

## CHEMICAL ENGINEERING, NIGERIA AND THE CHANGING SOCIETY

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### ABSTRACT

*This paper reviews the nature of Chemical Engineering, as well as its foundations and practice in Nigeria. It then examines the needs of contemporary society and economy and explores the role and relevance of Chemical Engineering in meeting the needs of the emerging society and economy. Subsequently, the peculiarities of future societal and economic challenges Nigeria will be faced with are highlighted. The question of training of Chemical Engineers to meet these future challenges in Nigeria then comes under focus. Suggestions are made for improved Chemical Engineering curriculum in the nation's universities which will enable the production of Nigerian Chemical Engineers who are well equipped to meet the needs of the future.*

**Keywords:** chemical engineering education; Nigeria developmental needs; curriculum.

### 1. CHEMICAL ENGINEERING

To put this discourse in proper perspective it may be helpful to explore the meaning and nature of Chemical Engineering.

Chemical Engineering may be defined as that aspect of Engineering concerned with development, design, construction, operation, maintenance and optimization of processes for transformation of materials from one form to another. In most cases it entails processes in which primary materials are converted to semi-finished products required as raw materials for industry or finished products for consumers.

The processes may or may not entail chemical conversion. The appellation, Chemical Engineering, therefore sometimes seems like a misnomer. No wonder therefore Chemical Engineers are often aptly referred to interchangeably as Process Engineers.

The traditional building blocks and tools of Chemical Engineering are Physics and Chemistry, robustly supported by Mathematics. Lately however, Biology is beginning to play a significant role as well.

### 2. CHEMICAL ENGINEERING IN NIGERIA

#### 2.1. Historical Prospective

The history of Chemical Engineering in Nigeria may be traced back to the mid- 1950s when the first Nigerian Chemical Engineers graduated from UK and USA universities. Even at that, there was only a handful of Chemical Engineers in the country subsequently and the

profession was virtually unknown till the late 1960s when several young overseas trained graduates had returned to the country. Most of these were beneficiaries of the US Government special scholarship assistance program at the time, the African Scholarship Program for American Universities (ASPAU), as well as the Commonwealth Scholarship Program, spearheaded by the government of UK.

#### 2.2 Education

The then University of Ife started an undergraduate Chemical Technology program in 1968. This metamorphosed into Chemical Engineering by 1972, paving the way for the graduation of the first set of 14 locally trained Chemical Engineers in the country from the university in 1973 (Erinne, 2021). The University of Lagos and Ahmadu Bello University followed suite, with their first Chemical Engineering graduates emerging in 1976 and 1977 respectively. Now Nigeria boasts of over 30 university and 10 polytechnic Chemical Engineering programs.

#### 2.3 Practice

It is estimated that there are 12-15,000 Chemical Engineers in Nigeria. They are involved in diverse sectors of society and economy. A recent survey (see Table 1) indicates however that up to 45% are engaged with the Oil & Gas Industry, while the Education Sector and Chemical/Process Industry account for 17% and 12% respectively (Dada, et al., 2013; Erinne and Ogbuigwe, 2018).

**Table 1. Engagement of Chemical Engineers in Nigeria by Sectors**

SECTOR	%
Oil & Gas	45
Education	17
Chemical/Process Industry	12
Government	8
Engineering/Construction/Consulting	5
Research	4
Other	9

Nigerian Chemical Engineers also serve in a full range of work functions. About 24% are estimated to be engaged in Production Operations, while about 16% and 15% are in the Academia and in Technical Services, respectively (Dada, et al., 2013; Erinne and Ogbuigwe, 2018).

**Table 2. Engagement of Chemical Engineers in Nigeria by Function**

FUNCTION	%
Production	24
Academia	16
Technical Services	15

FUNCTION	%
Projects/Construction	8
Design/Engineering/Consulting	7
Research & Development	6
General Management	5
Sales & Marketing	5
Others	14

### 3. CHANGING SOCIETY

Human society has undergone monumental changes over the decades. The trajectory of change in the last 60 years may be captured in the Global Change Snapshot shown in Table 3.

In 1962 the world population was only 3.2 billion. Today we have a global population of 8 billion to cater for, with accompanying huge challenges. Indeed massive changes have occurred in virtually all spheres of life, including communications, computation, information technology, manufacturing, etc. Whereas the leading global business corporations in 1962 were dominated by Oil & Gas and Chemical companies, now IT and healthcare companies have taken the center stage (Dada, 2019).

**Table 3. 60 Years of Global Change: Snapshot**

		1962 World	2022 World
1.	World Population	3.2 Billion	8 Billion
2.	US Gasoline Price	25 Cents/Gal	>\$2/Gal
3.	Telephony/Communication	Fixed Telephones	Mobile Telephones/Smart Phones/Internet/Worldwide Web
4.	Computation	Slide Rules/Mechanical Calculators/Primitive Computers	Electronic Calculators/Personal Computers/Super Computers
5.	Industry	Mechanized	Digitized/AI & Robotics
6.	Leading Global Corporations	Esso, Shell, Bp, Du Pont, Union Carbide, Hoechst, Basf, Ici, Gm, Ford, General Electric, Itt, Dow, Volkswagen, Mobil,	Apple, Google, Microsoft, Amazon, Facebook, Cvs, Exxonmobil, AT&T, Walmart, Tesla, Saudi, Aramco, Petrochina, Johnson & Johnson,

	Amdco, Gulf, Etc	Visa, Etc.
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#### 4. GLOBAL SOCIETY NEEDS

Social scientists consider that the most basic needs of man are: food, shelter and clothing.

They also consider that man has other basic needs, which include: energy, water, clean air, and healthcare.

Chemical Engineers are relevant to the delivery of these needs to society and shall be challenged to play even more important roles in providing and sustaining them in the evolving global society. In particular, it is envisaged that Chemical Engineers will play major roles in providing food, energy and clean air. They will also play an important part in the areas of clothing, water and healthcare (Dada, 2019; Wilspeywey, 2015).

**Table 4. ROLE OF CHEMICAL ENGINEERING IN DELIVERING THE BASIC NEEDS**

SN	Need	Extent Of Role	Nature Of The Role
1.	Food	Major Role	Efficient Process Technologies Biomolecular Technologies
2.	Shelter	Minor Role	Development Of Cheaper & Environmentally Friendlier Building Materials
3.	Clothing	Important Role	Improved Cotton & Synthetic Materials Processing Biomolecular Technologies
4.	Energy	Major Role	Development Of Renewable/Clean Energy (Solar/Wind/Biomass/ Hydroelectric/Hydrogen/Energy Storage/Etc)
5.	Water	Important Role	Improved Treatment Processes For Potable/Industrial/Effluent Water
6.	Clean Air	Major Role	Pollution Abatement Technologies
7.	Healthcare	Important Role	Life Systems Modelling New Biomedical Processes & Devices More Efficient Continuous Pharma Manufacturing Processes

#### 5. THE GLOBAL ECONOMY

Chemical Engineers shall also be challenged to sustain relevance in the emerging global economy. They will be expected to be highly relevant to the Petroleum, Power

& Energy and Manufacturing Sectors, but moderately relevant in the Agricultural, Mining, Engineering & Construction, ICT and Healthcare Sectors (Westmoreland and McCabe, 2018; Dada, 2019).

Table 5. Relevance Of Chemical Engineering In The Economic Sectors

Sector	Extent of Relevance	Nature Of Relevance
Agriculture	Moderate Relevance	Agric/Food Processing Technologies Biomolecular Technology
Mining	Moderate Relevance	Mineral Processing Technologies
Petroleum	High Relevance	Oil & Gas Processing Technologies Petrochemicals Processes
Power & Energy	High Relevance	Renewable/Clean Energy Technologies
Manufacturing	High Relevance	Process/Production Technologies In Chemical/Process Industries
Engineering & Construction	Moderate Relevance	Design & Construction Of Resource Improvement Systems Development Of Improved Construction Materials
Transport	Low Relevance	Improved Materials Components
ICT	Moderate Relevance	Improved Materials & Components
Banking & Finance	Not Relevant	---
Healthcare	Moderate Role	Life Systems Modelling Improved Biomedical Processes/Devices More Efficient Continuous Pharma Processes
Education	High Relevance	Education & Training of ChEs
Others	Not Relevant	---

## 6. THE NIGERIAN CHALLENGE

Nigeria is not insulated from the momentous societal and economic changes sweeping through the world. In addition to the general challenges associated with these changes, the country is also increasingly challenged by other peculiar afflictions, especially in the areas of:

- Catering to social needs of a very rapidly growing population: over 500 million by 2050
- Huge unemployment levels, over 30%

- Massive poverty levels, up to 40% of population
- Generation and conservation of valuable foreign exchange
- Provision of adequate public infrastructure

Consequent on these, it is considered that the challenges which will define the role of Nigerian Chemical Engineers and the solutions they are expected to bring to bear are as summarized in Table 6. The challenges will include: the Energy Transition, Oil & Gas Processing, Petrochemicals Development, Evolution of a Local

Chemical Industry, Food / Water Security, Resource & Development.  
Improvement, Environmental Protection and Research

**Table 6. Chemical Engineering Solutions for Future Nigerian Challenges**

SN	Challenge	Chemical Engineering Solution Input
1.	Energy Transition	Development & Deployment of Renewable/Clean Energy Technologies
2.	Oil & Gas Processing	Efficient Petroleum Processing & Oil Refining In The Transition Phase
3.	Petrochemicals	Rapid Petrochemicals Development Based On Abundant Natural Gas
4.	Local Chemical Industry	Development Of Chemical Intermediates Manufacturing: Aldehydes, Acrylates, Ethanolamines, Sulfonates, Acetic Acid, Etc
5.	Food/Water Security	Development Of Food Processing & Storage Technologies
6.	Resource Improvement	Development & Implementation of Technologies & Projects For Processing Abundant Natural Resources: Agricultural & Mineral
7.	Environmental Protection	Technologies For Air Pollution Control & Efficient Water Treatment
8.	Research & Development	Outcomes Across The Spectrum

## 7. FUTURE NIGERIAN CHEMICAL ENGINEERS

### 7.1 Imperative of Change

The Chemical Engineering curriculum in most Nigerian Universities are not considered adequate or appropriate for the coming challenges. The country needs Chemical Engineers of the future who will be globally competitive but relevant to local challenges. They will be expected to retain the traditional Chemical Engineering skills which define the profession, while in addition acquiring the new cutting edge technologies and skills, such as Artificial Intelligence, Data Analytics, Process Intensification, Nanotechnology, 3D-Printing, Gene Editing, etc. Considerable modifications are therefore desired, for improvement.

### 7.2 Towards a New Curriculum

The critical contents of a robust and adequate Chemical Engineering curriculum of the future, for Nigeria, should encompass a full range of key course groups and modules which include: Basic Sciences, Mathematics/IT, Basic Engineering, Chemical

Engineering Fundamentals, Applied Chemical Engineering, New Technologies, Sustainability, Economics/Management/Entrepreneurships, Humanities/Social Sciences (General Studies), Other Soft Skills, Electives, Design, Project and SIWES.

A typical 5-year university curriculum for Nigerian universities is proposed in the Appendix, based on these identified course modules, with summaries in Tables 7 and 8. The thrust and remedial features of the proposed curriculum include the following:

- Limiting course load per semester to a maximum of 18 credit hours.
- Creation of balance between critical thinking and learning.
- Equipping young Chemical Engineers with a combination of traditional skills and new technologies.
- Providing them with necessary soft skills.
- Infusing versatility while providing for specialization through the Technical Electives.
- Ensuring rational progression sequence and accommodating course pre-requisites.

Table 7. Typical Chemical Engineering Curriculum Summary

	Course Groups/Modules	Credit Hours Required	% of Total
1.	General Studies	8	4.9
2.	Basic Sciences (Physics/Chemistry)	26	16.0
3.	Maths/Computer Science	24	14.8
4.	General Engineering	16	9.9
5.	Chemeng Fundamentals	17	10.5
6.	Chemeng Practice	34	21.0
7.	New Technologies	4	2.5
8.	Sustainability	5	3.1
9.	Economics/Business/Management	10	6.1
10.	Capstone Projects	9	5.6
11.	Technical Electives	9	5.6
		<b>162</b>	<b>100.0</b>
12	SIWES	8	
		<b>170</b>	

Table 8. Progression of Course Group Credit Hours Through the Year Levels

SN		Year I (100 level)	Year II (200 level)	Year III (300 level)	Year IV (400 level)	Year V (500 level)	Total
1.	General Studies	4	4	---	---	---	8
2.	Basic Sciences	16	7	3	---	---	26
3.	Maths/Computer Science	11	8	5	---	---	24
4.	General Engineering	4	6	6	---	---	16
5.	Chemeng Fundamentals	1	10	6	---	---	17
6.	Chemeng Practice	---	---	11	12	11	34
7.	New Technologies	---	---	4	---	---	4
8.	Sustainability	---	---		3	2	5
9.	Economics/Business/Management	---	1	1	3	5	10
10.	Capstone Projects	---	---	---	---	9	9
11.	Technical Electives	---	---	---	---	9	9
		36	36	36	18	36	162
	SIWES			2	6		
		36	36	28	24	36	170

### 7.3 Other Considerations

In addition to curriculum reform, Chemical Engineering education in Nigeria can be enhanced through a number of other considerations, such as:

- **Class Size Control** - Class sizes in Chemical Engineering Schools should be limited to a maximum of 50, for proper learning and education. Larger class sizes are unwieldy and highly undesirable. Schools which wish to have large intakes in a year may establish parallel classes in order to keep class numbers in control.
- **Strengthening of Admission Requirement** - The practice of opening admission consideration to candidates with 5 School Certificate credits in two sittings is not helpful and should be discouraged. The intellectual rigour entailed in Chemical Engineering education dictates that candidates should make the credit requirement in one sitting only
- **Resourcing** - Without any doubt adequate staffing and provision of requisite facilities and infrastructure in the Chemical Engineering Schools is imperative for success
- **SIWES Program Reform** - The SIWES Program, as laudable as it is in concept, now seems to be crumbling. Students often do not find placement and even when they find they are not adequately supervised. The situation calls for major reforms and revitalization of the scheme, to reposition it to support the training of competent future Chemical Engineers

### 8. CONCLUSION

Consideration and adoption of new curricula along these lines, as well as the additional measures outlined in this study, should help overcome current deficiencies and should prepare young Nigerian Chemical Engineers for the coming challenges.

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### Appendix

#### Recommended Typical Chemical Engineering

##### YEAR I:

SEMESTER I	CU*	YEAR I: SEMESTER II	CU
Elementary (Basic) Mathematics I	4	Elementary (Basic) Mathematics II	4
General Physics I	3	General Physics II	3
General Physics Lab I	1	General Physics Lab II	1
General Chemistry I	3	General Chemistry II	3
General Chemistry Lab I	1	General Chemistry Lab II	1
Engineer in Society	1	Comp. Science for Engrs.	3
Introduction to ChemEng	1		
Engineering Drawing	2	Workshop Practice	1
General Studies I	2	General Studies II	2
	18		18

\* CU - Credit Units

## YEAR II:

SEMESTER I	CU	SEMESTER II	CU
Engineering Mathematics I	4	Engineering Mathematics II	4
Organic Chemistry I	2	Physical Chemistry I	3
Inorganic Chemistry I	2	ChemEng Thermodynamics I	3
ChemEng Fundamentals (Process Principles)	4	Transport Phenomena I	3
Strength of Materials	3	Mechanics & Machines (Basic Mech. Engrng)	3
Technical Communications I	1	General Studies IV	2
General Studies III	2		
	18		18

## YEAR III:

SEMESTER I	CU	SEMESTER II	CU
Chem Engn Thermodynamics II	3	Unit Operations I (Particulate Systems)	3
Transport Phenomena II	3	Unit Operations II (Fluid/Heat Operations)	3
Basic Electrical Engineering	3	Statistics & Data Analytics for ChEs	3
Physical Chemistry II	2	ChemEng Laboratory I	2
Chemistry Lab	1	Unit Operations III (Separation Processes I)	3
Adv. ChE Analysis/ Numerical	2	Engineering Materials Methods	3
Energy Systems/Technologies	1	Technical Communications II	1
Introduction to Artificial		Intelligence & Machine Learning	2
	18		18
SIWES	2		

## YEAR IV:

SEMESTER I	CU	SEMESTER II	CU
Process Dynamics & Control	2	SIWES	6
Process Economics	2		
Reaction Engineering	3		
Unit Operations IV (Separation Processes II)	3		
Project Management	1		
ChE Laboratory II	2		
Process Safety & Loss Prevention	3		
Process Simulation & Modelling	2		
	18		6

## YEAR V:

SEMESTER I	CU	SEMESTER II	CU
Plant/ Process Design Principles	3	Design Project	3
Product Design Principles	2	Research Project II	3
Research Project I	3	Entrepreneurship & Innovation	2
Process Instrumentation	2	Industrial/Business Management	3
Environmental ChE	2	Technical Elective II	3
Industrial Chemistry/Process Industries	3	Technical Elective III	3
Technical Elective I	3	ChemEng Practice & Ethics	1
	18		18



## ANALYSIS OF BARYTE FROM AZARA DEVELOPMENT AREA IN AWE LGA OF NASSARAWA STATE, NIGERIA

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### ABSTRACT

*Baryte serves as raw materials in the petroleum and chemical industries for production of several end-products. Its applications are based on its physiochemical and mineralogical characteristics. Still, the primary use of baryte is as a weighting agent for oil drilling operations. In spite of Nigeria's huge baryte reserves, the multinational oil companies operating in Nigeria neglect its use on the grounds that Nigerian baryte have lower quality compared to imported baryte. This study analyses the physiochemical and mineralogical properties of Azara baryte, located in Nassarawa State, Nigeria. These parameters were examined vis a vis the American Petroleum Institute (API) standards. Results of the XRF analyses reveals that the baryte sample has a BaO content of 8.595%, and SO<sub>3</sub> of 7.45%; Thus, equivalent to 15.885% BaSO<sub>4</sub> (BaO+SO<sub>3</sub>). Physiochemical analyses indicate that while the moisture content (0.02), pH (7.2), and metallic contents, were within API specification for drilling, its low specific gravity value of 3.45g/cm<sup>3</sup>, makes it unacceptable for drilling operations, as it compares unfavourably with the API standards. Mineralogical analysis shows quartz as the most predominant associated mineral. These results indicate the sample's suitability for paper, glass, paint, and rubber, production. However, it requires further processing to meet globally acceptable specifications for drilling operation.*

*Keywords: Baryte; Specific gravity; XRF; XRD; Azara.*

### 1. INTRODUCTION

Baryte is the natural barium sulphate (BaSO<sub>4</sub>) in its natural form, produced primary from baryte deposits. Baryte, which is readily found in limestone, lead and zinc veins, hematite ore and hot spring deposits are also repeatedly combined with gangue minerals such as arnglesite and celestine (Fatoye et al., 2014). In terms of usage, baryte is an essential industrial mineral used worldwide in oil and gas drilling operations. The global baryte estimate of 80% is used as a weighing agent in oil drilling operation. For that reason, baryte is often referred to as; drilling mud or heavy mud. Baryte equally serves as a source of raw mineral for barium-based chemicals such as glass, textiles, paints, rubber, explosives, brake-pads and radiation shields (Dibello et al. 2003).

The economical, efficient and resourceful use of barytes depends on its mineralogical and physiochemical characteristics and properties comparable to globally acceptable specification/standards. Naturally, baryte is brittle with a clear white colour. However, the presence of impurities or gangue minerals such as; hydroxides, sulphides and iron oxides, often results in the variation of its natural white colour to a host of colours, ranging

from brown, yellow, red etc. With a high value in specific gravity of about 4.2-4.5g/cm<sup>3</sup> baryte serves comfortably as a weighting agent in the formulation of drilling fluid for drilling operations. In addition, baryte is crystalline with tabular, prismatic, or radiating bladed shape; possess a low solubility, making it virtually insoluble in water, acid and most chemical solvents; it is non-toxic, as well as chemically and physically inert (Mathew, et al. 1971; Wood, 2005).

Though the international oil companies (IOC's) operating in Nigeria are largely dependent on imported baryte, the present drive in Nigeria's government's oil and gas industry local content policy, has made it necessary to assess the quality of the local Nigerian baryte deposits. According to Mgbemere, (2018), the international oil companies (IOC's) operating in Nigeria, import their baryte from multinational companies like Schlumberger and Halliburton. This importation translates to a loss of over ₦5billion annually in foreign exchange Association of Miners and Processors of baryte (AMAPOB, 2018).

In Abdullahi, and Lawal, (2010) research on the determination of the constituents and suitability of Azara

barytes from Nasarawa State Nigeria, the emphasis of the analyses, was on the chemical and elemental constituents of the Azara baryte sample. The mineralogical analyses were practically omitted.

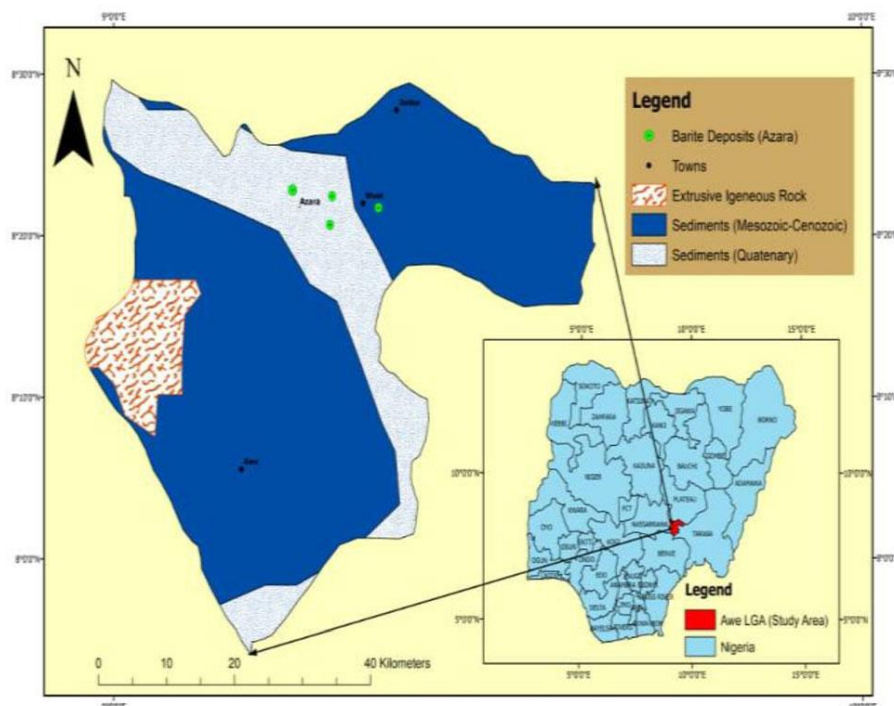
In contrast to Abdullahi, and Lawal, (2010) work, Salahudeen, et al., (2020) study on the characterisation of Azara baryte, focused on its mineralogical properties and application of baryte waste for drilling mud use.

Afolayan, (2021) carried out the characterisation of Nigerian baryte for drilling mud formulation. The baryte samples were taken from Torkula baryte deposit is located in Guma local government area of Benue State in Nigeria. In following up with Abdullahi, and Lawal, (2010) and Afolayan (2021) researches, this work seeks to characterise Azara baryte in other to determine the physiochemical and mineralogical fitness for use as drilling mud and other industrial applications.

## 2. MATERIALS AND METHODS

### 2.1 STUDY AREA DESCRIPTION

Azara (8.3661° N, 9.2491° E), which is located in Awe local government area of Nassarawa state of Nigeria, has an estimated baryte ore reserve of roughly 731,000 tonnes with about 71,000 tonnes of barytes (Obi et al., 2014). The Azara baryte deposit (see Figure 1) is found at roughly 98km south of the capital-Lafia. The baryte deposit which originates from the Benue valley system can be termed as a vein and cavity filling deposit, housing an estimated number of 18 veins. Precipitation from hot barium enriched fields in fractures and faults resulting in the mixing of fluid at reduced temperature or pressure, results in the formation of the baryte deposit. According to Smith (1978), the fluid often dissolves the surrounding host rocks resulting in the formation of irregular replacement of baryte deposits.



**Figure 1: Map of Nasarawa State of Nigeria with the study area in sight (red legend)  
(Source: Kolawole, et al., 2021).**

### 2.2 SAMPLE COLLECTION

About 7000g of Azara baryte ore sample was collected from Qualchem Global Limited, Port Harcourt in Rivers State, Nigeria. The collected sample was cleaned and then placed in polythene bags and sealed to prevent loss of moisture and transported to the PTDF Bio-oil research laboratory ETF Building, Abuja campus, University of Port Harcourt, Rivers State for further analyses.

A portion of the sample was first crushed manually using a locally fabricated hammer mill into large pieces before being subjected to further crushing and milling actions, using a combination of grinding and scrubbing actions with the cheek of the hammer mill. These actions transformed the baryte pieces into fine powder of 75µm or less in size. Figure 2 shows the pulverized sample of Azara baryte after crushing.



Figure 2. Pulverized sample of baryte from Azara

### 2.3 PHYSICAL ANALYSES

Specific gravity, pH value, hardness, moisture content and loss on ignition were the physical analyses carried out on the sample. The specific gravity was determined using the Le Chatelier Flask Method, while the XRF and XRD analysis were used for the elemental and mineralogical determination.

