Volume 39
No. 1

JNSChE
March/April 2024

ISSN
0794-6759

DOI: 10.51975/EFSD2591

JOURNAL OF THE NIGERIAN SOCIETY OF CHEMICAL ENGINEERS

Published by,
THE NIGERIAN SOCIETY OF CHEMICAL ENGINEERS
National Secretariat: Infinite Grace Hocuse, Plot 4, Oyetubo Street,
Off Obafemi Awolowo Way, Ikeja, Lagos State, Nigeria.
E-mail: nationalhqtrs@nsche.org, nsche_headquarters@yahoo.com
Website: https://www.nsche.org.ng; Journal URL: https://journal.nsche.org.ng
Submission of Manuscripts: nschejournal@yahoo.com and copy: stevmomoh@yahoo.com
The journal is indexed in International Scientific Indexing (ISI). The URL for journal in the ISI server is https://isindexing.com/isi/journaldetails.php?id=16333

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10.51975/24380101.som

Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

ASSESSMENT OF NIGERIA'S CASSAVA PRODUCTION POTENTIAL AND EVALUATING THE TECHNO-QUALITY ANALYSIS OF LOCALLY PRODUCED GARRI THROUGH EXPERIMENTAL STUDIES

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ABSTRACT

The global energy narrative is pushing for the deep decarbonisation of energy-consuming systems. This paper seeks to assess cassava (a staple food in West Africa) production potentials in Nigeria and to technically evaluate the garri production process as well as to study the effect of storage duration on the quality of the garri. Available data indicate that there were wide variation in cassava production potential in Nigeria between 1961 to 2020 with appreciable increase of cassava production occurring within the period 1981 to 2000. Identified factors such government policies and interventions; cassava virus and bacteria are responsible for this variation. Garri has several value chains with roasting playing a dominant role in the energy consumption and quality. Garri was produced using three fermentation pathways with each product stored in three different types of storage containers (plastic container with cover, airtight polythene bag and ordinary polythene bag) for 15 weeks and monitored regularly for quality purposes. Results obtained indicated that fermentation duration, with the exception of acid content, does not significantly affect the moisture content, swelling index and bulk density of garri within the period under investigation. These results will be useful for designing an improved environmental friendlier and climate responsive garri roasting process unit powered by solar energy in the context of energy efficiency and quality control.

KEYWORDS: *Cassava, Garri, Fermentation, Physiochemical properties, Roasting, Shelf life*

1. INTRODUCTION

Cassava (*Manihot esculenta Crantz*) is a drought-tolerant crop that can be grown in areas with uncertain rainfall patterns which usually results in unsuccessful cultivation of many other crops (Otekunrin and Sawicka, 2019). It is the most important tropical root crop, which is primarily grown for its starchy tuberous roots that are rich in dietary energy (FAO, 1995). Cassava is a major source of staple food in Africa, Asia, and Latin America with Africa producing about half of the world's cassava output. It provides around 20% of the total energy intake for many people in developing countries (Hillocks *et al.* 2002). The plant consists of 6% leaves, 44% stem, and 50% roots with the roots containing 60–66% moisture and 32–35% carbon hydrates, 0.4–0.6% protein and 0.1–0.3% fat (Felber *et al.*, 2017). Cassava tubers are very rich in starch and contain significant amounts of calcium (50mg/100g), phosphate (40mg/100g) and vitamin C (25mg/100g) (Adejuyitan *et al.*, 2018). Some of the key factors driving the cassava transformation in Nigeria are the high consumer demand for cassava by rural and urban households and, to an extent, the government's policy on cassava utilization.

Due to its high moisture content, cassava is normally processed immediately after harvest into different storable products; these include garri (cassava flakes), lafun (fermented cassava flour), fufu (steeped cassava starch dough), pupuru (fermented dried cassava) and cassava flour (Kuye *et al.*, 2015; Kuye *et al.*, 2017; Bechoff *et al.*, 2018; Oyeyinka *et al.*, 2019; Abiodun *et al.*, 2020). Cassava is also a source of bio-fuel as well as animal feed and raw material for industries (Ani *et al.*, 2018).

Over 70% of cassava production in Nigeria is processed into garri (Sanni and Olubamiwa, 2004). Garri is variable granule flour; it is a cream coloured, white or yellow partially gelatinized starchy food from cassava tubers and it is an easy to store product (Sanni *et al.* 2008; Samuel and Ugwuanyi, 2014; Oluwafemi and Udeh, 2016; Ezeocha *et al.*, 2019). Garri consumption cuts across all socio-economic classes (Edem *et al.*, 2001; Kostinek *et al.*, 2005; Jekayinfa and Olajide, 2007; Samuel and Ugwuanyi, 2014; Ogbonna *et al.*, 2017). Garri can be consumed without further cooking by soaking in water with sugar or common salt and consumed with either or a combination of smoked fish, roasted groundnuts, cooked beans porridge, coconut, palm kernel and groundnut cake (*kwuli-kwuli*). The garri granules can also be soaked and stirred in boiled water to produce a stiff paste eaten with soup or stew (*eba*) (Asegbeloyin and Onyimonyi, 2007; Adinsi *et al.*, 2019).

The main unit operations required for the conversion of cassava tubers into garri are peeling, washing and grating of the tubers and fermentation. Sometimes, red palm oil is added to improve the colour and nutrients (Ogbonna *et al*., 2017). This is followed by pressing, fragmentation, granulation, drying, sieving and suitable heat treatment (roasting) (CODEX STAN 151 - 1989). The roasting step is energy intensive (Felber *et al*., 2016), which is normally driven by the burning of firewood derived from the felling of forest trees. Garri production in Nigeria is still largely carried out traditionally mostly by village women with some efforts to mechanize some of the Adonis processes, e.g., the grating and pressing process units. However, the mechanization of the roasting unit operation, which is highly energy intensive, is still at its nascent stage. Several studies have evaluated the effect of unit operations and cassava genotypes on the quality of garri (Agbor-Egbe and Lape Mbome, 2006; Nwancho *et al*., 2014; Olaoye *et al*., 2015), in particular fermentation (Moorthy and Mathew, 1998; Iwuoha and Eke, 1996; Sokari and Karibo, 1996; Kostinek *et al*., 2005; Montagnac *et al*., 2009) and roasting/frying (Chuzel *et al*., 1995a; Igbeka *et al*., 1992; Sobowale *et al*., 2017; Ezeocha *et al*., 2019; Samuel *et al*., 2010). The combined effect of the successive unit operations on the final quality of garri under laboratory conditions was investigated by Escobar *et al*. (2018) while Escobar *et al*. (2021) characterized garri processing at two scales; conventional process by small scale processors at village level versus a smaller-scale process using laboratory equipment.

Garri quality is judged by its swelling capacity, colour, particle size, moisture content, and taste (Irtwange and Achima, 2009; Oduro *et al*., 2000; Agbetoye and Oyedele, 2013). The foregoing indicates that a lot of researches had been conducted on the effect of garri processing variables on the quality of garri, however, there is little information on pertinent process design variables for its production. The work, therefore, provides cassava production potentials and a technical evaluation of the garri production process as well as to study the effect of storage duration on the quality of the locally produced garri to determine the quality control measure to support the improvement of garri roasting/frying process unit driven by possibly environmentally friendly process.

2. MATERIALS AND METHODS

2.1 Cassava production in Nigeria

The data used for this work was obtained from Food and Agriculture Organization Statistics (FAOSTAT) for Nigeria during the period 1961–2020. During this period, various economic reform programs were implemented in Nigeria to boost the overall economic growth and transform the agriculture of the country. To assess the effect of these reform programs on the growth and instability of cassava production, the data for each studied variable (i.e., harvested area, production, and yield) in the entire study were divided into four periods: Period I (1961–1980); Period II (1981–2000), Period III (2001-2020) and Pool (1961–2020). The statistical tools used were descriptive statistics and annual compound growth rate (CGR) to estimate the trend for production, area harvested and yield.

The descriptive statistics computed were mean, median, maximum, minimum, standard deviation, coefficient of variation, skewness, kurtosis, and Jarque-Bera (JB) test and p-value for Jarque-Bera test. JB is a goodness-of-fit test that determines whether or not sample data have skewness and kurtosis that matches a normal distribution. This test statistic is defined by Jarque and Bera (1987) and Thadewald and Büning (2007)):

$$\mathrm{JB} = \frac{n}{6}\left(S^2 + \frac{C^2}{4}\right) \tag{1}$$

where n is the number of observations in the sample; S is the sample skewness = $\mu_3/\mu_2^{3/2}$; C is the sample kurtosis = $\mu_4/\mu_2^2 - 3$ with μ_j calculated using:

$$\mu_j = \frac{1}{n}\sum_{i=1}^{n}(x_i - \overline{x})^j;\ j = 2,3,4 \tag{2}$$

Under the null hypothesis of normality, JB is asymptotically chi-square distributed with two degrees of freedom.

