycerol started to separate within 25 min. The ester content increased with reaction time 25 min onwards and reached a maximum at a reaction time of 65 min. at 55^{o}C, and then decrease with increasing further the reaction time (Fig. 2). The results indicated that an extension of the reaction time from 65 to 105 min. had no significant effect on the conversion of triglycerides but leads to a reduction in the product yield. This is because longer reaction enhanced the hydrolysis of esters (reverse reaction of transesterification), resulted in loss of esters as well as causing more fatty acids to form soap.

Experimental results showed that the transesterification reaction could proceed within the temperature range studied but the

reaction time to complete the reaction varied significantly with reaction temperature as shown in fig. 3. It can be seen that a high product yield could be achieved at 65°C. With the temperature increase above 65°C, the product yield started to decrease. The reaction for this is that higher temperature accelerates the side saponification reaction of triglycerides.

Maximum ester content was obtained at a methanol amount of 6:1 for edible oil and 9:1 for non-edible oils. With further increase in the methanol to oil amount above 9:1, a very little effect on the biodiesel yield was observed (Fig. 4). Moreover, it was observed that for high alcohol amount added the set up required longer time for the subsequent separation stage since separation of the ester layer from the water layer becomes more difficult with the addition of a large amount of methanol. This is due to the fact that methanol, with one polar hydroxyl group, can work as an emulsifier that enhances emulsion. Therefore, increasing the alcohol amount to oil is another important parameter affecting the biodiesel yield and biodiesel purity, apart from catalyst concentration and reaction time. This result is in line with the report of many investigations based on neat vegetable oils (Meher etal, 2006; Senthil et al, 2003; Encinar et al, 2002). However, the non-edible like *Jatropha* and *neem* require 9:1 ratio of methanol to KOH to give maximum ester yield, possibly due to higher viscosity of non-edible oil than edible oils. In this case more amount of methanol is required to increase the solubility of the oil in the methanol. This leads to maximize the ester yield at high methanol concentration level. However, when compared to edible oil ester content yield was minimal in non-edible oil but glycerol yield was found to be more in non-edible oil when compared to edible oil.

4.0 CONCLUSION

The study on the biodiesel production process optimization of edible and non-edible oils showed that the quantity of catalyst, amount of methanol, reaction temperature and reaction time are the main factors affecting the production of methyl esters. From the results the following were drawn:

- Addition of excess catalyst causes more triglycerides' participation in the saponi? cation reaction leading to a marked reduction in the ester yield.
- Biodiesel production process is incomplete when the methanol amount is less than the optimal value. Operating beyond the optimal value, the ester yield would not be increased but will result in additional cost for methanol recovery.
- Higher reaction temperature decreases the viscosities of the oils and resulted in increased rate o f transesteri? cation and shortening of the reaction time. When the temperature increases beyond the optimum level it induces a negative impact on the ester yield due to acceleration of the saponi? cation of triglycerides.
- Suf? cient reaction time should be allowed to ensure complete conversion of triglycerides into esters. However, excess reaction time did not promote the conversion but favors the reverse reaction of transesteri? cation which resulted in a reduction in the ester yield.