#### 2.3.1 SPECIFIC GRAVITY DETERMINATION

The specific gravity was determined by weighing 80g of dried baryte with a weighing balance and carefully transferred into the Le Chatelier flask filled with kerosene. The mass was recorded, in grams, as  $m$ . The neck of the flask was carefully tap with the hands to dislodge any baryte clinging to the walls without allowing kerosene to come into contact with the glass stopper joint of the flask. The upright flask at the neck was vigorously twirled between the palms of both hands to remove entrained air from the baryte sample. This procedure was repeated until no more bubbles can be seen rising from the baryte. The flask was returned to the bath and left standing for 30 min. The flask was then removed from the bath, and the previous steps repeated to remove any remaining air from the flask. The flask was then immersed in the bath again for an hour. The final volume was recorded in milliliters as  $V_2$ , and the initial volume  $V_1$ . The baryte density,  $\rho$ , was calculated in grams per milliliter, according to Equation (1):

$$\rho = \frac{m}{V_2 - V_1} \quad (1)$$

Where,

$m$  is the sample mass, expressed in grams;  $V_1$  and  $V_2$  are the initial volume, and final volumes, respectively, expressed in milliliters.

#### 2.3.2 DETERMINATION OF pH

10grammes of the sieved baryte sample- placed in a beaker with 20ml of distilled water. The formed slurry was kept standing for an estimated period of one hour, after which it was stirred for about 5-10 minutes in order to estimate the pH value of the slurry (Ibe et al. 2016; Afolayan, 2021). The pH value was then measured repeatedly after a period of 24hrs (Washington State Department of Transportation (WSDT) 2009; Ibe et al. 2016).

#### 2.3.3 GRAIN SIZE DETERMINATION

Approximately 1100g of the 2 powdered baryte samples (obtained from the dried, crushed and grinded baryte ores) was cooled to room temperature in a desiccator. The samples were transferred in batches to a 75 $\mu$ m sieve. A small brush was used to agitate the baryte particles to ensure that particles of sizes below 75 $\mu$ m passes through the 75 $\mu$ m sieve into a clean pan positioned below the sieve. The baryte powder remaining on the sieve was transferred to a second clean pan. The process was repeated until all the baryte powder had been sieved. The total sieved baryte powder (less than 75 $\mu$ m) was added and weighed.

The mass fraction of baryte powder greater than 75 $\mu$ m,  $w_1$ , in percent, according to Equation (2):

$$w_1 = 100 \left( \frac{m_2}{m_1} \right) \quad (2)$$

Where,

$m_1$  is the sample mass, expressed in grams;  $m_2$  is the powder mass retained by 75 $\mu$ m sieve, expressed in grams.

#### 2.3.4 MOISTURE CONTENT

Samples of 30, 30, and 40 g of the 110g of 75µm baryte were introduced into a dry and clean crucible after weighing with a weighing balance. The sample was then carefully placed into a muffle furnace, at a temperature of 110°C. The samples were constantly removed from the muffle furnace at an interval of 30minutes and reweighed, until a constant weight was obtained (Ibe et al., 2016). After, roughly 3hours of heating, the samples were permanently removed from the muffle furnace and placed in a desiccator, to cool for an estimated 3hours. The baryte samples were then measured and calculated as the dried weight of the samples.

To determine the effect of long-time storage on the moisture content of baryte samples, some of the sample was left in the desiccator for a period of 30days. After which it was removed and reweighed with a weighing balance.

### 2.3.5 MOHR'S HARDNESS

The scratch test was used to determine the hardness of the un-pulverised form of the Azara baryte sample. This scratch test technique is based on relating the hardness of a mineral sample to the harness of commonly known items using the Mohr's hardness scale (Alan 1997). The Mohr's scale applies known reference objects of different hardness from 1 to 10 (in ascending order of hardness). Between the test material and reference mineral, the one that is able to leave scratch marks on the other, is termed the harder of the two. The sample was scratched with using the fingernail, and paper clip as test materials. The results were then appraised using Mohr's scale to determine the hardness of the baryte sample (Ibe et al. 2016; Omoniyi and Mubarak 2014).

### 2.3.6 DETERMINATION OF SAMPLE STREAK

The streak test was evaluated by scraping a portion of the un-pulverised baryte sample across a piece of ceramic pate, which served as the streak plate. The ceramic plate was firmly held with the left-hand while the scraping action was done. A tiny quantity of powdery substance known as "streak" was produced on the ceramic plate as a result of the streak test. The colour and powdery nature of the streak was determined by observation and touch of the test sample. The Streak test was repeated thrice as a confirmatory test.

### 2.3.7 LOSS ON IGNITION (LOI)

1g of the baryte sample was weighed and carefully poured into a porcelain crucible. The crucible was then carefully put in a muffle furnace. The temperature of the muffle furnace was gently increased from 320°C to

850°C and sustained for an estimated 2 hours. After the heating period (2hrs), the crucible was gently taken out of the muffle furnace, cooled in a desiccator and weighed with a weighing balance. After a period of approximately 10mins, the crucible was returned into the muffle furnace at 850°C for another 10 minutes and after which it was reweighed. The later process was repeated until a constant weight was achieved (Rowell, 1994). Using equation (3) below, the Loss on Ignition (LOI) was obtained.

$$LOI = \frac{M-m}{M} \times 100 \quad (3)$$

Where:  $M$  = mass of sample (g) and  $m$  = mass of sample after ignition (g)

## 2.4 CHEMICAL COMPOSITION ANALYSIS

The chemical composition in the baryte sample was determined using X-ray fluorescent XRF analysis at Spectral Laboratory Services, Kaduna State, Nigeria. After powering on the XRF Nitron 3000 the machine was allowed to stabilised for 5 minutes after initialization. The use of the Cu-Zn method is its ability to detect a greater number of sesquioxides as well as elements as a result of its intensity. After placing the baryte sample on the sample holder, the ray point was then positioned over it. To begin taking data the ray button was powered on. The data were obtained in triplicates and this automatically takes its mean value.

## 2.5 MINERALOGICAL ANALYSIS

An XRD EMPYREAN MALVERN PANALYTICAL diffractometer machine was utilised for the mineralogical analyses. The machine works by using the X-ray diffraction (XRD) technique. The process begins by powering the machine from the panel, before adjusting the current and voltage values to read 40mA and 45kV respectively. The temperature of the diffractometer was then adjusted to 21- 23°C. After a double lick of the "TUMI" (that is, the XRD software), The XRD empyrean malvern Panalytical diffractometer system was activated. To endure that all necessary settings relating to temperature and power matched those of the XRD, the dialogue box setting of the machine was clicked and compared with that of the XRD. The Azara baryte sample was carefully transferred into the machine's sample holder and carefully put in the sample chamber column. The sample chamber column was fully closed as confirmed by an indicator on the computer. The settings measurement knob was fixed for scan axis at "Gonio", "start" and "end" positions. The time and angle of scan were



equally set. The scanning process started and ended at the set time. Results at the end of the scan were further saved to a file. The result obtained was then matched with different library, such as the NIST and PubChem in order to get the chemical structure, name, and other physicochemical properties.

### 3. RESULTS AND DISCUSSIONS

#### 3.1 PHYSICAL PROPERTIES

The physical properties of the Azara baryte sample are shown in Table 1.

**Table 1: Physical properties of Azara baryte**

	Azara baryte	API Standard (Ibe, 2016; Afolayan, 2021)
Specific gravity	3.45	4.20
Moisture Content	0.02	0.02
pH value	7.2	7-12.5
Hardness	3.5	3.0-4.0
Streak test	White	White
Loss of Ignition	1.96%	0.5

#### MOISTURE CONTENT

Moisture content of Azara baryte sample shows the differential weight loss at 0.02%. After drying at a temperature of 110°C for a cumulative time of 12hrs, the average weight of the baryte slurry was 0.02g indicating a near total loss of moisture in the baryte. It should be noted that, the baryte sample was taken from the muffle furnace at a regular interval of 45mins and weighed using a weighing balance until 0.02g net weight loss was obtained, thus signifying the near absence of moisture in the Azara baryte. When compared with the API specification for moisture content, the result of the analysed baryte sample fell within the 1% maximum mark given by the API standard. According to Afolayan (2021), if the baryte moisture content is higher than the 1% API mark, it can lead to a breakdown or collapse of the borehole resulting from varying conditions of the downhole conditions-a product of the adverse consequence on the density of the mud. Previous researches carried out by Dhiman (2012); Osokogwu et al. (2014) and Ibrahim et al. (2017) resulted in related findings.

The effects of long-time storage of the baryte sample on its moisture content showed no significant change in the moisture content of the Azara baryte samples after the drying. This was probably as a result of storing the dried baryte particles in a desiccator. This result somewhat agrees with Afolayan (2021) assertion that long-term storage has a negligible effect on the properties of barytes which have undergone processing.

#### pH VALUE

The results in Table 1 shows that sample was basically neutral in the pH value. The results ascertain that the pH of the Azara samples was within standard API range of

drilling weighting agent (7-12.5) (Christ, 2006; Osokogwu et al. 2014; Ádewale and Salihu, 2014).

#### HARDNESS

The sample recorded a mean value 3.5 using the of Mohr's hardness scale. The hardness value can be attributed to the nature of the gangue or associated minerals in the baryte sample (as reflected in the varying colours and texture of sample). The practical implication of the Azara baryte sample having a Mohr's hardness between 3.5 means that it can satisfactorily give protection to the drilling bit as well as safeguard the circulation system oil and gas drilling operations. Furthermore, processing of baryte to eliminate its associated (gangue minerals) usually enhance improve its hardness (Afolayan, 2021). Failure to address the elimination of the gangue minerals before the commencement of drilling or cementing operation, results in an enormous increase in the wear debris as a result of an increase in the wear rate during drilling operations.

#### STREAK

The streak test resulted in a baryte powder with a white colour. This agreed with similar results obtained by Bassey, et al (2021).

#### LOSS OF IGNITION

The loss of ignition was approximately 1.96wt%. This value is above the maximum benchmark of 1.0% required for the formulation of drilling mud as given by the API standard.

#### 3.2 MINERALOGICAL ANALYSIS BY X-RAY DIFFRACTION (XRD)

The X-ray diffractogram of the Azara baryte sample as

shown in the qualitative and quantitative analysis report in Table 2, as well as figures 3 and 4, respectively, reveals the composition and crystallographic information of the Azara baryte sample respectively. The XRD identified sharp peaks in the XRD patterns of the Azara baryte sample showing the presence of a highly crystalline microstructure of the sample. The macrostructure of Azara baryte is expected to impact high acuity on the resulting drilling fluid as a result of sharp particle edges, provided by its high crystallinity microstructure. From the analysis of the XRD patterns of the Azara baryte sample as shown in figure 3, the highest peak value was from the quartz mineral at 28.45, 2 $\theta$  with 10100 cps intensity. The figures also revealed the presence of other lower peak values of quartz as well as several peaks values of baryte and other gangue minerals in trace proportion such as; garnet (8%), gypsum (6%), orthoclase (4%), davyne (1.4%) and Illite. Summarily, the overall level of gangue mineral (impurities) in the Azara baryte was high. This is in line with reports given in the literature Tanko et al. 2015; Aladesanmi et al. 2018; Mgbemere et al. 2018a, b).

The qualitative analysis result of the sample as given in Table 2 confirms the presence of; baryte, quartz, orthoclase, muscovite, gypsum, davyne, Illite and garnet as the mineral phases present in the analyzed sample. The presence of quartz, baryte, gypsum, orthoclase, davyne, and illite. Cronan, (1969), affirms that presence of gypsum, orthoclase, davyne, Illite and quartz is as a result of the association and occurrence of baryte in silicate carbonate rocks and siltstone-sandstone sequences. The presence of baryte has equally been located in residual deposits from weathered baryte-bearing limestone; low-temperature hydrothermal veins; an as an accessory mineral in igneous rocks; and in carbonatites. The prevalent impurities from the Azara samples are quartz and garnet which correlates the occurrence and association of the barytes to silicate carbonate rocks. According to Moore and Reynolds,

(1989), this is because quartz is by far the most common of the silica minerals in the sedimentary rocks, and its diffraction lines can be used as internal standard for the accurate and precise measurement for the inter-planar spacing. Quartz presence in baryte ore reduces its specific gravity and also affect its brightness and colour. Purer baryte range from colourless to white in colour and have a brighter appearance. The Azara barite sample was light brown in colour and brightness (due to the presence of the gangue minerals or impurities), Thus, making them less useful in the production of paints and medical applications.

The presence of quartz, which is a low temperature mineral in baryte, coupled with the coarse-grained texture of the baryte means that there is a possible relationship of the baryte formation process to low temperature (Offodile, 1976; Tanko et al., 2015). The invariable association of baryte with quartz, further suggest that the baryte veins of the studied area appear to have been formed during the later stage of the Cretaceous (Offodile, 1976; Tanko et al., 2015).

However, the presence of quartz can be separated from baryte samples using any of the gravity separation methods (Wang et al, 2014) as well as through bioleaching with the microbe, *Bacillus licheniformis* as a bio-collector (Wang et al, 2014; Ashkavandi, et al., 2022).

The pie chart representation of the quantitative result of the sample as shown in figure 4 further indicates that the gangue oxide quartz or silica oxide (SiO<sub>2</sub>), and sulfate of barium (BaSO<sub>4</sub>) are the most dominant compounds at 45% and 29% respectively. Other gangue minerals present in the sample include; garnet, gypsum orthoclase, davyne, illite and muscovite.

**Table 2: Qualitative Analysis Result of the Azara baryte Sample**

Phase name	Formula	Figure of merit
Baryte	Ba S O <sub>4</sub>	2.078
Quartz	Si O <sub>2</sub>	1.511
Orthoclase	Al <sub>2</sub> O <sub>3</sub> . K <sub>2</sub> O .6 Si O <sub>2</sub>	3.302
Muscovite	K Al <sub>2</sub> (Si <sub>3</sub> Al) O <sub>10</sub> (0 H, F) <sub>2</sub>	3.189
Gypsum	Ca S O <sub>4</sub> -2 H <sub>2</sub> O	3.273
Davyne	K <sub>2</sub> Na <sub>4</sub> Ca <sub>2</sub> Al <sub>6</sub> Si <sub>6</sub> O <sub>24</sub> Cl <sub>2</sub> ...	3.237
Illite	K (AlFe) <sub>2</sub> AlSi <sub>3</sub> O <sub>10</sub> (0 H...	2.837
Garnet	3 (Ca, Fe, Mg) O ■ (Al, Fe...	3.067

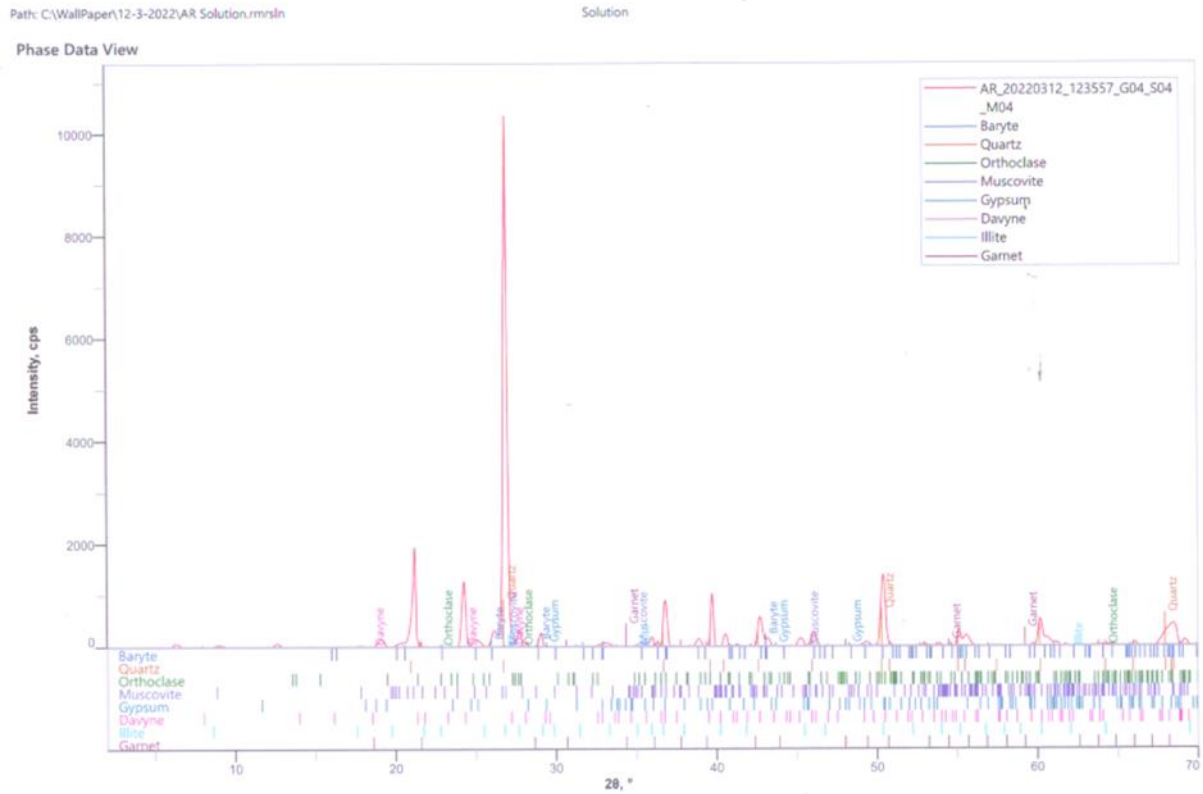


Figure 3: XRD patterns of the Azara baryte sample (Quantitative analysis report)

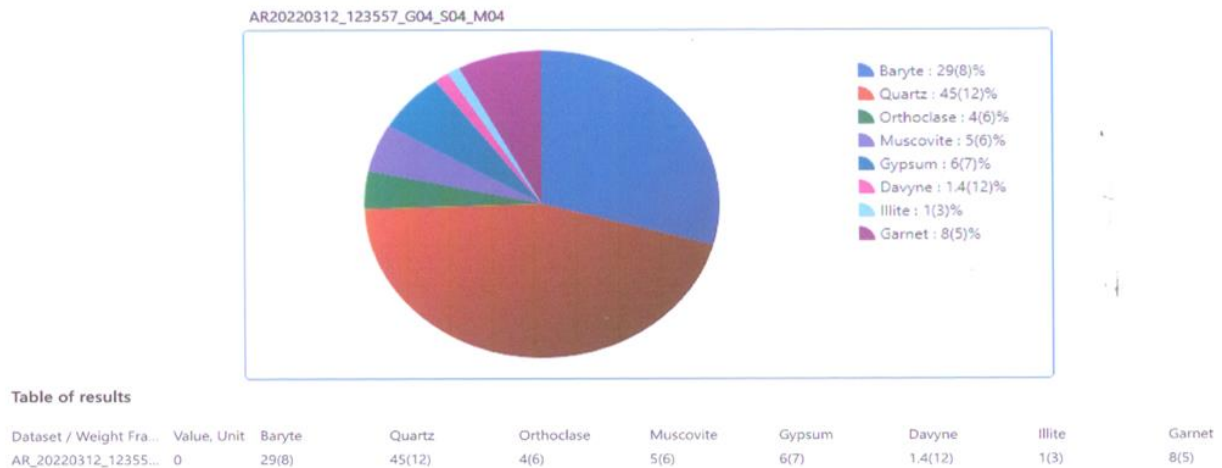


Figure 4: Pie chart representation of the XRD analysis result

### 3.3 CHEMICAL OXIDE CONTENT AND METALLIC CONTENT OF THE AZARA BARYTE BY XRF ANALYSES

The X-ray fluorescent XRF of the Azara baryte sample as shown in Table 3 and 4 gives the respective API

chemical oxide content and metallic content of the baryte compared with the standard baryte composition for use as a drilling mud.

**Table 3: Chemical Oxide Content of Azara Baryte Sample compared with Standard Baryte Composition for use as a drilling mud**

Composition	Azara Baryte (%)	API specification (API 2010) (%)
*BaSO <sub>4</sub>	15.885	95.00
Fe <sub>2</sub> O <sub>3</sub>	2.820	0.03
CaO	0.250	0.01
MgO	0.000	0.04
Al <sub>2</sub> O <sub>3</sub>	5.655	0.035
Silicate	32.990	0.01
Total heavy metals	ND	0.01
Water-soluble salt	ND	0.001
Specific gravity	3.45	4.2

N/B: The value was obtained by adding the BaO and SO<sub>3</sub> values as indicated in the XRF report.

BaSO<sub>4</sub> (15.885wt %) = BaO (8.595wt %) + SO<sub>3</sub> (7.290wt %)

\*ND: Not determined

**Table 4: metallic content of the Azara baryte for use as a drilling mud**

Metal	Concentration (mg/kg)	
	Azara Baryte	API maximum limit
Ca	0.179	250
Mg	0.000	250
Fe	1.973	Nil
Zn	0.001	140
Cu	0.047	36
Co	0.017	1000
Cd	-	5

Tables 3 and 4 indicates that the Azara baryte sample had an average specific gravity of 3.45. The average oxide values of BaSO<sub>4</sub> (BaO 8.595wt%+SO<sub>3</sub> 7.290wt% = BaSO<sub>4</sub> 15.885wt %), CaO (0.250wt %), MgO (0.00wt %), SiO<sub>2</sub> (70.576wt %), Fe<sub>2</sub>O<sub>3</sub> (2.820wt %), Al<sub>2</sub>O<sub>3</sub> (5.655wt %), K<sub>2</sub>O (2.697wt %). The combined BaSO<sub>4</sub> value of 15.885wt% placed Azara baryte far below the minimum required value of 96wt% which is required for any industrial application. The Fe<sub>2</sub>O<sub>3</sub> of 2.820wt% is far above the minimum of 0.03wt% needed for oil and gas operations. The high Fe<sub>2</sub>O<sub>3</sub> content can be said to be responsible for the grey colouration of the Azara baryte sample. The water-soluble element CaO is much higher than the API threshold of 0.01, indicating poor grade of baryte for drilling mud formulation. The concentrations of Al<sub>2</sub>O<sub>3</sub> is well above the API mark of 0.035, further indicating the inability of the baryte sample to be used in drilling purposes. The SiO<sub>2</sub> content of 70.576wt% is greatly beyond the API acceptable maximum limit of 0.01wt%. The specific gravity of 3.45g/cm<sup>3</sup> is well below the API requirement of 4.2g/cm<sup>3</sup>. However, the MgO content of 0.000wt. % is within the API tolerable limit of 0.04wt%. Thus, while the Azara baryte cannot

be used for the formulation of drilling mud (used in oil and gas operations), it is fitting for the production of papers of low-quality, fillers for rubber as well as sodalime glasses (Abudllahi and Lawal, 2010).

Furthermore, results of the metallic content of the Azara sample, shows that the element; Ca, Mg, Zn, Cd, Fe, Cu and Cd were all within API acceptable threshold for drilling use. The result equally shows an absence of Pb- a familiar metal in baryte samples (Afolayan, et. al 2021).

#### 4. CONCLUSION

The Azara baryte sample physiochemical analyses, obtained for Azara Development Area in Awe Local Government Council of Nasarawa State Nigeria, shows that the pH value, (7.2) hardness (3.5), moisture content (0.02%) as well as the streak test, meets the requirement for the formulation of drilling mud. However, the very low BaSO<sub>4</sub> content of 15.88wt% as well as other critical parameters such as its specific gravity (3.45), loss of ignition (1.96wt.%), makes it highly unsuitable for use



in drilling operation. The low specific gravity of the sample can be attributed to the high level of quartz (45%), as the presence of quartz in baryte ore has been known to reduce baryte specific gravity. Furthermore, the brown colouration of the Azara barytes samples is due to the high Fe<sub>2</sub>O<sub>3</sub> content (2.82 wt. %), as well as the presence of the gangue minerals; garnet, gypsum, orthoclase, davyne, illite and muscovite. These gangue minerals further lowered its brightness, thus making it less useful in the production of paints and medical applications. Thus, the Azara baryte sample can be best use in production of rubber (paper industries) as fillers or extenders and paper industries. Consequently, the Azara baryte sample needs to undergo processing operations in order to make it acceptable for use in the formulation of drilling mud for the oil and gas industry, paint production and medical applications.

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## LITHIUM EXTRACTION FROM SPENT LITHIUM-ION BATTERIES WITH GREEN SOLVENTS: COSMO-RS MODELING

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### ABSTRACT

*Lithium-ion batteries (LIBs) wide usage constitutes a disposal threat to the environment. As a result, several laws are being introduced to encourage the recycling of this waste, particularly, in lithium recovery. Deep eutectic solvent (DES) has been reported as an efficient solvent in valuable metal recovery from spent LIB. However, efficient deep eutectic solvent design requires a smart selection of components. This study developed a COSMO-RS model to screen several components as DES starting material in lithium extraction from spent LIB. The model consists of 188 different constituents. The model is developed using the cosmo therm software in the LIB application for the first time. The model uses lithium chemical potential to measure the affinity of lithium in the screened components. Overall, all the compounds show an affinity for lithium. The components are classified into ionic and non-ionic. The ionic compounds performed better than the non-ionic compounds. This is due to the coordinating ability of the ionic compounds' cations with lithium. Further, this study highlights other properties such as reducibility, toxicity, and viscosity as screening strategies in DES component selection for lithium extraction. This is to implement the full green chemistry principle essential for industrial application.*

*Keywords: Lithium-ion battery; Lithium; green technology; Deep eutectic solvents; COSMO-RS.*

### 1. INTRODUCTION

Lithium possesses excellent properties such as high redox potential, specific heat capacity, and charge-to-weight ratio. This enables its wide use as an important element in several industries such as energy security, environmental safety, daily life, industrial consumption, and mental health treatment (Swain, 2017). Among the end consumer use of lithium, lithium-ion batteries (LIBs) have the largest lithium market share value worldwide. This is owing to the increasing usage of LIBs for various applications such as portable electronic devices, stationary storage devices, and hybrid electric vehicles. It is estimated that the market value of LIBs would reach \$99.98 billion by the year 2030 (Fan et al., 2020). This poses a major disposal threat to the environment. Moreover, the recovery of lithium from LIB could decrease the reliance on environmentally harmful mining and dependence on geographically unstable countries. Therefore, recovery of lithium from

spent LIBs ensures twin benefits of conserving spent LIB and sustaining lithium production.