To calculate CGR, the trend is assumed to be represented by the equation:

$$Y = ab^t \tag{3}$$

where Y is the cassava production (tonne), area harvested (ha) or yield (t/ha); t is the year; a is the constant; b = (1 + r) is the slope coefficient that measures the instantaneous relative change in Y for a given absolute change in the value of an explanatory variable; r is CGR (Ayele *et al*., 2021). Linearizing Equation (3) gives

$$\log Y = \log a + t \log b \tag{4}$$

Thus, b is obtained from Equation (4) using linear regression and the percentage CGR is then calculated as 100r.

In addition, the arable land area available in Nigeria was obtained from FAO (2022) and the % of arable land area used for harvesting cassava (HC) was calculated using

$$\mathrm{HC} = 100\,\frac{\text{Cassava harvested area}}{\text{Arable land area}} \tag{5}$$

2.2 Cassava Processing

To technically monitor and analyse the small scale garri production process effectively, local garri production site managed by a dweller in Oduoha community was visited and the whole processes involved were accounted. The cassava variety used for this work is locally called *Welewo* which was obtained from a farm located in Rumuodogo (4.918ºN, 6.790ºE), Rivers State, Nigeria. The flow diagram for cassava roots processing is shown in Figure 1. The harvested cassava tubers were divided into three parts and were labelled as Samples A, B and C. Each sample was weighed, peeled and washed. Samples A, B and C were allowed to ferment for 18, 38 and 66 hours, respectively on a screw press. After fermentation, each sample was sieved using a local wooden sieve and then roasted in batches (1.7 kg of cassava mash/pulp per batch) over an aluminium roasting pan with temperature of the fire ranging between 200°C to 700°C, which was controlled by adding and removing some of the burning firewood; the frying pan temperature was maintained between 70°C to 90°C. The roasted garri were spread on a platform and allowed to cool down to ambient temperature (25ºC at the studied area). Thereafter, the samples were packaged in three different types of containers: Plastic Container with Cover (PCC), Airtight Polythene Bag (APB) and Ordinary Polythene Bag (OPB) and stored for analysis in the laboratory.

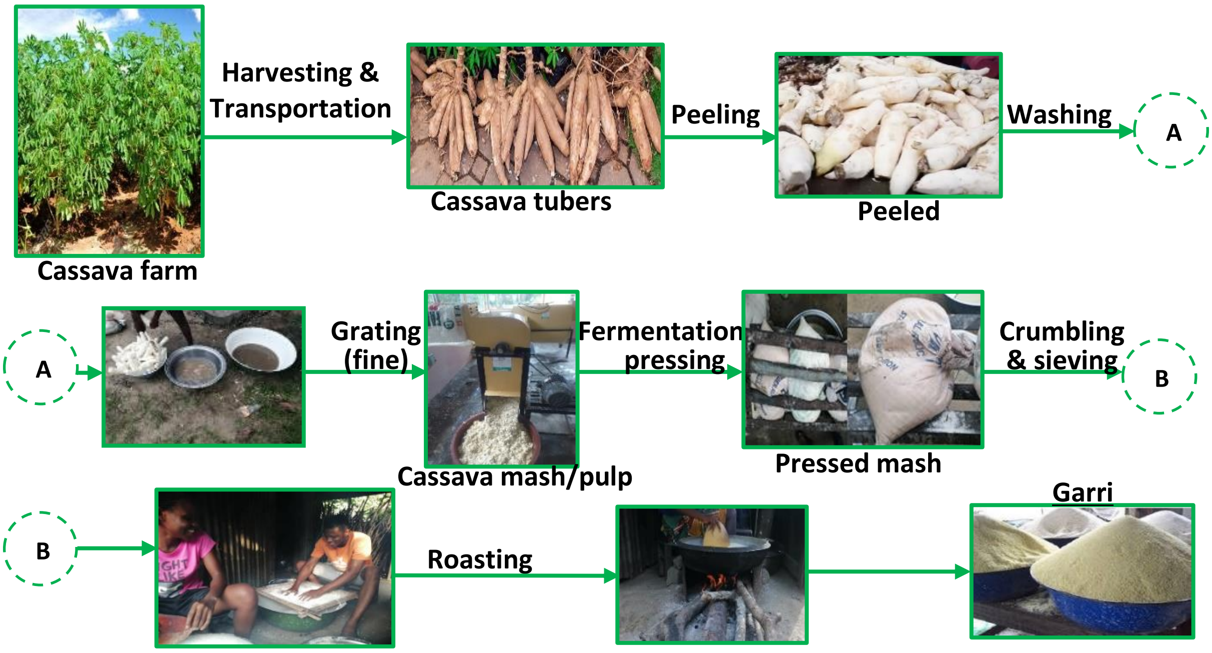

Figure 1: Process flow diagram for garri production from cassava roots

2.3 Analysis of Garri

The Samples A, B and C were sent to a laboratory for further analysis in the different containers on week 1 and were repeated for week 4, 7, 11 and 15. The analyses that were carried out in the laboratory included moisture content, acid content, swelling index, bulk density, and grain size. This was done to monitor the quality of the garri, that is its storability, in different storage bags over some time.

(a) **Moisture Content Determination:** The moisture content of the grated cassava samples was determined using handheld moisture meter (TK100, Model: VER170D08, Accuracy: ±0.5, Sensitivity: 0-84%) at different points within the sample.

(b) **Acid Content:** The acid content of the garri was measured by titration. 2.5 g of the ground sample was dissolved in 50ml of distilled water, agitated for 1 hour and allowed to rest for 24 hours. The solution was filtered and a volume of 20 ml of the filtrate was titrated with 0.05 M sodium hydroxide and using 2-3 drops of phenolphthalein as indicator. The titration is complete when the colour of the solution turns a pale rose. The acid content, A_c, is calculated using (Pearson 1973; Lees 1975 and AOAC 1990):

$$A_c = \frac{0.45V_B}{(1-h)} \tag{6}$$

where V_B is volume of Sodium Hydroxide used and h is moisture content of the sample.

(c) **Swelling Index:** 5.0 g of garri sample was measured into a measuring cylinder and the volume occupied was recorded as the initial volume. 40 ml of distilled water was added to it and the swollen volume was recorded at regular time intervals until the volume became constant (Figure 2). The Swelling index is the ratio of the swollen volume to the initial volume (Owuamanam *et al*., 2011).

Figure 2: Volume of garri sample in a measuring cylinder before and after swelling.

The rate of swelling, r, was modelled by assuming that the swelling rate is inversely proportional to time, that is,

$$r = \frac{dV}{dt} \propto \frac{1}{t} \qquad (7)$$

(d) Bulk Density: The bulk density was determined using a technique similar to the one used by Akpapunam and Markakis (1981). 5.0g of each sample was placed in a measuring cylinder and gently tapped for 5 minutes to determine its volume. The bulk density was calculated using:

$$\text{Bulk density} = \frac{mass}{volume} \qquad (8)$$

(e) Grain Size Distribution: The grain size distribution of each sample was determined using the screening method by Sahin and Sumnu (2006). Electromagnetic Sieve Shaker (Model: BA200N). The shaker comprises a set of stacked standard screens (1 mm, 710 µm, 500 µm, 425 µm and 300 µm). 50g of each sample was measured and put in the 1mm screen. After 15 minutes of agitation, the mass of each grain size retained in each screen was measured and recorded. The Fineness Modulus (FM) and the average particle size were then calculated (Sahay and Singh, 2001).

2.4 Material Balance of Garri Production

The corresponding block diagram for the garri production process shown in Figure 1 is presented in Figure 3. Thus, the input streams are Stream 1 and 4 while the output streams are 2, 5, 7, 9, 11, 13 and 14 as described in Table 1. At steady state it is expected that:

$$\text{Input} = \text{Output} \qquad (9)$$

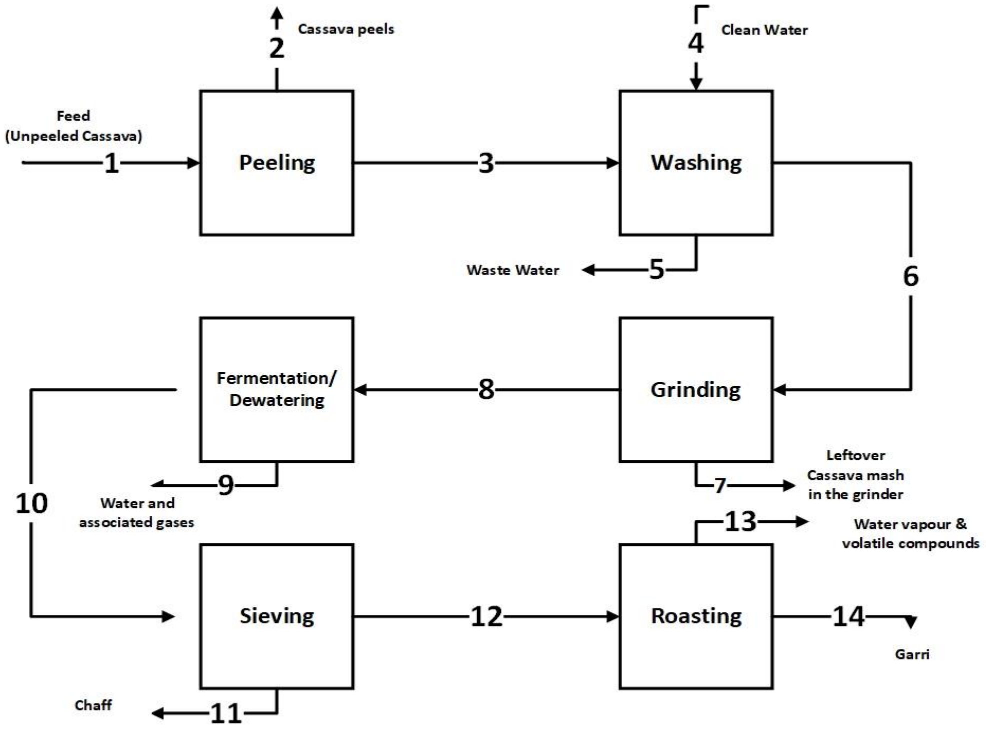

Figure 3: Block diagram for garri production

Table 1: Process streams and their descriptions

Stream	Description
1	Feed (unpeeled cassava)
2	Cassava peels
3	Peeled cassava
4	Clean water
5	Waste water
6	Washed cassava
7	Leftover cassava mash in the grinder
8	Ground cassava mash
9	Drained water and gaseous products of fermentation
10	Fermented cassava mash
11	Chaff and fell-off of the sieve tray
12	Sieved cassava mass
13	Water vapour, volatile compounds and fell-off from the frying pan
14	Garri

3. RESULTS AND DISCUSSION

3.1 Cassava production in Nigeria

The cassava data for Nigeria from 1961 to 2020 is shown in Figures 4 to 6. Figure 4 indicates that cassava production varied between 7.4 million tonnes in 1961 and 60 million tonnes in 2020. The increase in cassava production was about 4 million tonnes between 1961 and 1980, 21 million tonnes between 1981 and 2000, and 28 million tonnes between 2001 and 2020. The years between 1981 and 2000 featured the highest production increase. The cassava harvested area (Figure 5) appears to follow a trend similar to that for production. There is a big leap between 1990 and 1991 as well as between 2011 and 2012. The seemingly non-uniform increments in the cassava production and area harvested may be due to the various interventions and policy strategies put in place by the Nigerian Government and the negative impact of cassava virus, bacteria and other infections (Ikuemonisan *et al*., 2020).

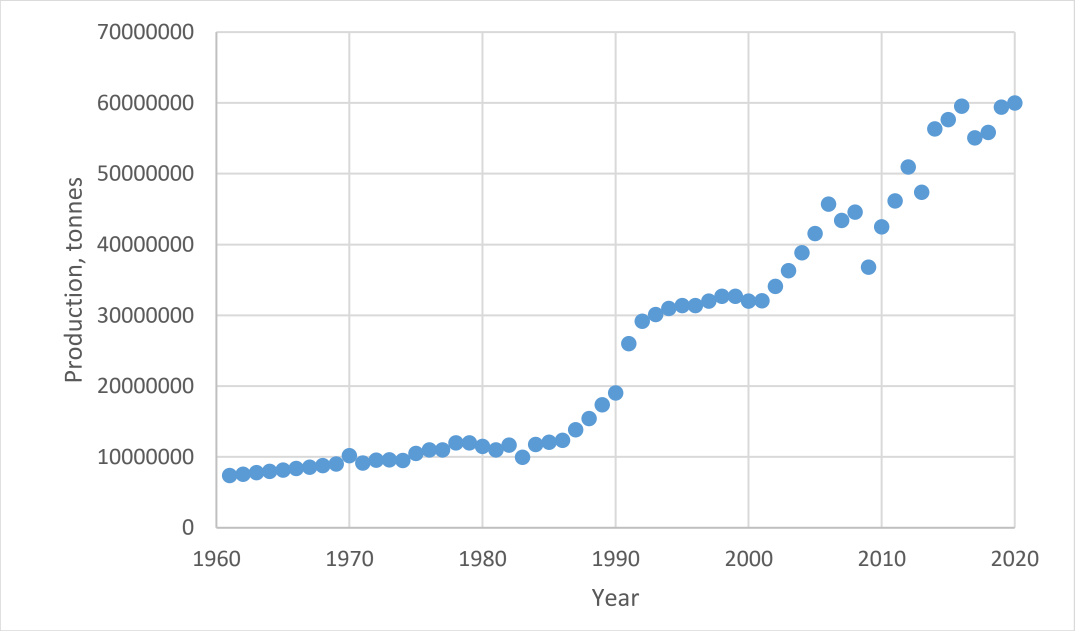

Figure 4: Nigeria cassava production from 1961 - 2020

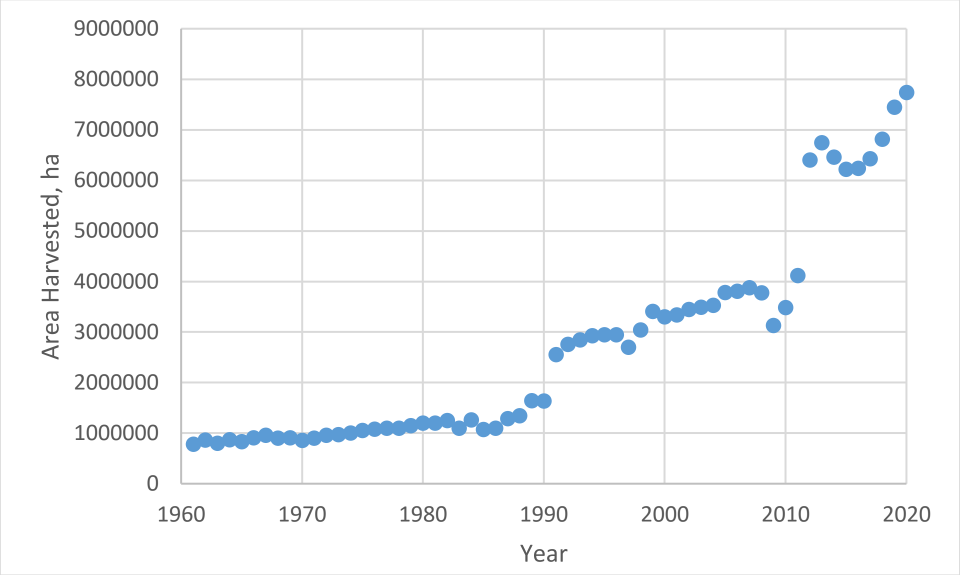

Figure 5: Nigeria cassava's area harvested from 1961 - 2020

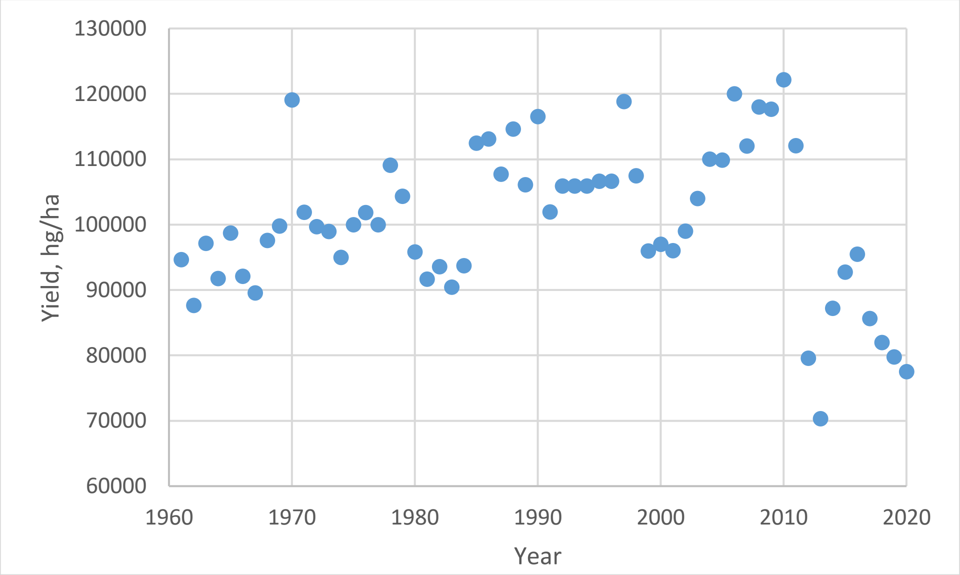

Figure 6: Cassava yield in Nigeria from 1961 - 2022

Between 1961 and 2020, the arable land area in Nigeria varied from 17.432 million hectares to 37 million hectares (FAO 2022). Figure 7 shows the percentage of this arable land that was used for harvesting cassava from 1961 to 2020. The values ranged from 3 to 7% for the period 1961 to 1982; 3.3 to 11.8 for the period 1983 to 2011; and 18.3 to 22.8% for the period 2012 to 2020. The lower yield observed during the period 2012 to 2020 (see Figure 5) may be due to more arable land being used that may not be very good for cassava cultivation.

