



**FEDERAL UNIVERSITY OF TECHNOLOGY  
MINNA**

**TRANSFORMING THE NIGERIAN  
ECONOMY TO BE LESS DEPENDENT  
ON FOSSIL FUEL: ADVOCACY FOR  
SUSTAINABLE ALTERNATIVE**

*By*

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*Professor of Chemical Engineering*

**INAUGURAL LECTURE SERIES 93**

**14<sup>TH</sup> OCTOBER, 2021**

ISSN 2550 - 7087



2550 7087

**Luradd Ventures Ltd**  
📞 08034505205, 08082545865



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*Published by:*

**University Seminar and Colloquium Committee,**

Federal University of Technology, Minna.

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## OVERVIEW

Nigeria is a key regional player in West Africa and accounts for about half of West Africa's population with approximately 202 million people and has one of the largest populations of youth in the world (Joseph-Raji *et al.* 2021). With an abundance of natural resources, it has the largest natural gas reserves on the continent (Joseph-Raji *et al.* 2021). However, since the discovery of oil, Nigeria's economy has been solely dependent on revenue from oil whereby agriculture which is *ab inito* the sustaining sector since independence was neglected.

## ECONOMIC OVERVIEW

It has been well established that Nigeria is highly vulnerable to the global economic disruption caused by COVID-19, particularly due to the pronounced decline in oil prices and thwarts in the threat to global capital markets. Nationally, 40 percent of Nigerians (80.8 million people) live below the poverty line, while another 25 percent (50.5 million) are vulnerable (Joseph-Raji *et al.* 2021). With COVID-19, many of these 50.5 million vulnerable people could fall into poverty. The magnitude of the health impact of the pandemic depends on the duration and the domestic spread of the outbreak, while the economic impact hinges on oil prices. The revenue from oil accounts for over 80 percent of exports, a third of banking sector credit, and half of government revenues from a recent survey (Joseph-Raji *et al.* 2021). The growth in non-oil industries and services is affected by oil prices also, with additional pressures arising from foreign portfolio investors' reassessment of risks and internal assets management.

The macroeconomic situation is more challenging now than in 2015-2016 when oil prices fell sharply and Nigeria experienced its first recession in 25 years. Currently, the situation in Nigeria

has less shield and few policy instruments to cushion adverse effects. In addition, the Excess Crude Account is depleted, external reserves are highly reliant on short-term flows, and policy uncertainty affects investor confidence (Joseph-Raji *et al.* 2021). Before the 2016 recession, Nigeria's economy was growing fast at 6.3%. By contrast, before COVID-19 struck, the economy was growing at 2.2%. Inflation was in single digits in 2014, compared to about 12% in 2019. The general government fiscal deficit was 4.4% of GDP in 2019, compared to 1.8% in 2014 (Joseph-Raji *et al.* 2021). Unemployment and underemployment are expected to soar, affecting poor households and increasing the share of the population vulnerable to falling into poverty. Only agriculture is expected to positively contribute to growth in 2020. However, the economy is expected to grow by 1.8% in 2021, though there is high uncertainty about the forecast (Joseph-Raji *et al.* 2021).

## **DEVELOPMENT CHALLENGES**

While Nigeria has made some progress in socio-economic terms in recent years, its human capital development remains weak due to under-investment. It ranked 152 of 157 countries in the World Bank's 2018 Human Capital Index (Joseph-Raji *et al.* 2021). The country continues to face massive developmental challenges, including the need to reduce the dependency on oil and diversify the economy, address insufficient infrastructure, build strong and effective institutions, as well as address governance issues and public financial management systems. These pre-existing structural challenges have left the Nigerian economy especially vulnerable to the COVID-19 outbreak and its consequences.

Inequality, in terms of income and opportunities, remains high and has adversely affected poverty reduction. The lack of job

opportunities is at the peak of poverty levels, regional inequality, and social and political unrest. Without the COVID-19 shock (the counterfactual scenario), about 2 million Nigerians were expected to fall into poverty in 2020 as population growth outpaces economic growth (Joseph-Raji *et al.* 2021). With COVID-19, the recession is likely to push an additional 5 million Nigerians into poverty in 2020, bringing the total newly poor to 7 million (Joseph-Raji *et al.* 2021). With concerted efforts, the narrative can be changed by fully engaging wholly in renewable and sustainable processes that will curb dependence on fossil and channel the available resources to biofuels obtained from biomass to power the Nigerian economy.

## **THE MAINSTAY OF THE NIGERIA ECONOMY BEFORE CRUDE OIL**

Nigeria has been sustained with her agrarian economy before the discovery of crude oil in commercial quantity in 1956 in a village called Oloibiri in present day Bayelsa State. Agriculture used to be the mainstay of the economy and for her foreign exchange earnings through the exportation of cash crops such as rubber from Delta State in South-south Region; groundnut, hide, and skin from the northern area; cocoa and coffee from the western territory; and palm oil and kernels from the eastern section of the country (Paul, 2015). The discovery of crude oil in Nigeria came with economic growth and infrastructural development, although some of the projects were abandoned and the funds for those projects embezzled, the major detriment was the destruction of the environment, that is the aquatic environment and affected fishing activities which is the primary source of livelihood of the population. Therefore, urgent need for diversification of the economy from the present crude oil dependent structure is required. Other sectors of the economy, such as agriculture and manufacturing, should be given an



opportunity to thrive and support revenue from petroleum, especially now that the price of oil is nose-diving.

## **THE FOSSIL FUEL AND THE ENVIRONMENT**

Fossil fuels is simply referred to as decomposing plants and other organisms, buried beneath layers of sediment and rock which have taken million years to become carbon-rich deposits (Christina, 2019). They are non-renewable fuels, which include coal, oil, and natural gas which supply about 80 percent of the world's energy. When they are put into use, it provides electricity, heat, and transportation (Christina, 2019). Unfortunately, when fossil fuels are processed, they release carbon dioxide and other greenhouse gases, which in turn trap heat in the atmosphere, making them the primary contributors to global warming and climate change as revealed in Figure 1.

## **REDUCING EMISSIONS FROM FOSSIL FUELS**

Governments around the world are now engaged in efforts to ramp down greenhouse gas emissions from fossil fuels to prevent the worst effects of climate change. At the international level, countries have committed to emissions reduction targets as part of the 2015 Paris Agreement, while other entities including cities, states, and businesses have made their own commitments (Christina, 2019). These efforts generally focus on replacing fossil fuels with renewable energy sources, increasing energy efficiency, and electrifying sectors such as transportation and buildings.

However, many sources of carbon emissions, such as existing power plants that run on natural gas and coal as depicted in Figure 2 are already locked in. Considering the world's continuing dependence on fossil fuels, all efforts are aimed at replacing them by sucking carbon from the air with technologies

such as carbon capture (sequestration), in which emissions are diverted to underground storage or recycled before they reach the atmosphere. Figure 3 is a representation of a handful of commercial-scale projects around the world already capture carbon dioxide from the towers or vents of fossil fuel-fired plants, and while its high costs have prevented wider adoption, advocates hope advances in the technology will eventually make it more affordable.

### **Fossil fuel causes deadly air pollution**

In addition to releasing planet-warming greenhouse gas emissions, burning fossil fuels also generate localized air pollutants such as soot (fine particulate matter) and smog (ozone) that increase the risk of death from stroke, heart disease, lung cancer, and respiratory illness among those exposed (Donaghy, 2019). Air pollution from fossil fuel production and consumption disproportionately affects communities of color and low-income perpetuating environmental injustices. The World Health Organization estimates that ambient exposure to fine particulate matter leads to around 4 million deaths worldwide every year excluding those exposed to indoor air pollution (Donaghy, 2019).

### **Fossil fuel causes water pollution**

Time and time again, oil spills and explosions have wreaked havoc on water, land, and homes and communities. Major incidents such as British Petroleum's Deepwater Horizon catastrophe, which spilled 4.9 million barrels of oil into the Gulf of Mexico, or Enbridge's massive tar sands spill into the Kalamazoo River (Donaghy, 2019) to mention but a few show that there is no guaranteed way to extract, process, or transport fossil fuels. Wherever there is drilling, inevitable spilling occur

and more often, these disasters have put marginalized communities under threat. In the last several years, an indigenous-led movement of water protectors and allies has risen up to oppose risky, climate-wrecking pipeline projects across the country and its expansion (Donaghy, 2019). Additionally, the oil and gas boom has put groundwater at risk from wastewater contamination and has consumed scarce water resources. Coal is not left out in this menace. It contaminates water resources during the mining process through unregulated coal ash activities (Donaghy, 2019).

## **THE SEARCH FOR RENEWABLE AND SUSTAINABLE ALTERNATIVES**

A renewable resource is a natural resource that can be replaced or replenished by natural processes at a higher or equal rate to its rate of consumption. The renewable resources are part of the natural environment and constitute what is known as the ecosystem. The factors of environment and the subject of thriving business have caused worldwide concerns and have resulted in the search of alternative source of energy which would be renewable and sustainable. One method of reducing the rise in energy-related emission of greenhouse gases (GHG) is to substitute the conventional fossil fuels with biofuels, which include any energy derived from biomass. The advantages of using biofuel can be summarized in three aspects: environment, energy security, and economy (Isah *et al.* 2020).

Table 1 shows the comparison between fossil fuel and fuel from biomass.

**Table 1: Comparison between fossil fuel and fuel from biomass**

Fuel from fossil	Fuel from biomass
<p>*Coal, crude oil, and natural gas are all considered fossil fuels because they were formed from the fossilized buried remains of plants and animals that lived millions of years ago. Thus, because of their origins, fossil fuels have a high carbon content.</p>	<p>*The term biofuel is referred to liquid, gas and solid fuels predominantly produced from biomass from a renewable source. Other sources of renewable energy are Solar, Wind, Hydro, and Geothermal.</p>
<p>*Fossil fuels can generate a large amount of electricity at a single location. They can be found very easily. They are cost-effective. Transportation of oil and gas can be done easily through pipelines. They have become safer over time. Despite being a finite resource, it is available in plenty</p>	<p>*Biofuels benefits include: energy security reasons, environmental concerns, foreign exchange savings, and socio-economic issues related to the rural sector.</p>
<p>*Fossil fuels can take thousands or even millions of years to naturally replenish. They are: Natural gas, Coal and Oil</p>	<p>*Biofuels include bioethanol, bio-methanol, vegetable oils, biodiesel, biogas,  bio-synthetic gas (bio-syngas), bio -oil, bio-char, Fischer-Tropsch liquids, and bio-hydrogen. Most traditional biofuels, such as ethanol from corn, wheat, or sugar beets, and biodiesel from oil seeds, are  produced from classic agricultural food crops that require high-quality agricultural land for growth.</p>
	<p>*Bio-methanol can be produced from biomass using bio-syngas obtained from</p>

Source: Adapted from Isah *et al.* 2020; Yusuff *et al.* 2019 and Olutoye *et al.* 2014

## **THE PICTORIAL REPRESENTATION OF NATURAL AND DEPLETED ENVIRONMENT: CONSEQUENCE OF GLOBAL WARMING**

The environment represents all the elements that affect a system or its inputs and outputs. It represents the circumstances, objects, or conditions by which one is surrounded. It refers to a complex of physical, chemical, and biotic factors (as climate, soil, and living things) that act upon an organism or an ecological community and ultimately determine its form and survival (Woma et al. 2019). It would appear that the public's awareness of the environment was only noticeable after a number of disastrous environment accidents. For example, the oil spills from the tankers Amoco Cadiz, off the coast of Brittany in 1978, Exxon Valdez, in Alaskan water in 1989, toxic gas release from a pesticide plant in Bophal, India in 1984, and the nuclear power station accident at Chernobyl in 1986. These events achieved great publicity and increased the awareness of environmental issues. Floods in Niger State have caused damage to hundreds of homes and farms, mostly across the Local Government Areas (LGA) of Mashegu and Kontagora. At least 200 homes and wide areas of farmland were destroyed in the floods (Richards, 2021). It was reported that at least 4 people died and many more people feared missing (Richards, 2021). Similarly, hundreds of families in the northern and northeastern states of Jigawa, Bauchi and Adamawa in Nigeria experienced flood in August, 2021 after days of heavy downpour in the region. Over 380 households have been affected or displaced across the 3 states since early August, 2021 (Richards, 2021).