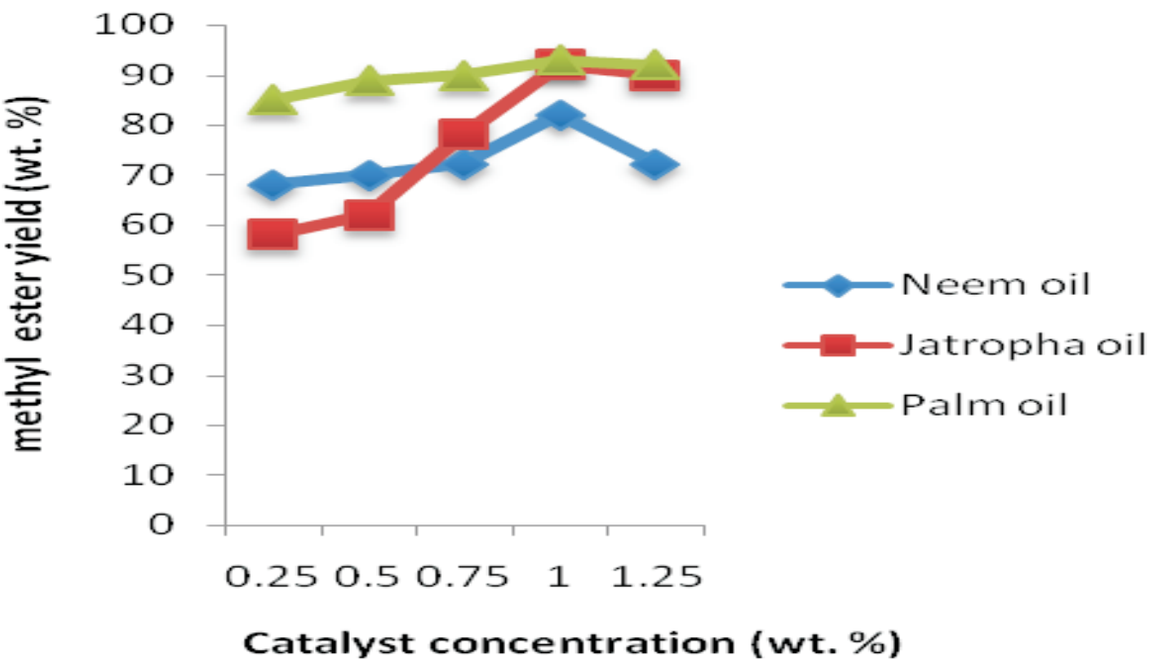

Fig. 1 Effect of catalyst concentration on methyl esters' yield

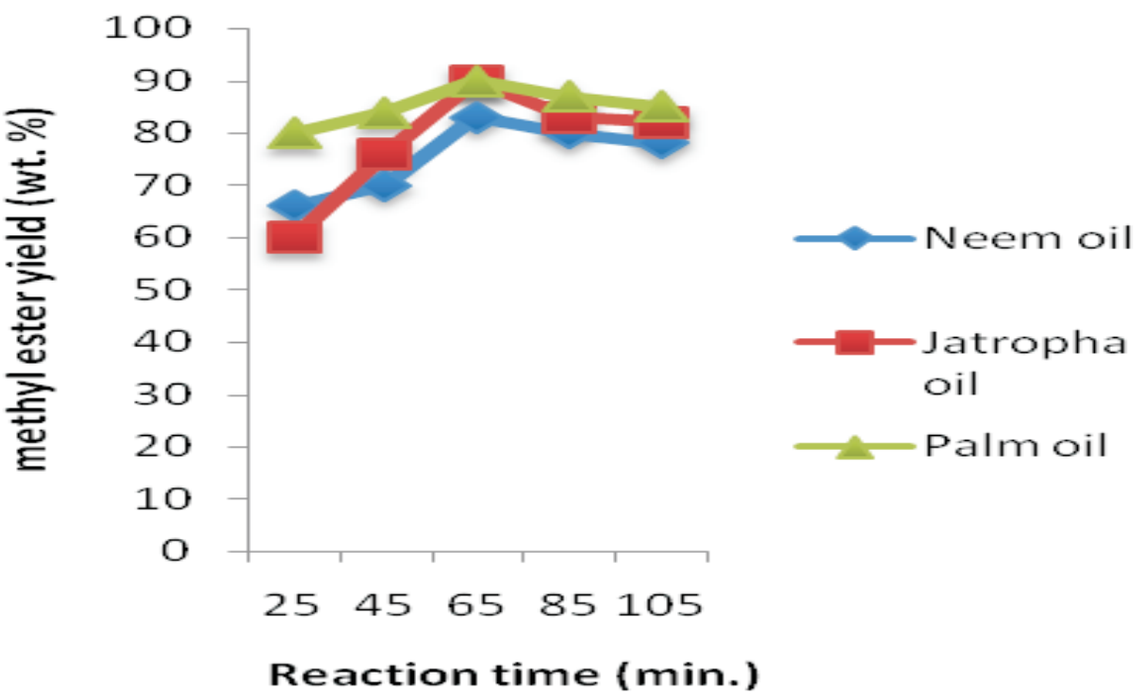

Fig. 2 Effect of reaction time on methyl esters' yield

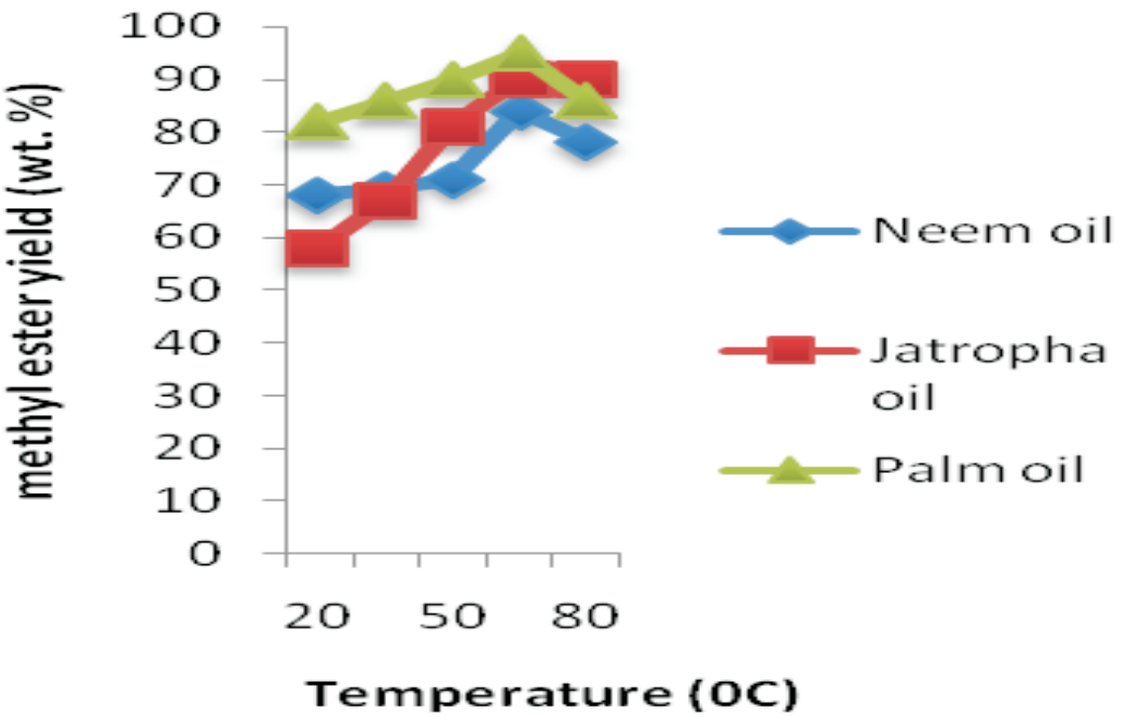

Fig. 3 Effect of temperature on methyl esters' yield

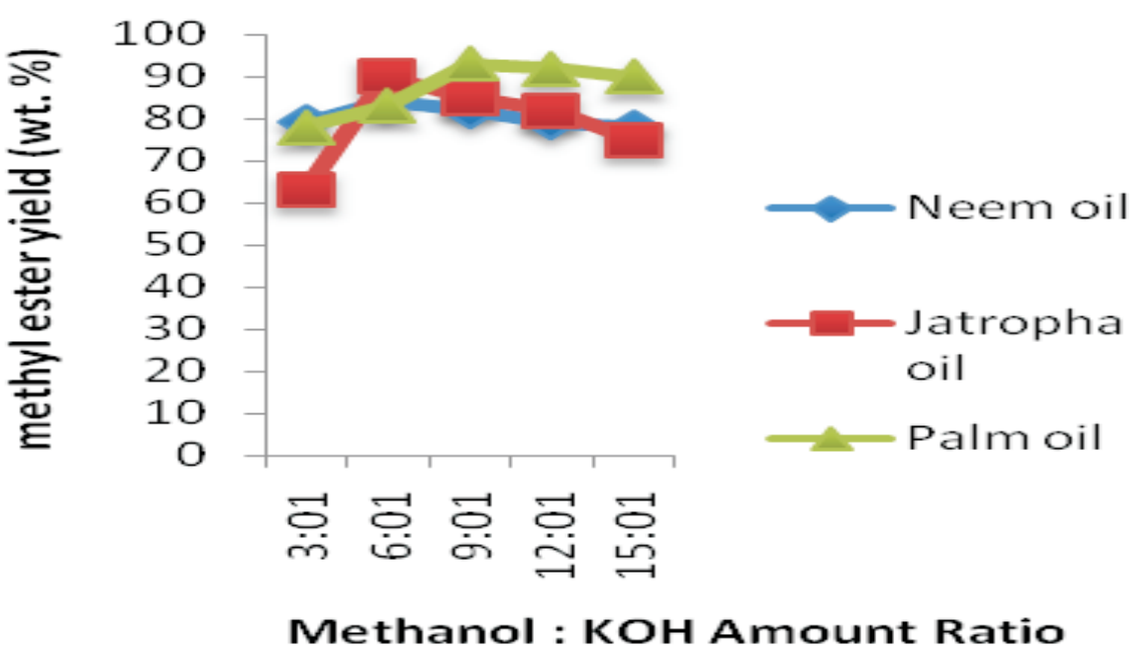

Fig. 4 Effect of methanol concentration on methyl esters' yield

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AN OVERVIEW OF NUCLEAR ENERGY ACTIVITIES IN NIGERIA

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ABSTRACT

This paper highlights an overview of nuclear energy activities in Nigeria from independence in 1960 to date. The introduction, energy in all its ramification as part of the landscape of national development was presented. A detailed discussion of historical developments of nuclear energy activities with emphasis on the quest by the country to join the race for peaceful uses of nuclear science and technology is presented. Furthermore, the current status of current nuclear energy acitivities beginning with the non-power applications and the current zest to add nuclear power to the energy mix of Nigeria are enumerated. In conclusion, solutions are proferred to problems slowing down the advancement of nuclear technology in the country.

1.0 INTRODUCTION