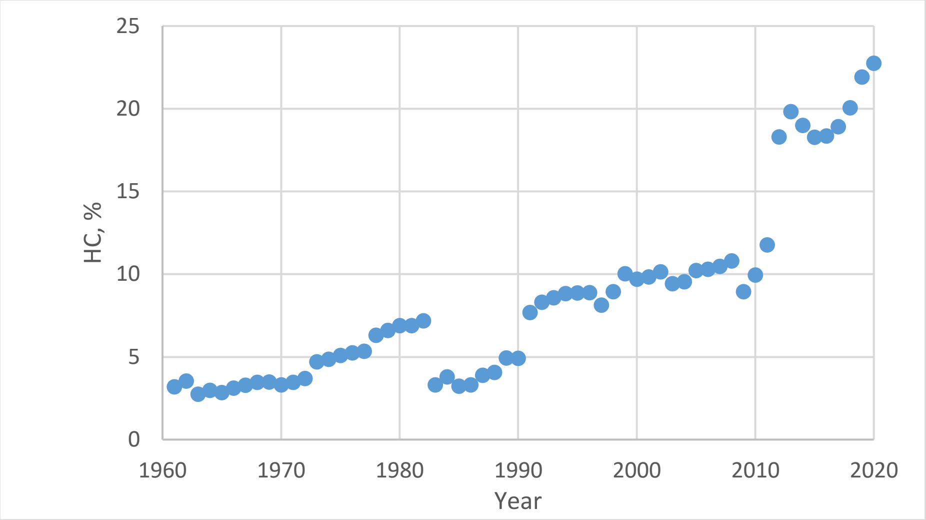

Figure 7: Percentage of arable land used for harvesting cassava from 1961 to 2020

The CGR is obtained from Equation (4) using linear regression for cassava production and harvested area with Table 2 showing the regression coefficients in parentheses for each of the four periods. The CGR for cassava within the period of 1961 to 2020 is 4.11% with the period 1981 to 2000 having the highest value of 7.67%, which also apply to the harvested area.

Table 2: Annual Compound Growth Rates for different periods

	1961-2020	1961-1980	1981-2000	2001-2020
Production	4.11 (0.9516)	2.54 (0.9452)	7.67 (0.9037)	3.17 (0.8667)
Harvested Area	4.18 (0.9388)	1.97 (0.8798)	7.23 (0.8678)	5.06 (0.8046)

Table 3 indicates that the average yield during the period under review was about 10 tonnes/ha while that for production and harvested area were 26 million tonnes and 2.7 million hectares, respectively. The coefficient of variation value was 65.88% for production; 11.29% for yield and 74.21% for the harvested area, which imply high variability in production and harvested area. The skewness of the distribution of cassava production and the harvested area were to the right whereas the yield was to the left. Positive skewness is expected for production and harvested area since Table 2 indicates that the respective means are higher than the corresponding medians (Sheskin, 2011). It also indicates that cassava farmers have harvested cassava from less than the average 2.7 million hectares than they have harvested more than the average (Ikuemonisan and Adebola, 2021) with no significant kurtosis problem. The Jarque-Bera test for cassava production and harvested area are far from zero, thus indicating that the data do not have a normal distribution. The JB test for production is significant at 0.05 level while that for the harvested area is significant at 0.004 level.

Table 3: Descriptive statistics for cassava production, yield and area harvested in Nigeria

Descriptive statistics	Production	Yield	Area Harvested
Mean	26290931.88	100639.18	2695995.33
Median	22523504	99889.5	2095389
Maximum	60001531	122155	7737846
Minimum	7384000	70323	780000
Standard Deviation	17320211.56	11359.95	2000882.65
Coefficient of Variation (%)	65.8790	11.2878	74.2168
Skewness	0.5366	-0.2641	1.0498
Kurtosis	-1.1017	-0.1382	0.0667
Jarque-Bera	5.9140	0.7452	11.0313
Probability (p-value for Jaque-Bera test)	0.0520	0.6889	0.0040
Observations	60	60	60

3.2 Cassava Processing

As shown in Table 4 there was a general reduction in the mass of the samples as they passed through the stages of processing. The removal of the external layer of the cassava, water, chaff and other unrecoverable losses which resulted during peeling, grating, dewatering, sieving and frying, are responsible for the mass reduction.

However, a slight increase in the mass of Sample C was observed between the peeling and grating processes. This increment might have resulted from the remains of Sample A and B in the grater which were first grated before Sample C.

Table 4: Mass Reduction Through Different Stages of Processing

Samples	Unpeeled	Peeled	Grated	Dewatered	Sieved	Roasted
A	36.11	26.32	23.85	17.45	17.10	10.15
B	33.73	23.99	23.70	16.87	16.60	9.70
C	35.80	26.46	27.10	17.68	17.55	9.55

Table 5 shows the moisture content of each sample through the processing stages. The moisture content of the dewatered cassava mash (Sample A, Sample B and Sample C) is slightly higher than the 47-50% idealized by the Food and Agriculture Organization of the United Nations. This slight disparity could be attributed to the difference in cassava varieties and soil moisture content.

Table 5: Moisture Content Reduction Through Processing Stages

Samples	Grated (%)	Dewatered (%)	Garri (%)
A	63.5.0	56.00	11.40
B	63.00	57.60	11.60
C	62.30	56.30	11.50

3.3 Garri physical properties variation with storage period

Figure 8 shows the moisture content of garri samples at different time intervals for the different storage materials with no remarkable change in the moisture content of garri samples throughout the 15 weeks. This can be attributed to the samples being kept in the same laboratory cardboard which provided a relatively controlled humidity. Also, the appearance of the garri samples remain unchanged; an indication of an absence of storage moulds (Mills, 1992), which agrees with the observations by Amadi and Adebola (2008).

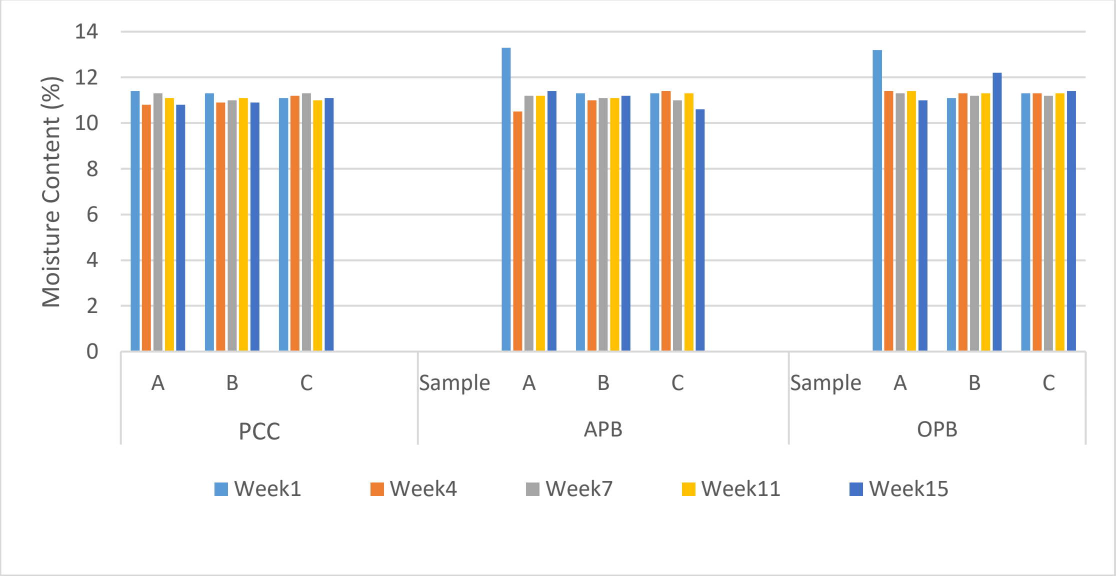

Figure 8: Moisture Content of Garri Samples for 15 Weeks

Figure 9 shows that the acid content increases with the period of fermentation; Sample A (18 hours) has lowest acid content whereas sample C (36 hours) has the highest acid content. This is responsible for the characteristic sour taste observed more in Sample C but mild in Sample B and weak in Sample A. The high acid content represents a 'good' garri with lower cyanide content. The acid content of Sample A corresponds to a typical market garri sample analysed by Ogbonna et. (2017), which suggests that some of the garri available in the market are products of a day or less fermentation process.