The pictorial representations shown in Figures 1, 2 and 3 depict few of the other environment disaster from various parts of the world.

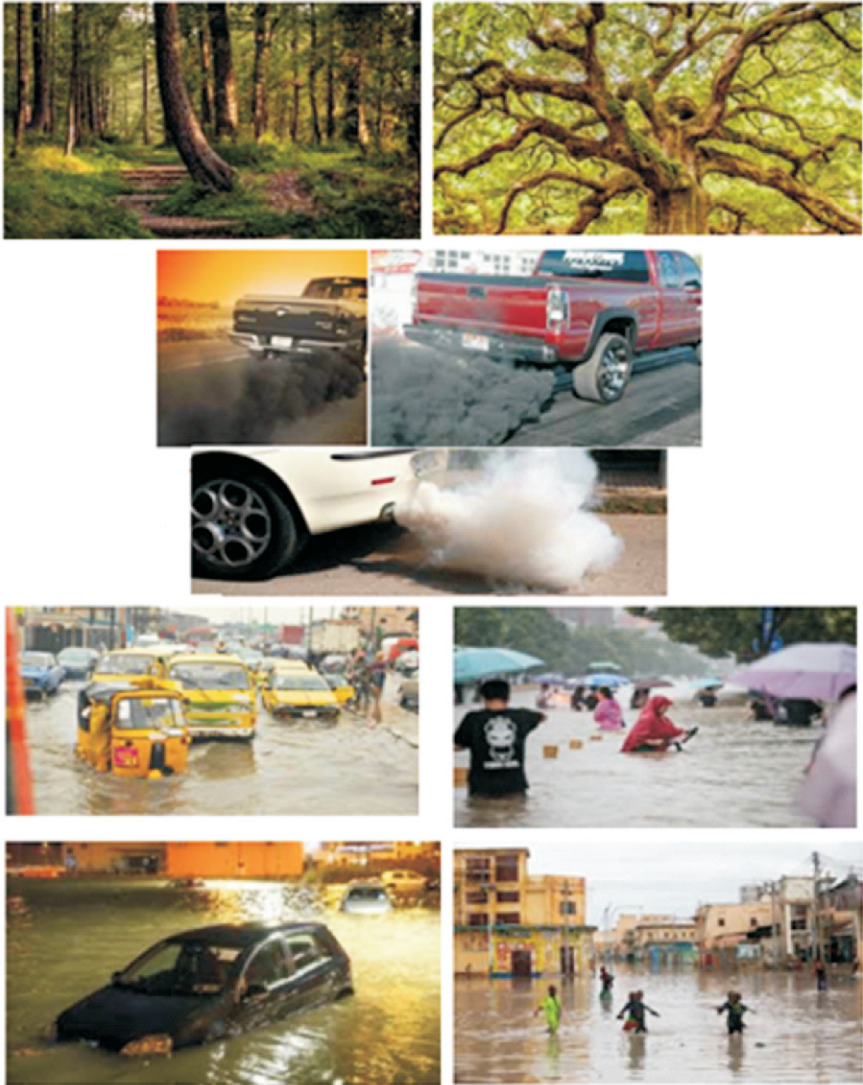


Figure 1: Photographs depicting natural and depleted environments

Sources: *ProStockMedia (2018); Odita Sunday (2019) and Environmental Pollution*



Figure 2: Smokestack of coal fired from a plant  
*Source: Environmental Pollution, 2019*



Figure 3: Massive environment pollution through gas flaring  
from oil  
*Source: Environmental Pollution, 2019*



## **THE LINK BETWEEN THE ENVIRONMENT AND THE ECONOMY: MY PERSPECTIVE**

Environment provides land, water, air, energy resources, coal, oil, forests, minerals and metals and so many other natural resources which are essential for the economic development of the country. The environment provides services which are directly used by both aquatic and terrestrial animals which is made of the air we breathe and the water we drink for life. All economic growth requires the transformation of the natural environment which can affect immediate quality of the environment in one of three ways viz: in the cases of air pollution, water pollution, and deforestation cum encroachment. There is little or no incentive for any individual to invest in maintaining the quality of the environment. Pollution has its economic consequences to bring down productivity, cause increase in health costs, and reduce crop yields which affects the biodiversity and ecosystems. However, clean air represents a benign environment where businesses thrive and the economy grows in leaps and bounds.

Thus, companies saddled with the design and installation, maintenance, and operate pollution-reducing processes create thousands of high-paying green jobs in engineering, manufacturing, construction, materials, operation, and maintenance. It thus imply that cleaner air and a growing economy can go hand in hand.

The role of environmental policy is to manage the provision and also the use of environmental resources in a way that supports improvements in prosperity and wellbeing for current and future generations. Therefore, there are a number of reasons why government intervention is needed to achieve a clean environmental policy. In particular, market failures in the provision and use of environmental resources mean that natural assets would be over-used in the absence of government



intervention. Illegal mining operations are testament of this fact in our immediate environment.

Environmental policy, including infrastructure and other investments, can reduce how vulnerable the economy and businesses are to adverse environmental events – both by reducing environmental risk and by increasing the economy's resilience to these risks. For example, not just investments that facilitate emissions reductions to avoid dangerous climate change, but also those investments that help the economy adapt to climate impacts already locked-in by past and current emissions.

### **GLOBAL DEMAND FOR ALTERNATIVE ENERGY SOURCE**

Recent research showed that there is increased global awareness in the utilization of alternative (renewable) sources of energy (Adepoju *et al.* 2020). The reason for this has been the increase in the cost of energy produced from fossil fuels coupled with the fact that it is a finite resource; it grossly affects the environment. There is the need to evolve new measures, in terms of appropriate technology and resources, to promote a shift from fossil fuels. Due to astronomical growth in world population and rapid industrialization which are direct consequence of increased technological breakthrough in almost all spheres of life, it is believed that the global energy demand will definitely increase. Thus, the search for the renewable resources such as solar, wind, water, biomass and other clean energy sources will surge in demand and eventually account for the vast majority of overall energy usage in the near future.

The increasing decline in crude oil reserves has made alternative energy sources inevitable and of great importance. This is in the light of increasing campaign for cleaner burning fuels in order to safeguard the environment and protect man from inhalation of

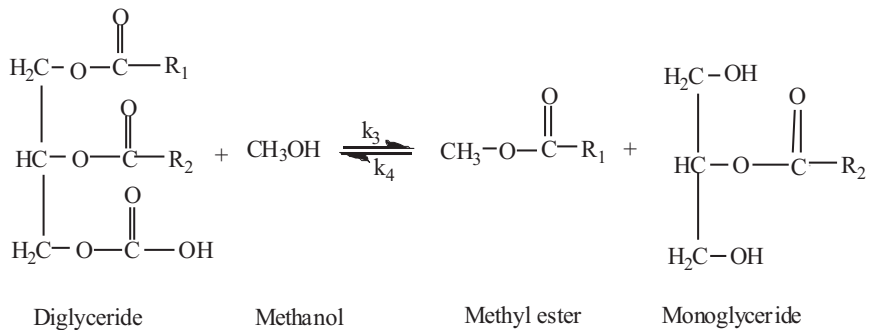
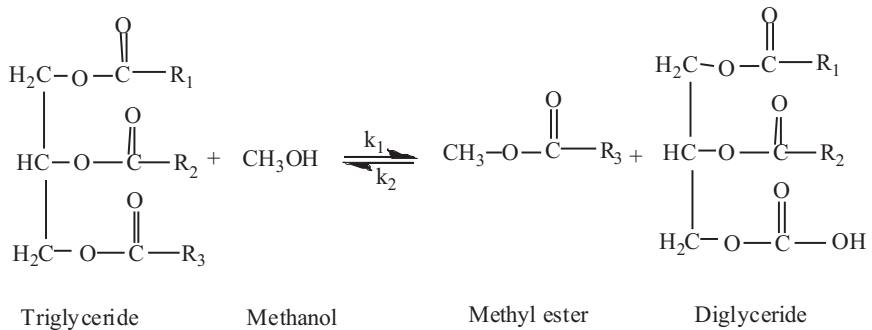
toxic substances. The exhaust from petroleum diesel is known to be carcinogenic in nature, since they contain polycyclic aromatic hydrocarbons and nitrated polycyclic aromatic hydrocarbons (PAHs and NPAHs), carbon monoxide, sulphates, and particulate matter (Olutoye *et al.* 2014).

A renewable resource is a natural resource that can be replaced or replenished by natural processes at a higher or equal rate to its rate of consumption. The renewable resources are part of the natural environment and constitute what is known as the ecosystem. The world-wide energy demand is quite huge and more than 80% of this energy is currently supplied by fossil fuels, coal, oil or gas (Adepoju *et al.* 2020). Fossil fuels are non-renewable finite resource. The shrinking supply of this resource globally will not make it possible for continued dependence on it for a longer time because they will be used up within the next decades. Besides, the drastic increase in the emission of carbon dioxide into the environment, when fossil fuels (oil, gas, petrol, kerosene, etc.) are combusted in engines and automobiles as shown in Figure 1 has been identified as the major cause for the change of temperature in the atmosphere generally referred to as global warming. Thus, the search for an alternative that could replace fossil fuels in the short or medium term has become imperative.

## **TRANSESTERIFICATION PROCESS**

Transesterification is a chemical reaction used for the conversion of triglycerides (fats) contained in oils, (Feedstocks) into usable fuel (biodiesel). Biodiesel produced by the process of transesterification has a much lower viscosity, making it capable of replacing petroleum diesel in diesel engines. Transesterification is a reversible reaction and the tendency to attain equilibrium depends on the operating variables. The presence of excess alcohol in the reaction mixture and a catalyst (acid or base) could accelerate and control the equilibrium to achieve a high yield of the ester or product.