Among the spent LIB recycling technology, Hydrometallurgy is a promising lithium production technique. This is due to its low energy requirements and high recovery rate. Unfortunately, this technique is limited by toxic and environmentally harmful solvents such as sulfuric, nitric, and hydrochloric acid in the leaching operation (C. Liu et al., 2019). Hence, the need for a green solvent.

Deep eutectic solvent (DES) arises as a green solvent for efficient leaching of LIB cathode materials. Since their discovery in 2003, several studies have been published on this green solvent for myriads of applications (Ramón & Guillena, 2019). The use of DES as a cathode material leaching agent started in 2019 when Tran et al. (Tran et al., 2019) used DES as a leaching agent for spent LIB cathode material. Since then, more studies

have emerged in this area (Peeters et al., 2020; Roldán-Ruiz et al., 2020; Tang et al., 2022). Deep eutectic solvents are obtained in the laboratory by incorporation of two or more components that exit as eutectic (Abbott et al., 2006). This eutectic has a lower melting point than the primary compounds that make up the eutectic. Owing to their designers' nature, there are vast possible combinations of the DES constituents. Therefore, it is reasonable to devise a means to efficiently screen the solvent base on their affinity for metal recovery from spent LIB cathode materials. This is to save researchers valuable time and experiment costs. In this regard, the application of computational models and molecular-level chemistry analyses is pertinent to suggest potential deep eutectic solvents for lithium extraction from spent LIBs.

The Conductor-like screening model for realistic solvation (COSMO-RS) is a method based on quantum chemistry and a statistical model to screen solvents for different applications (Hou et al., 2016). For example, Adeyemi et al used COSMO-RS to screen several probable deep eutectic solvents for CO<sub>2</sub> capture (Adeyemi et al., 2018). It has also been used as a solvent screening tool in several liquid-liquid extraction processes (Jiang et al., 2021; Khan et al., 2021; Ozturk & Gonzalez-Miquel, 2019). In metal extraction, the use of COSMO-RS has been reported in a few studies. In one instance, Zhao et al. (Zhao et al., 2018) utilized COSMO-RS to predict and screen probable ionic liquids (ILs) constituents that show an affinity for lithium from brine. In the study, the model was used to optimize the probable IL in extracting Li. In another investigation, Olea et al. (Olea et al., 2022) used COSMO-RS to propose new ILs based on the experimental findings and correlations with COSMO-RS prediction. The two mentioned studies were based on lithium production from brine. To the best of the authors' knowledge, no prior study has been carried out on the use of COSMO-RS to predict and design deep eutectic solvents in lithium extraction from spent LIB.

Thus, this work aims to use COSMO-RS to screen several DES constituents for lithium extraction from spent LIB. The chemical potential of lithium was obtained by sigma profile analyses. Further, selected best DES constituents with strong affinity would be used to design novel DES over different parameters such as toxicity, viscosity, reducibility, and ability to form chelating compounds with spent LIB cathode material metals.

## 2. MATERIALS AND METHODS

The COSMO-RS workflow is in two phases (Fig 1.) 1. COSMO (uses quantum chemistry programs like TURBOMOLE) and 2. COSMO-RS (COSMOtherm). The COSMO surface can be used to qualitatively determine molecular properties such as activity coefficient at infinite dilution and chemical potential. Since Li is a cation, the model works to determine the chemical potential of lithium on DES constituents. Detailed information on the COSMO-RS working procedures can be found elsewhere (Adeyemi et al., 2018).

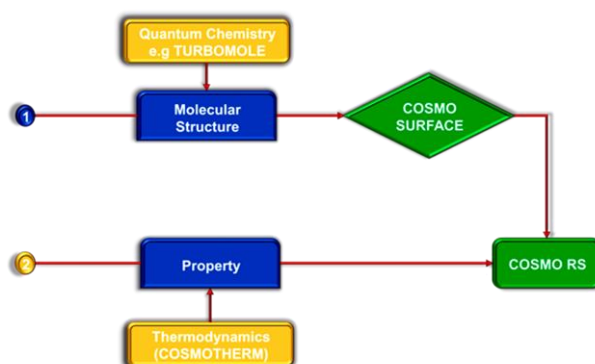


Figure 1: COSMO-RS workflow

### 2.1 Computation details

To develop the COSMO-RS model used in this study, the Turbomole software version TmoleX19 4.5.1 was first utilized to draw the geometry of each DES component. Subsequently, the density functional theory (DFT) calculations were used to optimize the geometrical structures of the components and create a cosmo file. The created cosmo file was then imported into the 2022 BIOVA COSMOtherm version software. The software assumes that each surface structure consists of a segment area  $a_i$  and characterizes the interaction between molecules as hydrogen bond interactions, van der waal interactions, or having surface charge densities with misfit electrostatic charge. In COSMO, the interaction is expressed mathematically as follows (Eqn 1 -4):

$$\varepsilon_{vdW} = a_{eff} \cdot (\tau_{vdW} + \tau'_{vdW}) \quad (1)$$

$$\varepsilon_{hb} = a_{eff} \cdot c_{hb} \min(0; \min(0; \sigma_{donor} + \sigma_{hb}) \max(0; \sigma_{acceptor} + \sigma_{hb})) \quad (2)$$

$$\varepsilon_{mf} = a_{eff} \cdot \frac{\alpha'}{2} (\sigma + \sigma')^2 \quad (3)$$

$$\mu_S(\sigma) = \frac{-RT}{a_{eff}} \ln \left[ \int P_S(\sigma') \exp \left( \frac{a_{eff}}{RT} (\mu_S(\sigma') - \varepsilon_{mf} - \varepsilon_{hb} - \varepsilon_{vdW}) \right) d\sigma' \right] \quad (4)$$

Where ( $\mu_S$ ) is the DES constituents potential(kJ/g),  $\sigma$  and  $\sigma'$  represents the two surface segments( $e/\text{nm}^2$ )  $a_{eff}$  is the area of effective contact( $\text{nm}^2$ ),  $\tau_{vdW}$  is the strength coefficient of dispersion,  $\sigma_{hb}$  is the screening charge density threshold for hydrogen bonding (set to  $0.008 \text{ e}/\text{\AA}^2$  by default),  $c_{hb}$  is the strength coefficient of hydrogen bonding,  $\sigma_{donor}$  and  $\sigma_{acceptor}$  are the surfaces of hydrogen bond donor and acceptor ( $e/\text{nm}^2$ ),  $\alpha'$  is a general interaction fitting parameter, and  $P_S$  the screening charge density distribution ( $e/\text{\AA}^2$ ).

### 3. RESULTS AND DISCUSSION

#### 3.1 COSMO-RS analyses

COSMO-RS analyses are based on two main principles—the signal profile, and the DFT structure of the molecules. The signal profile represents the degree to which a molecular surface segment gives a positive, negative, or neutral charge. Essentially, the signal profile provides information on the types of interaction in molecules. This is represented through the height, width, and location of the peaks in the signal profile. Fundamentally, the signal profile categorized molecules into three groups: region of positive polarity, negative polarity, and neutral surfaces. Further detail on the significance of the signal profile can be found elsewhere(Lemaoui, Darwish, Attoui, et al., 2020; Lemaoui, Darwish, El, et al., 2020). The signal profile also reveals which constituents make a hydrogen bond donor or acceptor. Compounds that give peaks on both sides can act as both hydrogen bond acceptors (HBAs) and hydrogen bond donors (HBDs). The signal potential reinforces the signal profile results. Negative potential indicates HBA while positive potential reveals HBD.

#### 3.2 Classification into ionic and non-ionic species

Figure 2 shows the dissolution of all the studied DES constituents (numbered 1 to 188 in descending order of Li chemical potential) on lithium benchmarked against water. Overall, all the constituents can dissolve lithium. The screened constituents are broadly classified into ionic (HBAs) and non-ionic (HBDs). As shown in the figure, both the hydrogen bond donors and hydrogen bond acceptors can dissolve lithium. Therefore, both constituents can influence the performances of the

resulting DESs in the lithium leaching process(Y. Wang et al., 2016). The ionic compounds generally perform better than the non-ionic compound in their ability to dissolve lithium. This may be due to the presence of different anions and cations which aids the formation of coordinating compounds between the ionic compound and lithium. The next section expatiates on the influence of the different anions and cations in the ionic compounds.

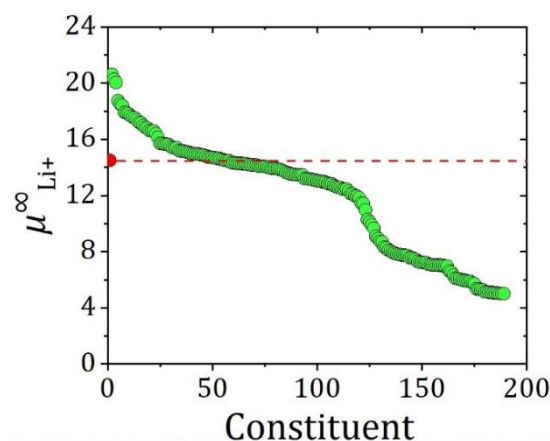


Figure 2: Chemical potential of Li on DES constituents

#### 3.3 Influence of HBAs

The HBAs studied in this work are mainly based on organic salts, and natural-based HBAs (Table S1 of the supplementary document). Regarding quaternary-based compounds, the central atom in the HBA cation plays a crucial role in their extractability. Generally, the order of chemical affinity is higher in ammonium-based compared to phosphonium-based compounds. Several reasons may be the outcome of this observation. It was reported that phosphonium-based compounds showed higher densities than ammonium-based compounds(Bahadori et al., 2013; Wazeer et al., 2021). This higher density translates to a lower leaching rate and hence lower efficiency. In addition, the chemical structure of the HBA also affects their dissolution rate. This is because the chemical structure of the HBAs relates to their charge density. Consequently, this influences their binding energies. For example, the charge density in the P=O group is influenced by the presence of radicals in the vicinity of the energy bonding(Blundell & Licence, 2014). The high performance of the ammonium-based HBAs may also be ascribed to the high polarity and small volume of the central nitrogen atom in ammonium-based HBAs. Furthermore, the length of the alkyl chain in the quaternary-based compounds also contributes to their

dissolution ability. The chemical potential tends to increase with an alkyl chain length with Tetrapentylammonium (TPA) > Tetrahexylammonium (THA) > Tetraoctylammonium (TOA). However, for the lower alkyl group, the chemical potential decreases with alkyl chain length in the order Tetrabutylammonium (TBA) > Tetraethylammonium (TEA) > Tetramethylammonium (TMA). For phosphonium-based compounds, the result shows similar trends as in ammonium-based compounds. However, comparing both types, ammonium-based compounds perform better than phosphonium-based molecules as discussed earlier. Several ring/phenyl group compounds were also predicted for their ability to dissolve lithium. In general, the HBA having rings shows a lower affinity for lithium compared to the straight-chain organic salts. This result may stem from the large steric hindrance between the ring-based compounds and the target metals (Braun et al., 2020).

The HBA containing halide plays a crucial role in the metal dissolution. This is because the leaching of metals could occur via coordination with halide ions in the HBA. For the halide compounds, the chlorine-containing compounds perform better than their bromine-containing counterparts. This attribute can be ascribed to the more electronegativity in chlorine compared to bromine (Cassol et al., 2007; Warrag et al., 2017).

In this study, three compounds based on carnitine, betaine, and choline chloride could lead to the group of DES called natural deep eutectic solvents (NADES). This is because the appropriate combination of HBDs with these compounds could exhibit all the principles of green chemistry (Dai et al., 2015; Paiva et al., 2014). Among these compounds, those containing oxygen anion perform better than the cholinium-based HBA. Although betaine and choline chloride possess similar structures, the higher solubility in betaine may be attributed to the presence of the oxygen anion. This is because oxygen is a better electron acceptor compared to chlorine.

Overall, the best DES design based on HBA presents an interesting analysis. However, the choice of selected HBD is also an important consideration in optimum DES design. Hence, the next discussion presents the hydrogen bond donor influence on lithium extraction from spent lithium-ion batteries.

### 3.4 Influence of HBDs

The several HBDs (Table S2 of the supplementary document) studied also showed a great ability to solubilize lithium. This may be explained due to the dipole moment theory (Antušek & Repisky, 2020). That is because like like repel while unlike charges repel. Since lithium has a lone outermost shell electron, it is expected that it would readily be solubilized by non-ionic molecules. In HBD analysis, the alkyl chain length, functional group, and the presence of a ring or cyclic molecules further influence the extractability of the studied constituents.

Based on the functional group present in the HBD, no rigid trends were observed regarding the superior performance of a specific functional group. It appears that several other factors influence the performances of the solvents. However, the compounds containing multifunctional groups tend to perform better than compounds with a single functional group. This behavior may be attributed to the specificity of each functional group's ability to solubilize lithium. Essentially, the amino-alcohol groups perform better than the other studied functional groups. This high performance may be related to the presence of the amine and alcohol groups. The literature also supported this finding for ethanolamine as an instance; Ethanolamine has been reported as a great lixiviant in metal extraction (Kamariah, Nor; Binnemans, Koen; Spooren, 2021). The metal extraction mechanism of ethanolamine could occur via metal amine complexes due to the interaction of metal and amine. The extraction mechanism could also be due to metal amine-hydroxyl complexes owing to the interaction of the metals and the two functional groups. Nevertheless, more studies are needed to decipher the extraction mechanism of ethanolamine in metal extraction from spent lithium-ion batteries. Furthermore, ethanolamine has been used to reduce several metallic salts from an aqueous solution (Hossain et al., 2021; Y. Liu et al., 2019; Sahu et al., 2018). Other amino alcohol groups- diethanolamine and triethanolamine also show a great affinity for the metals. The trends are ethanolamine (EA) > triethanolamine (TEA) > diethanolamine (DEA). Therefore, insights into the mechanisms of DES formation from different amino alcohols in metal extraction may be an interesting future research direction.

In addition, it is important to also investigate HBD compounds' properties such as reducibility, viscosity, and proton exchangeability. This is to assess and compare their metal extractability. For instance, it was



established that for the same HBA, the more reducing HBD forming the DES has the highest extraction efficiency. For example, Wang et al. (S. Wang et al., 2020) compared the performances of choline chloride/urea and choline chloride/ethylene glycol. It was shown that choline chloride/urea performs better due to the more reducing ability of urea compared to ethylene glycol. Interestingly, our model agreed with their findings. This suggests the reliability of the model. To this end, other components with known reducing ability may be the desired starting material as leaching agents in spent LB. These components include fructose, glucose, sucrose, and most carboxylic acid compounds. The terpenoid ketones have also been reported to be a suitable candidate in metal extraction technologies (Swain, 2016; Zhang et al., 2017). To this end, other compounds with strong reducing ability may be promising hydrogen bond donors in lithium recovery from spent LIB.

Strong acidity is considered a favorable trait in metal extraction from spent LIB (Golmohammadzadeh et al., 2018). In this study, different carboxylic acid groups are identified based on the acid group number such as mono, di, and tricarboxylic acids; the presence of rings such as cyclic or phenolic compounds; and the presence of other functional groups, for instance, hydroxycarboxylic acids. Carboxylic acid performance is also influenced by the length of the alkyl chain. The higher the length of the alkyl chain, the more viscous the HBD tends to behave (Florindo et al., 2014; Li et al., 2022). Consequently, this limits the extractability of the acid. Except for propionic acid, the COSMO prediction agrees with this relation.

#### 4. CONCLUSIONS

A COSMO-Rs model has been developed to predict lithium extractability with several DES components. The model can determine the affinity of any DES component comprising of hydrogen bond for lithium. The only input of the model is the chemical structure of these hydrogen bond-forming compounds. Among the 188 studied components, non-ionic compounds gave Li high chemical potential. This phenomenon is attributed to the coordination of lithium with these compounds. In addition, the presence of functional groups in the non-ionic compound is influential in the ion exchange mechanism with lithium and target metal. Thus, the COSMO-RS model can guide the appropriate selection of probable DES constituents from a pool of possible

DES components for metal dissolution. Therefore, our future study will focus on the application of this model to other valuable metals in spent LIBs. Further, novel DES would be synthesized based on other desirous properties such as reduction ability, transport property, health, and ecological concerns.

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## OPTIMIZATION AND KINETIC STUDY OF THE HYDROTHERMAL PRETREATMENT OF COCOA POD HUSKS AND UNRIPE PLANTAIN PEELS

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### ABSTRACT

Cocoa pod husks and unripe plantain peels are mass flows that are hugely available in Nigeria without any known industrial application. This study involves the kinetic analysis of the decomposition of hemicellulose into xylose and furfural during the hydrothermal pretreatment of cocoa pod husks and unripe plantain peels within a temperature, time, and solid concentration range of 110 - 140 °C, 5 - 10 minutes and 60 - 100 g/dm<sup>3</sup> respectively. The experimentally obtained kinetic constants determined the optimal time for the desired product's (xylose) maximum yield. At the process temperature of 110, 125 and 140 °C, the optimal times were 332, 140 and 79 minutes respectively for the cocoa pod husks, and 210, 117 and 29 minutes respectively for unripe plantain peels. The kinetic models developed from the Response Surface Methodology show that the formation of xylose and furfural from cocoa pod husks and unripe plantain peels depends only on the process temperature and time but not on the concentration of biomass in the reactor.

Keywords: lignocellulosic biomass; severity factor; response surface methodology

### 1. INTRODUCTION

The use of lignocellulosic biomass (LCB) for biofuel production has received a lot of attention from researchers. These biomasses consist of lignin, cellulose and hemicellulose (Tu and Hallett, 2019). A reason for using LCB for biofuel production is that they are mostly wasted and readily available. However, unlike substrates like maize grain, cassava tubers and sugar cane juice, LCB are naturally recalcitrant. Therefore, they require pretreatment (Soltanian et al. 2020), without which their enzymatic saccharification will be difficult (Pandey et al. 2015). Different pretreatment methods exist to alter

the recalcitrant nature of LCB. These methods include chemical pretreatment involving the use of alkalis (Sindhu et al. 2015) and acids (Dahunsi et al. 2019), biological pretreatment where microorganisms like bacteria and fungi are used (Sharma et al. 2019), and physical pretreatment like milling and grinding (Kumar et al. 2009). Irrespective of the method used for pretreatment, the goal is to make the biomass porous and accessible to enzymatic attack as shown in Figure 1.

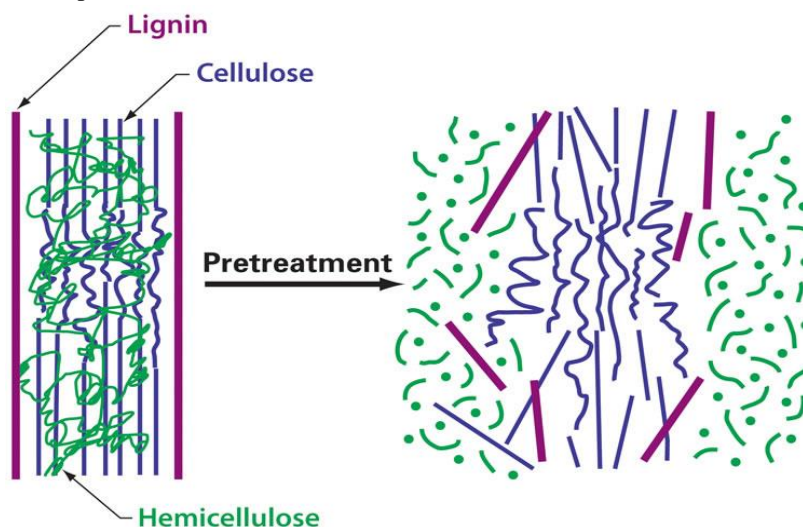


Figure 1. Effect of pretreatment on a lignocellulosic biomass (Kumar et al. 2009)

Pretreatment methods all have their advantages and disadvantages. About 40 % of the total cost of biofuel and bioproducts production from LCB is for pretreatment (Ingle et al. 2018). A good practice, therefore, is to choose a pretreatment method that is cheap, effective and efficient. The Hydrothermal pretreatment method offers the advantage of not requiring the use of chemicals and corrosion-resistant materials, which can add to the cost of the process (Eskicioglu et al. 2017) as well as forming lower inhibitory products compared to chemical pretreatment (Yang et al. 2017). During hydrothermal pretreatment, conditions like operating temperature, process time and the quantity of substrates per unit volume of water in a reactor are varied to achieve optimal results (Ruiz et al. 2020). Water is maintained in the liquid phase at temperatures above 100 °C under appropriate pressures (usually between 5 – 40 MPa). The water ionizes into hydronium ( $H_3O^+$ ) and hydroxide ( $OH^-$ ) ions, making it acidic and thereby enabling it to break up the biomass molecules with the solubilization of hemicellulose and lignin (Ahmed et al. 2019).

Like most pretreatment methods, one disadvantage of hydrothermal pretreatment is the formation of chemicals that can serve as inhibitors during the production of biofuels or other bioproducts. These inhibitors, such as furan derivatives, are usually produced from decomposing desired products like reducing sugars at elevated temperatures (Lee and Park 2020). Therefore, a good practice is to determine the optimal operating conditions that will enhance the production of reducing sugars and limit their degradation into inhibitors.

Cocoa pod husks (CPH) and unripe plantain peels (UPP) are some of Nigeria's most abundant LCB. In 2019, Nigeria produced about 0.35 million tons of cocoa beans and 3.1 million tons of plantain (FAOSTAT 2020). According to Oddoye et al. (2013), the cocoa bean makes up only about 33 % of the cocoa fruit. Therefore, in 2019, Nigeria generated an estimated 0.71 million tons of CPH. Similarly, about 0.93 million tons of UPP were generated from plantain production in Nigeria in 2019, since peels make up about 31 % of the plantain fruits (Agama-Acevedo et al. 2016). These massive wastes are usually left on the farmlands or disposed inappropriately, where they constitute a nuisance and serve as vehicles for transmitting pathogens of public health importance. Moreover, the waste contributes immensely to environmental pollution since they have no known industrial application (Olatundun et al. 2020). One way of using these mass flows is for biofuel

production. Since they are LCB, CPH and UPP must be pretreated before they can be used as substrates for biofuel production. There is available literature on the chemical pretreatment of CPH (Dahunsi et al. 2019a; Sandesh et al. 2020). However, chemical pretreatment requires equipment that can withstand corrosion in addition to requiring downstream refining (Tian et al. 2018). Similarly, available literature on the pretreatment of UPP is limited, hence the choice of hydrothermal pretreatment for both biomasses. This study aims to determine the optimal conditions for the hydrothermal pretreatment of CPH and UPP using the Response Surface Methodology. In addition, the kinetics involving the pretreatment process was investigated to enhance the control of the process parameters.

## 2. MATERIALS AND METHODS

### 2.1 Preparation of raw materials

Cocoa pod husk and unripe plantain peels were obtained from plantations in Boki, South-Southern Nigeria. The biomasses were then cut into smaller pieces, sun-dried to get a constant mass, milled using a commercial grinder (Avamix BX1100K, USA) and sieved to obtain particle sizes of less than 2 mm. Finally, these substrates were stored at 4 °C until further use.

### 2.2 Proximate analysis of substrates

Ash and moisture contents of the substrates were determined following the method developed at the National Renewable Energy Laboratory (NREL) (Sluiter et al. 2008a). The percentage composition of cellulose, hemicellulose and lignin were determined using the neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL) methods as described by van Soest and Wine (2020) using Equations 1-3.

$$\% \text{ cellulose} = \% \text{ ADF} - \% \text{ ADL} \quad 1$$

$$\% \text{ hemicellulose} = \% \text{ NDF} - \% \text{ ADF} \quad 2$$

$$\% \text{ lignin} = \% \text{ ADL} \quad 3$$

Xylose and furfural were determined with the aid of an HPLC using the method of NREL as outlined by Sluiter et al. (2008b).

### 2.3 Hydrothermal Pretreatment

Hydrothermal pretreatment of the substrate was carried out according to the method described by Kouteu Nanssou et al. (2016). The autoclave batch reactor used (Parker, USA) had a working volume of 2L, was made

of stainless steel and contained a magnetic stirrer as well as a temperature controller. The reactors were capable of withstanding an operating temperature and pressure of 510 °C and 345 bar respectively. Operating temperature for the substrates was between 110 and 140 °C while the heating time was varied between 5 and 10 minutes. Process time started from the time of attainment of the desired temperature. Each desired temperature was kept constant by the controller installed with the reactor. Since hemicellulose degrades faster than cellulose (Jin 2014), the concentration of xylose and furfural were taken as the response variables of the process.

#### 2.4 Design of Experiments

All experiments were designed using the Box-Behnken response surface methodology of the Design Expert software (version 12). Data from each experiment were used to fit the quadratic equation (equation 4) generated by the software.

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \sum_k \sum_{j=2}^k \beta_{ij} X_i X_j \quad 4$$

where Y is the response of interest,  $\beta_0$  is the intercept,  $\beta_j$  and  $\beta_{ij}$  are quadratic coefficients.

The independent variables are shown in Table 1. The range of process parameters were chosen to ensure that the severity factor is kept below 3.0. The stirrer revolution rate and the heating rate of the reactor were kept constant for all the batches. The hydrolysis of hemicellulose was modelled as a consecutive reaction as shown in equation 5 (Fogler, 2016).



where A is hemicellulose, B is xylose, I is furfural.  $k_1$  and  $k_2$  are first-order rate constants of the formation of the respective products. The severity factor, which is a function of temperature and time, was evaluated using equation 6 (Yang et al. 2017).

$$R_0 = \log\left[t \times \exp\left(\frac{T-100}{14.75}\right)\right] \quad 6$$

where  $R_0$  is the dimensionless severity factor, t is time (min) and T is operating temperature (°C).