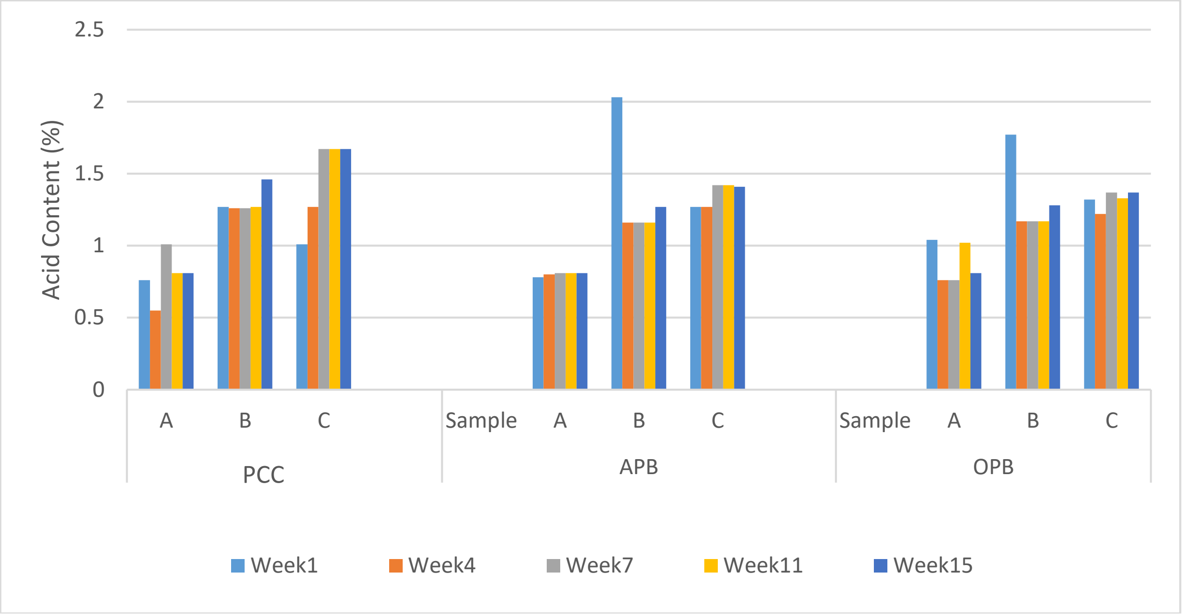

Figure 9: Acid Content of Samples over a Period of 15 Weeks

The swelling index and bulk density of the samples did not vary remarkably within the period under investigation; which are within the neighbourhood of 3 and 500 kg/m^3, respectively (Table 6 and Figure 10). A good garri has been reported as that which is capable of swelling at least 3 times its original volume (Ukpabi and Ndimele, 1990). Sanni *et al*., (2008) had reported a garri bulk density of 519 kg/m^3.

Table 6: Swelling Index of Samples over a Period of 15 Weeks

Container	Sample	Week1	Week4	Week7	Week11	Week15
PCC	A	3.05	2.90	3.30	3.30	3.30
	B	2.95	3.47	3.47	3.30	3.30
	C	3.00	3.50	3.30	3.30	3.30
APB	A	3.05	2.90	3.30	3.30	3.30
	B	2.95	3.47	3.47	3.30	3.30
	C	3.00	3.50	3.30	3.30	3.30
OPB	A	3.05	2.90	3.30	3.30	3.30
	B	2.95	3.47	3.47	3.30	3.30
	C	3.00	3.50	3.30	3.30	3.30

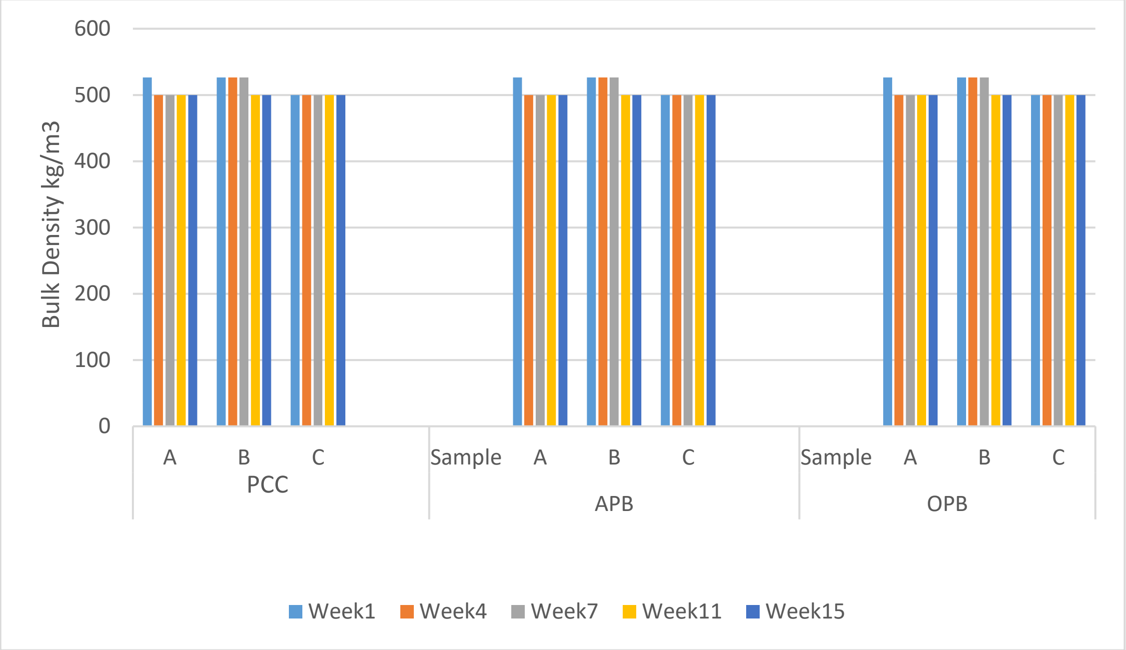

Figure 10: Bulk Density of Samples over a Period of 15 Weeks

The plot of swelling volume versus ln (Time) for Sample A, shown in Figure 11, is a straight line with regression coefficient (R^2) of 0.9764; similar to samples B and C. The slope of the plots is the swelling rate constant and is a is given, respectively, as 4.7452, 4.5756 and 4.3766 for Samples A, B and C; consumers demand garri with good swelling capacity (Owuamanam *et al*., 2011).

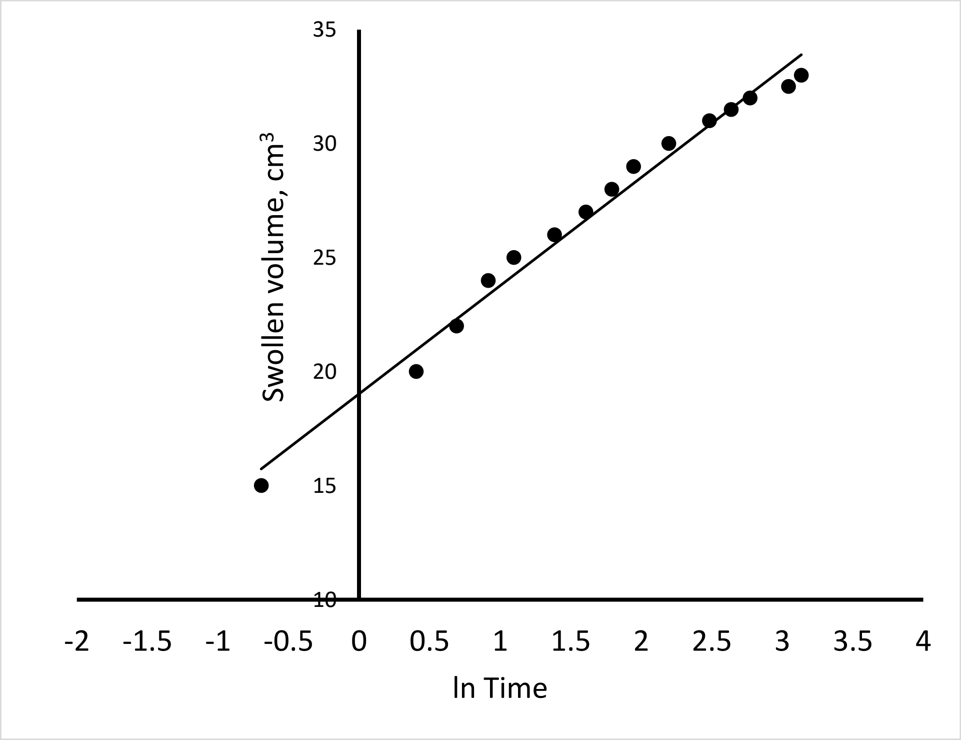

Figure 11: Swollen volume of garri versus ln time for Sample A

According to Tables 7 - 9; the Fineness Modulus (FM) of Sample A is between 4.19 and 4.48; Sample B falls between 4.21 and 4.44 and Sample C ranged from 4.69 to 5.09 which is similar to the range reported by Olaosebikan *et al*., (2016). The average particle size of A fell between 0.50 mm and 0.55 mm. Sample B maintained particle size between 0.50 mm and 0.54 mm, while

Sample C ranged from 0.58 mm to 0.66 mm. These results agree with Olaosebikan *et al*., (2016) who reported an average particle size range of 0.53 mm and 0.63 mm for a garri produced from a Conductive Rotary Dryer

(CRD) and 0.70 mm for a garri produced using the traditional fryer. Agbetoye and Oyeleke (2013) also reported a particle size of 0.553 mm for garri in Nigeria.