Usually, an excess of the alcohol is used to increase the yield of the methyl esters and to allow its physical separation from the glycerol formed. Investigations have shown that transesterification reaction depends on some number of parameters which enhances the product conversion. In other words, the extent of the reaction will depend on the type of catalyst (acid or base), alcohol to vegetable oil molar ratio, temperature, purity of the reactants and free fatty acid content (Olutoye et al. 2016a; Yusuff et al. 2018c). Methyl esters of fatty acids are produced by alcoholysis (transesterification) of triglyceride with methanol in the presence of an acid or base catalyst as illustrated by the reaction in Figure 4. Complete conversion of the triglyceride involves three consecutive reactions with monoglyceride and diglyceride intermediates.



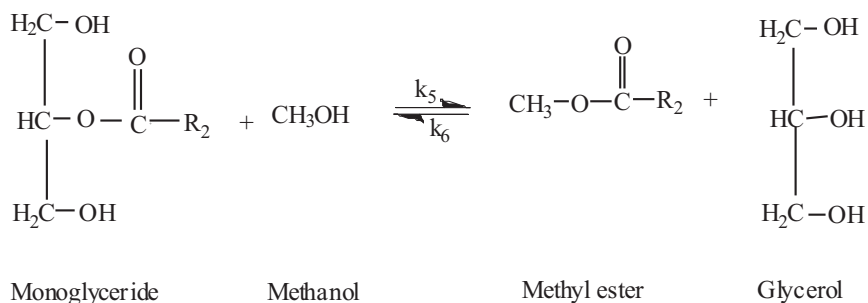


Figure 4: 3-step reversible reactions of triglyceride where  $R_1$ ,  $R_2$ , and  $R_3$  are long chains of carbons and hydrogen atoms (fatty acid chains) (Balakrishnan, Olutoye and Hameed, 2013)

### HOMOGENEOUS TRANSESTERIFICATION PROCESS

Transesterification reaction as mentioned earlier requires a suitable catalyst for the conversion of the triglyceride (vegetable oil) to fatty acid methyl esters (FAME). The use of homogeneous catalyst where the catalyst and the reactants are in same liquid-liquid phase for the production of FAME is among the various technologies that emerged over decades and has gained increased acceptability because it is widely known to give higher conversion to methyl esters but not without some shortcomings. These include free fatty acid in feedstock which formed saponified products, water in raw materials interferes with the reaction, glycerol separation is usually difficult, and purification of methyl esters require repeated washing with water which when discharged make the environment unsafe (Balakrishnan, Olutoye and Hameed, 2013).

The current industrial production of FAME basically employed homogeneous alkali-catalyzed transesterification of vegetable oils with methanol. This is because of the fast kinetics of the reaction that is involved by the use of homogeneous catalysts. For example, NaOH, KOH,  $(\text{K}^+ \text{ or } \text{Na}^+) \text{OCH}_3$  are most often used. It is also a known fact that, though the reaction involving these

catalysts is fast, saponification (a side reaction) in the system considerably reduced FAME yield, the product requires repeated washing with water to remove glycerol. Also, in homogeneously catalyzed process, fats and alcohols are not totally miscible and vigorous mixing is required to increase the area of contact between the two immiscible phases and a kind of emulsion is produced which reduce the yield of fatty acid methyl ester (FAME) product. Moreover, the catalyst is not reusable and soap is formed. The glycerin by-product that is contaminated with the alkaline catalyst also has little market worth and disposal problem becomes aggravated. The conventional production of FAME relies on soluble sodium and potassium hydroxide catalysts; however, removal of these catalysts is technically difficult and brings extra cost to the final product.

In the same vain, acid transesterification allows formation of ester without by-products. The main disadvantage of this process lies in the fact that the acid homogeneous transesterification involve the application of corrosive catalyst such as  $H_2SO_4$ ,  $H_3PO_4$ ,  $HNO_3$ , and  $HCl$ . In addition, the reaction rate is generally slow. However, at high operating temperatures and pressures, the rate and yield could be improved but this will add extra cost to the final product. All the limitations mentioned here make the homogeneous process cumbersome and uneconomical. Thus, this makes investigation into suitable acid or basic heterogeneous catalysts, or heterogeneous catalyst with dual sites, that is, with both acid and basic functions for the process imperative.

## **HETEROGENEOUS TRANSESTERIFICATION PROCESS**

Heterogeneous catalysts (where the catalyst and the reactants are in different phase, that is, solid-liquid phase) could be employed in the transesterification process to improve the yield of fatty acid methyl ester (FAME) at lower cost. There is

significant demand in energy consumption worldwide and to meet this challenge, a new and efficient catalyst for fatty acid methyl ester (FAME) production which possesses criteria such as good activity and selectivity, low cost, ease of separation and environmental friendliness is required.

Heterogeneous catalysts have been used in various processes, for example, alkali metal (Li, Na, and K) promoted alkali earth oxides (CaO, BaO, and MgO), as well as  $K_2CO_3$  supported on  $Al_2O_3$  ( $K_2CO_3/Al_2O_3$ ), has been used for transesterification of different vegetable oils. However, there was potassium leaching into the product during the reaction and require catalyst modification to obtain higher yield and eliminate or reduce leaching (Adepoju *et al.* 2020).

The utilization of heterogeneous catalyst in transesterification reactions appeared very promising because they could become cheaper materials in substitution of noble metal supported on alumina, silica and other inert solids in addition to the ease of separation of the products. Various heterogeneous catalysts have been used in transesterification of different oils. For example, nano crystalline metal oxides, combusted oyster shell, Mg-Al hydrotalcites, Silica-supported solid acid catalysts, basic solid Mg/Zr catalysts have been applied on edible and non-edible oils.

## **GENERAL USES AND PROPERTIES OF BIODIESEL**

Biodiesel, a renewable alternative fuel to diesel from fossil, will be extensively used in the near future due to its many benefits. Biodiesel can be used in the blended form-B20 (20 % of biodiesel with 80 % petroleum diesel) or in the pure form (B100) depending on the feedstock from which it is derived or the quality of the finished fuel. In the blended form, it can be used in

unmodified diesel engines. The flow and combustion properties of biodiesel are similar to petroleum-based diesel and thus, it can act as a substitute for diesel fuel and justify its suitability as blends with fuels. Higher blends, or even pure biodiesel (100% biodiesel or B100), can be used in some other engines, for example as in aviation with little or no modification.

In terms of efficiency, biodiesel has positive performance attributes such as increased cetane, high fuel lubricity, and high oxygen content, which may make it a preferred blending stock with future ultra-clean diesel. It is a performance enhancer in terms of operations to conventional diesel. There is an increase in engine life span because biodiesel is more lubricating than diesel fuel. The increased lubricity will enhance engine performance and reduce the frequency of engine parts replacement. Thus, it can serve as a replacement to sulphur (a lubricating agent) in blends. Sulphur dioxide is produced during combustion of sulphur containing diesel fuel which is a primary component in acid rain. Pure biodiesel carries about 90% of the energy content of the normal diesel and hence it can be expected that the engine performance can be nearly the same.

Emissions of nitrogen oxides increase with the concentration of biodiesel in the fuel and the increase is roughly 2% for B20. Some biodiesel produces more nitrogen oxides than others, and some additives have shown promise in reducing the increase production of these oxides in the fuel. In the case of biodiesel, NO<sub>x</sub> emissions are a function of combustion temperature. The higher the heat of combustion the greater is the NO<sub>x</sub> emissions. Also, because biodiesel contains more oxygen than diesel fuel, the heat of combustion is slightly higher (Adepoju *et al.* 2020).

Biodiesel is biodegradable and this feature makes it ideal for use in fragile areas such as natured reserves, water reserve, forestry estates, bodies of water, inland waterways and coastal waters,

and in urban agglomerations. Production and use of biodiesel are environmentally friendly because of their proximity to the feedstock. Besides, biodiesel is safe to transport because it has a high ignition temperature (higher flash point than normal diesel). No danger of explosion is associated with this fuel.

The production of biodiesel is relevant for most industrialized nations where energy demand is quite huge. For example, the demand for transport fuels is going to increase to a great extent as the world population increases. Biodiesel will enable the development and the support for sustainable society projects that are of strategic importance. Similarly, the economic and social aspects of development which aims at greater energy self-sufficiency, a more secured environment (by decreasing the air pollution from transportation and mitigating greenhouse gas emissions) and socio-economic benefits of the bottom billion will be promoted.

The increased utilization of biodiesel will help to develop the economy and provides significant results in microeconomic growth for both the urban and rural sectors. In addition, it is hoped that the research will generate employment opportunities, thereby providing livelihood support (economic empowerment). It is expected that more plantations of oil yielding plants such as palm tree (cash crop) and others for the production of biodiesel will help to create eco-restoration and environment sanity vis-à-vis environment security and reduce drought. The long term benefits derivable from using biodiesel are improvement in national security, environmental protection, guarantees public health cum safety, and a source of income to farmers. The Figure 5 represents the biodiesel production process.



## SOLID CATALYSIS IN BIODIESEL PRODUCTION

The production of fatty acid methyl esters (biodiesel) generally depended on the application of catalysts for transesterification of vegetable oils. The process of biodiesel production using homogeneous catalysts is accompanied with several disadvantages making the product expensive and this poses a major barrier to large scale commercialization. Among these include the formation of saponified products because of the presence of free fatty acid in feedstock, interference of water (in raw material) with the reaction, difficulties of separating glycerol and the repeated washing of methyl esters needed for its purification and quality. Apart from the alkali catalyst, other catalysts such as acids and enzymes can be used for transesterification. The acids mostly used to catalyze the transesterification process are sulphuric acid, nitric acid and hydrochloric acid.

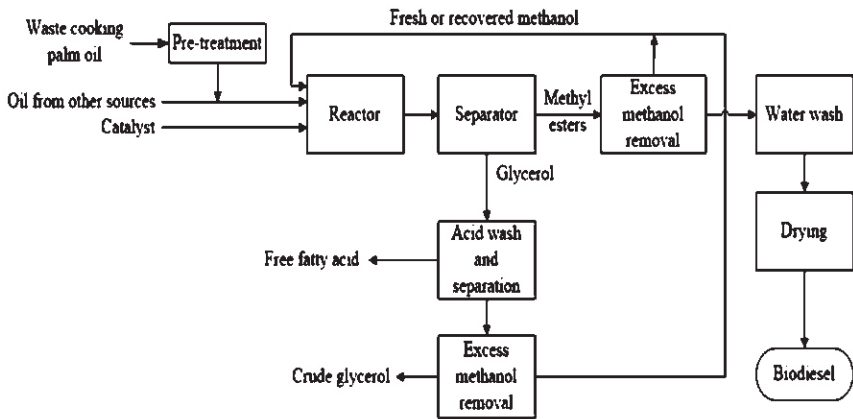


Figure 5: Typical process flow diagram for biodiesel production

Source: Isah *et al.* 2020

In recent times, attention of researchers has shifted away from making biodiesel using homogeneous catalyst because of the limitations enumerated. However, production costs are still rather high, compared to petroleum-based diesel fuel. There are two main factors that affect the cost of biodiesel: the cost of raw materials and the cost of processing. Processing costs could be reduced through simplified operations and eliminating waste streams. The partial solution to these problems could be achieved by transesterification in supercritical methanol without using any catalyst. As a matter of fact, in this case, the reaction is very fast (less than 5 min) but in my opinion, it is expensive, unsafe and cannot be commercialized.