**Table 1. Box-Behnken design of the hydrothermal process**

Factor	symbol	Coded levels		
		-1	0	+1
Temperature (°C)	$X_1$	110	125	140
Time (min)	$X_2$	5	7.5	10
Mass of substrate in water (g/dm <sup>3</sup> )	$X_3$	60	80	100

### 3 RESULTS AND DISCUSSION

#### 3.1 Physicochemical parameters of substrates

**Table 2. Characteristics of Biomass**

Parameter	Unit	CPH	UPP
Moisture content	% TS	12.18	11.54
Ash content	% TS	13.33	15.01
Hemicellulose	%	26.72	29.2
Cellulose	%	31.25	37.9
Lignin	%	20.15	8.7

TS, total solids

Table 2 shows the composition of the biomass used in the study. Cellulose was the major component of both substrates while lignin was the least, as is common with most lignocellulosic biomass (Xu and Li 2017). Sandesh et al. (2020), Dahunsi et al. (2019) and Nazir et al. (2016) have also reported a higher composition of cellulose than hemicellulose and lignin in CPH. A higher cellulose content compared to hemicellulose and lignin has also been reported in UPP by Agama-Acevedo et al. (2016). The percentage of the cellulose, hemicellulose and lignin content of CPH in the present study are similar to the report of Dahunsi et al. (2019) but different from those reported by Nazir et al. (2016). Geographic and climatic factors contribute to diversity in the physicochemical parameters of plants biomass (Dong et al. 2019).

### 3.2 Effect of Process Parameters on Responses

The highest yield of xylose was obtained at the maximum severity factor of 2.18 for both the CPH and the UPP. Although there were degradation products at 140 °C at all pretreatment durations, it is unlikely that these degradation products will significantly affect biofuel production (Antwi et al. 2019). At temperatures below 140 °C, both substrates' degradation products were below detectable limits. For both substrates, temperature and time were the only factors that influenced the formation of xylose and degradation products. The effect of the amount of biomass per unit volume of water in the reactor was not significant, contrary to the report of Kouteu Nanssou et al. (2016) in the hydrothermal pretreatment of cassava stems and peelings. However, according to Fogler (2016), the maximum concentration of the desired product in a series reaction taking place in a batch reactor is dependent more on temperature and time than on the initial concentration of the reactant substrate. The concentration of the desired product can change even at a constant initial concentration of a reactant specie, provided the process temperature is changing and time is varied, as shown in equation 7.

$$C_B = \frac{k_1 C_{A0}}{k_2 - k_1} \left[ \left( \frac{k_1}{k_2} \right)^{k_2 - k_1} - \left( \frac{k_1}{k_2} \right)^{k_2 - k_1} \right] \quad 7$$

where  $C_B$  and  $C_{A0}$  are the concentrations (g/L) of B and A respectively in equation 5.

The adequacy of the models describing the formation of xylose during the hydrothermal pretreatment of CPH and UPP were significant at  $R^2$  values of 0.9985 and 0.9971, respectively. These models are represented by equations 8 and 9, respectively. In both models, temperature had more effect on xylose formation than time.

$$Y = 3.69 + 2.09T + 5.46t + 1.99Tt + T^2 \quad 8$$

$$Y = 4.54 + 2.69T + 6.83t + 2.68Tt + 3.05T^2 \quad 9$$

where Y is the concentration of xylose. The 3D diagram of the interaction of temperature and time in the formation of xylose is shown in Figure 2.

The concentration of degradation product was also affected by the interaction between temperature and time. Substrate concentration was not significant in forming fufural from both substrates. The models of their formation are shown in equations 10 and 11 for CPH and UPP respectively, where X is the concentration of fufural.

$$X = 0.02T + 0.07t + 0.04Tt + 0.07T^2 \quad 10$$

$$X = 0.028T + 0.12t + 0.05Tt + 0.12T^2 \quad 11$$

The 3D response surface plots for the formation of fufural is shown in Figure 3.

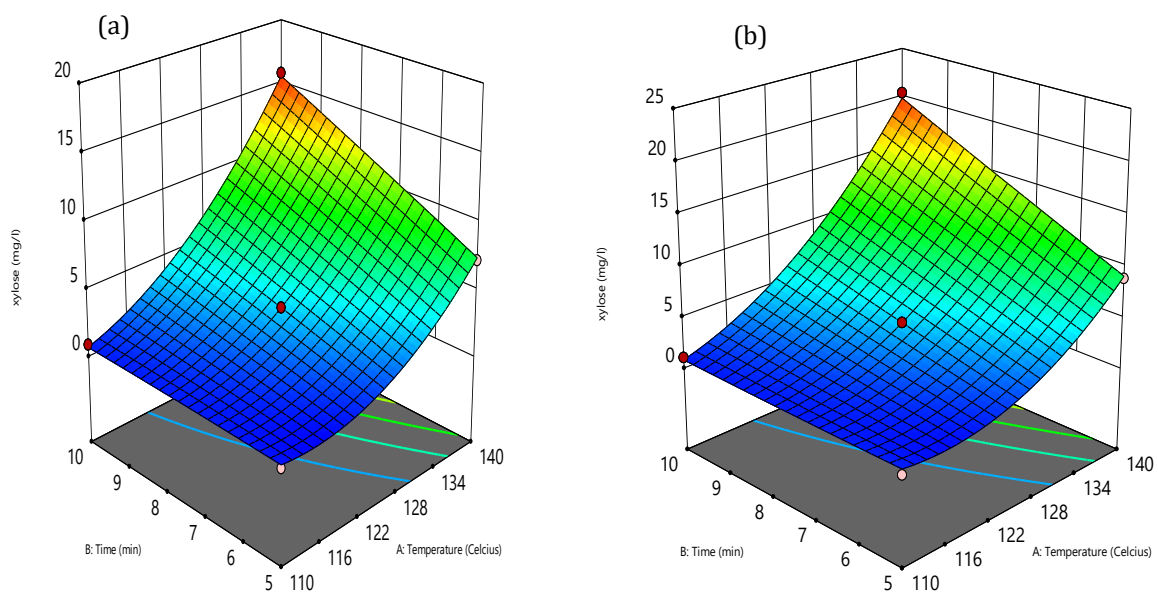


Figure 2. 3D diagram of xylose formation in cocoa pod husk (a) and unripe plantain peels (b)



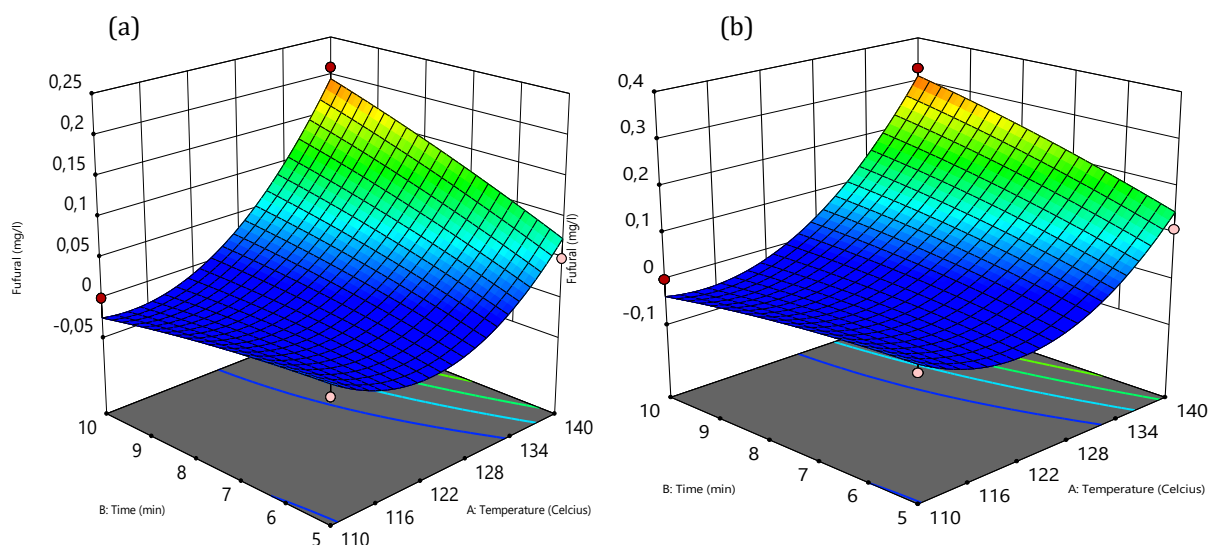


Figure 3. 3D diagram of furfural formation in coco pod husk (a) and unripe plantain peels (b).

### 3.3 Kinetic Evaluation

The series reaction of the degradation of hemicellulose into simple sugars and degradation products has been presented in equation 5.

Applying mole balance and rate laws on each of the reacting species, the yield,  $Y_B$  was obtained from Equation 12

$$Y_B = \frac{k_1}{k_2 - k_1} (e^{-k_1 t} - e^{-k_2 t}) \quad 12$$

Equation 12 was used to obtain the values of  $k_1$  and  $k_2$  using linear regression. These values are presented in Table 3. Equation 12 also confirms that the yield of xylose is independent on the concentration of substrates.

Table 3. Rate constants for the degradation of hemicellulose in the substrates

Temp. (°C)	Cocoa pods		Plantain peels	
	$k_1 \text{ min}^{-1}$	$k_2 \text{ min}^{-1}$	$k_1 \text{ min}^{-1}$	$k_2 \text{ min}^{-1}$
110	0.0112	0.0003	0.0174	0.0005
125	0.0281	0.0006	0.0323	0.0008
140	0.0453	0.0014	0.0572	0.0193

As seen in Table 3, the rate constants increased with an increase in pretreatment temperature. At all temperatures, the rate constant of xylose formation ( $k_1$ ) was higher than that of furfural ( $k_2$ ). If the reaction temperature is continually increased, a point will reach where the rate of formation of inhibitors will be higher than the rate of formation of reducing sugars, as reported by dos Santos Rocha et al. (2017). The rate constants presented in Table 3 estimate the optimal time required for producing reducing sugars during hydrothermal

pretreatment at different temperatures using equation 13 (Fogler 2016).

$$t_{\max} = \frac{1}{k_2 - k_1} \ln \frac{k_2}{k_1} \quad 13$$

The calculated optimal time for the yield of xylose was 332, 140 and 79 minutes at 110, 125 and 140 °C respectively for CPH, and 210, 117 and 29 minutes at the respective temperatures for UPP. The maximum severity factor was 3.1 for CPH and 2.80 for UPP.

An inverse relationship existed between the optimal time required to form the desired product and the process

temperature. This optimal time varies from one substrate to another. Although the calculated optimal times will produce the maximum amount of desired products, there is also the possibility of forming a significant amount of degradation products. In practice, if the amount of product degradation will have a significant effect on biofuel production, there will have to be a trade-off between having a mixture of the maximum amount of the desired product with a significant amount of degradation or having a yield of the desired product below the maximum with little or no degradation products.

The process' activation energies for both substrates were estimated using the Arrhenius equation. The activation energy,  $E_a$  of the formation of furfural was higher than that of the formation of xylose. The implication is that a higher temperature is required for the decomposition of xylose to degradation products than for the decomposition of hemicellulose to form xylose. Generally, The  $E_a$  of the formation of responses in the CPH was higher than the  $E_a$  of responses in UPP (Table 4). This higher activation energy may be due to the higher lignin content (Table 1), which is responsible for structural rigidity (Zoghalmi and Paës 2019) in lignocellulosic biomass.

**Table 4. Activation energy of formation of responses**

Rate constants ( $\text{min}^{-1}$ )	Cocoa pods		Plantain peels	
	$k_1$	$k_2$	$k_1$	$k_2$
$R^2$	0.9876	0.9840	0.9979	0.9726
$E_a$ (kJ/mol)	6.01	7.52	5.06	6.97

## CONCLUSION

The formation of xylose and furfural during the hydrothermal pretreatment of cocoa pod husk and unripe plantain peels is influenced by the interaction of the operating temperature and time but not by the concentration of substrates in the reactor. The optimal temperature and time depends on the nature of the biomass that is being pretreated. Degradation products were not formed at temperatures below 140 °C for both substrates. At an experimental temperature and time of 140 °C and 10 minutes, respectively, the degradation products formed from the cocoa pod husk were lower than the degradation products formed from the unripe plantain peels.

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## STUDENT PERCEPTION OF RESEARCH CAPACITY OF CHEMICAL ENGINEERING DEPARTMENTS IN SELECTED NIGERIAN UNIVERSITIES

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### ABSTRACT

*This research work is aimed at assessing the research capacity of chemical engineering departments in selected Nigeria Universities. Thirty-three universities were selected for the survey based on the criteria of running accredited chemical engineering programs. A framework designed to measure specific KPIs or dimensions was developed and adapted to a questionnaire. The 37- item questionnaire was administered to students online with responses downloaded from the web portal for cleaning, coding and analysis using Microsoft excel software program. The coded responses were analyzed using percentages, mean, variance, and ANOVA. 56 out of the 60 responses received were found to be valid. The responses received were grouped into undergraduate and postgraduate respondents. The administered questionnaires had high internal consistencies with Cronbach alpha values of 0.8898, 0.8525 and 0.8994 for all, undergraduate and postgraduate responses. The responses were analyzed using three key performance indicators (KPIs) namely: (a) research infrastructure, dissemination, (b) knowledge translation and research applicability and (c) empowerment, skills, and confidence. The means for research infrastructure range from 3.6607 to 4.0714, 3.6176 to 3.8529 and 3.7273 to 4.5455 for all, undergraduate and postgraduate respondents respectively. Quite a number of the undergraduate respondents are neutral with respect to each dimension of the research infrastructure while the postgraduate respondent's perception is that achieving peer recognition is not a motivation for conducting research. On average, the respondents agree with all the dimensions for knowledge translation and research applicability. The empowerment, skills, and confidence mean values range from 3.4643 to 4.2500, 3.2353 to 4.1765 and 3.7727 to 4.4091 for all, undergraduate and postgraduate respondents respectively. These imply that, on average, the undergraduate and postgraduate respondents view of this KPI differ.*

*Keywords: Research capacity; Key performance indicators; Questionnaire; Cronbach Alpha; Analysis of variance (ANOVA)*

### 1. INTRODUCTION

Around the world, knowledge is recognised as the critical factor to global competitiveness in the 21st century. Successful societies are those most able to exploit knowledge for competitive advantage and performance, attract international talent, new business and investment. This requirement for a knowledge-based society is central to most national governments strategies for sustainable economic and social development, especially as a response to the global economic crisis (European Commission, 2010). University-based research is the primary domain for the production of new knowledge. It is critical to research-innovation eco-system, providing human capital through

education and training, attracting high-skilled talent and investment, actively engaging with local and regional community through knowledge and technology transfer, and laying a solid foundation for nations and regions to be globally competitive.

Universities are at the vanguard of the effort to build a knowledge-based society and economy that is competitive. In an effort to build a competitive knowledge-based society and economy, Universities are undergoing significant changes in order to meet the challenges associated with absorbing a constantly increasing student intake without sacrificing the quality inherent in third-level education, relevance of studies, balancing the basic functions of teaching, research

and service, improving the administration of higher education for the sake of better performance of its basic functions of teaching, research and extension, knowledge generation, internalization of higher education. The methods for evaluating and assessing the activities and results of higher education, with a particular focus on the assessment of university-based research (AUBR), have been steadily increasing over recent years. The unavoidable result of globalization is a cross-national or global comparison of research performance and quality. Research assessment can be an important part of performance and quality improvement, institutional autonomy and strategic planning support, talent attraction and research mission differentiation. It must be noted that the indicators are not unbiased since it can easily distort to measure the wrong thing.

In Nigeria, chemical engineering education began in the late 1960s with the kick off of an applied chemistry programme at the then University of Ife now Obafemi Awolowo University (OAU) at Ile-Ife, south western Nigeria. This was later upgraded to chemical technology and finally chemical engineering. In 1973, the first chemical engineers with local training graduated. The University of Lagos and Ahmadu Bello University (ABU), Zaria in northern Nigeria, took a cue from OAU with parallel chemical engineering programmes that resulted to the first graduates from the two institutions in 1976 and 1977 respectively (Erinne, 2015). There are 34 universities offering COREN accredited chemical engineering degree programmes in Nigeria with 16, 10 and 8 of them respectively in the Federal, State and Private Universities. A typical chemical engineering curriculum in Nigerian universities is generally consistent with international standards, particularly in the UK and US. They incorporate modules from the basic sciences, particularly physics and chemistry, sometimes biology, as well as modules from mathematics and computing. They also include general engineering modules (graphics, workshop practice, mechanics of machines and basic electrical circuits), as well as basic chemical engineering courses like process principles, thermodynamics, transport processes, and materials technology.

Research ecosystems in universities in low and middle income (LMIC) countries like Nigeria face many challenges. These challenges translate into a much lower level of research outputs from universities in low- and middle- income countries compared to that of developed countries (Duermeijer, 2018; Gonzalez-Brambila et al., 2016; Ngongalah et al., 2018). The challenge of research

translation is also pervasive (Gonzalez-Brambila et al, 2016). Not only is there inadequate data and tools to inform development decisions among development practitioners and funders in LMICs, but involvement of LMIC universities in generating this data is low compared to the level of need for such evidence. Furthermore, many research products are not adequately translated and disseminated to end-users, policy makers and development partners. There is a need to build research capacity for the universities in order to expand development research outputs from LMIC universities. This necessitates identifying key capacity strengths and gaps in these ecosystems, so that new mechanisms can be designed to address some of them especially in vital areas.

The National University Commission (NUC) reported that, up until the late 1980s, the research performance of Nigerian tertiary institutions was among the best in sub-Saharan Africa in terms both of quality and quantity (Chiemeké et al, 2009). Good research training and motivation, as well as availability of equipment and library facilities are the foundations for research. By 1996, both the amount and quality of research had reached an all-time low. Okebukola (2002) lists the following factors as contributing to the decline from late 1988 to 1996 and subsequent collapse from 1997 to date:

- a) Deficient skills in modern research methods
- b) Lack of equipment for conducting cutting-edge research
- c) Overloading teaching and administration schedule which leaves little time for research
- d) Difficulty in accessing research funds
- e) Dwindling ability of experienced and senior researchers to mentor junior researchers due to intellectual emigration (brain drain)

The 2016 Bibliometric review of Nigerian publications indexed on the Scopus database was completed by Salisu and Salami (2020) over 115 years (1901-2016). The study tried to expound Nigerian research achievements, developments in publishing, patterns of publications and patterns of cooperation at national and international level. The underlying bibliometric metrics and figures have reviewed 95,304 publications in general. Results show a steady growth in Nigerian post-independence publications; prevailing post publications in conferences and analyses compared with other national collaborations, as well as in most academic papers in Nigeria compared with foreign countries. The

findings indicate a steady growth in Nigerian publications since independence; a preference for publications in articles over conference papers and reviews, along with regular local cooperation in comparison to foreign cooperation; and that universities produce the majority of research publications in Nigeria. However, there exists a scarcity of capacity assessment frameworks and tools that are tested and applied to assess research capacity in African university settings (Jessani et al., 2014). This study is therefore aimed at assessing the student perspective on research capacity of chemical engineering departments in selected Nigerian universities using a developed template with key performance indicators and measures.

## 2. MATERIALS AND METHODS

### 2.1 Sample and procedure

This research work was designed to assess the research capacity of chemical engineering departments in selected Nigerian universities using descriptive survey design. The sample comprises 33 universities in Nigeria offering accredited chemical engineering programmes (COREN, 2020). The survey comprises of questionnaires containing thirty-seven items for the students. The questionnaire was divided into two sections (A and B). Section A captures demographic data like age, gender, level of education, current academic qualification, research portfolio and others while section B is aimed at needs assessment. The key performance indicators (KPIs) for measuring the various dimensions of the needs assessment are shown in Table 1.

**Table 1: Dimension measuring KPIs and their corresponding items**

Dimensions	Items in the questionnaire
Research infrastructure	10, 13, 14, 18, 22, 28, 30 and 36
Dissemination, Knowledge translation and research applicability	15, 17, 20 and 24
Empowerment, Skills, and confidence	11, 16, 19, 21, 23, 25 - 27, 29, 31, 32, 33 - 35 and 37

The questionnaire was converted to google forms and then administered to departments, running duly accredited chemical engineering programs in the selected universities, after obtaining their permission to participate in the research. The completed questionnaires were downloaded, cleaned (removal of multiple responses from respondents) and coded. Data coding was carried out by assigning numerical values to responses from the participants (see Table 2) and the computations were done using Microsoft Excel 2016. The coded data was then used for statistical analysis.

**Table 2: Coding scheme for Likert scales**

Likert scales	Code
Strongly disagree	1
Disagree	2
Neutral	3
Agree	4
Strongly agree	5

The Cronbach's alpha was used to analyse the questionnaire instrument's reliability and validity and calculated using eqn 1 (Mondal and Mondal, 2017):

$$\alpha = \frac{K-1}{K} \left( 1 - \frac{\sum s_i^2}{s_t^2} \right) \quad (1)$$

where  $\alpha$  = Cronbach alpha, K = Number of items in questionnaire,  $s_t^2$  = Standard deviation of sum scores and  $s_i^2$  = Standard deviation of ith item.

The instrument validity is an essential factor used in research in order to yield beneficial results (Surucu et al., 2020). Validity is concerned with whether the research instruments measure the quality or behaviour that it is supposed to measure. Additionally, validity determines whether the research instrument expresses the scale that is suitable to its function and measures its desired results. A panel of experts in the study area authenticated the items in the questionnaire to ensure that they properly reflect topics that addressed all applicable aspects to determine the internal relevance and whether or not it was appropriate for use as an instrument to achieve the research's goals and objectives.

### 2.2 Data Analysis

Frequencies and percentages were used to describe the profile of the respondents regarding personal and social factors and the number of completed, disseminated, and utilized researches. Mean and standard deviation were used to ascertain the respondent's degree of perception to the three KPI's stated in Table 1. A developed

Microsoft Excel 2016 template was used to code the questionnaire responses and to perform the following calculations: Number of Responses; % Valid Responses; gender and qualification distribution of respondents; Cronbach Alpha; the mean, variance and ANOVA for each of the three Key Performance Indicators.

### 3. RESULTS AND DISCUSSION

#### 3.1 Response analysis and Questionnaire reliability

A total of 33 universities were contacted but responses were obtained from only 10. These responses were from all the six geo-political zones in Nigeria (1 each from North Central, North East and North West; 2 from South East; 3 from South South; 2 from South West). Also, 1 out of the 10 universities run chemical engineering programs with interim accreditation while the remaining 9 universities run a fully accredited program in chemical engineering (COREN, 2020). Seven of these universities are owned by the Federal government, 1 is owned by a

State government and the remaining 2 are private universities.

Table 3 shows the summary of the responses obtained for the administered questionnaires. A total of 60 responses were received with 4 of them invalidated due to duplications. This represents 93.33% valid responses. Out of the 56 valid responses, 34 were undergraduate students while 22 were postgraduate students. Roughly about 20% of the respondents were females. It is also noteworthy that 11.76% of the undergraduate respondents hold a first degree. 18.18% of the postgraduate students were PhD students.

The calculated Cronbach's Alpha coefficients for the 37 items measured were respectively 0.8898, 0.8525 and 0.8994 for all undergraduate and postgraduate respondents. Since these values are greater than 0.7, we can infer that these items have adequate internal reliability (Taber, 2018).

**Table 3: Summary of Questionnaire responses**

	All	Undergraduate	Postgraduate
Responses	60	38	22
Valid Responses	56	34	22
Valid Responses	93.33%	89.47%	100.00%
Male	80.36%	79.41%	81.82%
Female	19.64%	20.59%	18.18%
with SSCE	53.57%	88.24%	0.00%
with First degree	39.29%	11.76%	81.82%
Master's degree	7.14%	0.00%	18.18%
Cronbach Alpha	0.8898	0.8525	0.8994

#### 3.2 Key Performance Indicators

##### 3.2.1 Research infrastructure

The coded mean and variance for each item used to assess the research infrastructure are given in Table 4. The means range from 3.6607 to 4.0714, 3.6176 to 3.8529 and 3.7273 to 4.5455 for all undergraduate and postgraduate respondents respectively. The postgraduate respondents appear to agree more with each dimension of the research infrastructure when compared with the undergraduate respondents. This may be due to the fact that the former respondents are more mature professionally. The range of the mean values for the

undergraduates being less than 4 indicate that quite a number of the respondents are neutral with respect to each dimension. To investigate whether the means for the different dimensions are different, a one-way analysis of variance (ANOVA) was carried out. The

ANOVA results are shown in Table 5. Clearly, there is significant difference at 5% level between the means for the dimensions for postgraduate respondents. It would appear that, for postgraduate respondents, achieving peer recognition is not a motivation for conducting research.