Since the dawn of the industrial age, the ability to harness and use different forms of energy has transformed living conditions for billions of people, allowing them to enjoy a level of comfort and mobility unprecedented in human history and freeing them to perform even more productive tasks. For most of the last 200 years, steady growth in energy consumption has been closely tied to rising levels of prosperity and economic opportunity in much of the world.

Energy has a major impact on every aspect of our socio-economic life. It plays a vital role in the economic, social and political development of our nation. Inadequate supply of energy restricts socio-economic activities, limits economic growth and adversely affects the quality of life.

Improvements in standards of living are manifested in increased food production, increased industrial output, the provision of efficient transportation, adequate shelter, healthcare and other human services. These will require increased energy consumption. Thus, our future energy requirements will continue to grow with increase in living standards, industrialization and a host of other socio-economic factors.

In elementary physics, energy is defined as the capacity to do work. This means that inadequate supply or lack of energy will invariably translate to diminshed or lack of capacity to do work. In the physical sense, energy appears as mechanical, thermal, chemical, electrical, nuclear among others. The quest by man to utilize the enormous energy in the nucleus of atom began with the discovery of the neutron in 1932 by Chadwick as a very powerful projectile for exploring the nucleus. Nuclear fission was later discovered through the analysis of the end products of neutron-induced reactions. This discovery opened up the prospect of obtaining limitless energy from the nucleus of uranium and thus the quest for the technology to exploit this new source of energy began in earnest.

In the early days, soon after the first critical assembly was operated by Enrico Fermi and others in 1942 at the University of Chicago, nuclear energy activities was dominated by the quest for military applications because of the fact that nuclear fission chain reactions had military significance. Today, the peaceful uses of nuclear energy are the main goals of all. This stems from the “Atoms for Peace” speech by the US President, Mr. Dwight Eisenhower while addressing the 470th plenary meeting of UN general assembly in December, 1953, after the World War II. To attempt a discussion of the military option is risky because of the emotional nature of subject and the impossibility of doing justice to the complex problems involved. Nevertheless, a complete neglect of the military issue in this paper does not imply that nuclear technology is entirely

benign. This overview of nuclear activities in Nigeria will include the historical developments of the nuclear science and technology in the country and the status of progress made by stakeholder organizations with regards to the introduction of nuclear option in Nigeria's energy mix.