The above mentioned points made the development of heterogeneous catalyst for the transesterification of vegetable oils to produce biodiesel imperative (Olutoye, Lee, and Hameed, 2011) and has attracted extensive research in the field. The application of heterogeneous catalysts has gained prominence because it can be separated more easily from reaction products and could address some drawbacks of other processes, they are noncorrosive and environmentally benign and they can be designed to give higher activity, selectivity and longer catalyst lifetime. Also, the reaction conditions could be less drastic than the methanol supercritical process.

Temperature is a critical parameter in transesterification process as reported by Olutoye, Lee, and Hameed (2011) and other authors Yusuff et al. (2018b); Osakwe et al. (2018). Leaching and reusability of heterogeneous catalyst are important factors which determine the quality of FAME. Hence, from information available in the literature, most researchers failed to stress on these parameters in their various studies. Thus, to achieve the sustainable and the renewable fuel goal, my contributions emphasized and developed various catalysts

effective for the production of biodiesel from various feedstock that is able to solve the leaching problem encountered during the production and obtain an environmentally benign fuel for use in automobiles, energy and industrial application for economy growth.

## **MY CONTRIBUTIONS**

Several heterogeneous catalyst were developed and are reported Synthesis of mixed oxides catalysts. Prior to the catalyst synthesis, a chemical formulation was derived for the combination of the elements in different ratios. Nitrates compounds of all the metals (K, Mg, Zn and Al) were used in the different stages of the catalyst synthesis. Similarly, operating procedure in terms of treatment methods was established. This was done to guide subsequent preparation/synthesis for all the catalyst developed.

### **Basis for catalyst preparation/treatment was established**

Different synthesis routes were followed to obtain two groups of catalyst, each group consists of five samples differing as to the calcination temperature. The role of different treatment methods was investigated using sets of experiments in which the composition and the combination ratio were varied. The theoretical amount of  $\text{KNO}_3$  was fixed at 50.0 wt % in all the preparation method used. All the solid catalysts were prepared by co-precipitation method at controlled pH of 10 which was determined by the indicator method. In a typical batch, using a basis of 20.0 g sample preparation, 3.5 g  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 6.5 g  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and 10 g  $\text{KNO}_3$  was dissolved in 50.0mL of distilled water to obtain a mixed nitrate solution. Aqueous solution

containing 5.6g of KOH in 50.0mL was added drop wise to precipitate the metal hydroxides and the mixture was stirred vigorously for 6 h to allow it to homogenize. At the end of mixing, the precipitate obtained was filtered, dried overnight at 85°C, and it was calcined at 460±1 °C for 4.5h to obtain the synergetic mixed oxide catalyst. This procedure was repeated for the catalysts prepared by co-precipitation with hot treatment. In this case constant heat was employed at 80°C while being stirred continuously during precipitation. In their various combinations for the hot and cold treatments, the catalysts were employed for transesterification of typical oil with methanol as synthesized. Design of experiment software was used to obtain optimum condition for the catalyst. Table 2 shows the levels of the factors considered for this study.

**Table 2: Factors and corresponding levels of experimental design used for the catalyst preparation**

Factors	ID	Units	Low Actual	High Actual	Low Coded	High Coded
Mg/Zn ratio	A	-	1.2	19	-1.000	1.000
Calcination Temperature	B	°C	400	700	-1.000	1.000
Calcination Time	C	h	4.00	6.00	-1.000	1.000

*Source: Olutoye et al. (2013)*

### **Preparation of KMgZnO**

This catalyst, KMgZnO, was prepared by co-precipitation method when accurately weighed amounts of their nitrate salts were dissolve and mix together in 1000mL glass beaker. In a particular batch preparation, a basis of 20.0g of all samples was chosen at first thoroughly mixed in 50mL of distilled water. The

mixture is equivalent to 1.66M of  $Zn(NO_3)_2$ , 0.34M of  $Mg(NO_3)_2$  and 2.00M of  $KNO_3$  calculated according to the ratio of their combination in the formula expression, that is,  $K_yMg_{1-x}Zn_{1+x}O_3$  and corresponds to  $x=0.3$ . The mixture was precipitated using 2.00M KOH. The beaker placed on a hot plate and its contents were stirred thoroughly at 600rpm with magnetic stirrer suitable to achieve avoid mass transfer limitations. The temperature of the mixture was maintained at 80°C under continuous stirring for 6h until the solution was homogenized. Basic strength was determined by using pH indicator strip and the solution pH was maintained in the range of 9-10 throughout the experiment. At the expiry of the mixing period, the solution was filtered and dried at oven temperature of 85°C for 12h. This was followed by calcination at  $460 \pm 1^\circ C$  for 4.5h. The catalyst as synthesized was employed for the transesterification activity immediately using RPO and some part was kept in the desiccator in a moisture free atmosphere for preservation.

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### The basis for the catalyst screening

Various metals that have potentials for the production of FAME ranging from alkali metals (Li, Na, K, and Cs) to alkaline earth metals (Be, Mg, Ca, Sr, and Ba), transition metals (Ti, Fe, Co, Ni, Zn, W, Nb, and La) and lastly Aluminium (Al) are considered as options in the preparation of the different catalysts used. However, based on literature where some few combinations of these metals have been employed (Yusuff *et al.* 2017), it thus suffix therefore to embark on screening and elimination to narrow down these options to the metals used for the study. The

synthesis of heterogeneous catalyst from combination of metals such as Mg, Zn, Al and K as fluxing material with the empirical formula  $K_yMg_{1-x}Zn_{1+x}O_3$ ,  $Mg_{1-x}Zn_{1+x}O_2$  and  $Mg_{1-x}Zn_{1+x}Al_{(2-y)/3}O_3$  structures have not been studied. Although, their independent structures and properties have been investigated and are chosen in the study because they are of interest in a large field of applications (Yusuff *et al.* 2017). In addition, the factors of cost, availability of materials and the simplicity in preparation method (that is non-corrosive) are the reasons which motivate the selection of K, Mg, Zn and Al as suitable metals for the synthesis of the catalyst used in the research.

The above findings motivated the development of catalyst in three main stages designated as catalyst 1, catalyst 2 and catalyst 3. The chemical formulation for catalyst 1 is given as  $K_yMg_{1-x}Zn_{1+x}O_3$ , catalyst 2 is written as  $Mg_{1-x}Zn_{1+x}O_2$  and finally catalyst 3 as  $Mg_{1-x}Zn_{1+x}Al_{2/3}O_3$ . Table 3 shows the various chemical formulae of the possible combinations of the metals considered for catalyst 1. Each of the developed catalysts were prepared, tested, optimized and finally characterized.

## **INVESTIGATION INTO THE EFFECTS OF CATALYST PREPARATION/TREATMENT CONDITIONS**

The suitable preparation condition for the catalyst formulated in Table 3 was established with some selected range of values for x and y variables which are chosen for the preliminary experimental investigation and prepared using the hot and the cold treatment methods. All samples were weighed accurately according to the interaction ratio by mass of the metals and

prepared by co-precipitation of the metal hydroxides from the mixed solution of their nitrate compounds. Table 4 shows the samples prepared according to their chemical formulae and the treatment method employed. The activity of selected catalyst 1 was tested with refined palm oil (RPO) as feedstock.

**Table 3: Various levels of chemical combinations of metals employed for the preparation of catalyst 1.**

No.	x	y	Chemical formula	Amount of each element in 20 g sample (g)			Mg:Zn ratio by mass
			$K_yMg_{1-x}Zn_{1+x}O_3$	Mg	Zn	K	
1.	0.1	2	$K_2Mg_{0.9}Zn_{1.1}O_3$	4.5	5.5	10	1:1.2
2.	0.2	2	$K_2Mg_{0.8}Zn_{1.2}O_3$	4.0	6.0	10	1:1.5
3.	0.3	2	$K_2Mg_{0.7}Zn_{1.3}O_3$	3.5	6.5	10	1:1.86
4.	0.4	2	$K_2Mg_{0.6}Zn_{1.4}O_3$	3.0	7.0	10	1:2.33
5.	0.5	2	$K_2Mg_{0.5}Zn_{1.5}O_3$	2.5	7.5	10	1:3.0
6.	0.6	2	$K_2Mg_{0.4}Zn_{1.6}O_3$	2.0	8.0	10	1:4.0
7.	0.7	2	$K_2Mg_{0.3}Zn_{1.7}O_3$	1.5	8.5	10	1:5.67
8.	0.8	2	$K_2Mg_{0.2}Zn_{1.8}O_3$	1.0	9.0	10	1:9.0
9.	0.9	2	$K_2Mg_{0.1}Zn_{1.9}O_3$	0.5	9.5	10	1:19.0
10.	0.7	2	* $K_yMg_{1+x}Zn_{1-x}O_3$	8.5	1.5	10	5.67:1
11.	-	-	MgO	10	0	0	-
12.	-	-	ZnO	0	10	0	-

Reverse empirical formula of catalyst 1 where Mg and Zn were swapped.  
*Source: Adapted from Olutoye et al. (2011a, b and c)*

The results of the tested activities for these various synthesized catalysts during transesterification of RPO with methanol to produce fatty acid

**Table 4: Catalyst formulation and the treatment methods employed during synthesis**

No.	Treatment effect during co-precipitation	Catalyst Formulation
1 <sup>a</sup> .	Hot	$K_2Mg_{0.3}Zn_{1.7}O_3$ ; x = 0.7
2.	Hot	$K_2Mg_{0.7}Zn_{1.3}O_3$ ; x = 0.3
3.	Hot	$K_2Mg_{0.9}Zn_{1.1}O_3$ ; x = 0.1
4.	Hot	$K_2Mg_{0.2}Zn_{1.8}O_3$ ; x = 0.8
5 <sup>b</sup> .	Hot	$K_2Mg_{1.7}Zn_{0.3}O_3$ ; x = 0.7
6.	Cold	$K_2Mg_{0.3}Zn_{1.7}O_3$ ; x = 0.7
7 <sup>b</sup> .	Cold	$K_2Mg_{1.7}Zn_{0.3}O_3$ ; x = 0.7
8.	Cold	$K_2Mg_{0.7}Zn_{1.3}O_3$ ; x = 0.3
9.	Cold	$K_2Mg_{0.9}Zn_{1.1}O_3$ ; x = 0.1
10.	Cold	$K_2Mg_{0.2}Zn_{1.8}O_3$ ; x = 0.8

5<sup>b</sup> (hot) and 7<sup>b</sup> (cold) is reverse of 1<sup>a</sup> of catalyst 1

*Source: Adapted from Olutoye et al. (2009)*

methyl esters (FAME) are presented in Table 5. The conditions (catalyst loading of 1.404 wt % based on oil, methanol/oil ratio of 6:1, temperature 150°C and reaction time of 12h) employed in transesterification for these preliminary tests were based on several experimental runs, and minimum value obtainable from literature (Olutoye *et al.* 2016b, 2016c). The results presented in the table are the FAME content obtained within 360min of reaction for the different catalysts. Transesterification of vegetable oil consists of a sequence of three reversible reactions, in which the triglyceride is successively transformed into di-glyceride. mono-glyceride and finally