**Table 4: Mean scores for research infrastructure**

No	Item	All		Undergraduate		Postgraduate	
		Mean	Variance	Mean	Variance	Mean	Variance
10	The chemical engineering curriculum has developed my skill in analysing data for patterns.	3.7857	0.5714	3.6176	0.6676	4.0455	0.3312
13	Achieving peer recognition motivates me to conduct research.	3.6607	0.9919	3.6176	0.6070	3.7273	1.6364
14	The chemical engineering curriculum has helped in figuring out the steps involved in a research work which has in turn helped develop my research skills.	4.0714	0.7221	3.7647	0.7308	4.5455	0.3550
18	Satisfying a need for creativity and or curiosity motivated my foray into research.	3.8929	0.7519	3.8235	0.7558	4.0000	0.7619
22	Satisfying the need to stay current in the field of chemical engineering is a motivation to carry out research work.	3.9286	0.8312	3.8529	0.9777	4.0455	0.6169
28	The chemical engineering curriculum has greatly improved my skill in the area of writing scientific reports and papers.	3.9643	0.7987	3.8235	0.8164	4.1818	0.7273
30	The chemical engineering curriculum has through research improved my ability to defend an argument with scientific facts when asked questions.	3.9464	0.7789	3.6471	0.7807	4.4091	0.4437
36	The chemical engineering curriculum has helped improve my statistical skill in data analysis in relation to research activities.	3.8214	0.9130	3.6471	1.0838	4.0909	0.5628
	Average	3.8839	0.8924	3.7241	0.8893	4.1307	0.8417

Table 5: ANOVA Results

	F <sub>cal</sub>	F <sub>critical</sub>	p-value	Interpretation*
Research infrastructure				
All	1.1168	2.0304	0.3512	No significant difference
Undergraduate	0.4392	2.0444	0.8770	No significant difference
Postgraduate	2.0729	2.0645	0.0491	There is significant difference
Dissemination, Knowledge translation and research applicability				
All	0.6253	2.6456	0.5994	No significant difference
Undergraduate	1.5663	2.6732	0.2006	No significant difference
Postgraduate	1.0309	2.7132	0.3832	No significant difference
Empowerment, Skills, and confidence				
All	3.2434	1.6778	0.0000	There is significant difference
Undergraduate	2.7637	1.6854	0.0004	There is significant difference
Postgraduate	1.1541	1.6962	0.3068	No significant difference

\* at 5% Confidence Level

### 3.2.2 Dissemination, knowledge translation and research applicability

The dissemination, knowledge translation and research applicability mean values are shown in Table 6. The

mean values are approximately 4, which implies that, on the average, the respondents agree with each dimension of this KPI. The ANOVA result shown in Table 5 confirms this.

Table 6: Dissemination, knowledge translation and research applicability

No	Item	All		Undergraduate		Postgraduate	
		Mean	Variance	Mean	Variance	Mean	Variance
15	Satisfying a need to contribute to or fill certain gaps in knowledge motivates me to conduct research.	4.0000	1.1273	4.0588	0.4207	3.9091	2.2771
17	The chemical engineering curriculum has helped in and enhanced my ability to identify limitations of research methods and designs.	3.9107	0.8101	3.6471	0.7807	4.3182	0.6082
20	Receiving mentorship from experienced lecturers and or researchers is an extra motivation to indulge in research work.	4.1429	0.8156	3.9706	0.9991	4.4091	0.4437
24	Satisfying a need to contribute to the field of chemical engineering or to fill certain gaps in knowledge has motivated me to go into research.	3.9821	0.6360	3.8529	0.5535	4.1818	0.7273
	Average	4.0089	0.8430	3.8824	0.6972	4.2045	1.0152

### 3.2.3 Empowerment, skills, and confidence

The empowerment, skills, and confidence mean values are shown in Table 7. The mean values range from 3.4643 to 4.2500, 3.2353 to 4.1765 and 3.7727 to 4.4091 for all, undergraduate and postgraduate respondents respectively. These imply that, on average, the undergraduate and postgraduate respondents' views of this KPI differs. The respondents do not agree that getting a lecturing job was a motivation for conducting research (see question 12). Response to question 19 indicates that the chemical engineering curriculum may need to be strengthened to improve the students' research data analysis skill. Responses to question 29

tend to suggest low interaction with academics from within and outside the immediate school environment. The undergraduate's responses to question 33 show that the chemical engineering curriculum should concentrate more on understanding the theory and concepts guiding research. Furthermore, question 34 indicates the need to use research activities as a way of improving oral presentation skill for the undergraduates. These observations are confirmed by the ANOVA results which indicate there are significant differences in the means for the dimension responses by the undergraduate students.

**Table 7: Empowerment, skills, and confidence**

No	Item	All		UG		PG	
		Mean	Variance	Mean	Variance	Mean	Variance
11	Research gives me confidence in my ability to contribute to science and technology.	4.2500	0.5182	4.1765	0.6952	4.3636	0.2424
12	Getting a lecturing job motivates me to conduct research.	3.4643	1.5260	3.2353	1.2763	3.8182	1.7749
16	Research gives me the confidence to work collaboratively with others.	4.1429	0.5974	4.0294	0.7567	4.3182	0.3225
19	Formulating a research question that could be answered with data is a skill that has been strengthened in me through the chemical engineering curriculum.	3.6071	1.0065	3.5000	0.8636	3.7727	1.2316
21	Understanding the relevance of research to course work is an important skill developed by the chemical engineering curriculum.	3.8214	1.0221	3.6765	0.9528	4.0455	1.0931
23	Research has contributed to my thinking creatively about any project.	4.1429	0.3429	4.0294	0.3324	4.3182	0.3225
25	Research has contributed to my feeling responsible for projects which I partake in.	3.9286	0.8675	3.8824	0.5312	4.0000	1.4286
26	My involvement in research has given rise to a feeling of being part of a research or scientific community	4.0714	0.4675	3.8529	0.4323	4.4091	0.3485
27	My general problem solving skills has been deepened by research.	4.0357	0.7987	3.9706	0.5143	4.1364	1.2662
29	Research activities have greatly improved my interaction with academics from within and outside my immediate school environment.	3.7679	0.7997	3.6765	0.7709	3.9091	0.8485



		All		UG		PG	
31	Being part of research activities has increased my understanding about what everyday research work is like.	3.8393	0.8282	3.7647	0.6702	3.9545	1.0931
32	Understanding journal articles is a research skill that has become enhanced by indulging in research activities.	3.9107	0.4464	3.8235	0.4528	4.0455	0.4264
33	Understanding the theory and concepts guiding research is one of the research skills that have been deepened by the chemical engineering curriculum.	3.7143	0.7169	3.5294	0.8021	4.0000	0.4762
34	Indulging in research activities has greatly improved my oral presentation skill.	3.7500	0.9182	3.5000	0.9848	4.1364	0.5996
35	The ability to conduct observations in the laboratory or the field as a skill has greatly been improved by being part of research activities.	4.0000	0.6182	3.7941	0.7745	4.3182	0.2273
37	Research activities have greatly enhanced my time management skill.	3.9107	0.6646	3.8824	0.7736	3.9545	0.5216
	Average	3.8973	0.7872	3.7702	0.7593	4.0938	0.7690

#### 4. CONCLUSIONS

The research capacity of selected chemical engineering departments in Nigeria Universities were assessed using three key performance indicators (KPIs) namely: (a) research infrastructure, dissemination, (b) knowledge translation and research applicability and (c) empowerment, skills, and confidence. The means for research infrastructure range from 3.6607 to 4.0714, 3.6176 to 3.8529 and 3.7273 to 4.5455 for all, undergraduate and postgraduate respondents respectively. Quite a number of the undergraduate respondents are neutral with respect to each dimension of the research infrastructure while the postgraduate respondent's perception is that achieving peer recognition is not a motivation for conducting research. On the average, the respondents agree with all the dimensions for knowledge translation and research applicability. The empowerment, skills, and confidence mean values range from 3.4643 to 4.2500, 3.2353 to 4.1765 and 3.7727 to 4.4091 for all, undergraduate and postgraduate respondents respectively. These imply that, on average, the undergraduate and postgraduate respondents view of this KPI differ.

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## OPTIMIZATION OF RISERBASE GAS INJECTION FOR SLUG FLOW ATTENUATION

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### ABSTRACT

*A new approach for the optimization of riserbase gas injection for slug flow attenuation has been proposed. Riserbase gas injection is an established method for slug flow mitigation, however it can be expensive due to the high volume of gas required and the cost of compression. In this study, optimum gas volumes required to stabilise a range of unstable slug flow conditions were obtained using bifurcation maps. The results showed that at the bifurcation point, minimum gas injection volume is required to achieve flow stability. This study established that the optimum gas volume required is dependent on the type of slug flow. For a very high frequency slug flow at high flow rate more volume of gas up to 100kg/s is required to achieve stability while slug flow at moderate flow rate could need up to 30kg/s and slug at low flow rate required about 6 kg/s to achieve stability. The developed approach in this work can help reduce the overall cost of gas injection for slug flow mitigation.*

Keywords: Slug attenuation; Bifurcation; Gas injection; slug flow; multiphase flow.

### 1. INTRODUCTION

The search for mitigation strategies for slug flow began in the early 1970s and has continued till date. Considerable advancements have been made ranging from manual topside choking, slug catcher, automated choking, gas injection, dual riser, intermittent absorber, and so on. These techniques could be deployed singly or in a combined mode to achieve more stabilising benefits (Ehinmowo *et al.* 2019). More recently efforts are now concentrated on developing slug flow mitigation techniques that can serve the dual purpose of stabilising slug flow and positively impacting production (Ehinmowo, 2015).

Gas injection involves the injection of compressed gas into the pipeline and has been shown to possess the capacity for slug stabilisation and improved production albeit at prohibitive cost. These cost components include the cost for the procurement of compressors and injection pipeline Schmidt *et al.* (1985) as well as additional energy for the operation of this system. Alvarez and Al-Malki (2003) discussed the results of field testing gas injection in a hilly terrain pipeline. The test proved that gas injection helps to stabilize slug flow. Additionally, it was observed that gas injection is effective in the reduction of wellhead back pressure and pipeline pressure losses thereby resulting in increased production.

Gas injection has a vast application in the petroleum industry. It serves as a secondary recovery method and reservoir maintenance technique. Hill (1989) experimental studies proved that gas injection is a slug mitigation technique and can also help to produce from a dead well. Schmidt *et al.* (1980) recommended gas injection as a mitigation technique for severe slug flow phenomenon but acknowledged the cost as an operational disadvantage. Also, some authors have suggested the combination of gas lifting and choking to be a very effective approach of slug flow mitigation. Jansen and Shoham (1991) performed a detailed investigation on a pipeline-riser, 12.1m long and 2.54cm diameter for the mutual benefits of two severe slug elimination methods: choking and gas lift. Although, choking effectively reduced slug effect but with attendant backpressure increase. On the other hand, gas injection achieved stability but at large volume. The deployment of the two methods were reported to provide an effective means for slug flow control. There was significant reduction in pressure and gas volume required.

Krima *et al.* (2012) also investigated these combined methods for hydrodynamic slug flow mitigation using an industry software package-OLGA. He confirmed reduction in gas volume required and a larger valve opening. This complimentary effects has also been reported in Ehinmowo (2015).

Apart from the large volume of gas required for slug flow stabilisation, the optimum point of injection is another major factor to be considered in the gas injection process. The gas can be injected either upstream or downstream of the riser base. Several authors have proven that upstream gas injection perform better if it is carried out close to the riser base. Henriot *et al* (1999) studied the effect of gas injection on slug flow mitigation. It was reported that at high flow rates, the gas injection upstream of the riser base attained better stability than the gas injection at the riser base. Pots *et al* (1987) also carried out a small scale experiment to investigate gas injection for slug flow control. It was reported that at a very high injection rate, the injection upstream of the riser base performed better than the riser base injection. Additionally, Meng and Zhang (2001) investigated severe slugging in a 25km pipeline in about 700m water depth. It was observed that injecting the gas close to the well formation required a much less (about one quarter) volume of gas.

The flow regime at the point of injection must be given due consideration. Introducing gas injection to a stratified flow can result in flow instability therefore it is important to carry out a proper assessment of the flow regimes and conditions present in the pipeline before carrying out gas injection. The slug flow reported in Al-Kandari and Koleshwar (1999) was as a result of transition from stratified flow to severe slugging as a result of gas injection in an onshore multiphase pipeline.

In a more recent study, Ehinmowo (2015) proved using a stability map that an increase in gas flow rate results in

a corresponding increase in friction loss and riser base pressure. The stability map showed that the flow will attain stability only at substantially high gas flow rate which is a downside of gas injection as a slug flow mitigation. Although gas injection has proven to be an effective technique for slug flow attenuation, the volume of gas required and cost is excessively high. More recently Igbokwe (2020) also reported that insignificant slug attenuation occurs at low gas injection rate but observed considerable mitigating effect at a very high gas volume. There is therefore the need to seek ways to optimize this technique to achieve slug flow stability economically. In this study, gas injection was investigated for some slug type reported in large pipe diameter (Ehinmowo *et al.*,2018).

## 2. MATERIALS AND METHODS

Ehinmowo *et al.* (2016) and (2018) investigated slug flow in pipeline-riser system using experimental and numerical approaches respectively. Three distinct type of slug flow (HIV) was reported. The characteristics of these slug flow and the use of choking for their attenuation investigated and have been reported. Following Ehinmowo *et al.*(2018), in this study, LedaFlow, an industrial software package was employed to investigate attenuation of these types of slug flow using gas injection in a 3.83 km pipeline-riser system (3.7 km and 0.13 km for the pipeline and riser respectively) with an internal pipe diameter of 17'' . The diagrammatic representation of the pipeline-riser system is shown Figure 1

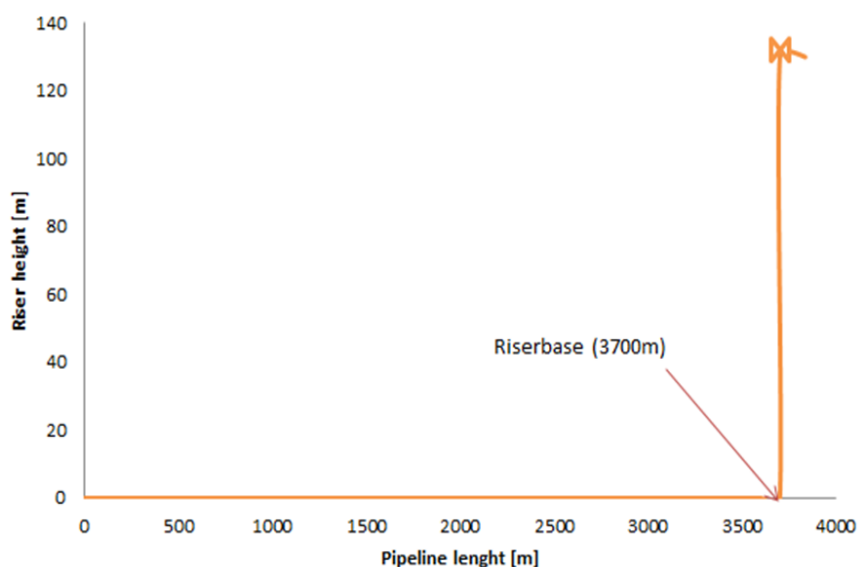


Figure 1. Geometry of the pipeline-riser system.

During the pre-processing, the fluid property was created, pipe materials are stated, meshing and mesh

sensitivity studies were conducted. The mesh convergence showed that 1800 cells was optimum and was adopted for this study. Tables 1 and 2 show the fluid properties and pipe materials properties respectively used in this study.

**Table 1. Fluid properties employed for this study**

Component	Gas	Oil	water
Density [kg/m <sup>3</sup> ]	23	780	1000
Viscosity [kg/m-s]	$1.3 \times 10^{-5}$	$1.1 \times 10^{-3}$	$3.5 \times 10^{-4}$

**Table 2. The properties of pipes and insulation materials for the pipeline and riser**

Material	Density [kg/m <sup>3</sup> ]	Specific heat [j/kg C]	Thermal conductivity [W/m C]
Steel Pipe	7850	500	50
Insulation	2500	880	1

Selected data points in the slug regions already identified in Ehinmowo et al. (2016) and (2018) were investigated for the efficacy of gas injection in this study. Tables 3, 4 and 5 show the summary of the cases.

**Table 3. Flow properties of case study in the H-type slug**

Total mass flow [kg/s]	600
Gas mass fraction[-]	0.01
Oil mass fraction [-]	0.239
Water mass fraction [-]	0.751
Inlet Temperature [°C]	90
Outlet Temperature [°C]	40
PR outlet Pressure [bar]	22.5
Horiz outlet Pressure [bar]	27.95

**Table 4. Properties of case study in the I-type slug**

Total mass flow [kg/s]	120
Gas mass fraction[-]	0.007
Oil mass fraction [-]	0.239
Water mass fraction [-]	0.754
Inlet Temperature [°C]	90

Outlet Temperature [°C]	40
PR outlet Pressure [bar]	22.5
Horiz outlet Pressure [bar]	27.95
Vert outlet pressure [bar]	22.5

**Table 5. Properties of case study in the V-type slug**

Total mass flow [kg/s]	19
Gas mass fraction[-]	0.04
Oil mass fraction [-]	0.239
Water mass fraction [-]	0.721
Inlet Temperature [°C]	90
Outlet Temperature [°C]	40
PR outlet Pressure [bar]	22.5
Horiz outlet Pressure [bar]	27.95

### 3. RESULTS AND DISCUSSION

Gas injection was used to achieve stability for the slug flow conditions. This technique has been widely employed in the petroleum industry for severe slug flow elimination. However, in this study, the identified slug type HIV in Ehinmowo *et al.*(2018) was investigated for the optimisation of gas injection. Bifurcation maps were produced for various illustrative slug flow situations.

#### 3.1 Gas injection bifurcation map for H-type slugging

The riser base pressure bifurcation map for the H-type slugging is shown in Figure 2.

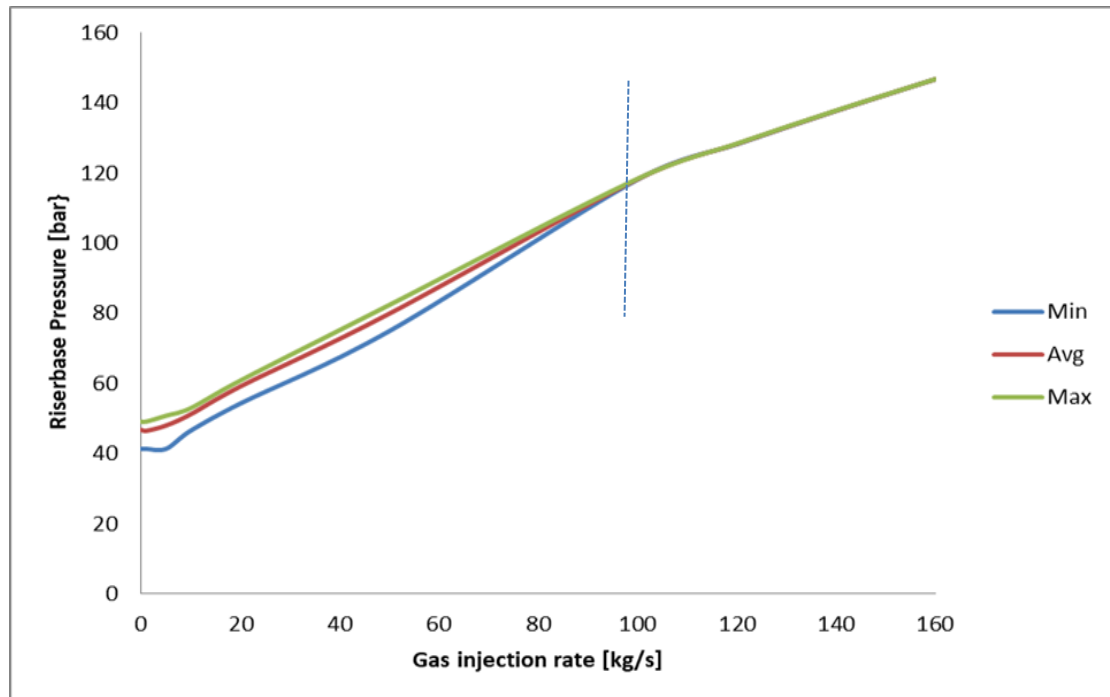


Figure 2. H-region gas injection bifurcation map

The unstable region is characterised by fluctuating riser base pressure. Figure 2 shows a fluctuation bounded by the green line and blue lines for various gas injection rates and the red lines represent the corresponding minimum and average pressures respectively. The bifurcation point demarcated by a dotted blue line occurs at 100 kg/s gas flow rate and at 118.37 bar pressure. The points before the bifurcation point is the unstable region while stability occurs beyond the bifurcation point. It is shown that between below 100 kg/s gas injection rate, various degree of instabilities were experienced. From injection rate of 80kg/s and above, the degree of instability has reduced significantly. This shows that that for the H type of slug which is typical of hydrodynamic slugging, significant volume of gas would be required. This is in agreement with the observation of Krima *et al.*(2012) that proposed the combination of gas injection and choking to solve this problem.

### 3.2 Gas injection bifurcation map for I-type slugging

The bifurcation point is at 30 kg/s gas flow rate and at 30.33 bar. The points before the bifurcation are unstable while the stable regions occur downstream the bifurcation point. It is shown that below 30 kg/s gas injection rate, various degree of instabilities were experienced. However, there is significant reduction in the degree of fluctuation from 20 kg/s injection rate. The shape of the bifurcation map shows something interesting. The pressure were fluctuating at lower pressure compared to when there was no injection. This shows that the injected gas helped to reduce the pressure head contributed by the slug liquid column from the riser. However, beyond the bifurcation point, the pressure value increased. This behaviour can be traced to the unique characteristics of the I-region where various slug types interact. This region has been reported to be complex and difficult to control as there would be interaction between dissimilar contrivances (Ehinmowo *et al.*,2018).

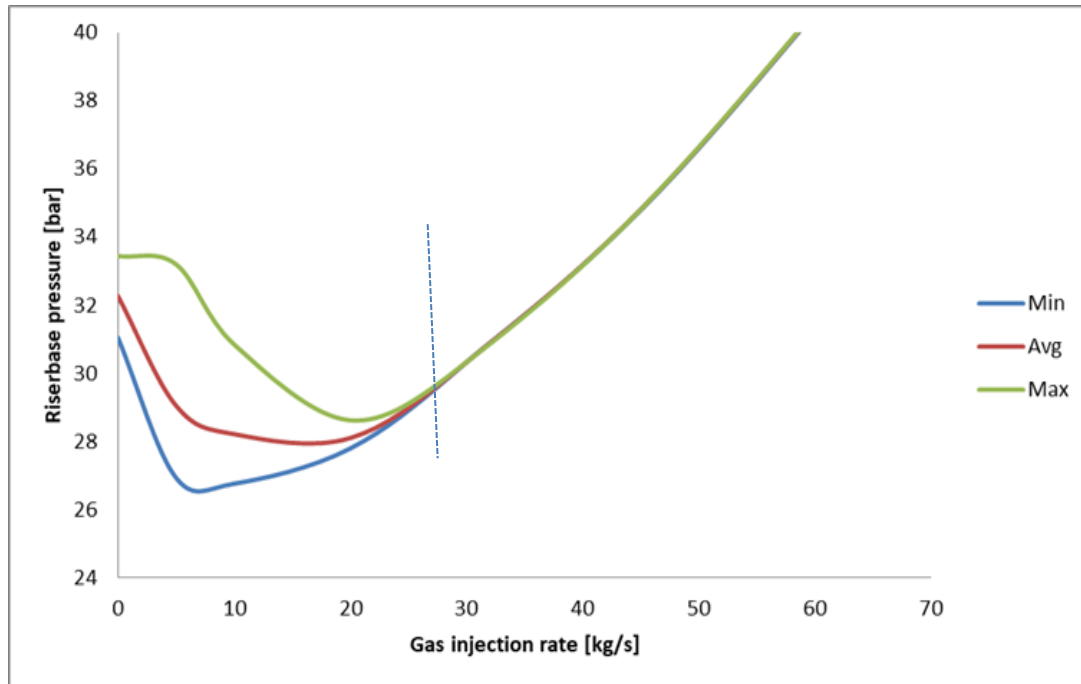


Figure 3. I-region gas injection bifurcation map

### 3.3 Gas injection bifurcation map for V-type slugging

Figure 4 illustrates the V-region gas injection bifurcation map. The bifurcation point is at 6 kg/s gas flow rate and

at 25.10 bar. The region before the bifurcation point is unstable while the flow is stable at the bifurcation point and beyond. It is shown that between below 6kg/s gas injection rate, various degree of instabilities were experienced.

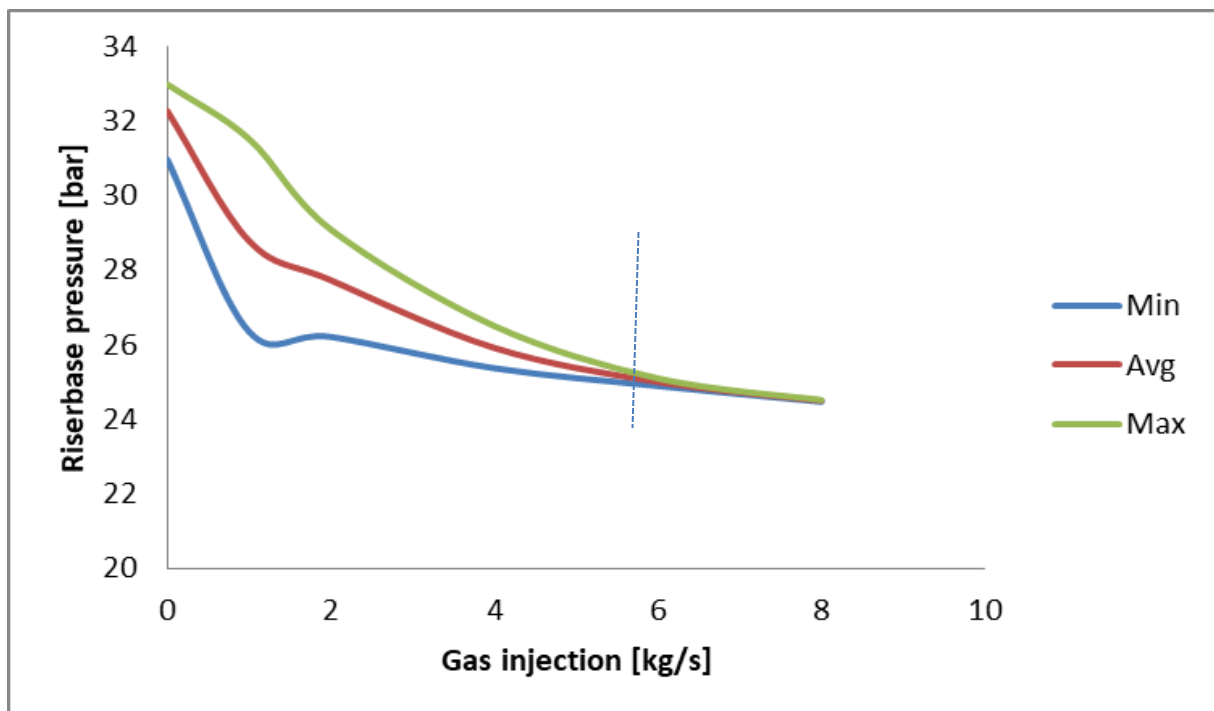


Figure 4. V-region gas injection bifurcation map



Small amplitude fluctuations were experienced between 4 kg/s and 6 kg/s injection rates. Though about 5 Bar maximum pressure fluctuation was observed, the gas injection volume required to overcome this slug type is very small compared with other regions of 100 kg/s and

30 kg/s for H-type and I-type respectively. It is commonly recognised that an inclined pipeline leading to the riser pipe is a precursor to severe slugging (Schmidt *et al.*,1980) However, in this study a pure upstream horizontal pipe was used. This proposes that with or without an inclined upstream pipe, severe slugging can still occur. This view has been reported in earlier works Fabre *et al.*(1990). This type of severe slugging may be due to the continued growth and merger of smaller slugs in a large diameter pipes. The V-region slug type is a kind of this severe slugging.