2.0 HISTORICAL DEVELOPMENTS

Nigeria took the first step in 1976 towards a coordinated and orderly application of nuclear energy in the country, when the Nigeria Atomic Energy Commission (NAEC) was created by Act 46 as a specialized agency to promote and streamline its implementation. However, NAEC was not activated and inaugurated until the year 2006. Also in 1976, two nuclear research centres, namely, Centre for Energy Research and Training (CERT), and Centre for Energy Research and Development (CERD) were established in Zaria and Ile-Ife, respectively. The two university-based research centres were later brought under the supervision of the Energy Commission of Nigeria (ECN). These two centres alongside the Nuclear Technology Centre (NTC) at Sheda Science and Technology Complex (SHESTCO), Abuja, were eventually transferred to NAEC in 2007. SHESTCO was established in 1991, with the mandate to, among others, conduct research for the development of nuclear energy. These research centres maintain various nuclear research and development facilities including neutron generators, isotopic neutron sources, X-ray Fluorescence facilities, a nuclear research reactor, a gamma irradiation facility and a linear accelerator. These facilities have impacted positively on the socio-economic development of the nation and contributed significantly in creating and promoting awareness in the peaceful uses of nuclear energy in the country. In order to ensure proper regulation and control in the nuclear technology sub-sector, the Nigerian Nuclear Regulatory Authority (NNRA) was established by Act 19 of 1995 and inaugurated in 2001. To address the environmental challenges (including those that may come with nuclear technology and other human activities) the National Environmental Standards and Regulations Enforcement Agency (NESREA) formerly known as Federal Environmental Protection Agency (FEPA), was established as a parastatal of the Federal Ministry of Environment.

Prior to the 1976 date, Nigeria became aware of the threats of nuclear radiation and so had monitored the French nuclear tests in Sahara desert in the early 1960's. To do this, the Federal Government had to set up the Federal Radiation Panel in 1961 and the Federal Radiation Protection Service (FRPS) at the University of Ibadan (UI) in 1964. This same year, Nigeria also became a member of the International Atomic Energy Agency (IAEA), thus opening up an avenue for obtaining technical assistance in the nuclear field and also signaling the country's interest in joining the race for the peaceful uses of nuclear energy. Before these events, Nigeria has had pockets of individuals and institutions applying one form of nuclear technology or the other, in their research and professional activities. Radium needles and X-ray machines have been used in medical application by some hospitals across the country. Similarly, caesium (Cs) sources were being used for cancer treatment as far back as 1960 at the University College Hospital (UCH), Ibadan. A close evaluation of our experience indicates that whatever progress had been made could have been many more times greater than it is if there had been able leadership, proper planning and coordination in our quest for peaceful applications of nuclear technology.

3.0 CURRENT STATUS OF NUCLEAR ENERGY ACTIVITIES

As contained in the National Energy Policy (NEP) “the nation shall pursue the exploitation of nuclear energy for peaceful purposes”. Therefore, in addition to the generation of electricity, nuclear energy finds many other peaceful applications. In fact, it had been used in the country for decades for various other applications in health care delivery system, petroleum industry, agriculture, food preservation, animal husbandry, water resources management, pest control, industry, materials analysis, and mineral exploration.
These non power applications of nuclear

energy currently dominate the present activities of the nuclear industry coordinated by NAEC. The three nuclear energy research Centres operate under the supervision of NAEC and are involved in manpower training and capacity building. Two of the Centres are university-based, one at the Obafemi Awolowo University, Ile-Ife (i.e. the Centre for Energy Research and Development, CERD) and the other at the Ahmadu Bello University, Zaria (i.e. Centre for Energy Research and Training, (CERT), respectively. The third Centre was established in 1991 at Sheda Science and Technology Complex (SHESTCO) in Abuja. The first nuclear research reactor in the country is sited at CERT, Zaria and has been in operation since 2004. It is a 30 kW nuclear research reactor, the Miniature Neutron Source Reactor (MNSR) and codenamed **N**igeria **R**esearch **R**eactor**-1** (NIRR-1). In addition to this, a 1.7 MV TANDEM, charged particle accelerator is in operation at CERD, Ile-Ife and was commissioned in 2008. These facilities are complemented by the Gamma Irradiation Facility currently in operation at SHESTCO, Abuja. Recently, NAEC has created two additional nuclear energy research centres at the Universities of PortHarcourt and Maiduguri, respectively. In the area of nuclear regulation, NNRA became operational in 2001. It was established to develop and enforce all the needed guidelines on radiation protection, nuclear safety, nuclear security and safeguards.