**Table 5: Preliminary activity test of selected catalyst 1 subjected to dual treatment at different calcination temperature**

Catalyst sample	Calcination Temperature (°C)	Calcination Time (h)	%FAME yield	
			Hot method	Cold method
$K_2Mg_{0.3}Zn_{1.7}O_3^a$	400	6	73	57
$K_2Mg_{0.3}Zn_{1.7}O_3^a$	600	4	42	30
$K_2Mg_{1.7}Zn_{0.3}O_3^b$	400	6	28	19
$K_2Mg_{1.7}Zn_{0.3}O_3^b$	600	4	22	13
$K_2Mg_{0.7}Zn_{1.3}O_3$	400	6	46	33
$K_2Mg_{0.7}Zn_{1.3}O_3$	600	4	38	27
$K_2Mg_{0.9}Zn_{1.1}O_3$	550	6	64	60
$K_2Mg_{0.2}Zn_{1.8}O_3$	700	4	41	35

b is for the reverse formula of a  
*Source: Adapted from Olutoye et al. (2009)*

glycerol and FAME. The yield of these primary products depends largely on the operating parameters which increases with most of the parameters tested. A decrease in the yield of FAME was observed with further increase in the calcination temperature, indicating the role that catalyst preparation/treatment method, calcination time and calcination temperature played in the ester production. The observed trend is corroborated with the work reported by Yusuff et al. (2019) and the performance of the catalyst investigated in this study. Figure 6 shows a plot of the product conversion to FAME for catalyst 1 and it revealed the difference in activity for the hot and cold precipitated samples during the catalyst synthesis. It was also observed that

increasing the calcination temperature of catalyst with the chemical formula  $K_2Mg_{0.3}Zn_{1.7}O_3$  and  $K_2Mg_{0.7}Zn_{1.3}O_3$

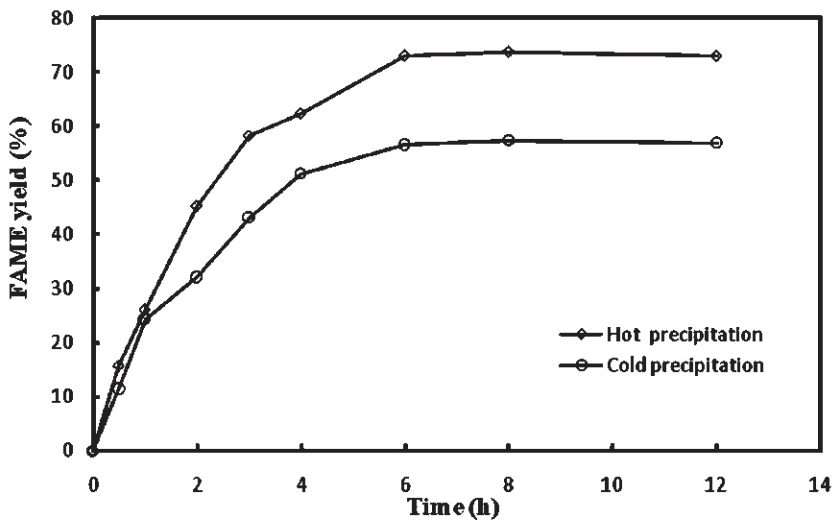


Figure 6: FAME yield (%) versus transesterification time (h) during test of activity with as synthesized catalyst  $K_2Mg_{0.3}Zn_{1.7}O_3$  at different treatment methods: Hot and cold treatment with methanol/oil ratio 6:1, catalyst loading 1.404 wt %, reaction time 12h and temperature 150 °C

Source: Adapted from Olutoye et al. (2013)

did not improve the FAME content. It is believed that pore constriction could have occurred when gases such as nitrous oxides and ammonia were diffusing out of the pore structure into the gaseous mainstream during heat treatment. The observed activity of the hot treated samples could also be due to the release of gases such as NO, H<sub>2</sub>O, and O<sub>2</sub> which usually induced strong inhibiting effect depending on the surface properties of the solids particularly the surface mobility of oxygen from  $KMgZnO_3$ . This observation is corroborated by the work of Yusuff

*et al.* (2019). This effect may have contributed to reduction in catalytic performance for the cold precipitated samples.

Based on the above argument and presentation, the catalyst formulation,  $K_yMg_{1-x}Zn_{1+x}O_3$ , with  $x = 0.7$  and  $y = 2$  was chosen for further investigation and characterization as it proved to be better than other formulations in the preliminary investigations. The catalyst treatment conditions were optimized using design of experiment software with high and low levels of the factors (Mg/Zn ratio, calcination temperature and time) as presented in Table 2. An independent confirmation of the effect of reaction period on the yield of FAME was obtained. Figure 6 shows the plot of time versus FAME which indicates that the yield increased with time until a point at which time is no longer significant justifying the reaction period of 6h which will later be investigated on other oils. The time  $t = 0$  was determined by the direct GC determination of blank refined palm oil (after duly observing sample preparation procedures) and as would be expected, it gave 0 % FAME. The trans-esterification conditions employed in the study of effect time are: catalyst amount (1.404 wt % of oil equivalent to 1.2g catalyst), methanol/oil molar ratio (6:1), temperature (150°C) and was run without the addition of external pressure reduce the cost of biodiesel production.

### **Preparation of MgO alone with KOH**

The exact contribution of individual components that make up the composite catalyst labeled “MgO alone” was determined. It was synthesized by weighing accurately the amount of magnesium nitrate contribution in the catalyst formulation and used it for the catalyst preparation. The particular amount of Mg-nitrate used was based on the catalyst screening previously carried out whereby ( $y=2$  and  $0.1 = x = 0.9$ ) and the combination from the catalyst formula which gave the highest FAME yield. Within this range, when  $x = 0.66$ , it corresponds to interaction of

Mg/Zn ratio 1:4.81. It was precipitated with 1.00M KOH to maintain a pH in the range of 9-10. The content in a 1000mL glass beaker was placed on hot plate where it was thoroughly mixed for 6h. The content was later filtered and dried at 85°C for 12h. This was followed by calcination at 460±1 °C for 4.5h. The catalyst as synthesized was employed for the transesterification activity immediately using RPO and some part was kept in the desiccator in a moisture free atmosphere for preservation.

### **Synthesis of ZnO alone with KOH**

Catalyst zinc oxide alone was synthesized by weighing accurately the amount of zinc nitrate contribution in the formulation and was used for the catalyst preparation. The particular amount of zinc nitrate used was based on the preliminary screening of the catalyst when ( $y = 2$  and  $0.1 = x = 0.9$ ) and the combination from the catalyst formula which gave the highest FAME yield. Within this range, when  $x = 0.66$ , it corresponds to interaction of Mg/Zn ratio 1:4.81 based on the preliminary investigation. It was precipitated with 1.50M KOH to maintain a pH in the range of 9-10. The content in a 1000 mL glass beaker was placed on hot plate where it was thoroughly mixed for 6h. The content was later filtered and dried at 85°C for 12h. This was followed by calcination at 460±1 °C for 4.5 h. The catalyst as synthesized was employed for the transesterification activity immediately using refined palm oil (RPO).

### **Preparation of KMgZnO with reverse composition**

The quest for the performance of the synthesized catalyst was extended by the investigation to its activity if the composition were to be reversed. Thus, KMgZnO composition was reversed with the interchange of Mg/Zn combination to obtain a catalyst with a formula  $K_yMg_{1-x}Zn_{1+x}O_3$ . As usual, accurately weighed amounts of their nitrate salts were dissolved and mixed together in 1000mL glass beaker and were precipitated by co-

precipitation method. The batch of catalyst during the screening when  $y = 2$  and  $0.1 = x = 0.9$  which gave the highest FAME yield was used in the reverse synthesis. Within this range, when  $x = 0.66$ , it corresponds to interaction of Mg/Zn ratio 4.81:1 based on the preliminary investigation. The mixture was precipitated using 2.00M KOH. The beaker placed on a hot plate and its contents were stirred thoroughly at 600rpm with magnetic stirrer. The temperature of the mixture was maintained at 80°C under continuous stirring for 6h until the solution was homogenized. Basic strength was determined by using pH indicator strip and the solution pH was maintained in the range of 9-10 throughout the experiment. At the expiry of the mixing period, the solution was filtered and dried at oven temperature of 85 °C for 12h. This was followed by calcination at  $460 \pm 1$  °C for 4.5h. The catalyst as synthesized was employed for the transesterification activity immediately using RPO and some part was kept in the desiccator in a moisture free atmosphere for preservation.

### **Synthesis of MgZnO catalyst precipitated separately with KOH and NH<sub>4</sub>OH**

In the preliminary experimental runs, homogeneous contribution of potassium incorporated into the catalyst structure at the developmental stage was substantial as large amount of potassium leached into the final product FAME. In order to address this problem, the potassium component of the composite catalyst was removed (dropped). The resultant (remaining) species were thereafter treated with two alkali media using the co-precipitation method. First, KOH was used and followed by ammonia water (NH<sub>4</sub>OH). Thus, the new catalyst formulation was obtained as Mg<sub>1-x</sub>Zn<sub>1+x</sub>O<sub>2</sub> where  $K_y=0$ . This was synthesized based on the preliminary investigation of the interaction effects of Mg-Zn ratio 1: 4.81. In the first case 2.00M KOH was used for precipitation, while in the second case, 5.00M

$\text{NH}_4\text{OH}$  was used. The metal hydroxides precipitated from their nitrate compounds were carried out in separate batches under constant stirring. Basic strength was determined by using pH indicator strip and the solution pH was maintained in the range of 9-10 throughout the experiment. At the expiry of the mixing period, the solution was filtered and dried in the oven at  $85\text{ }^\circ\text{C}$  for 12h. This was followed by calcination at  $460\pm 1\text{ }^\circ\text{C}$  for 4.5h. The catalyst as synthesized was employed for the transesterification activity immediately using RPO and WCPO, some portion was kept in the desiccator in a moisture free atmosphere for preservation.

The reusability tests aimed at establishing the extent of leaching was run when the first cycle of transesterification reaction was completed. The catalyst was collected, washed thoroughly with methanol and dried in an oven at  $100\text{ }^\circ\text{C}$  for 48 h. It was weighed and reused for second, third and fourth times. Comparison was made with reference to the two methods employed for the catalyst preparation which are  $A_{\text{pptd}}$  (precipitation with  $\text{NH}_4\text{OH}$ ) and  $K_{\text{pptd}}$  (precipitation with  $\text{KOH}$ ). Table 6 shows the development and empirical formulation of catalyst 2. It has been reported that disposal of this WCPO from vast majority of restaurants into the drainages constitute environment nuisance (Yusuff *et al.* 2019).