For each of the conditions investigated in this study, slug mitigation occurs at a minimum gas injection rate. This is the bifurcation point. Below this rate, slug attenuation would be insignificant. At the bifurcation point, the slug flow is totally eliminated and beyond this point, excess gas injection would translate to increased cost at no significant value. Generally, the results showed that without a method to establish the minimum gas required, operators are likely to employ large volume of gas more than required for a particular slug flow condition. This is in consonance with Igbokwe (2020) who systematically increased gas injection rate until the volume of gas was large to change slug flow regime to annular flow. This huge volume is a major reason gas injection could be unattractive. Thus this study provides a milestone in addressing the optimisation of gas injection for slug flow attenuation.

#### 4. CONCLUSIONS

The need to seek attenuation strategy for hydrodynamic slugging in large pipeline made this study compelling. Bifurcation maps were created using an industrial multiphase commercial code-LedaFlow for three distinct slug regions (HIV). Gas injection was shown to possess capacity for attenuating these types of slugging but at substantial cost. The results showed that the required gas volume is a function of the flow conditions (the slug type). It has been shown that huge amount of gas volume was necessary to ensure stability for slug flow at high flow rate compared to other regions. This unfortunately translate to high operational cost. This study has presented an approach to achieving stability

and at the bifurcation point where the cost is least. The study has established that indeed the various slug types can be mitigated using gas injection. In this study, the results have established that bifurcation maps can help establish minimum volume of gas required to mitigate slug flow in pipeline-riser system. An experimental study to validate this is desired and this is a subject for further studies.

#### 5. ACKNOWLEDGEMENTS

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## PYROLYSIS OF SPENT LUBRICATING OIL USING SIMPLE RECYCLING TECHNIQUE TO REDUCE ENVIRONMENTAL HAZARDS

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### ABSTRACT

*The recycling of spent lubricating oil would reduce the environmental concerns associated with its indiscriminate disposal. Since temperature influences the properties of lubricating oil, this study investigated the effect of temperature on the properties and metals composition of used lubricating oil using simple recycling techniques. The spent lubricating oil after pyrolysis yielded 79.1 % liquid fraction, 18.2 % gaseous fraction and 2.7% solid fraction or char. The properties and composition of the pyrolysis oil were determined using the Gas Chromatography Mass Spectrophotometer (GCMS), this shows that the major composition of the refined oil is 34.037 % Octamethyl-Cyclotetrasiloxane (C<sub>8</sub>H<sub>24</sub>O<sub>4</sub>Si<sub>4</sub>). Results indicated that since the properties of viscosity, pour point and metals composition in the pyrolysis oil were improved upon at a high temperature of 420°C, waste or spent oil is a viable feedstock for lubricant production.*

*Keywords: lubricating oil; pyrolysis; fuel properties; temperature; spent oil.*

### 1. INTRODUCTION

Lubricating oils are viscous fluids used to reduce friction during relative motion of surfaces in contact. They are mainly obtained from petroleum, which consists essentially of complex mixtures of hydrocarbon molecules. They are composed of isoalkanes having slightly longer branches and the monocycloalkanes and monoaromatics which have several short branches on the ring (Dygula et al., 2022). These hydrocarbon molecules generally range from low viscosity oils having molecular weights as low as 250, up to very viscous lubricants with molecular weight as high as 1000 (Conrad et al., 2021). The carbon atoms range from 20 to 34. Lubricating oils are viscous liquid and are used for lubricating moving parts of engines and machines. Grease, which is a semi-solid, also belongs to this group. There are three major classes of lubricating oils, namely: lubricating greases, automotive oils and industrial lubricating oil. When lubricating oils are used in service, they help to protect rubbing surfaces and promote easier motion of connected parts. In the process, they serve as a medium to remove high buildup of temperature on the moving surfaces (Decrozant-Triquenaux et al., 2021). Further buildup of temperature degrades the lubricating oils, thus leading to reduction in properties such as: viscosity, specific gravity, and others. Dirt's and metal parts worn out from the surfaces are deposited into the lubricating oils. With increased

time of usage, the lubricating oil loses its lubricating properties as a result of over-reduction of desired properties, and thus must be evacuated and a fresh one replaced (Raina et al., 2021).

Preserving the internal combustion engines in motor vehicles and powered equipment is an important area of applying lubricating oil (Cekerevac et al., 2022). Spent lubricating oil refers to the engine oil, transmission oil, hydraulic and cutting oils after certain period of application. It also refers to the degradation of the fresh lubricating components that become contaminated by metals, ash, carbon residue, water, varnish, gums, and other contaminating materials, in addition to asphaltic compounds which result from the bearing surface of the engines. These oils must be changed and removed from the automobiles, different machineries or utility equipment after certain period of application because they become inadequate from serious deterioration in service (Wolak et al., 2021). Typically, lubricating oil contains ninety percent base oil, most often petroleum fractions called mineral oils and less than ten percent additives. The chemical breakdown of these additives during use results in the buildup of halogenated hydrocarbons in the oil. Polycyclic aromatic hydrocarbons (PAHs) as well as other polycyclic compounds are generated and accumulated in the oil (Nakano et al., 2021) together with metals from wear and tear of the engine being lubricated (Sentanuhady et

al., 2021) These compounds (metals chips, sludge, water, dust, etc.) gradually reduced its quality leading to change in its physical and chemical properties and thereby deteriorated (Sang et al, 2021). Components of used lubricating oil are highly toxic upon release to the environment and it poses harmful effects to both humans and the biophysical environment. The indiscriminate disposal of used lubricating oil can pollutes environment to a great extent and a volume of it can contaminate up to two hundred and fifty thousand volumes of water (Kaithari et al., 2021). Proper management of this hazardous material is therefore important in order to make it a valuable product by greatly reducing the quantity being disposed indiscriminately (Usman et al, 2021).

Management of waste oils is a growing concern particularly in industrial and urban areas. Generation of waste oil is closely linked with increase in population of automobiles and industries (Mishra et al., 2021). The management of waste oil is particularly important because of the large quantities generated globally through transport and Industrial activities. These waste oils may have detrimental effect on the environment if not properly handled, treated or disposed (Ali et al., 2021). In recent decades a number of innovative treatment technologies have been developed that promise to solve technical, economic and environmental problems associated with used oil recycling. The efficient recycling of waste lubricant could help reduce both the environmental pollution and gas emission from greenhouses, thus creating an environmental and economic benefit (Mishra et al., 2021).

Several techniques are available for the regeneration of used lubricating oil, among which are chemical treatment (Hani and Al-Wedyan, 2011) acid activated process that is similar to acid-clay process with little modification (Sanchez-Alvarracin et al., 2021) physical treatment by distillation and thin film evaporation (Criollo-Bravo et al., 2021) and solvent extraction (Katiyar and Husain, 2010). However, cost, low recovery, high energy demand and poor lubricating properties have been the major setback to its acceptability. This study intends to improve on the existing pyrolysis process of recycling used lubricating oil. These negative environmental impacts associated with the disposal of the used (waste) lubricating oil necessitated the research into the recycling of waste lubricating oil. This study aimed at investigating the possibility of recycling spent lubricating oil through pyrolysis process. Therefore, we report the recycling

process of spent lubricating oils using a simple technique which includes cheaper chemicals, utilities and equipment relative to other recycling or re-refining options for protecting the environment and conserving resources.

## **2. MATERIALS AND METHODS**

The used oil sample was collected from an internal combustion engine of a car under repair at a mechanic shop located along the University of Ilorin road, Tanke, Nigeria.

### **2.1. Characterization Procedures**

#### **2.1.1. Specific Gravity**

This was investigated as the ratio of sample density to that of water using analytical balance and density bottle.

#### **2.1.2. Kinematic Viscosity (ASTM D445)**

This was examined using a commonly reported technique obtained from literature (Mekonnen and Ababa, 2014). The viscosities of oil at 40°C and 100°C were determined using the falling ball method, for 100°C the sample was heated in a water bath tub at 100°C. In the falling ball method (Mekonnen and Ababa, 2014), the sample was poured into a graduated tube channel containing the ball, the time taken by the ball to fall through the medium was recorded.

The relation in Eqn. 1 is used to calculate the viscosity using the time data. Finally, the kinematic viscosity was obtained by dividing the dynamic viscosity with the respective density of the oil.

#### **2.1.3. Flash point**

The flash point was measured using a Stanhope–Seta Pensky –Marten closed cup flash point tester (Model 13661) consisting of a closed copper cup, heater, and a source that gives continuous sparks. The sample was heated, and its vapour accumulated inside the sample holder until it was sufficient to ignite. The temperature measured immediately ignition occurred [Babatunde et al, 2020].

#### **2.1.4. Pour point**

The pour point was determined according to the ASTM D97 method.

#### **2.1.5. Cloud point**

Test method according to ASTM D5773 using constant cooling method was used to determine the cloud point.

## 2.2. Pyrolysis of Used Lubricating Oil

The reactor is a batch type consisting of an inner and outer cylinder separated by glass wool lagging material serving the purpose of an insulator to prevent heat losses. An elliptical convergent top, exhaust chute, a condenser, an indicator coil, contractor, and two thermocouples for controlling temperature of the reactor heating element and reactor interior. The reactor is constructed of a stainless steel material and powered electrically with temperature rising to about 800°C. The reacting vessel was cleaned and 1L of the used lubricating oil was introduced and uniformly heated from all sides. The Pyrolysis experiment was carried out at temperatures ranging from 350°C to 500°C with a heating rate ranging from 10°C/min to 30°C/min.

A drum containing ice cold water is connected to the reactor condenser to supply cooling water that aids the recovery of oil from overhead vapour, the condensed oil was collected from the outlet of the condenser in a collecting jar and then measured and weighed. The remaining residue collected and weighed after cooling the reactor. The weight of the non-condensable gases was measured by mass balance.

## 2.3. Analysis of the pyrolysis oil

The pyrolysis oil was analyzed using the Gas Chromatography Mass Spectrophotometer (GC-MS) by Agilent Technologies GC system 7820A, USA, using a DB-23 wax column with N<sub>2</sub> as a carrier gas, to test for the chemical composition of the liquid. Characterizing tests were also carried out to determine the following properties: Yield, density, kinematic Viscosity, colour, flammability and pH. The colour of the pyrolysis oil was determined by visual inspection.

## 3. RESULTS AND DISCUSSION

The Product yield from pyrolysis experiment is presented in Table 1. This shows that the spent lubricating oil that was pyrolysed produced highest liquid fraction with 79.1%, low solid fraction or char of 2.7% and 18.2 % gaseous fraction. The result shows a very good oil yield from lubricating oil pyrolysis at a pyrolysis temperature of 420°C and a residence time of 1 h. Other researchers obtained similar oil yields to that of current study. Manasomboonphan and Junyapoon (2012), obtained a  $\geq 50\%$  wt liquid yield at 350°C and Selukar and Wagh (2014) obtained 83% oil yield at 430°C. The disparity in the results is as a result of the differences in reactor configuration and experimental

process variables. The low char produced is characteristic of pyrolysis products from a hydrocarbon base feedstock. Biomass pyrolysis consequently produces far more char than hydrocarbon pyrolysis for similar temperatures (Jahirul *et al.*, 2012).

The high gas yield is an indicator that there was a significant occurrence of secondary reactions in the pyrolysis process. Gas yields are high in high and medium temperature pyrolysis and in high residence time pyrolysis. The results obtained in this study were due to the reactor configuration. The reactor design determines the directional pattern of heat flow, product vapour and the effectiveness of condensation of product vapour. Also the high gas yield can also be a result of the cooling system which was insufficient to cool and condense the escaping vapour. The minimization of heat losses from the system is another key factor that is controlled by the reactor design. The oil yield from the process can be improved by the utilization of a catalyst in the process.

**Table 1: Product yield from pyrolysis experiment.**

PRODUCT	YIELD (%)
Liquid	79.1
Char	2.7
Gas	18.2

The investigated fuel properties and metals in the different categories of lubricating oil are reported in Table 2. From the experimental analysis carried out, pyrolysis oil obtained properties. Flash point, kinematic viscosity at 40°C and 100 °C, viscosity index (VI), pour point, fire point, and metal contents (Cu, Pb, Fe) are the additional characteristics tested. This characterization helps to explain the quality improvement of the pyrolysis oil as compared to the properties of the used oil sample and fresh oil presented in Table 2. The flash point of the recovered oil is 98°C which is different from those of the spent lubricating oils (used oil [124°C] and fresh oil [246°C]). The oils were characterized for density because air-fuel ratio and energy content within an engine's combustion chamber are evidently influenced by fuel density. Also, fuel density does affect engine performance because fuel injection pumps metre fuel by volume. The pyrolysis oil showed an increase in density and a simultaneous decrease in viscosity. This implies that the density is independent of its viscosity and vice-versa. This is in agreement with the findings of

Akhihero *et al.* (2019) that with high density, incomplete combustion resulting in carbon deposits do occur because of the consequent low volatility and poor atomization of the fuel. This may be due to the presence of contaminants in the fuel.

Decrease in viscosity at different temperature values (40 °C and 100 °C) indicates that though temperature may have influenced the extent of viscosity of the lubricating oil, viscosity generally reduces with temperature decrease. Therefore, increase in temperature decreases the viscosity of lubricating oil due to the action of heat. Since previous research has shown that viscosity is improved when diesel fuel is blended with lubricating oil, blending therefore would significantly reduce the viscosity of lubricating oil. Kinematic viscosity is important because it influences the performance and efficiency of engines (Akhihero *et al.*, 2019). Both viscosity and density are physical properties which determine engine performance more than other properties. However, increase in kinematic viscosity does affect engines because of the presence of unreacted triglycerides in highly viscous oil. Similarly, Babatunde *et al.* (2020) reported that high viscosity is a problem because it influences the flow of fuel and spray characteristics.

The result of -28°C pour point of pyrolysis oil is close to the pour point of fresh oil, this shows a significant improvement as compared with the 5°C pour point of

spent lubricating oil. Due to the presence of contaminants, an increase in pour point of lubricating oil which reduced the lubricating oil quality was reported. However, temperature has a more significant effect on pour point because the pyrolysis process significantly improved the point due to the action of heat on the intermolecular forces of the oil. It is important because it is one of the cold flow properties which determine the flow-ability of oil. A recent study conducted on pour point reduction found that as an additive, 20% ethanol-fuel mixture accomplished 30°C temperature reduction in the fuel pour point (Dehaghani and Rahimi, 2018).

Flash and fire points are properties that indicate the safety of fuel relating to its' flammability. This implies that the higher these properties are, the better the quality of the fuel. However, result in Table 2 shows that there was decrease in both properties after pyrolysis of the lubricating oil. This reduction probably implies the presence of contaminants in the oil (Akhihero *et al.*, 2019).

The presence of metals in the spent oil could be attributed to high friction between the moving parts of an engine caused by lost in the lubrication properties of the oil over prolong use. Also, the metal composition result of pyrolysis oil presented in Table 2 reveals a general decrease after pyrolysis reaction. This shows a significant improvement in lubricant properties as compared with the used oil.

**Table 2: Fuel properties and metal constituents in lubricating oils**

Fuel Properties	Fresh Oil	Spent Oil	Pyrolysis Oil
<b>Density (Kg/m<sup>3</sup>)</b>	890	860	870
<b>Kinematic Viscosity (cSt)</b>	@40°C	246	177.9
	@100°C	18	20.9
<b>Flash Point (°C)</b>	246	124	98
<b>Pour Point (°C)</b>	-27	5	-28
<b>Viscosity Index</b>	120	161.2	152.9
<b>Fire Point (°C)</b>	-	134	105
<b>Cu (ppm)</b>	-	1.79	0.51
<b>Pb (ppm)</b>	-	0.6	0.1
<b>Fe (ppm)</b>	-	36	0.81

The GCMS analysis shown in Table 3 presents the chemical compounds found in pyrolysis oil, which are a mixture of low and high molecular weight aliphatic and aromatic hydrocarbons.

The chemical compound present are the siloxane (Octamethyl- Cyclotetrasiloxane 34.0%), (Decamethyl- Cyclopentasiloxane, 10.3%), and (Dodecamethyl-

Cyclohexasiloxane , 5.9%) which is made up of silicon, oxygen, and alkane. (silicon is a non-metallic element abundant on the earth). They are silicon based ingredients used in the cosmetics, deodorant, lubricants and soaps to soften, smoothen and moisten, similarly, Azulene which is also present, can be used for razor burns, redness and irritation.( Udonne, 2011)

**Table 3: Chemical Compounds obtained in the Pyrolysis oil**

Retention Time	Name of Compound	% Composition	Chemical Formulae
13.150	Octamethyl- Cyclotetrasiloxane	34.037	C <sub>8</sub> H <sub>24</sub> O <sub>4</sub> Si <sub>4</sub>
19.243	Decamethyl- Cyclopentasiloxane	10.361	C <sub>10</sub> H <sub>30</sub> O <sub>5</sub> Si <sub>5</sub>
19.874	Azulene	2.435	C <sub>10</sub> H <sub>8</sub>
23.915	2,4,4,6-Tetramethyl-6- phenyl-2-heptene	3.902	C <sub>17</sub> H <sub>26</sub>
25.260	Dodecamethyl- Cyclohexasiloxane	5.942	C <sub>12</sub> H <sub>36</sub> O <sub>6</sub> Si <sub>6</sub>

#### 4. CONCLUSION

The effect of temperature on the fuel properties and metal constituents of diesel engine lubricating oil has been reported. Since the lubricating oil was categorized on the basis of usage and pyrolysis, these processes were noted to have occurred at different temperatures. The fresh lubricating oil was at normal room temperature, while the spent and pyrolysis oils were 300°C and 420°C respectively. The pyrolysis spent lubricating oil yielded 79.1 % liquid fraction, 18.2 % gaseous fraction, and 2.7% solid fraction or char. The significant improvement in the properties of refined lubricating oil showed an increase in viscosity index and density for the pyrolysis oil, while a reduction in kinematic viscosity, pour, flash, and fire points were also reported from the experiment. This indicates that spent (used) lubricating oil undergoes pyrolysis, since most of the properties of the pyrolysis oil were improved, high temperature can be said to significantly enhance the fuel properties of lubricating oil. The GCMS analysis shows that the major components of the refined oil is 34.037 % Octamethyl-Cyclotetrasiloxane

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## STRATEGIES FOR TEACHING-FOCUSED INDUSTRY-ACADEMIA COLLABORATION IN ENGINEERING

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### ABSTRACT

*Industry-academia collaboration largely depends on the way the nexus is defined as well as the extent of formalization of the relationship. The process of teaching and learning are a lot better when there is a concerted unison of research and knowledge transfer from the higher institutions to the industry and vice versa. Jointly organized course-based and project-based teaching collaborations have become increasingly crucial in guaranteeing that the student benefits from both sides of the theoretical and practical divide. This paper presents a review of research literature on the possible industry-academia success factors and seeks to highlight avenues through which this bond can be better strengthened. In a bid to close this lacuna between academia and industry, infusing industry experts to supervise research projects on one hand and improving teaching methods via curricula improvement on the other hand will prove beneficial.*

**Keywords:** Industry-Academia collaboration; teaching; engineering education; joint-based research; curriculum development.

### 1. INTRODUCTION

Partnerships between industry and academia is not necessarily a novel concept as it has been proven to be beneficial to both the industry partners and the academic institution when appropriately applied. This emphasizes the need to prioritize impactful collaborations between the industry and academia especially in the wake of the fast-paced modernization and global technological surge. Recent advances in technology as well as industry-specific processes now mean that institution syllabuses and teaching methods must be adapted or reconfigured to match the realities of the business and industrial world. There exist several institutions of higher learning already offering industry-tailored courses as part of their curriculum which points to the reality that such synergy is paramount in closing the gap between the classroom and the workplace.

The ultimate motive behind the push for better teaching strategies to enhance this industry-academia synergy is to connect students with real-world projects that enhances their education and tackles the increasing engineering and societal challenges (Diane, 2016). In examining the historical development of the industry-academia relationship, it is pertinent to note that the industry perceives the partnerships from the angle of how their profits can be positively impacted while the

academia finds the collaboration beneficial to the overall growth of the students and the research quality.

One of the most crucial benefits of collaboration between the industry and the research community is the capacity to engage in academic research which positively impacts both economy and the society (Perkmann, 2013). This also means that the institutions are availed the opportunity to stay updated on current industry trends resulting in graduates that are ready for the workplace; significantly lessening the learning curve required when they arrive in the industry. A plan of action is thus critical in ensuring that teaching-focused industry-academic collaboration is sustained and consistently improved upon.

### 2. TEACHING-FOCUSED I-A PARTNERSHIPS

While it is true that teaching is a paramount mission of higher institutions, there is not so much known about the industry's influence on the school's teaching methods. Good teaching plays a critical role in enhancing the quality and reputation of a university. With an appropriately designed curricula, the school can ensure that students are ready for the industry upon graduation (Dhruba, et al., 2020).

Existing studies on teaching-focused partnerships offer in-depth analysis on the organizational aspects of such

collaborations, i.e., how these collaborations occur and the incentives for both industry and universities to develop such collaborations (Orazbayeva et al., 2019).

It is important for universities to formulate strategies to support their teaching performance and impact. The core of the strategy is to develop teaching-focused industry–academia collaborations, which refer to collaborations between universities and corporations for teaching activities, curriculum review or development, training students and academics, and setting up laboratories etc (Orazbayeva et al., 2019).

Despite the importance of teaching for universities, and the wider socioeconomic environment, little research has been conducted on teaching-focused industry–academia collaborations. Instead, university–industry linkages have been mainly discussed in the context of partnerships for joint research, contract research, consulting, technology transfer, and creating spin-offs (Perkmann et al., 2013).

Perkmann et al. (2013) conducted a systematic review of 109 studies on university–industry collaborations published during the period 1990–2014, and he reported that none of the reviewed studies addressed the consequences of the engagement on teaching and learning experience of students affiliated to universities that engaged with the industry.

In Nigeria, there are several Engineering graduates produced each year, however the quality of the graduates has remained a source of concern as several of them have been reportedly noted to lack employability competencies.

### **3. STRATEGIES TO STRENGTHEN I-A PARTNERSHIP**

Several researchers have chronicled different strategies and methods that can improve the relationship between the industry and academia. These methods are widely used in more developed countries as opposed to the developing ones. While it is factual that some developing countries are now catching up with the trend, there is still a lot to be done to reach that needed peak. According to Dr. Akuma Oji, Director of the NLNG Centre for Gas Refining and Petrochemical Technology, Rivers state, Nigeria - which is a postgraduate institute that has institutionalized partnership with the industry, the essence of such collaboration in his view is to provide graduates with a broad-based training necessary

to guarantee their acceptance and success in the workplace afterwards.

The following strategies will better ensure that this relationship is improved upon:

#### **3.1 Course-based and Project-based Teaching Collaborations**

The Course-based collaboration mainly entails the application of industry input into the academia module framework. This can either be in the form of theoretical additions or practical inputs. Project Based Learning on the other hand, is noted as one of the desirable methods for engineering education. It is based on using practical projects where students work in groups to evolve scientific theories on actual problems. The students are divided into separate groups where each group works on a certain project (Ahmed, et al., 2015). This enhances the learning potential of the students.

#### **3.2 The Role of Policymakers**

A key policy priority endeavors to see that graduates are well equipped with the necessary employability competencies. Increased involvement in university education, focus on skills, entrepreneurship, employability of higher education graduates and bringing education and business closer together are policy initiatives pursued in several European countries and regions (Thune, et al., 2011). The consequence of such policies in this regard is that education and training systems become major parameters when the policy makers seek to stimulate engineering and technological innovation.

#### **3.3 Mutually Shared Course Development and Teaching**

For a cordial relationship between the institution and the industry, regular updates and improvements need to be shared from the companies to the universities and vice versa. This means that the industry should be consulted to support in the development of the curriculum as well as in its teaching. Seeking the opinion of the industry professionals will go a long way in enshrining the needed trust for the industry-academia collaboration.

#### **3.4 Governmental Intervention**

Governments may also establish intermediary structures such as research parks, consortia, university research centers and grooming centers (Hayter, 2016; Kodama, 2008; Suvinen et al., 2010). These intermediary organizations act as border spanners (Wright et al., 2008), linking universities with potential industry

partners and vice versa, and thereby contribute towards reducing search and bargaining costs for both universities and companies (Kodama, 2008). They also offer a common geographical space to industrial and academic organizations to work together on research and commercialization projects (Massa and Testa, 2008; Suvinen et al., 2010). Thus, having access to such government-supported organizations may better the universities' propensity to forge that nexus with the industry.