Table 7: Grain Size Distribution Sample A

Container	1 mm	710µm	500µm	425µm	300µm	Pan	FM	Average (mm)
PCC	17.00	10.00	13.00	2.30	5.05	2.34	4.4792	0.55
APB	12.90	9.80	14.05	3.50	6.85	3.00	4.194	0.50
OPB	13.30	10.45	15.60	2.71	6.05	2.17	4.3074	0.52

Table 8: Grain Size Distribution Sample B

Container	1 mm	710µm	500µm	425µm	300µm	Pan	FM	Average (mm)
PCC	12.95	11.49	15.82	2.49	5.36	1.81	4.3702	0.53
APB	13.90	11.53	15.68	2.43	4.41	1.51	4.4386	0.54
OPB	11.47	10.70	15.93	2.75	6.45	2.50	4.2138	0.50

Table 9: Grain Size Distribution Sample C

Container	1 mm	710µm	500µm	425µm	300µm	Pan	FM	Average (mm)
PCC	18.57	11.28	12.6	2.23	4.08	1.38	4.6862	0.58
APB	23.4	11.27	10.81	1.81	2.18	0.39	5.0202	0.65
OPB	25.47	10.80	9.84	1.60	2.0	0.46	5.0884	0.66

3.4 Material Balance for Garri Processing

Table 10 shows the input and output streams for the three samples. Out of 36.11kg, 33.73kg and 35.80kg of feed (unpeeled cassava), of Sample A, B and C, 10.15kg, 9.70kg and 9.55kg of garri was produced which represents 28.11%, 28.76% and 26.68% yield respectively. These results agree with the minimum of 25% reported by Sanni (1990).

Table 10: Material Balance for Garri Processing

Stream	Sample A		Sample B		Sample C	
	Input (kg)	Output (kg)	Input (kg)	Output (kg)	Input (kg)	Output (kg)
1	36.11		33.73		35.80	
2		9.79		9.74		9.34
4	20.00		20.00		20.00	
5		20.00		20.00		20.00
7		2.47		0.29	0.64	
9		6.40		6.83		9.42
11		0.35		0.27		0.13
13		6.95		6.90		8.00
14		10.15		9.70		9.55
Total	56.11	56.11	53.73	53.73	56.44	56.44

4. CONCLUSIONS

Political and economic decisions and ecological factors have been identified as among the critical factors that affect the cassava production potential of Nigeria. A significant increase in cassava production potential occurred within the period 1981 to 2000, which could be attributed to positive government policies and interventions. This research also showed that the duration of fermentation does not significantly affect the moisture content of garri and hence its storability. The moisture content of garri is roughly 11%. A garri with moisture content around this value and stored in a container with controlled relative humidity would be stable over several months of storage. However, the acid content varies with the fermentation duration while the swelling index and bulk density of the samples did not vary remarkably within the period under investigation. The roasting process plays an important role in its preservation. The results obtained will be useful to design an improved environmental friendlier and climate responsive garri roasting process unit powered by solar energy in the context of energy efficiency and quality control.

5. ACKNOWLEDGEMENTS

The authors acknowledge the Tertiary Education Trust Fund (TETFUND) Institution Based Research 2020 Grant for this work. However, neither the findings nor the views expressed necessarily reflect the policies of TETFUND. We are also grateful to Mrs. Virginia Chidadi Owhorji for her local knowledge of garri production process.

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10.51975/24380102.som
Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

DIGESTATE VALORIZATION: A VALUE ADDITION TO ANAEROBIC BIODIGESTION TECHNOLOGY

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ABSTRACT

Anaerobic digestion technology transforms diverse bio-sourced feedstocks into valuable products including biofertilizr., . Digestate, the leftover product after biogas generation, is mostly discarded as its valorization is domiciled within fertilizer application. The consumption rate for fertilizer generation is reported to be one-tenth of its production rate. This review found that large-scale bio-fertilizer production is confronted with high operational and transportation costs, greenhouse gas emissions during storage, and improper nutrient content. It becomes necessary to examine the potential of digestate beyond bio-fertilizer application. It was found that the digestate liquor is suitable for microalgae cultivation because of its high nutritional content. Due to its rich cellulose content, the digestate filtrate is a suitable culture medium for bioethanol production with the advantages of about 33% energy reduction with fewer inhibitory compounds. In the case of the digestate residue, strong complementary role of thermochemical process was showcased. It was found that several valuable products with superior properties were produced. These valuable products are exploited as: soil amender, adsorbent, activated carbon, particle board, and nanocellulose. However, the processes are still in their infancy and, as expected, have some drawbacks. Consequently, more studies to overcome these challenges are recommended.

Keywords: *DigestateValorization, Biofertilizer, Biochar, Bioethanol, Activated Carbon.*

1.0 INTRODUCTION

The growing interest in meeting the energy needs globally leads to a shift from fossil fuels to biofuels while keeping the environment pollution free (Turkiewicz*et al.*, 2013; Obonukut and Inyang, 2022). Studies, (Kozo *et al.*, 1996; Oparaku*et al.*, 2013), showcased the anaerobic digestion process to proffer a solution to not only the energy and environmental needs of the society but also an attractive "waste-to-wealth" strategy via digestate valorization, as shown in Figure 1.

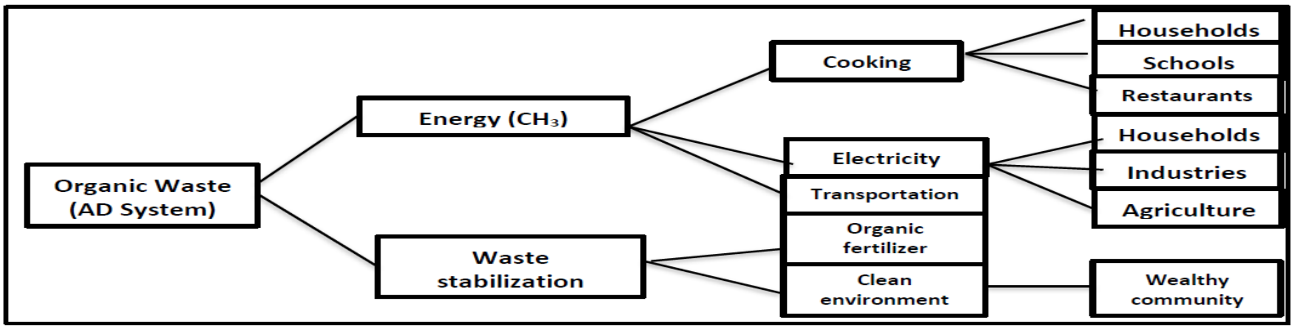


Figure 1: Anaerobic Digestion Technology: A waste-to-wealth strategy

Anaerobic Digestion (AD) Technology involves the degradation and stabilization of organic materials under anaerobic (oxygen-deficient) conditions by a microbial consortium leading to the production of an energy rich biogas and digestate. However, AD technology has been be devilled by the Dutch disease/Gregory effect. The Dutch Disease/Gregory Effect describes a situation in which growth in one sector causes neglect in the other. In other words, the strong demand for a particular resource causes a knock-on effect on the other (Warr, 2006).The impacts of the Gregory effect on AD technology not only defeat the essence of the technology but also adversely affect its profitability. This is critical and should be urgently addressed, hence the need for this review.

Specifically, AD technology is widely known because biogas generation is designed to convert biomass into recoverable methane-rich gas and a stabilized digestate using a complex anaerobic biological process. Biomass refers to biodegradable organic materials of plant and animal origin. It extends beyond the remains/residue of animals/plants to their waste, especially agricultural waste products, which are principally used as the feedstock for the AD process (Eboibi*et al.*, 2020). The advances in technological development reveal that these biological materials are highly valuable feedstock for the industrial production of chemicals, fuel, and other related products. Hence, they are regarded as bio-sourced feedstocks (Edensetinget *al.*, 2020; Issah*et al.*, 2020)).

AD produces two valuable outputs: biogas and digestate. However, biogas is considered more valuable and highly sought-after than its counterpart (Abila, 2012). Akinbami *et al.* (2001) reported that Methane, the primary component of natural gas, is present in high concentrations in biogas (50 to 75 percent). Biogas, like natural gas, is energy-rich (fuel) and provides heat, generate electricity, and power cooling systems, among other uses (Akinbami *et al.*, 2001). Moreover, biogas, when purified by removing inert or low-value constituents, is considered cleaner and more environmentally friendly than natural gas (Andrade *et al.*, 2019). Consequently, most AD plants are widely known as biogas plants, as if biogas is the only product generated from this technology (Dariusz *et al.*, 2012).