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**Table 6: Development and empirical formulation of catalyst 2**

No.	x	y	Chemical formula	Amount of each element in 10 g sample (g)			Mg:Zn ratio by mass
				Mg	Zn	K	
			$Mg_{1-x}Zn_{1+x}O_2$				
1	0	0	$MgZnO_2$	5.0	5.0	0	1:1.0
2	0.1	0	$Mg_{0.9}Zn_{1.1}O_2$	4.5	5.5	0	1:1.22
3	0.2	0	$Mg_{0.8}Zn_{1.2}O_2$	4.0	6.0	0	1:1.50
4	0.3	0	$Mg_{0.7}Zn_{1.3}O_2$	3.5	6.5	0	1:1.86
5	0.4	0	$Mg_{0.6}Zn_{1.4}O_2$	3.0	7.0	0	1:2.33
6	0.5	0	$Mg_{0.5}Zn_{1.5}O_2$	2.5	7.5	0	1:3.0
7	0.6	0	$Mg_{0.4}Zn_{1.6}O_2$	2.0	8.0	0	1:4.0
8	0.7	0	$Mg_{0.3}Zn_{1.7}O_2$	1.5	8.5	0	1:5.67
9	0.8	0	$Mg_{0.2}Zn_{1.8}O_2$	1.0	9.0	0	1:9.0
10	0.9	0	$Mg_{0.1}Zn_{1.9}O_2$	0.5	9.5	0	1:19.0

*Adapted from Olutoye et al. (2009; 2011a, b and c)*

### Characterization of the synthesized catalyst 2

The catalyst with the formula  $Mg_{0.34}Zn_{1.66}O_2$  when  $x = 0.66$  was developed and characterized to study the textural properties which include the surface area, pore size distribution, average pore diameter, particle size distribution and surface morphology. The catalysts crystal phase compositions as well as surface functional groups were also determined.

### Catalyst development challenges and comparative advantage of catalyst 2

The activity of catalyst 2 on both RPO and WCPO produced good yield of FAME with all the synthesized catalysts. From the results obtained, about +10 % in amount of FAME higher than catalyst 1 with respect to RPO. The optimum value for the factor of methanol/oil ratio also dropped from as high as 16:1 to 10:1 for

catalysts 1 and 2, respectively. This is considered to be an advantage in terms of overall cost of the process. The factors of temperature, time and catalyst loading seems to be close in range at optimum conditions for the two catalysts. Although, the development of catalyst 2 is quite simple and with reduced cost (input) of materials used in the synthesis, further modification could be made in its formulation to achieve improved FAME yield. A careful selection was made in terms of a component that could be integrated to make the catalyst possess a dual function (with both acidic and basic active sites) as well as improve the textural properties. It is a well-known fact that the rate of a chemical reaction increases with increase in the surface area of the participating solid catalyst as a result of increase in the number of active surface available for reaction. The surface areas obtained for both catalysts were quite low.

The more critical factor of leaching encountered in catalyst 1 was therefore given priority in the development of catalyst 2. First, by removing K from the formulation of the catalyst and second, by introducing another alkali ( $\text{NH}_4\text{OH}$ ) as precipitating medium and comparison was made with similar catalyst formulation in which KOH was used as the medium for precipitation. The observed leaching dropped as expected to a minimal value in the range of 0-1% for catalyst  $A_{\text{pptd}}$ . This seems quite good and satisfactory. Thus, one of the research goals has been successfully accomplished.

On the other hand, the leaching from  $K_{\text{pptd}}$  catalyst produced a value in the range of 8-13%. Close observation revealed that despite the removal of K from the catalyst formulation that comes from  $\text{KNO}_3$  and was only precipitated with alkaline KOH; the leaching value was still high and falls within the range of first generation catalyst that is 4-13%. Therefore, it was established in this thesis that  $A_{\text{pptd}}$  catalyst is very stable and addressed the leaching concerns during transesterification. From the above



mentioned points, in order to improve on the properties of catalyst 2, addition of Al component was considered to be favourably disposed to improve the textural and acidic properties of the catalyst since in many applications it is being used in the form of  $\text{Al}_2\text{O}_3$  and as support in some catalysts synthesis (Babatunde *et al.* 2020). In the current research,  $\text{Al}_2\text{O}_3$  synthesized from its nitrate compound by co-precipitation of its hydroxide with other metal hydroxides was integrated into the catalyst composite and its activity investigated to cover a wider range of application for the transesterification reaction using refined palm oil, waste cooking palm oil, coconut oil, palm kernel oil and non-edible, high fatty acid content, jatropha oil. Thus, the new development makes Al to be occluded in the catalyst formulation which resulted into the emergence of catalyst 3.

### **Preparation of MgZnAlO catalyst**

In all the synthesized catalysts, the surface area was a major concern. Although, the catalyst activity is high, nevertheless, the conversion to FAME could be improved if the surface area is improved upon. To this end, aluminium was considered for integration into the catalyst structure to correct this defect, that is, low surface area of the other synthesized catalyst. The formula representation of the various synthesized catalyst in this category with a defined range for x and y is given by  $\text{Mg}_{1-x}\text{Zn}_{1+x}\text{Al}_{(2-y)/3}\text{O}_3$  i.e.  $0.1 = x = 0.9$  and  $y = 0$ . The MgZnAlO modified catalyst was prepared by co-precipitation using the nitrates of the metals. Using a basis of 20.0g for sample preparation, 50mL of the mixed nitrates salt solution containing 0.66 M of  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 0.41M of  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and 0.27 M of  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  were prepared and placed in a 1000mL conical flask. The contents were continuously stirred at 800rpm with magnetic stirrer on a hot plate. Each batch preparation were precipitated using 9.61mL  $\text{NH}_4\text{OH}$  equivalent to 5.00M solution and the mixture

was maintained at a temperature of 80°C for 6h until completely homogenized milky solution was obtained. The basic strength was determined by the pH indicator strip which gives a value in the range of 8-9. This was maintained throughout all the catalyst preparation. The solution was filtered and gradually dried initially at oven temperature of 70°C for 8h for ease of handling from the filter paper into a glass dish. Thereafter, the catalyst was dried in glass dish when temperature was regulated to 100°C and was maintained for 6h. This was followed by thermal treatment at  $460 \pm 1^\circ\text{C}$  for 4.5h. Parameters for the synthesis were based on the preliminary investigation. All the catalyst as synthesized were employed for the transesterification using RPO, WCPO, CCO, PKO and CJO, some portion was kept in the desiccator in a moisture free atmosphere for preservation.

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### **The development and synthesis of catalyst 3**

In this section, based on the argument presented on catalyst 2 where detailed explanation was given on development, challenges and comparative advantage of the catalyst, it became imperative therefore that for the complete achievement of this research goal will require to develop a reusable catalyst with minimal leaching and improve on the % FAME yield during transesterification reaction be developed. Three factors were quickly considered as paramount for modification of catalyst 2. First, is the inclusion of Al into the catalyst composite matrix in the form of stable aluminium compound ( $\text{Al}_2\text{O}_3$ ). This will provide the catalyst with dual sites suitable for the transesterification of oil with high fatty acid in one pot synthesis (e.g jatropha oil). The second consideration is improvement on the surface area as a consequence of the inclusion of Al into the matrix of the solid catalyst to increase the number of available active sites during reaction. Third, since  $\text{NH}_4\text{OH}$  precipitation has

proved to be better in the previous catalyst development in terms of textural properties, reduction in leaching and % FAME yield; thus, it was used throughout the synthesis of catalyst 3.

Synthesis of catalyst 3 was carried out in the same vein as for the preparation reported for catalysts 1 and 2. The metal hydroxides were co-precipitated from the mixture solution from the nitrate compounds of all participating species. Thus, catalyst 3 was developed with the chemical formula  $K_yMg_{1-x}Zn_{1+x}Al_{(2-y)/3}O_3$  where  $y=0$  and  $x$  take values in the range of 0.1-0.9 ( $0.1=x=0.9$ ). Table 7 shows the possible catalyst combination.

**Table 7: Various levels of chemical combination of the metals employed in catalyst synthesis for catalyst 3**

No.	Chemical formula		Amount of each element in 20g sample (g)				Mg:Zn ratio by mass	
	x	y	Mg	Zn	K	Al		
		$K_yMg_{1-x}Zn_{1+x}Al_{(2-y)/3}O_3$ ; total atomic number of metals						
1.	0.1	0	$Mg_{0.9}Zn_{1.1}Al_{2/3}O_3$ (2.667)	6.75	8.25	0	5.00	1:1.22
2.	0.2	0	$Mg_{0.8}Zn_{1.2}Al_{2/3}O_3$ (2.667)	6.00	9.00	0	5.00	1:1.50
3.	0.3	0	$Mg_{0.7}Zn_{1.3}Al_{2/3}O_3$ (2.667)	5.25	9.75	0	5.00	1:1.86
4.	0.4	0	$Mg_{0.6}Zn_{1.4}Al_{2/3}O_3$ (2.667)	4.50	10.50	0	5.00	1:2.33
5.	0.5	0	$Mg_{0.5}Zn_{1.5}Al_{2/3}O_3$ (2.667)	3.75	11.25	0	5.00	1:3.00
6.	0.6	0	$Mg_{0.4}Zn_{1.6}Al_{2/3}O_3$ (2.667)	3.00	12.00	0	5.00	1:4.00
7.	0.7	0	$Mg_{0.3}Zn_{1.7}Al_{2/3}O_3$ (2.667)	2.25	12.75	0	5.00	1:5.67
8.	0.8	0	$Mg_{0.2}Zn_{1.8}Al_{2/3}O_3$ (2.667)	1.50	13.50	0	5.00	1:9.00
9.	0.9	0	$Mg_{0.1}Zn_{1.9}Al_{2/3}O_3$ (2.667)	0.75	14.25	0	5.00	1:19.00

*Source: Adapted from Olutoye et al. (2011 and 2013)*

The catalyst synthesis was done with a random selection of the low, the center point and the high values of the variable  $x$  as presented in the Table 7, that is,  $x=0.1, 0.5,$  and  $0.7$ . This was done to establish a trend in the catalytic performance. The test of activity of the developed catalyst was carried out by transesterification of WCPO with methanol using the condition for one batch run for the tests. The conditions are methanol to oil ratio of 9:1, catalyst loading of 2.55 wt % (based on weight of oil), and temperature of 188°C and at a reaction time of 4.25h.