### 3.5 Quality and Research Strength of University Lecturers

It is a reality that high engagement in academic research and extensive production of academic publications and patents within a particular field indicate high quality of a university's knowledge and expertise in that field which helps the university to establish legitimacy in the academic and industrial community (Karlsson and Wigren, 2012), which ultimately attracts industry collaborators.

### 3.6 Geographical Location of the Institutions

The location of universities also plays a central role in developing research and commercialization focused institution-industry collaborations (Youtie and Shapira, 2008). Finding industrial partners for research collaborations and an investor for university spinouts becomes easier if the university is domiciled in an industrial cluster (Storey and Tether, 1998). Furthermore, geographical nearness offers the necessary continuous and regular interactions between the involved partners, helping the partners to develop trust and overcome barriers brought about by the differences in the respective institutional setup.

## 4. BEST PRACTICES AND FUTURE REQUIREMENTS

The implementation of teaching-focused collaboration between the industry and academia is hinged on the adoption of the right tutoring techniques which emphasizes curriculum development, revolutionized trainings, and simulation of industry-type laboratories. One major impediment to industry-academia collaboration is the fact that academics are quick to publish the results of their research whilst the industry guards the proprietary information of their research or findings.

Another crucial point of note is that whilst academics focuses on basic research and the formulation of new hypothesis, the business place investors are mainly concerned with applied research that improves products and maximizes profits in the short term (M.M. Gandhi,

2014). There is no gainsaying that irrespective of the number of engineering graduates churned out by universities, if they are deemed unemployable by companies, it reflects poorly on the institutions as well as on the teaching methods employed.

Therefore, an appropriate institutional setting that fosters a positive industry-focused learning where the employability of the graduate is bettered is critical to the collaborative success of the university-academia partnership (Dhruba, et al., 2020). A major incentive for institutions to involve industry in their teaching activities is to enhance the industrial relevance of education which can then amplify the student's experiences as well as their work prospects (Plewa et al., 2015).

Looking forward, as we know, technology is improving rapidly and so is the skill required to follow up. This means that courses need to be updated and taught as per increasing innovation and societal needs. Faculty bureaucracy, with respect to curriculum modification are often impediments; however, a way around this is to teach some of these industry-based courses as off-curriculum courses.

## 5. CONCLUSIONS

A healthy nexus between the industry and academia ensures a balanced and well-rounded output from the institutions since the students will be adept at both theoretical skills as well as the practical knowhow. Thus, the methods adopted by the institutions in teaching is very vital in ensuring that this objective is realized.

Appropriately designed curriculum and the training of academics with industry-relevant competencies will go a long way in concretizing this synergy. An increased number of industry-oriented thesis or projects that are co-supervised by academics and industry personnel are also critical in fostering this relationship. It is therefore imperative that academia must strive to strengthen its bond with the industry such that the seamless transfer of technology from the workplace to the classroom becomes a consistent phenomenon.

## 6. RECOMMENDATIONS

The following are recommendations based on the review done in this work:

- A more robust approach to teaching and learning which adopts a combination of field work, internships and industry-specific projects should be

employed to better the industry-academia partnership.

- The adoption of a problem-based learning strategy which is also student-centered and imbibes the tenets of groupworks where the students work as a team and attempt solutions to real-life challenges.
- The assessment of student's industrial/internship attachment projects or reports should be more than a tick-the-box exercise. Faculty should invest in adequate monitoring of participation with emphasis on learnings acquired by the students whilst on the internship program.
- Curriculums and syllabuses should be tweaked to include more of the industry-tailored educational programs with a principal focus on current engineering and societal trends.
- An increased number of industry-oriented projects or thesis which would be co-supervised by faculty and experienced industry staff.
- Governmental interventions mandating industries that recruit certain field of Engineering graduates to be compulsorily invested in the provision of support to the institutions in the form of grants, scholarships, research aids and other related support mechanisms.
- Institutions to invest in the creation of a liaison department with the sole purpose of dialoging with industry experts and company reps to understand their research needs and how to optimize their businesses.
- Inclusion of Engineering entrepreneurship and sustainability modules in education at all levels to impart business competencies as well as boost their sustainability awareness.

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## CHARACTERIZATION OF METAKAOLIN PREPARED FROM UN-TAPPED KAOLIN DEPOSIT AS ALTERNATIVE TO CEMENT IN CONSTRUCTION INDUSTRY

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### ABSTRACT

*In this work, the production and characterization of metakaolin from un-tapped kaolins deposit were carried out and their pozzolanic reactivity in cement replacement were evaluated between 28days and 56days accompanied by a steep decrease of  $\text{Ca}(\text{OH})_2$  content. The physiochemical properties of the two kaolins and metakaolins were studied by X-ray fluorescence (XRF), thermogravimetry analysis (TGA) and X-ray diffraction (XRD) analysis. The properties of cements containing metakaolin were monitored for period up to 56days. Two metakaolins derived from indigeneous Ahoko and Aloji Kaolins were used. Cement mortars and concretes with 0%, 5%,10%, 15%, 20%,30% and 35% metakaolin's were studied. Water demand and setting time were determined in all samples. The blended cements of the metakaolins demand significantly more water than the relatively pure cement. 10% replacement has the highest water demand percentage .it is concluded that a 10% metakaolin content of Ahoko and 10% metakaolin content of Aloji seems to be generally favorable than the other replacement percentages. The produced metakaolins derived from indigenou kaolins are good pozzolans with respect to setting time and water demand.*

**Keyword:** cement; metakaolin; dehydroxylation; pozzolan; setting time

### 1.0 INTRODUCTION

Concrete is the building material to meet housing demand. However, cement is the central and main raw material which is regularly costly in developing countries because of the significant energy consumption related with its manufacturer (Fernandez, 2009). Scrivener and (Nonat, 2011), reported that, concrete is the widely produced and used artificial material, compare with other building materials for sustainability. The main constituent of concrete is cement, and it accounts for almost 8% of worldwide artificial  $\text{CO}_2$  emissions. These occur due to high temperature of production, and carbonation of limestone which lead to the production of clinker, the major constituent of cement. Blending of conventional Portland cement with pozzolanic materials is the most appropriate option to reduce the costs and environment impact. Pozzolans are found in natural deposit or can be gotten as by products in agro-industrial applications. The attention of cement manufacturers was drawn, towards pozzolans materials for their good execution as cement replacement materials (Neville, 1996). There is need to assess the availability of supplementary cementing materials locally. Fly ash and blast furnace slags have been extensively used as supplementary cementing materials

(SCMs) but forecast production of these materials did not meet growth in demand for cement. The volume of production of the fly ash and blast furnace slags and their material characteristics are subjected to a technological process not controlled by the cement industry; their availability is dependent on the degree of economic development (Schneider et al., 2011). The most common cementitious materials that are used as concrete constituents, in addition to Portland cement, are fly ash, ggbs (ground granulated blast-furnace slag) and silica fume. They save energy, conserve resources and have many technical benefits (Neville, 1996). Metakaolin, produced by controlled thermal treatment of kaolin, can also be used as a concrete constituent, since it has pozzolanic properties (Makavicky, 1994). Kaolin Clay undergoes three stages of thermal behavior which includes: dehydration, dehydroxylation and recrystallization (Mohammed, 2017). (Kakali et al;2001), reported that, the end of dehydration stage occurred at  $100^\circ\text{C}$ , whereas (Mohammed, 2017) mentioned that it can extend up to  $200^\circ\text{C}$ . The temperature ranges for the dehydroxylation and recrystallization stages changes, depending on the clay structure and mineral present. Thermal dehydroxylation of kaolinite has been studied and investigated by numerous numbers of researchers, and some of them

discussed the kinetic description of the observed reaction rate (Shani, 2010). Dehydroxylation of kaolinite using molecular dynamic study and pozzolanic behavior of metakaolin and effect (Dunster, 1993)

This paper is focused on the properties of cements containing metakaolin monitored for periods up to 56days. Two metakaolins, derived from indigenous kaolins were used to form part of research project, which aims to exploit Ahoko and Aloji indigenous kaolins in concrete technology.

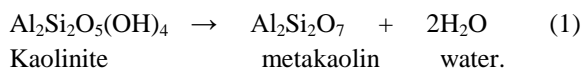
## 2.0 materials and methods

### 2.1 purification of kaolin

An indigenous raw kaolin samples were collected from Ahoko and Aloji site in Northern part of Nigeria in Kogi State. The two kaolin samples were mechanically treated using manual crushing. Thereafter, purified by sedimentation. The kaolin samples were oven-dried at 125°C for 12 hours to less than 0.5% moisture content.

### 2.2 Metakaolin Production

The kaolins, Ahoko and Aloji were thermally treated in a pro-pilot plant furnace at 550°C for 1hour. The complete transformation of kaolinite to metakaolinite was confirmed by X-ray diffraction (Siemens D5000 diffractometer—nickel-filtered Cu Ka<sub>1</sub> radiation k<sub>1</sub> 1:5405 Å). The metakaolins derived from Ahoko and Aloji were referred to as MK1 and MK2 respectively.



Equation 1 represents dehydroxylation reaction of kaolinite to metakaolin

### 2.3 Production of Mortar and Concrete.

The cements were mixed with water at water-to-cement ratio (w/c) of 0.52 to produce mortar and concrete. The concrete produced by the two kaolins, Ahoko and Aloji samples were cast in cubic mould. (100.100.100 mm) each, and mortar produced by the samples were cast in cubic mould (50.50.50 mm) each. The specimens were left in the moulds for 5 hours, then were put in polythene containers, sealed hermetically and water-cured at 20°C. Samples hydrated for periods of 7, 28, 56days (Kakali et al;2004). Blended cements were produced by replacing Portland cement with 5% w/w, 10% w/w, a and 15% w/w, of Mk1 and MK2 for production of mortar. And 5% w/w, 10% w/w, 15% w/w, 20% w/w, 30% w/w and 35% w/w for the production of concrete. The setting time and water

demand of mortar and concrete, cured for up to 56days were determined.

## 3.0 RESULTS AND DISCUSSION

### 3.1 Oxide Composition of Kaolin

Two kaolins, K1 (Ahoko) and K2 (Aloji), having varying chemical composition were examined and their compositions are stated in Table 1.

**Table 1: Chemical composition of kaolins (% w/w)**

	K1(/Ahoko)	K2(Aloji)
SiO <sub>2</sub>	54.039	54.05
Al <sub>2</sub> O <sub>3</sub>	40.827	26.21
Na <sub>2</sub> O	0.109	0.04
K <sub>2</sub> O	0.537	0.18
Fe <sub>2</sub> O <sub>3</sub>	2.009	-
MgO	0.177	0.22
Feo	-	2.94
CaO	-	3.61
TiO <sub>2</sub>	-	2.37
L.O.I.	2.07	10.37

The result shown in Table 1. is of Aloji refined kaolin, and is based on the phase analysis. The titanium oxide in the kaolin emanate from anatase, potassium and sodium are closely related to muscovite and illite. The Feo is suspected to be from iron containing mineral like goethite, or colloidal particle from kaolin crystal structure (Hu & Liu, 2003). The alumina and silica contents are 26.21% and 54.05% respectively. The alumina-silica ratio is 0.48. The chemical composition is similar to that of kaolin deposits studied by Hu and Liu (Hu & Liu, 2003), and Dai and Huang (Dai & Huang, 1999). The fluxes (TiO<sub>2</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, CaO and MgO) are suspected to have lowered the refractory value of the kaolin, but because the alumina content is relatively high, this effect is minimal. The result in table 1, of the Ahoko refined kaolin shows a significant reduction in the percentage composition of SiO<sub>2</sub> and corresponding increase in the percentage composition of Al<sub>2</sub>O<sub>3</sub> for the refined. A high percentage of Al<sub>2</sub>O<sub>3</sub>(>30%) in the purified sample also indicates that Ahoko kaolin clay can be classified as kaolinite rich clay.

### 3.2. Oxide Composition of Produced Metakaolin

Table 2, is the result of oxide composition of metakaolins. Metakaolin is an amorphous aluminosilicate structure produced by control calcination of origin kaolin which break off -OH chain from the kaolinite to become pozzolan (Mohammed,



2017). Pozzolans have the ability to react chemically with calcium hydroxide in the presence of water to form cementitious compounds (Neville, 1996). However, metakaolin obtained from dehydroxylation of kaolinite comprised majorly oxide of aluminium and silica. From the table below, it can be seen that the sum of percentage composition of silica and Aluminium for Ahoko metakaolin is 95.965% and 82.259% for Aloi metakaolin respectively. This indicates that the metakaolin produced is a good pozzolana since these values are greater than 70%.

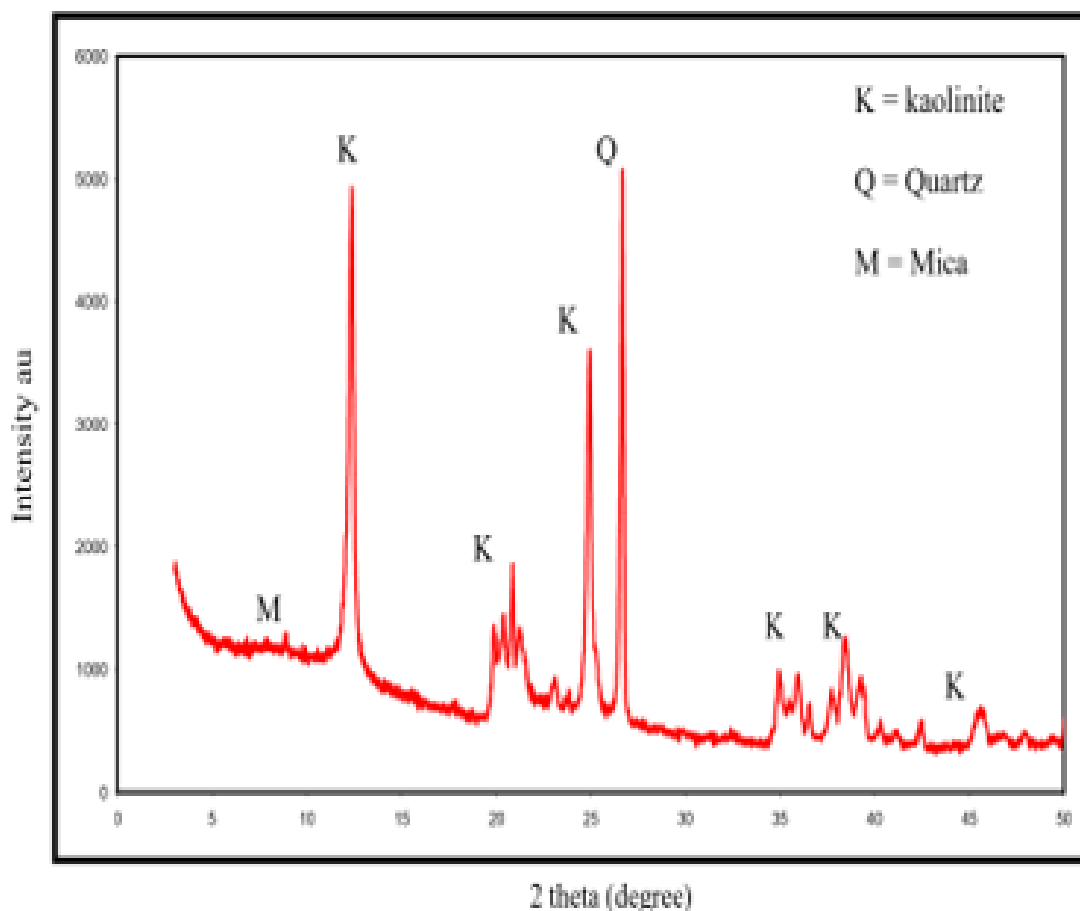
**Table 2: chemical composition of produced metakaolins. (% w/w)**

	MK1(Ahoko)	MK2(Aloji)
SiO <sub>2</sub>	54.195	54.15
Al <sub>2</sub> O <sub>3</sub>	41.477	28.11
Na <sub>2</sub> O	0.066	0.04
K <sub>2</sub> O	0.537	0.18
Fe <sub>2</sub> O <sub>3</sub>	0.887	-
MgO	0.54	0.22
Feo	-	0.84
CaO	-	3.61
TiO <sub>2</sub>	-	2.47

### 3.3. X-Ray Diffraction Analysis

#### 3.3.1 X-Ray Diffraction of Refined Kaolin

Figure 1 is a result of X-Diffraction of refined kaolin. XRD is a test used to confirm the structural characteristics of crystalline samples because it gives a unique fingerprint of samples under investigation. XRD is based on the principle of scattering phenomena whereby crystals perform the function of diffraction grating toward an incident X-ray. The atoms in the crystals scatter the X-rays in all directions and in a way unique to specific material (Kovo, 2010). The XRD pattern of the refined samples of kaolin reveals the degree of crystallinity and crystallite size. It indicated a sharp and narrow peak with a basal reflection at  $2\theta = 12.37^\circ$  ( $d = 7.15 \text{ \AA}$ ). This is also similar to the peak position in the refined sample of Aloi kaolin. This was carried out to determine the level of quartz, kaolinite in the sample. The amount of non-clay mineral (quartz) is almost the same level with the kaolinite.



**Figure 1. XRD pattern of Refined Ahoko kaolin**

## 3.3.2 X- Ray Diffraction of Metakaolin

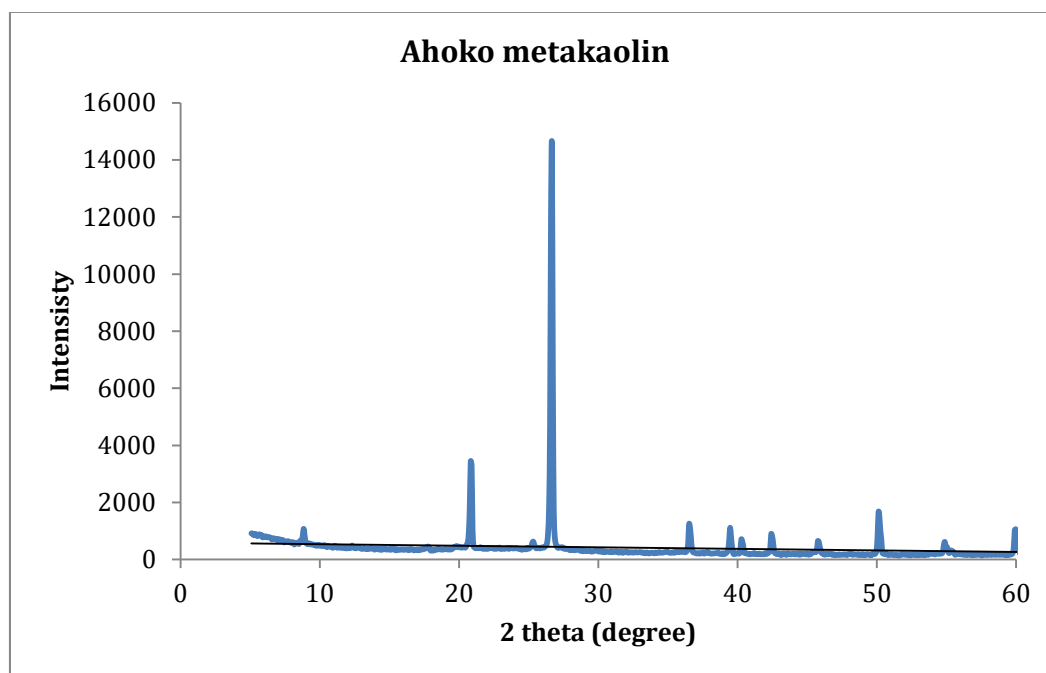


Figure 2. XRD pattern of Ahoko Metakaolin

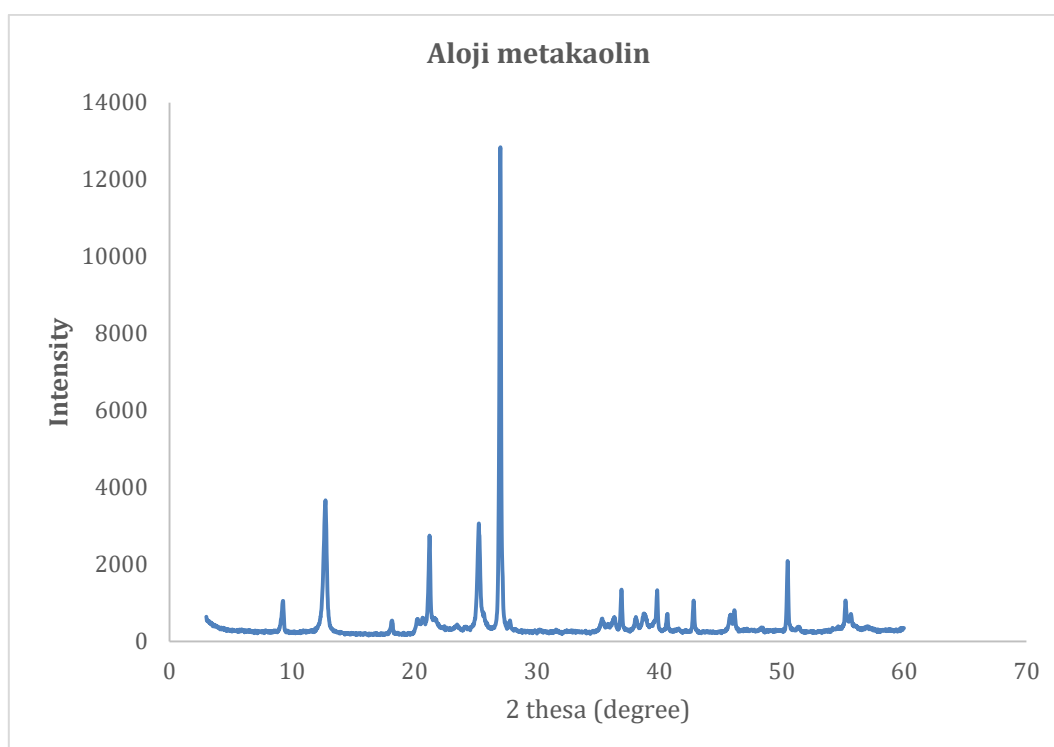


Figure 3. XRD pattern of Aloji metakaolin

Figures 2 and 3 show the XRD pattern of Ahoko and Aloji kaolin sample from room temperature to 550°C caused an increased intensity of the main reflections. According to (Franco *et al.* 2003), this is probably due to the loss of the hydration water which results in a better ordering in the structure of the kaolinite molecule. However, there was complete loss of the main reflection (12.3°) at 550°C leaving only a small halo which indicates the formation of a non-crystalline phase. Therefore, metakaolin based on the temperature programmed XRD result can be described as an amorphous material containing free silica, free alumina as reported by (Brindley *et al.* 1998).

### 3.4 Setting time and water demand of metakaolin blended mortar

Table 3: gives the water demand and the setting times of the cement mortar. The “water demand” is the quantity of water that is required in order to prepare a cement paste or mortar of standard consistency as specified in EN 196-3. The blended cements demand significantly more water than the relatively pure cement. With a metakaolin content of 5%, the water demand varies from 28.0% to 25% for Ahoko and 27% to 24% for Aloji, while the Portland cement has a water demand of 25%. With a metakaolin content of 5% to 10% for Ahoko sample, the water demand increases from 28% to 29%. MK1 and MK2 show the best behavior from 5% to 10%. The initial and final setting time of metakaolin cements is affected by the metakaolin content. Cements with 10% metakaolin, generally, exhibit similar initial setting times but different final setting time with delay. while for 12.5% metakaolin content there is not delay in the setting time.

**Table 3: Physical properties of metakaolin cements**

Sample	Metakaolin (% w/w)	Water demand (% w/w)	Initial setting time	Final setting time
PC	-	25	40	256
Mk1-5	5	28	112	200
Mk1-10	10	29	110	260
Mk1-12.5	12.5	27	50	180
Mk1-15	15	25	40	250
Mk2-5	5	27	80	243
Mk2-10	10	29	110	310
Mk2-15	15	23	131	263
Mk2-20	20	24	121	269

### 4.0 CONCLUSION

The following conclusions can be drawn from the present study:

- The produced metakaolins derived from indigenous kaolins, when combined with PC to produce blended cements, show a good pozzolanic behavior.
- The studied metakaolins have a very positive effect on the cement strength after 7 days and specifically at 28 and 56 days. The blended cements demand significantly more water than the relatively pure cement and the water demand increase is higher at 10% replacement.
- The pozzolanic reaction of metakaolins is accelerated between 7 and 28 days, accompanied by a steep decrease of Ca(OH)<sub>2</sub> content.
- Based on the mechanical and physical properties of metakaolin cements, a 10% metakaolin content seems to be more favorable than the other percentages replacement.