With regards to the power applications of nuclear energy activities in Nigeria, the Federal Republic of Nigeria has shown the political will to add nuclear to energy mix of the country through the activation of NAEC in 2006. This came as result of the visit of immediate past Director-General of the IAEA, Dr Mohammed El-Baradei to CERT, Zaria in 2005, following the successful commissioning of NIRR-1. The former President, Chief Olusegun Obasanjo requested the technical assistance of the IAEA for the country in her quest to add nuclear to the energy base of Nigeria. NAEC is the National Focal Point for the promotion and development of atomic energy and charged with the primary responsibility for nuclear power plant (NPP) programme implementation. In pursuant of the Federal Government's intention of adding nuclear to its energy mix, a National Road map, tagged the "Technical Framework for the Deployment of Nuclear Power Plants for Electricity Generation in Nigeria" and its "Strategic Implementation Plan" have been developed by NAEC and approved by Government for implementation. The technical framework is a three-phase plan, which is aimed at positioning Nigeria to generate electricity from nuclear reactors by the year 2020 with considerable national participation. The three phases are: (a) Manpower and infrastructure development; (b) Design certification, regulatory and licensing approvals; and (c) Construction and start-up. The programme is in the first phase with emphasis on the training of requisite human capital and the involvement of existing expertise in the implementation of the second phase. Some of the challenges confronting the programme are: Long-term national (political) commitment, and sustainability of programme over a gestation period of at least 20 years; Training of requisite restrictive manpower and long time required to build critical mass; Development of appropriate infrastructure needed to support the implementation of the programme; Development of requisite industrial capacity to gradually domesticate nuclear technology; Development of the requisite financing plan, catalyzed by Government, with the private sector as a partner; and Motivation and sustenance of the interest of the Nigerian Public to develop a positive attitude in the country, while maintaining the confidence of our partners.

4.0 CONCLUSION

For the past 10 to 15 years, the energy sectors in most developing countries have been in turmoil. Many of these countries including Nigeria have been attempting to restructure their energy sectors but are finding it difficult to implement reforms for a host of reasons, including the multiplicity of actions involved, which include changing perceptions of the relative roles of the market and Governments, and the baggage of accumulated policies of the past few decades—many of which may have made sense, when they were proposed but now impose unsustainable burdens. The provisions of the NEP with respect to nuclear energy sources are adequate for achieving sustainable

development, if faithfully implemented. The needed infrastructures have been put in place but require strengthening through proper supervision and funding. The only way to ensure the attainment of Vision 20:2020 is to emulate the developed and fast growing economies of the world by mainstreaming energy planning into overall national plans. The deployment of nuclear power for electricity generation globally has achieved good degree of success and it is part of the energy mix of the leading economies in the world. Even disregarding the current "nuclear renaissance" in the world, the acute power shortage in Nigeria, nuclear is no longer an option but a necessity that must be added to the nation's energy mix in the medium and long term timelines. **Over the past 40 years, nuclear power has presented less risk to the public and workers than the other major sources of electricity generation (coal, gas and hydro). Regarding environmental impact, nuclear power is a clean source of energy with very low GHG emissions.** With the activation of NAEC in 2006, the Federal Government of Nigeria has launched a nuclear electricity programme, and has approved the roadmap for its implementation. Government should restructure the focal agencies for the prosecution of the national nuclear power programme vis-à-vis other stakeholder institutions and international development partners such as the IAEA. Nigeria has frittered away several years through bad leadership and lack of coordination when compared with smaller and poorer neighbouring countries whose nuclear activities are rated higher by the IAEA. It is not too late to put our acts together so as to enable our trained nuclear scientists contribute to the programme, for there is room for all to do so. This will fast track the manpower development programme and create the requisite enabling environment for the successful implementation of the nuclear energy programme of Nigeria in partnership with the private sector.

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