The choice of WCPO and the selected conditions on which the test run was based without prejudice but rather to ascertain its performance and trend using the condition at which FAME content was highest during transesterification of WCPO. Further verification of selected catalyst 3 activity was also performed at other conditions different from above using WCPO and RPO as model oils. The results show that it yielded FAME of 88% for refined RPO after purification and 87% for WCPO at the same experimental conditions of methanol/oil ratio of 16:1, catalyst loading of 4.10 wt % (based on weight of oil), and temperature of 188°C at reaction time of 6h. The conditions here were chosen because higher yield of FAME was targeted above all that has previously been obtained. The outcome of these results was used as a guide in the design of experiment table in which the selected catalyst 3 was used for transesterification of five different types of oils feedstock viz: RPO, WCPO, palm kernel oil (PKO), coconut oil (CCO) and crude jatropha oil (CJO). Apart from the target for higher yield of FAME, the reasons also include establishing the catalyst effectiveness in transesterification of feedstock with high % FFA and moisture contents. In addition, testing the catalyst on other feedstock which includes non-edible oil will give the catalyst wide applicability and put to rest the food for oil debate. Similarly, utilizing waste oil from restaurants will minimize a disposal problem which is currently a huge task in

many countries. The continuous and large-scale production of biodiesel from edible oils without proper planning may cause negative impact to the world, such as depletion of food supply leading to economic imbalance (Babatunde *et al.* 2020). A possible solution to overcome this problem is to use non-edible oil or used edible oil (that is WCPO). The utilization of used edible oil will not only help in cleaning up the waste but by converting them into value added chemical products. WCPO from restaurants and household are inexpensive compared with RPO and other sources of oil. Thus, it is a promising alternative to RPO for biodiesel production. Reducing the cost of the feed stocks is necessary for the process to be commercially viable to compete with the petroleum diesel. The consistent supply of these materials should not pose any problem in the near future. However, the desired product and the properties of biodiesel from WCPO would largely be dependent on the physico-chemical properties of the feedstock (Khayoon *et al.* 2012, Nur Ashikin Ab Rahman *et al.* 2011). Of course, in the search for higher % FAME yield, high conversion to FAME was recorded for all the oils. Consequently, the variable when  $x=0.3$  was chosen, synthesized and its activity was tested. Thereafter, comparison was made with the results of their % FAME yield. The reason for settling for variable  $x=0.3$  as catalyst 3 among others considered is because a decline in trend of FAME yield was observed for the variable  $x$  as it tends towards the higher values.

## **FURTHER CONTRIBUTIONS**

### **Emission Characteristics of the renewable fuel produced was studied with or without blends with fossil fuel using Compression Ignition Engine**

Other catalysts obtained were oxide of calcium from cowbone treated at 900 °C was supported on silica obtained from clay by dealumination process designated as SC9 was functionalised with Cs/Zr metal oxides for improved performance and referred

to as CsZr/SC9. The ratio of Cs and Zr were 40% and 60% respectively and the silica to CaO was 50:50 throughout the experiment. Four different catalysts were obtained namely catalyst A (CsZr/SC9), catalyst B (CsZr/SC9)<sub>KOH</sub>, catalyst C (CsZrO<sub>2</sub>) and catalyst D (SC9). Carbon monoxide emission (CO) is produced by the incomplete combustion of carbon-containing substances. During burning of fuel in the presence of oxygen within the cylinder, the carbon present in any fuel is converted to CO (product of incomplete combustion). There are variations in the CO emission as the concentration of biodiesel in its blends increased. Figure 7 shows the CO emission at various blends. It was observed that as the concentration of FAME in its blends increased, the CO emission decreased. This occurs as a result of complete oxidation of FAME than that of mineral fuel. Some of the CO formed during combustion was further oxidized and converted to CO<sub>2</sub> due to the presence of certain amount of oxygen in FAME itself. Thus, it was concluded that the oxidation process of CO to CO<sub>2</sub> was enhanced due to the presence of oxygen in biodiesel fuel.

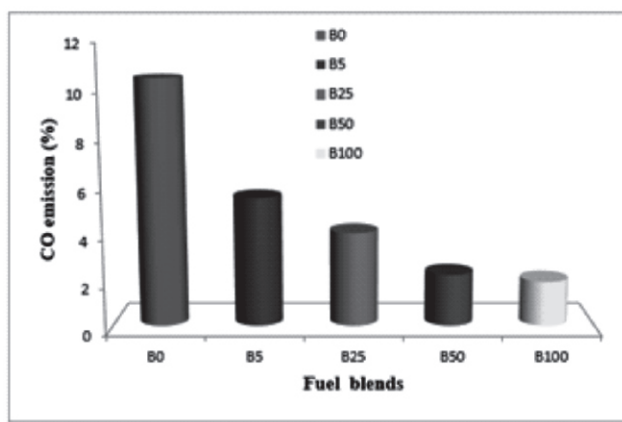


Figure 7: Effect of FAME on CO Emission in Compression Ignition Engine

Source: Babatunde et al. (2020)

## Carbon dioxide emission (CO<sub>2</sub>)

Naturally CO<sub>2</sub> is obtained from combustion of fuel. It has no negative influence on human but it is a greenhouse gas that combined with the earth's heat to cause global warming. Figure 8 illustrates the CO<sub>2</sub> emission using different blends of FAME and conventional diesel. It was observed that the CO<sub>2</sub> emission increased with increase in the FAME content in its blends. This was justified by the complete combustion in the combustion chamber due to the presence of oxygen in FAME which allowed the CO to oxidize into CO<sub>2</sub>. Likewise, the lower elemental carbon to hydrogen ratio in the molecular structure of FAME can be identified to be possible reason to lower CO<sub>2</sub> emission. This result was similar to the findings of Yusuff *et al.* (2019).

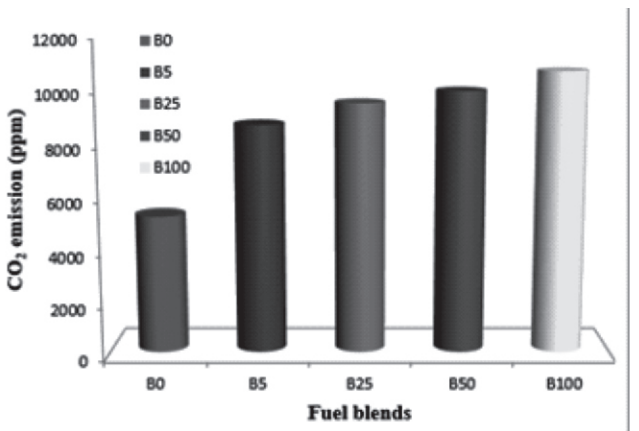


Figure 8: Effect of FAME on CO<sub>2</sub> Emission in Compression Ignition Engine Oxygen emission (O<sub>2</sub>)

Source: Babatunde *et al.* (2020)

The effect of FAME on O<sub>2</sub> emission in compression ignition engine was also studied. It was noted that O<sub>2</sub> emission of FAME blends is higher than that of the conventional diesel. This is

because FAME is an oxygenated fuel which contains oxygen of about 11% by volume, as a result of higher density. Thus, high oxygen content leading to complete combustion (Yusuff et al. 2017 and Woma *et al.* 2019a; 2019b). The presence of oxygen in methyl ester resulted in higher heat release during combustion. Also, from Figure 9, it can be observed that  $O_2$  emission increased with increase in FAME content. This is due to the lower elemental carbon to hydrogen ratio in FAME according to the work of Yusuff *et al.* (2019).

### **Comparison of Emission from FAME and Petrodiesel**

The substances emitted by FAME, 25% FAME blend and petrodiesel are presented in Figure 9.  $NO_x$  is formed by chain reactions involving Nitrogen and Oxygen in the air. These reactions are highly temperature dependent. Since diesel engines always operate with excess air,  $NO_x$  emissions are mainly a function of gas temperature and residence time. It was observed that harmful substances emitted by 100% FAME are far less than the petro diesel. Higher percentage of CO was released when the diesel engine was fueled with 100% petrodiesel and 25% FAME blend but in case of 100% FAME, there was a low CO released into the environment. In case of  $O_2$  emission for the blend of FAME and 100% FAME is higher than that of petrodiesel. This may be due to the fact that FAME is an oxygenated fuel which contain about 11% by volume as a result of higher density of FAME that led to complete combustion (Krishna *et al.* 2016). However, it was observed that  $CO_2$  poisoning is rare but a high concentration of it in a confined space can be toxic. The symptoms of mild carbon dioxide poisoning include headaches and dizziness when the concentrations less than 30000ppm. Thus, at 80000ppm,  $CO_2$  can be life threatening with a permissible exposure limit (PEL) of 5000ppm over an 8h period and 30000ppm not exceeding ten minutes from standard occupational safety and health administration.



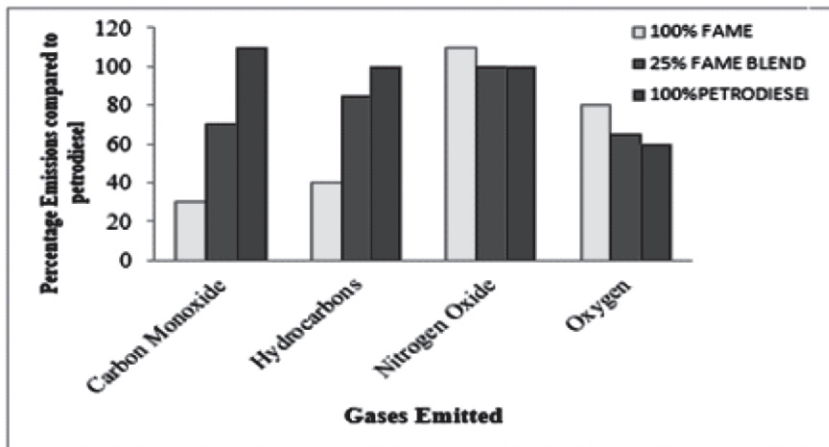
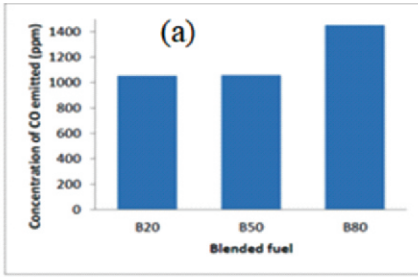


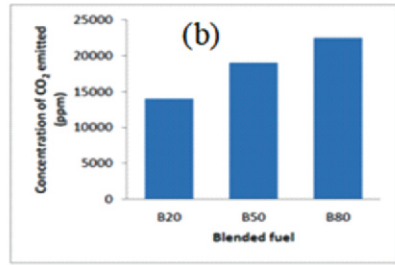
Figure 9: Comparison of Emissions from FAME and Petrodiesel  
 Source: Babatunde et al. (2020)

### Anthill eggshell promoted Ni-Co mixed oxides (NiCoAE) catalyst

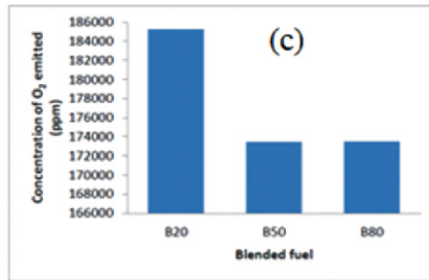
This was prepared and the emission performance tested in ignition engines. The performance and emission characteristics of different blended fuels in diesel engine whereby the gas exhaust emissions are compared for different biodiesel blends: B20, B50 and B80 at the engine speed of 2,000 rpm. The gas emissions measured included carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>) and oxygen (O<sub>2</sub>) using gas analyzer to monitor the gases. Comparison of Carbon monoxide (CO) Emission from the exhaust emissions for the three biodiesel blends are presented in Figure 10. Larger concentration of CO was emitted when the diesel engine was fueled with B80 fuel, followed by B50 which released 1,055ppm and least CO was emitted from diesel engine when it was fueled with B20 blended fuel. Although, the amount of CO emitted for every blended fuel was found to be very small.