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## CRITICAL THINKING APPROACH TO CHEMICAL ENGINEERING EDUCATION IN NIGERIA

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### ABSTRACT

*All learning activities are geared towards not what the teacher is going to teach, but what the outcome of that teaching should be, what the learner is supposed to do, and at what level. Education plays a main role in shaping the learner to be a better, responsible citizen and an active member of the society. It provides the ladder for achieving success in life as well as constructive utilization of acquired skills and knowledge. Critical thinking should be taught in schools so that students may learn to think for themselves and make their own choices, as well as to prepare them for the difficulties of adult life and to help them live a full and happy life that includes critical thinking. The instructor is at the core of traditional education, which does not foster inventiveness or originality. Critical thinking approach will erase traditional teaching deficiencies and will breed professional competence, cognitive transformation, and self-confidence. Various strategies that can foster the development of critical thinking across disciplines in education have been explored by researchers recently, and despite the acknowledged importance, critical thinking skills are frequently not taught explicitly; teachers are not intentional in the teaching and learning process, the assumption being that students will learn from the implicit values buried deep within our teaching philosophies. It is essential that teachers understand critical thinking processes and employ instructional activities to build these processes to teach critical thinking abilities. Researching critical thinking is a continuous process since it is a contentious and extensive topic in terms of definition and progress.*

**Keywords:** Critical thinking skills; Teacher-centered; Strategies; Cognitive transformation.

### 1. INTRODUCTION

Education, possibly the most fundamental human need, is the process through which humans grow and evolve. It's only a matter of learning and understanding. The beginning of a child's education is in their mother's arms. Families and parents teach their children proper conduct and cultivate civic virtues in them. Meyer (1976) argues that the goal of education is to help the person grow and develop his or her inherent abilities to reach their fullest potential. In schools, this nurturing process is known as teaching. Home is called first school but formal education starts in school where teachers teach students and guide them through the process of learning. It has to be organized, systematic and interactive. Education is very important for every human being because it empowers the individual to understand what is happening around him or her in a logical and clear manner. It plays a main role in shaping an individual to be a better, responsible citizen and an

active member of the society. Education provides the ladder for achieving success in life as well as constructive utilization of acquired skills and knowledge.

Since ancient times, people have believed that education and thought are two intertwined, ever-evolving processes (Chaffee, 1994). This is backed up by (Bailin & Siegel, 2003), who proposed that critical thinking should be the primary goal of education. Many factors contribute to the importance of critical thinking in education, including the capacity to think for oneself and make judgments, preparation for maturity, and the ability to conduct a life that needs smart and analytical thinking (Siegel, 2010). Developing critical thinking skills in young people is a short-term aim, but the long-term goal is to develop high-functioning and creative individuals who can help a society flourish (Abrami, et al., 2008). For centuries, schools were seen

as places where children's minds were nurtured, not merely as an afterthought (Lipman, 2003).

Many educators and ideologies across the world have characterized critical thinking, along with evaluation standards, abilities, and proclivities (Siegel, 2010). Critical thinking, according to Abrami, et al. (2008), is the capacity of a person to participate in an intentional, self-regulatory thought process. Consideration of alternate viewpoints and the exploration of probable assumptions that may underlie a problem are all part of Halvorsen's definition of critical thinking in his 2005 study. If students want to be successful in a highly technological culture, they must be prepared with the lifelong learning and thinking abilities required to collect and analyse knowledge in an ever-changing environment," according to Cotton, (1991). Students need to know what and how to study in order to get a high-quality education. Students' critical thinking skills are on display as they reflect on what they've learned and how they've learnt it (Emir, 2009).

Science, mathematics, and technology all have a role in engineering. The word "techne" is the creative ability that differentiates an engineer from a scientist; to design, to make, to conceive, and to actually bring to fulfilment. It is important to recognize that engineering is more than to simply understand the rudiments of science, it is basically a vocational subject, which depends on the sound understanding of scientific principles as well as appropriate mathematical skills, the modelling language, and vital communication skills.

Students in Chemical Engineering face a wide range of difficulties that are frequently intertwined in nature. Undergraduate chemical engineering courses include topics that treat thermodynamics, fluid-mechanics, heat and mass transfer, chemical process engineering, chemical process control, plant design and analysis, and unit operations. Chemical Process Engineering's three pillars of design, control, and optimization are supported by each of these components (Cocke et al., 2003). The development of optimal solutions to Chemical Engineering challenges usually depends on organized and complicated mental processes that need assessment, interpretation and opinion. Developing good critical thinking skills is essential for dealing with these situations because of their complexity. Academic studies have proven the importance of teaching and developing critical thinking in the field of engineering. Most of them have theoretical and intellectual foundations. Less prevalent are practical and inventive techniques to teaching critical thinking in Chemical Engineering

curriculum. Goals, frameworks, and pedagogy are all important components of an effective curriculum (Cameron, et al. 2019).

## **2. NEXUS BETWEEN OUTCOME BASED LEARNING AND CRITICAL THINKING**

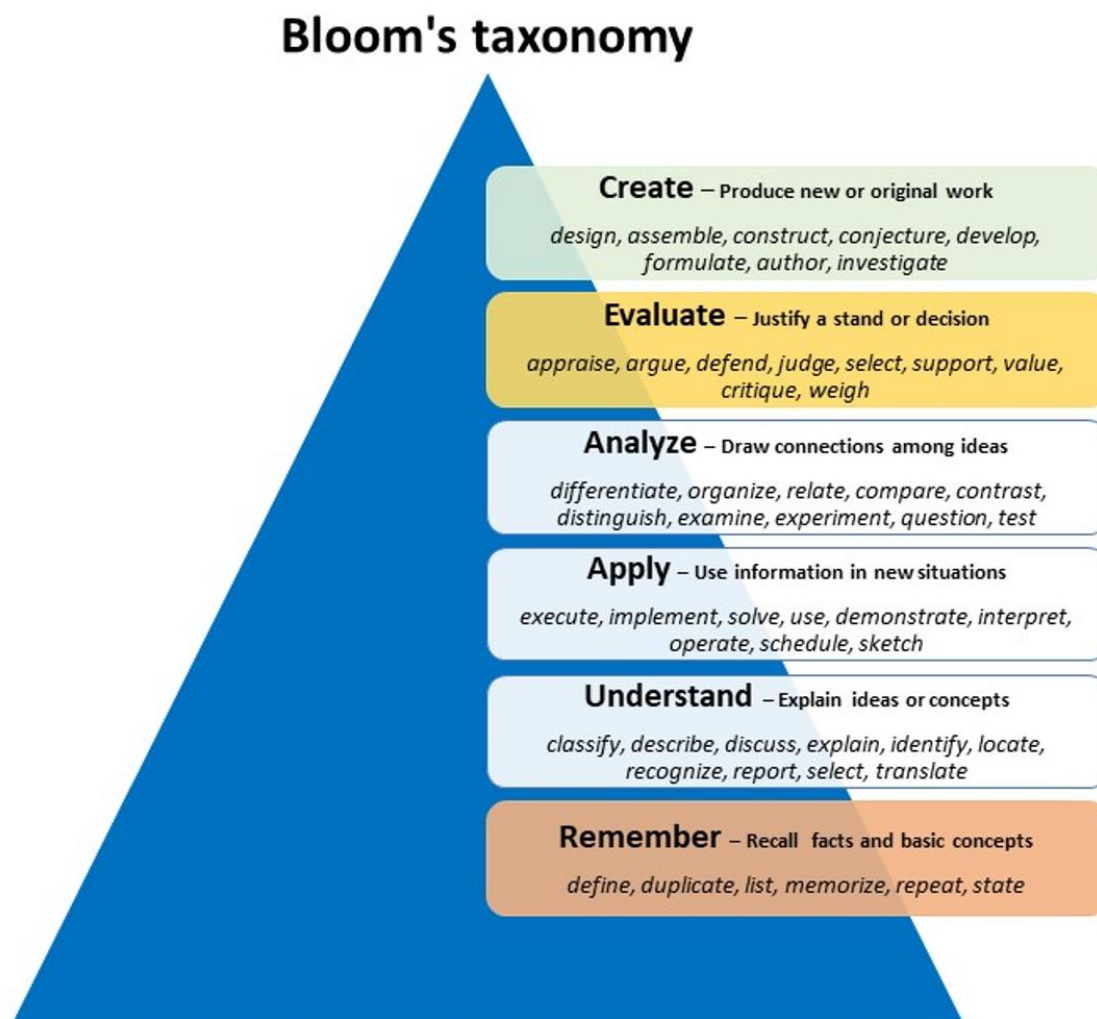
All learning activities are geared towards not what the teacher is going to teach, but what the outcome of that teaching should be, what the learner is supposed to do, and at what level. The Outcome-Based Education, a student-centered strategy aiming to enhance the quality of teaching and learning, was launched in 1997 as a major initiative among higher education institutions (Mouton, Louw, & Strydom, 2012). It is designed to assist in determining whether the methods and resources used are successful in helping students to meet their learning objectives (University Grants Committee of Hong Kong, 2008). Students must know what they are required to learn; secondly, students must show their progress; and thirdly, different teaching and assessment methodologies must be accessible to accommodate the specific requirements of each student. An Outcome-Based Learning (OBL) programme must have these three components: One of the most important aspects of OBL, according to Tucker (2004), is a shift away from the acquisition of course credits toward the attainment of higher-order learning (critical thinking) and mastery. OBL leads to educational change, which Tucker (2004) supports.

## **3. COMPONENTS AND DIMENSIONS OF CRITICAL THINKING**

### **3.1 Bloom's Taxonomy**

Bloom's taxonomy comprises three learning domains: the cognitive, the affective, and the psychomotor. There are two broad sorts of educational initiatives (education and training). Learning is divided into two parts: Cognitive and Psychomotor/Emotional/Affective (PME/A) domain. Critical thinking, problem solving, and the generation of new information are all part of the cognitive domain. It includes anything from basic memory to the creation of new knowledge based on previously acquired material. A linear progression from "remember" to "create" is anticipated of learners in this domain, starting with "remember" and ending with "create" (see Figure 1). First three are regarded to be lower levels of cognitive activity, while the other three are believed to be higher. They demand and cultivate the ability to think creatively, critically, and innovatively.





**Figure 1: Bloom's revised taxonomy (adapted from the Vanderbilt University Center for Teaching).**

Bloom sees learning as a pyramid, believes that it is only after a student completes one level of learning goals, through formative assessments, corrective activities, and other enrichment exercises, can they then move onto the next level, in other words, each level subsumes the levels that come before it. In essence, Bloom's taxonomy reveals:

- (a) Instructors were not fulfilling the needs of each student and instead depended on a single uniform curriculum. It was hypothesized that providing students with personalized educational programmes will help them learn more effectively and achieve their objectives;
- (b) Can be used as a potential model for framing educational objectives within a course and as a guide to structure activities and assessment based on learning goals. We may use Bloom's taxonomy not just to construct curriculum and instruction, but also to build

relevant test questions for students at different stages of cognitive development. The focus of the cognitive domain is on the task at hand. By emphasizing its significance to pupils and explicitly outlining academic standards, it shows the teacher's personal devotion and emphasis on the subject matter Requires dedication to achieve mastery in every area of expertise if duly followed.

### 3.2 Instructional approaches to Critical Thinking

The learning process consists of the way in which learning takes place and involves primarily the capacities of the activities by means of which knowledge is gained, skill is produced, and habits, attitudes, and ideas are acquired. Knowing the learning approach of the Chemical Engineering student will help

the students and lecturers identify areas or strategies that are facilitative to learning and enable the students to adopt a more meaningful learning approach. Strategies to learning are considered important elements.

Critical thinking and creative skill are important, but they are not enough; competent thinkers must also have a set of motives, values, and habits in addition to their talents. (Tishman & Andreade, 1995). In Al-Ghadouni's (2021) critical thinking methodology, there are six methods:

- a. Disposition approach: It's important to develop the disposition approach, which involves cultivating a habit of critical thinking as part of one's everyday life (Profetto-McGrath, 2003). Someone with a critical thinking disposition is more likely to use their critical thinking skills, which include curiosity, enthusiasm, dedication to reason, and a thirst for reliable information than those without it (Zhang, 2008). Dispositions are more important than skills when it comes to cultivating pupils' critical thinking abilities, which may be taught.
- b. General approach: Lessons are established in advance and taught in conjunction with the current curriculum to improve general thinking abilities. For teaching critical thinking, this is one of the most well-known and well-tested methods (Sedaghat & Rahmani, 2011). Apart from the particular content of a topic, the general approach emphasizes the teaching and formation of critical thinking.
- c. Infusion approach: An infusion strategy involves teaching students to think critically about their subject matter in a deep, deliberate, and well-understood way. There is also a focus on the broad concepts of critical thinking abilities and attitudes (Abrami, et al., 2014). There must be an emphasis on critical thinking in each subject area where the broad principles of critical thinking are properly defined.
- d. Immersion approach: Here the tenets are not explicitly communicated to the students, as opposed to the infusion approach (Abrami, et al., 2014). Students are engaged in the subject content without being exposed to it in any way, shape, or form directly. It's not always clear to students that they're being taught to think critically when they're taught this manner.
- e. Mixed approach: It combines the general strategy with either an infusion or an immersion approach. There is a distinct section for teaching fundamental concepts of critical thinking as well as topic-specific critical thinking education under it (Abrami, et al., 2014); critical thinking is taught as a different track inside a particular subject matter.
- f. Holistic approach: When it comes to developing critical thinking abilities, a holistic approach is the best way to

do so. The effectiveness of an educational programme in fostering critical thinking is evaluated by administering a pre-test and a post-test to students. A "holistic approach" was defined by Niu, Behar-Horenstein, & Garvan (2013) as the whole academic degree programme. It usually lasts for a year or two, but it might be as long as three or four. There is a growing consensus among researchers that the duration of the programming method compromises its validity, and that it also acts as an interfering element and might have unintended consequences for its outcomes. Few have attempted to deal with these dangers by programmatic intervention, according to Behar-Horenstein, L.S. & Niu, L. (2011). Programmatic intervention may have a good influence on critical thinking growth notwithstanding the intrinsic effects of an educational programme, according to the holistic approach.

Research on ways to increase college students' critical thinking abilities using instructional-interventions was evaluated by Behar-Horenstein, L.S., & Niu, L. (2011), who looked at 61 empirical research published between 1994 and 2009. Among the research analysed, immersion was determined to be the most commonly employed strategy (52 percent), followed by a comprehensive approach (19 percent), and the other three approaches (general, infusion, mixed; each with 9.5 percent). Immersion is least beneficial from the students' critical thinking point of view (Wang, 2017; Al-Ghadouni, 2021), and the combined method is most effective. As stated by Oermann (1997) research, critical thinking is not formed in a single lecture. Rather, it is built via a variety of experiences, and so, students must actively engage in their own learning to enhance their abilities.

#### **4. CRITICAL THINKING AND CURRICULUM EVALUATION IN CHEMICAL ENGINEERING**

The old-style (traditional) undergraduate chemical engineering curriculum allows students to cover core topics through a series of courses. Students start with basic concepts such as material and energy flows in chemical processes, followed by thermodynamics and transport of mass, momentum and heat. Courses in chemical separations, reaction-engineering, process control, and a capstone design course are required in the last two years of a degree programme. These core courses are supplemented with various technical courses and some electives that depend on the professional track the student will follow. Typically, little attention is

given to how the knowledge and skills in one course will be used in subsequent courses or its contribution to the whole curriculum. Implicitly, there is no systematic emphasis on the linkages unless an individual instructor focuses on it at his or her own discretion.

#### **4.1 Review of Current Chemical Engineering Curriculum with Critical Thinking and Outcome-Based Learning Embedded**

Chemical Engineering's curriculum is being overhauled to better serve the educational needs of its students. Multidimensional spiral Chemical Engineering curriculum with critical thinking and outcome-based learning like the one suggested by Bloom's taxonomy is better from a student's perspective. This arrangement splits the core curriculum into smaller units, exposes students to each unit and level, gives a better understanding of each stage or level gradually, and requires mastery of each level before moving to the next. This is a superior learning approach as it ensures that students have mastered each increment of a subject in hierarchical sequence before going on to the next. There is a progression from basic steady state systems and equilibrium units, which are theoretically easy, to the more difficult challenge of large-scale systems integration in this curriculum; while in the second direction the student will learn the technical concepts for analysis and design that are essential for a professional Chemical Engineer. In this spiral approach, links between different traverses of the spirals are emphasized which allows students to reinforce the prior concepts and begin grasping the application to new knowledge. A steady and continuous deepening of knowledge is incorporated as students move from simple to complex concepts. Students are encouraged to work together as a team in order to accomplish their goals. When students analyze, evaluate, interpret, or synthesize information and use creative thinking to build an argument, solve a problem, or arrive at a conclusion, they are promoting critical thinking.

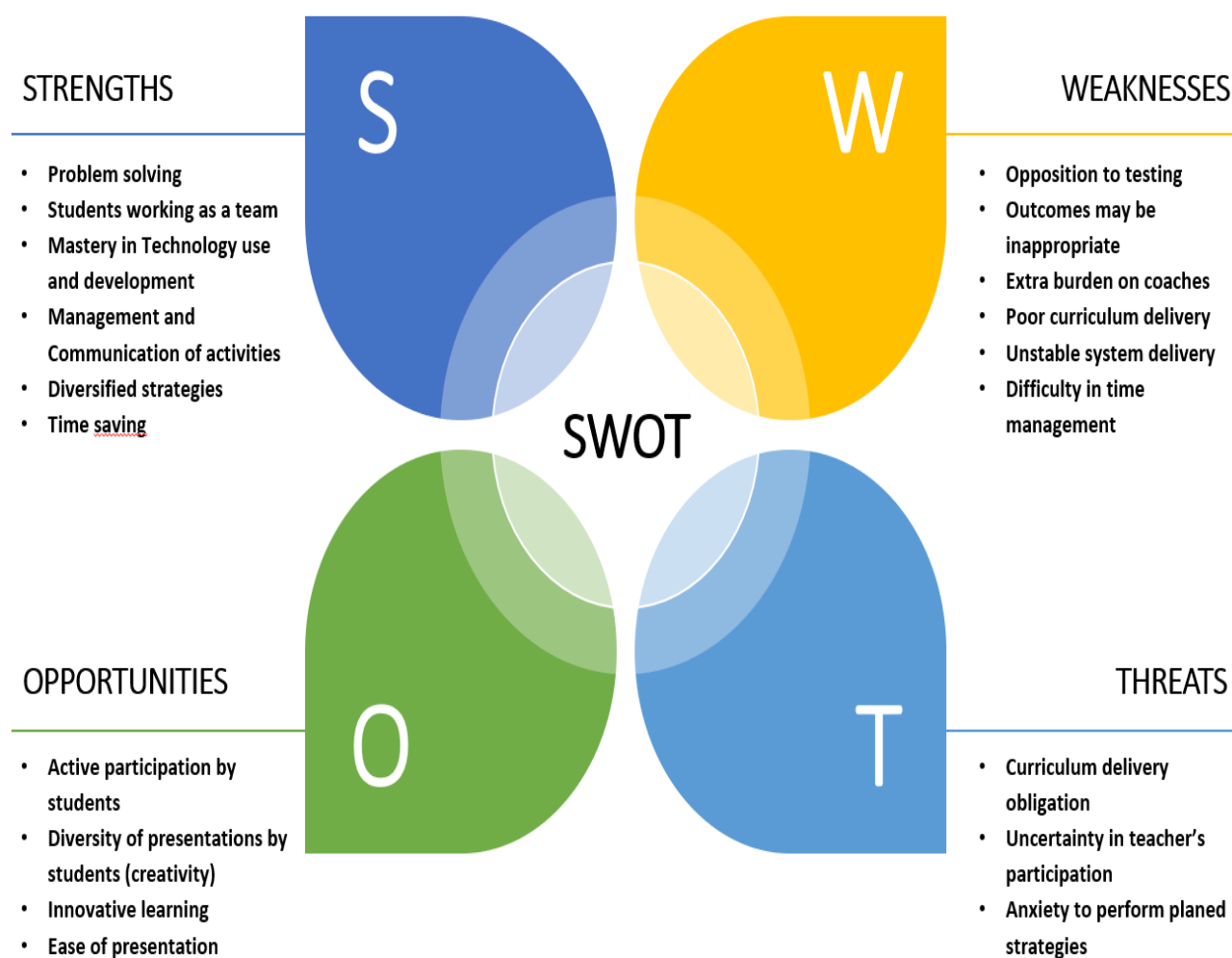
#### **4.2 Student Assessment: Traditional and Modification**

The problem-based learning (PBL) technique may be used to teach critical thinking in practical fields like Chemical Engineering by allowing students to undertake research, integrate theory and practice, and use their newly acquired knowledge and abilities to solve a specific issue (Savery, 2015). Using PBL, students are given a problem to solve, they work together in groups, and the challenge drives their learning attitude and motivates them to use their cognitive processes to solve

the issue. Setting objectives for students is a key component of outcome-based learning (OBL). Student learning is structured in such a manner that it provides a ladder for achieving defined objectives; it guides students on the skills and information they need in order to prepare them for life after graduation. Every learning experience must have a result or outcome. In both circumstances, the student is at the centre of the learning and must be in command, the student must work as part of a team, and the student must be self-motivated.

Students in this course will study about David Kolb's experiential learning theory, which claims that "learning is best understood in terms of processes, not in terms of results" (Kolb & Kolb, 2005). This concept of learning defines the evaluation of students, which is dispersed across the whole learning process rather than merely reviewing the final report, notably during exams, alone. When students are allocated to a project, they are given information about the system they will be working with, and they begin thinking about what they want to accomplish and how to deal with any potential technical difficulties. To put it another way, students get first-hand knowledge and experience through participating in class activities. An academic supervisor or lecturer conducts a fair and transparent formative evaluation while doing the summative assessment (literature search, work plan, report, and brief interview). The latter is used to gauge students' degree of topic knowledge at various phases of the project. They are provided with assessment and criterion techniques right from the start of the assignment. Performance indicators must be used to measure learning outcomes since every learning result is deliberate. Students' Knowledge, Skills, and Attitude will be addressed as part of the program's objectives.

### 4.3 SWOT Analysis of Outcome-Based Education and Critical Thinking in Chemical Engineering



**Figure 2: SWOT Analysis of outcome-based education and critical thinking framework.**

Higher education's current focus is on implementing a learning outcomes-based approach. It is a method of teaching in which curricular choices are based on what students should be able to demonstrate at the conclusion of the course. Outcome based education helps chemical engineering students to initiate and accept changes, it exposes them to team work which is needed in the industry, it saves time. Students are also exposed to innovative learning as an opportunity (See figure 2). The chemical engineering student requires ability to think independently to be able to identify and solve plant problems. Alternatively, the content of the curriculum cannot be delivered under Outcome-based education, the Chemical Engineering lecturer concentrates on what the student can gain and not what the curriculum dictates.

## DIFFERENCES BETWEEN TRADITIONAL AND OUTCOME BASED LEARNING

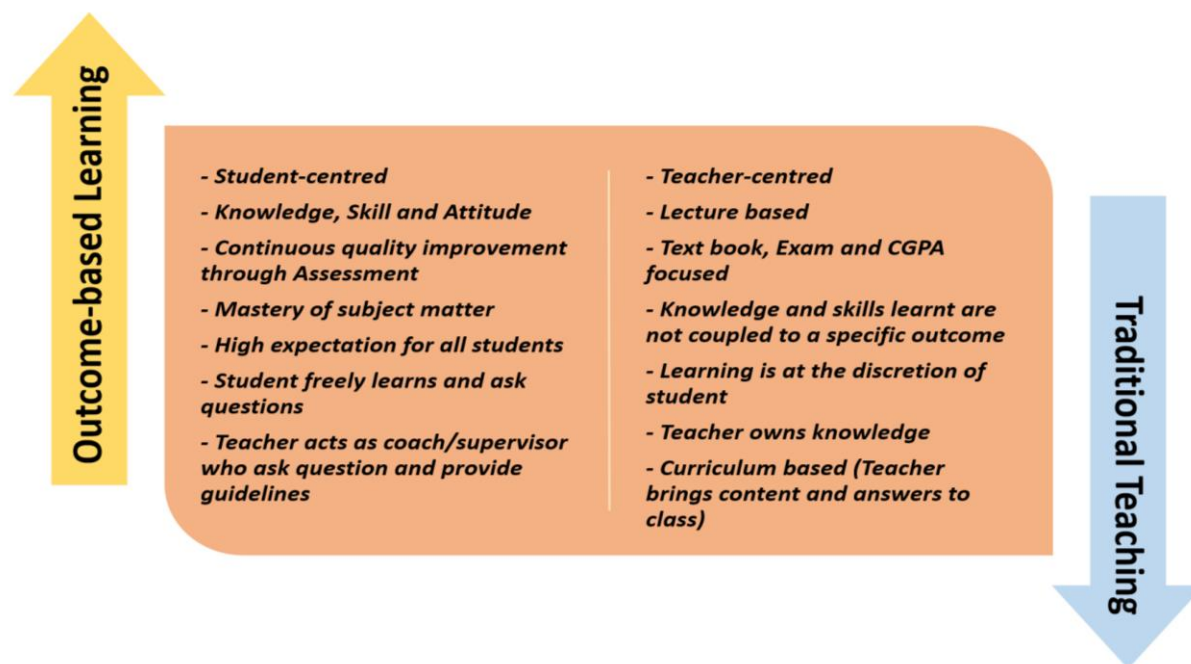


Figure 3: Differences between the traditional teaching methods and the outcome-based learning.

The old method of teaching also known as traditional teaching is teacher centred and doesn't encourage students' creativity and innovation (Figure 3). Critical thinking will overwrite all the deficiencies associated with the old fashioned teaching method, and will rather breed professional competence, self-confidence and logical transformations if duly implemented.

## 5. CONCLUSION

It is generally agreed that critical thinking requires the ability to think independently, clearly, and rationally. It entails the ability to think about a problem or a concept, use reasoning, and connect ideas logically. The critical thinking skill is an important part of student development and it is intrinsically linked to both problem-solving and decision-making skills. To ensure that Nigerian Chemical Engineers are equipped with critical thinking abilities, a paradigm shift in the way they are trained and educated is necessary. Educators throughout the world must respond to the need for a paradigm change in higher education from conventional teaching to learning. Students and graduates in Chemical Engineering will not only be able to solve issues, but they will also be able to come up with fresh and innovative solutions. The analysis is based on the components of reasoning. Employability of Chemical

Engineering graduates will be guaranteed because of exposure to innovative skills, which will also enhance the visibility of our institutions as a result of proficiency in skills and professional knowledge. Government and academic leaders in universities (especially Chemical Engineering Departments) need to show concerns for and enforce the quality of teaching/learning. Chemical Engineers with innovative culture will play a major role in providing sustainable solutions to national issues as it relates to other sectors of the economy.

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