Profit maximization of any process depends on the total valorization of all its products, ensuring zero wastage. Of all AD products, digestate is one of the most neglected and unexploited (Szwaja*et al.*, 2019). In this paper, digestatevalorisation for fertilizer application and its challenges were reviewed (section 2). Thermochemical processes with a complementary role in the digestate residue waspresented (section 3). This was followed by a detailed description of alternative routes of digestate valorization other than fertilizer applications discussed insection 4

2.0 DIGESTATE VALORISATION FOR FERTILIZER APPLICATION AND ITS CHALLENGES

Digestate is mechanically separated into liquid and solid fractions that are stored separately for easy handling and transport. The liquid fraction (digestate filtrate) contains a large part of N and K, whereas the solid fraction (digestate residue) is composed of a large amount of residual fiber and phosphorous (Lied *et al.*, 2006). These are the reasons behind their applications as fertilizer or soil improvers (Tambone *et al.*, 2010; Rodriguez-Navas*et al.*, 2013).

Clearly, as a stabilized and homogeneous product from the anaerobic digestion of organic matter, the mineralization of fertilizing elements in the digestate liquor (filtrate) makes it a product of choice for soil nutrition (Insam *et al.*, 2015). The utilization of digestate for agricultural purposes represents economic and environmental benefits. Biofertilizer is of prime importance in the recycling of limiting nutrients (e.g., phosphorous) and is a better substitute for commercial fertilizer (Suominen *et al.*, 2014). Walsh *et al.* (2012) indicated that, contrary to commercial fertilizers, digestate improves yields from grassland cultures and concurrently reduces nutrient losses to the environment (Monlau*et al.*, 2015). Studies have shown that digestate (bio-fertilizer) from the AD process is superior to that of aerobic digestion in terms of bioavailability as its nutrients are more accessible to crops (Manlay, 2000; Dioha*et al.*, 2005). Despite the edge over others, less than one-tenth of the digestate produced is utilized for bio-fertilizer, and the remaining is discarded due to the following drawbacks attributed to fertilizer application.

Firstly, storing digestate for future application is challenging as its consumption rate is limiting the growth stages of crops, soil size/type, and stabilization levels (Ndiaye*et al.*, 2010). In most cases, the digestate is stored in uncovered tanks releasing greenhouse gases into the atmosphere (Sambusiti *et al.*, 2013). The gases (CH_4, CO_2, and N_2O), affect the global environment and climate, while NH_3 contributes to general atmospheric pollution (Bonetta*et al.*, 2014). Secondly, commercial production of digestate is challenged by the high cost of transporting the digestate (Ndiaye*et al.*, 2010). The quantity of bio-fertilizer supplied per farm is small according to regulations on nutrient loading per hectare based on fertilization plan (Holm-Nielsen *et al.*, 2009). Once the costs of transportation and spreading are taken into account, the value of the digestate can be close to zero and it may even become an expense for the farmer

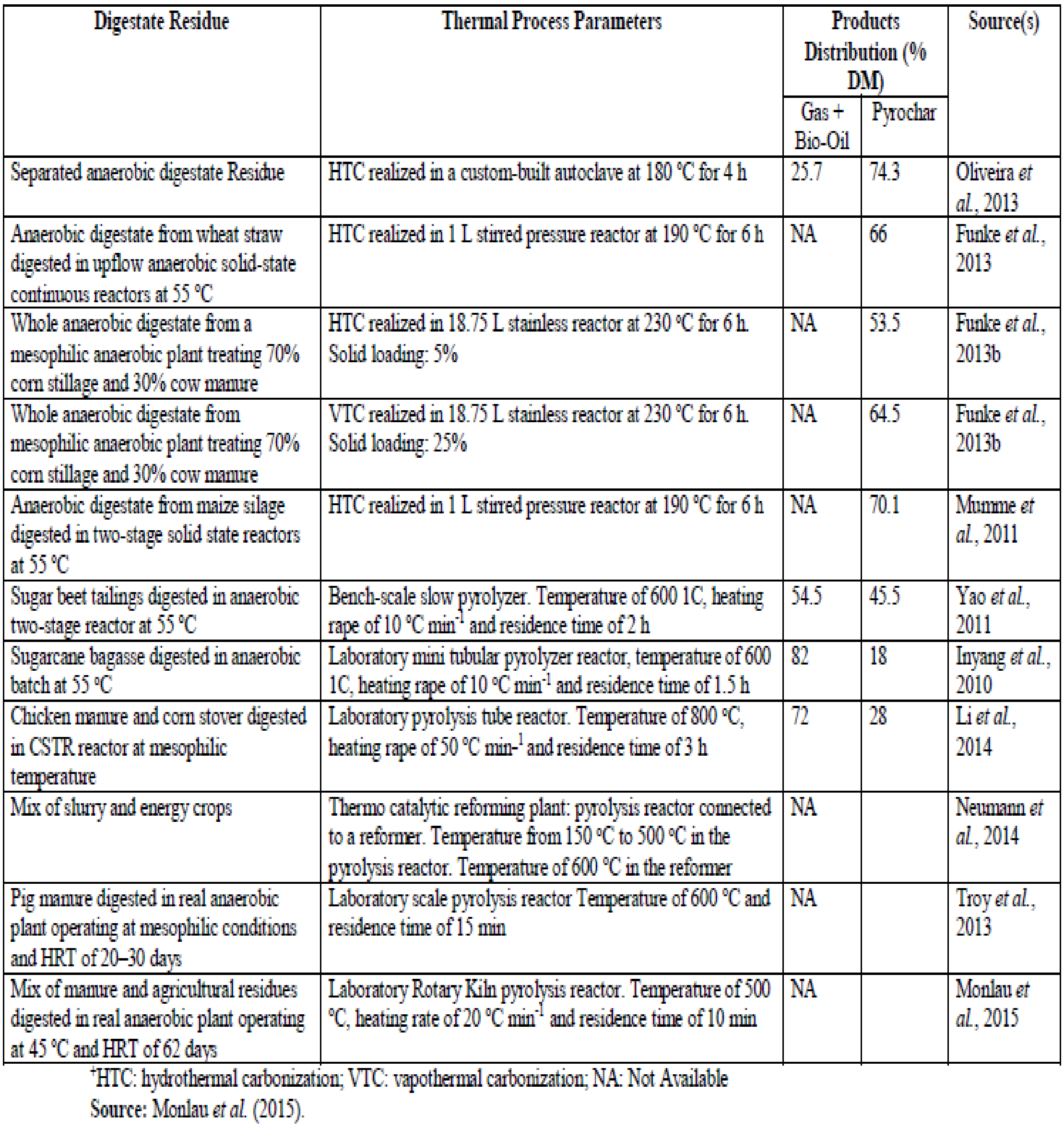
(Monlau *et al.*, 2015). Thirdly, environmental pollutants (pathogens, heavy metals, pesticides, steroid hormones, and organic compounds) may be found in bio-fertilizers (Bonetta*et al.*, 2014; Insam*et al.*, 2015). This is one of the challenges negating its application, making it unattractive for storage. However, with good agricultural practices, optimal environmental and economic benefits can be achieved using bio-fertilizers (Holm-Nielsen *et al.*, 2009).

Proper characterization of the digestate before its application as fertilizer would prevent contamination of soil, which would eventually lead to food chain contamination (Zirkler *et al.* 2014). This would reveal the irregularity attributed to an unbalanced nutrient as well as the presence of contaminants.

3.0 COMPLEMENTARY ROLE OF THERMOCHEMICAL PROCESSES ON DIGESTATE RESIDUE

Thermochemical processes (including combustion, hydrothermal carbonization, and pyrolysis) have played a complementary role in various biological processes; including anaerobic digestion process (Szwaja*et al.*, 2019). Several researchers have investigated the impacts of thermal processes on anaerobic digestate and found that valuable products can be produced, which eventually improves the overall energy efficiency of AD processes translating to increased profitability (Hoffman *et al.*, 2013; Li *et al.*, 2014; Monlau*et al.*, 2015; Szwaja*et al.*, 2019). Table 1 shows these thermochemical processes and digestates exploited.