CO emission pattern for B20, B50 and B80 blended fuel



CO<sub>2</sub> emission pattern for B20, B50 and B80 blended fuel



O<sub>2</sub> emission pattern for B20, B50 and B80 blended fuel

Figure 10: Emission characteristics of various blends of biofuel (a) CO, (b) CO<sub>2</sub> and (c) O<sub>2</sub>

Source: Yusuff et al. (2017)

## CONCLUSIONS

The current economic system is undermining our prosperity as it is destroying the natural foundations of our commercial activity. This calls for a transformation to a sustainable and renewable options in line with nature and the environment. The continuous dependence on oil during processing and combustion of fuel in industries is not sustainable for the economy. It is imperative that a transformation is necessary for a benign environment to preserve the natural resources. This will improve the well-being of people.

In order to transform the economy, to be less dependent on fossil, the energy sector is of particular interest because of its size and complexity. Advocacy for renewable energy policies for the energy sector can achieve important outcomes such as better resource management, innovation and productivity gains, reducing environmental damage and creating new markets and industries. Given the preponderance of fossil fuel in the current energy mix, reduction in gas emission plays a centrally important part of the economic transformation. Thus, it is believed that the renewable energy economy will break with the path dependency of existing technologies and will require innovative ideas with new technologies. Also, environmental protection and economic development is not a contradiction of terms but in fact depend on each other. Environmental protection will reduce insecurity and create jobs. Failure to protect the environment will generate high costs to the society through environment-induced health problems and material damages. There is need for deliberate and determined environmental policy to reduce the cost implications through the use of sustainable and renewable fuel in the transport, energy and industrial sectors for economic transformation that will set the pace for the journey to our Eldorado.

## **RECOMMENDATIONS**

- There should be deliberate government policy to create incentives for any individual to invest in maintaining the quality of the environment
- Other sectors of the economy, such as agriculture and manufacturing, should be given an opportunity to thrive and support revenue from petroleum, especially now that the price of oil is nose-diving
- Require a boost in research and development of the energy sector for innovative transformation from the present structure

## ACKNOWLEDGMENTS

First, I wish to thank God, The Almighty, The Most High (*Jehovah-Elyon*-Lord Most High God) for uncountable reasons. He alone giveth knowledge and wisdom to whom He pleases. I worship Him with these few words: Who is this? Great and Mighty One; Who rules the earth; Who sets above; HE is my GOD and HE IS GREAT. May His glorious name be praised forever- Amen

I wish to acknowledge the management team: the Vice-Chancellor, Professor, Abdullahi Bala; Deputy Vice-Chancellors (Academic & Administration); the Registrar; the Bursar and the Librarian. I thank you all for your immense support especially the Vice-Chancellor, who found me capable; appointed me the Director of Directorate for Research, Innovation and Development (DRID) and also the Coordinator of the Intellectual Property and Technology Transfer Office (IPTTO). It is a privilege to serve under him in these two offices. Similarly, I wish to thank Professor Adewumi Akanji (former Vice-Chancellor, FUT, Minna) under whom I was promoted to the ranks of Reader and Professor and served as Deputy Director in the DRID office.

I thank Professor Bassim H. Hameed (my PhD supervisor) for his guidance, great inspiration and support. It is a great opportunity to have worked with him. Also, I acknowledged gratefully Professor Abdul Latif Ahmad (my PhD co-supervisor) for his encouragement throughout my stay in Universiti Sains Malaysia (USM). I thank you both.

I wish to acknowledge my Lecturers beginning with Prof. K. R. Onifade (Father of Engineering at FUT, Minna), Professor F. Aberuagba, Professor S. Sadiku, Professor Emmanuel Udensi (DVC Admin), Professor Oyedum, Professor Abubakar Ndaman Saidu, Professor Mrs. H.O. Akanya, Dr. M. B. A. Wuna –Director of Works, Professor Alfred A. Susu (my MSc Supervisor), University

of Lagos etc. They started with me and made me who I am today. I appreciate you ALL

I wish to acknowledge ALL my Friends. They are too numerous to mention. But of note are:

Professor. B. O. Aderemi and Professor Mohammed Tijani both of Chemical Engineering Department, and Professor. Abdul-Raheem Giwa of Department of Textile Science, Ahmadu Bello University (ABU), Zaria, Nigeria. In addition, appreciation goes to the following (without much elaborate encomium, they know their contribution and impact in various capacities). They are: Professor Uduak George Akpan (Tqvm), Dr. Manase Auta, Dr. O. S. Azeez, Engr. Dr. E.J. Eterigho, Engr. Dr. E.A. Afolabi, Engr Shehu and other friends in my research group; I thank you all for your advice, kindness, help, concern, motivation and moral support. I appreciate all your efforts, my dear friends. To those who indirectly contributed to the success of this lecture, your kindness means a lot to me. Thank you very much.

I wish to acknowledge the Church: Mountain of Fire and Miracles Ministries (MFM) and The Apostolic Church (TAC). Words will not be enough for me to express my heartfelt gratitude to all members of The Apostolic Church, Minna Area, Nigeria for your love well expressed. THANK YOU. The Regional Overseer (MFM), Pastors and all members, The TAC, Minna Area Supt, others too numerous to mention, most importantly Pastor G.A. Adeniyi and family (1st Minna Area Supt.)

I wish to acknowledge ALL Chemical Engineering staff. I am indebted to my colleagues at the Chemical Engineering Department, My sincere thanks go to all. In the same vein, I wish to acknowledge ALL SIPET & SEET staff. My sincere thanks go to all. Thankyou

I acknowledge immensely the contributions of the following staff from the Directorate for Research, Innovation and Development (DRID), they are: Professor C.E Chinma (Deputy Director, DRID), Mr. K. Ndayako, Mrs H. Abdulrahman, Mrs. E. Gana, Mrs. J.A.Bechila, Miss E.A. Nwankpa and Mal. D. A. Baba and more importantly, Professor Hussaini Anthony Makun (former Director, DRID), and wife. I appreciate you in many ways. Thank you

I wish to acknowledge my parents & relations

Parents: My gratitude goes to my parents (all late): Pa Joseph Oyeluyi Alonge Olutoye- you are a GREAT MAN even in the grave. Thank you for your investment on me. My mother Mrs Grace Ola-Ashake Olutoye (Eye-Aanu) and Mrs Grace Oluwawo Olutoye in Erio-Ekiti, Ekiti State, Nigeria (my origin). I thank them for their support, encouragement, understanding, concern and for standing by me all through to this day. Thank you

Siblings: My thanks go to my siblings: Mr. & Mrs. Ajibola Olutoye (London, UK), Mr & Mrs. Olanrewaju Olutoye (NSCDC, Ado-Ekiti, Nigeria) Mr. and Mrs. Adaramola Olutoye (Abuja, Nigeria). Others are Pastor & Mrs. Samuel Olutoye (Ayobo, Lagos) Mr. & Mrs. Oluwasesan Olutoye, Mr. & Mrs. Kehinde Olutoye. I thank you all for being there always. God will reward you bountifully. Amen!

SPECIAL THANKS GOES TO MY AMIABLE WIFE. (I am loaded with story but not today! She has done what Napoleon could not do. Who can find a virtuous woman? For her price is far above rubies (Proverb 31vs10). I FOUND ONE!!! She has managed me too well from inception till date. My Dear Faith Olunike Ronke (FOR Olutoye or SM as I fondly call her), A MILLION THANKS TO YOU. Same goes for my adorable children whom the LORD has

blessed us with. They are Miracle Omowumi, Israel Gift Ifeoluwa, Deborah Abiodun Oluwaseyi Praise, and Ephraim Boluwatife Oluwatobi Oluwatimilehin. Thank you the shining stars.

I wish to acknowledge my research students: Postgraduate & undergraduate. Worthy of note are Engr Dr. Mrs Esther Olubunmi Babatunde of the University of Ilorin, Nigeria and Engr. Dr A.S. Yusuff of Afe Babalola University, Ado-Ekiti, Nigeria, and others too numerous to mention

I wish to acknowledge members of University Seminar and Colloquium Committee. I thank the Chairman of Committee, Professor B.A. Ayanwale and his team (Professor Jude T. Kur and others) for making this inaugural lecture a success. I will end this lecture with the quote I found so intriguing “OUT OF THE POND OF KNOWLEDGE, THE IGNORANT REMAINS ATLANTIC”- Anonymous

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## THE BRIEF PROFILE OF THE INAUGURAL LECTURER

**P**rofessor Moses Aderemi Olutoye was born on 29<sup>th</sup> May, 1964 in Erio-Ekiti, Ekiti West Local Government Area of Ekiti State, to the family of late Mr and Mrs Joseph Oyeluyi Alonge Olutoye. He attended Saint Paul's Primary School, Idi-Araba, Palmgrove, Lagos State and Saint Charles' Grammar School, Oshogbo in Osun State. He obtained Bachelor of Engineering Degree in Chemical Engineering from the Federal University of Technology, Minna (1991), and his second degree in Chemical Engineering (1998) from the University of Lagos, Nigeria. He later proceeded to the Universiti Sains Malaysia (USM), Penang, Malaysia for his PhD in Chemical Engineering (2012).

Professor Moses Olutoye was posted to Borno State for his National Youth Service in 1991 but later redeployed to Yobe State and served at Government Girls' College, Damaturu and also as the first President for the Nigeria Christian Corpers' Fellowship (NCCF), Yobe State after election at the Maiduguri Orientation Camp from 1991 – 1992. He joined the services of Federal University of Technology, Minna as Assistant Lecturer in December 1992 and rose through the ranks to Professor of Chemical Engineering in 2016

He is actively involved in research and his interest is in Catalysis and Renewable energy, especially in areas related to Energy and Environment, Waste Management, Environmental Engineering, Modelling and Simulation. He publishes regularly with his research students in ISI journals. Professor Olutoye has supervised several undergraduate and postgraduate students (Masters and PhD). He has assessed many candidates for promotion to professorial ranks at many universities.



He has held several administrative positions in the Department (such as Academic Adviser for various Levels, Research project and Plant Design Coordinator among others), in the School (Student Work Experience Programme (SWEP) Coordinator, Engineering Staff Welfare Coordinator and the **first Sub-Dean** for the Old School of Engineering and Engineering Technology (SEET)) and the University. Presently, he is the Director, Directorate for Research, Innovation and Development (DRID) of the Federal University of Technology, Minna and the Coordinator of the Intellectual Property and Technology Transfer Office (IPTTO) of the institution.

He is a recipient of the Best Graduating Student, Chemical Engineering award by Federal University of Technology, Minna (1992) and Nigerian Society of Engineers Award (Niger State Chapter) for Best Graduating Student. He also bagged Best PhD Thesis award of the School of Chemical Engineering, Universiti Sains Malaysia (2013).

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

Professor Olutoye has published over 70 articles in high impact international and national journals and conference proceedings. His publications have been cited over 1,052 times (Google scholar database). He has an author h-index of 18 (Google scholar database) and h-index of 15 (Scopus database), and an i-index of 24 in the period of 2009 — 2021. He is highly ranked among the top ranking scientists in the world according to AD Scientific Index Ranking for Scientists, University, Subject, Country, Region and the world.