Table 1: Thermochemical processes of anaerobic digestates⁺



Digestate Residue	Thermal Process Parameters	Products Distribution (% DM)		Source(s)
		Gas + Bio-Oil	Pyrochar	
Separated anaerobic digestate Residue	HTC realized in a custom-built autoclave at 180 °C for 4 h	25.7	74.3	Oliveira *et al.*, 2013
Anaerobic digestate from wheat straw digested in upflow anaerobic solid-state continuous reactors at 55 °C	HTC realized in 1 L stirred pressure reactor at 190 °C for 6 h	NA	66	Funke *et al.*, 2013
Whole anaerobic digestate from a mesophilic anaerobic plant treating 70% corn stillage and 30% cow manure	HTC realized in 18.75 L stainless reactor at 230 °C for 6 h. Solid loading: 5%	NA	53.5	Funke *et al.*, 2013b
Whole anaerobic digestate from mesophilic anaerobic plant treating 70% corn stillage and 30% cow manure	VTC realized in 18.75 L stainless reactor at 230 °C for 6 h. Solid loading: 25%	NA	64.5	Funke *et al.*, 2013b
Anaerobic digestate from maize silage digested in two-stage solid state reactors at 55 °C	HTC realized in 1 L stirred pressure reactor at 190 °C for 6 h	NA	70.1	Mumme *et al.*, 2011
Sugar beet tailings digested in anaerobic two-stage reactor at 55 °C	Bench-scale slow pyrolyzer. Temperature of 600 1C, heating rape of 10 °C min^{-1} and residence time of 2 h	54.5	45.5	Yao *et al.*, 2011
Sugarcane bagasse digested in anaerobic batch at 55 °C	Laboratory mini tubular pyrolyzer reactor, temperature of 600 1C, heating rape of 10 °C min^{-1} and residence time of 1.5 h	82	18	Inyang *et al.*, 2010
Chicken manure and corn stover digested in CSTR reactor at mesophilic temperature	Laboratory pyrolysis tube reactor. Temperature of 800 °C, heating rape of 50 °C min-^{1} and residence time of 3 h	72	28	Li *et al.*, 2014
Mix of slurry and energy crops	Thermo catalytic reforming plant: pyrolysis reactor connected to a reformer. Temperature from 150 °C to 500 °C in the pyrolysis reactor. Temperature of 600 °C in the reformer	NA		Neumann *et al.*, 2014
Pig manure digested in real anaerobic plant operating at mesophilic conditions and HRT of 20–30 days	Laboratory scale pyrolysis reactor Temperature of 600 °C and residence time of 15 min	NA		Troy *et al.*, 2013
Mix of manure and agricultural residues digested in real anaerobic plant operating at 45 °C and HRT of 62 days	Laboratory Rotary Kiln pyrolysis reactor. Temperature of 500 °C, heating rate of 20 °C min^{-1} and residence time of 10 min	NA		Monlau *et al.*, 2015

⁺HTC: hydrothermal carbonization; VTC: vapothermal carbonization; NA: Not Available

Source: Monlau *et al.* (2015).

4.0 EMERGING OPPORTUNITIES FOR DIGESTATE VALORIZATION

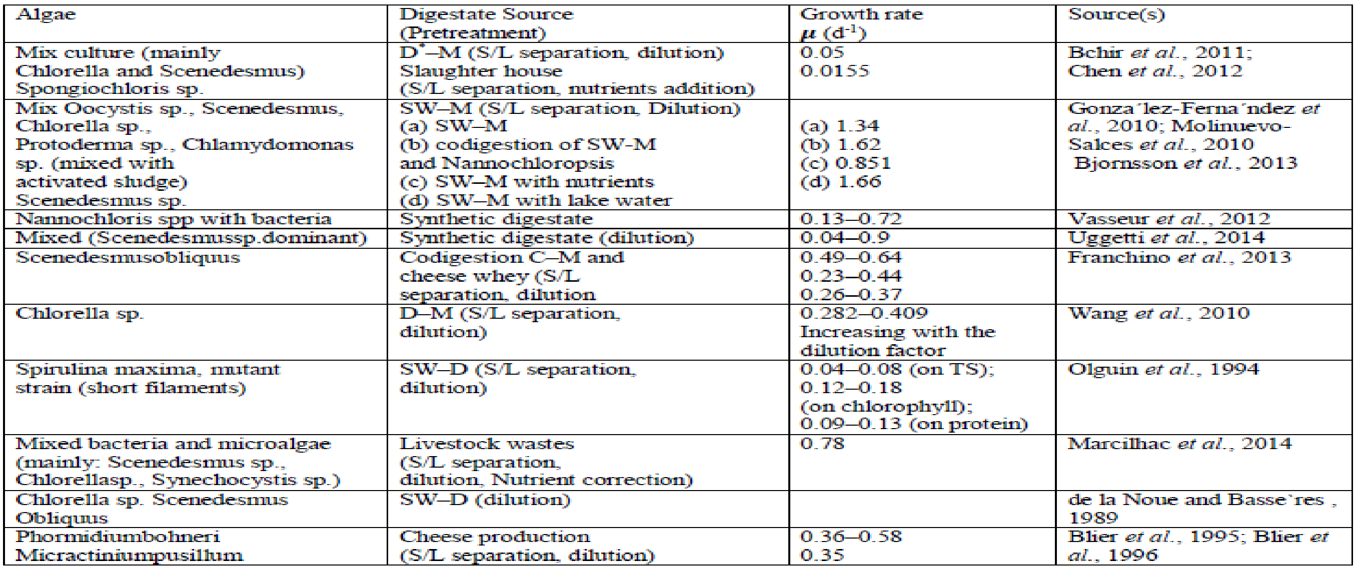
Clarification (i.e., dewatering) and further thermal processing of digestate have been investigated and promisingvalorization alternatives are reported as presented in this section.

4.1: Digestate Valorization: Emerging Opportunities for Microalgae Cultivation

The impact of anaerobic digestion on algae growth dates to the 1950s. Golueke*et al.* (1957) suggested that the anaerobic digestion process would promote the conversion of sunlight into chemical energy. However, no further research was conducted to investigate the mechanism until recently when experiments were carried out to evaluate the feasibility of digestates as a nutrient source in microalgae cultivation was investigated and the outcome was promising (Monlau*et al*., 2015).

The hike in food prices promoted algae cultivation especially when algae (i.e.4th generation bio-sourced feedstock) was found to be a suitable alternative to first-generation counterparts (i.e., edible crops) for biofuel production (Edensetinge*t al*., 2020). However, algae cultivation for biofuel production was initially uneconomical as production and extraction costs were found to be too high (Monlau*et al*., 2015). Several strategies geared towards cost reduction and possibly improving the viability of algae cultivation economically have been exploited. Alternative valorization of anaerobic digestate via nutrient recovery has been well-reported as the most attractive strategy (Norsker*et al*., 2011). Specifically, microalgae were assessed for their ability to grow on digestates and the most prominent ones among them are presented in Table 2.

Table 2: Digestates valorization for algae cultivation



Algae	Digestate Source (Pretreatment)	Growth rate μ (d^{-1})	Source(s)
Mix culture (mainly Chlorella and Scenedesmus) Spongiochloris sp.	D*–M (S/L separation, dilution) Slaughter house (S/L separation, nutrients addition)	0.05 0.0155	Bchir *et al.*, 2011; Chen *et al.*, 2012
Mix Oocystis sp., Scenedesmus, Chlorella sp., Protoderma sp., Chlamydomonas sp. (mixed with activated sludge) Scenedesmus sp.	SW–M (S/L separation, Dilution) (a) SW–M (b) codigestion of SW-M and Nannochloropsis (c) SW–M with nutrients (d) SW–M with lake water	(a) 1.34 (b) 1.62 (c) 0.851 (d) 1.66	Gonza´lez-Ferna´ndez *et al.*, 2010; Molinuevo-Salces *et al.*, 2010 Bjornsson *et al.*, 2013
Nannochloris spp with bacteria	Synthetic digestate	0.13–0.72	Vasseur *et al.*, 2012
Mixed (Scenedesmussp.dominant)	Synthetic digestate (dilution)	0.04–0.9	Uggetti *et al.*, 2014
Scenedesmusobliquus	Codigestion C–M and cheese whey (S/L separation, dilution	0.49–0.64 0.23–0.44 0.26–0.37	Franchino *et al.*, 2013
Chlorella sp.	D–M (S/L separation, dilution)	0.282–0.409 Increasing with the dilution factor	Wang *et al.*, 2010
Spirulina maxima, mutant strain (short filaments)	SW–D (S/L separation, dilution)	0.04–0.08 (on TS); 0.12–0.18 (on chlorophyll); 0.09–0.13 (on protein)	Olguin *et al.*, 1994
Mixed bacteria and microalgae (mainly: Scenedesmus sp., Chlorellasp., Synechocystis sp.)	Livestock wastes (S/L separation, dilution, Nutrient correction)	0.78	Marcilhac *et al.*, 2014
Chlorella sp. Scenedesmus Obliquus	SW–D (dilution)		de la Noue and Basse`res , 1989
Phormidiumbohneri Micractiniumpusillum	Cheese production (S/L separation, dilution)	0.36–0.58 0.35	Blier *et al.*, 1995; Blier *et al.*, 1996

However, turbidity of the media due to dissolved and suspended materials in the digestates is one of the major drawbacks to digestates' valorization via algae cultivation (Wang *et al*., 2010). Ammonia inhibition is another critical issue of concern as microalgae are known to use ammonium as a source of nitrogen (Uggetti*et al.*, 2014). More so is bacterial contamination, as it may induce sanitary issues for downstream biomass valorization (Chen *et al*., 2012). The contamination is inevitably due to microbe-medium interactions, either symbiotically or competitively. It is believed that bacterial growth will alter the turbidity, pH, dissolved oxygen, nutrient apportionment (mineral or organic), and chemical nature (oxidation state) of the media.

4.2 Digestate Valorization: Emerging Opportunities in Soil Amendment

The endearing properties (stability, porosity, mineral content, etc.) of pyrochars are exploited to improve soils with fragile structures, waterlogged soil, and attenuate the drought stress of semi-arid regions of the world. The porous structure of pyrochars not only improves soil structure but also provides shelter for beneficial soil microorganisms and, as well, influences positively the binding effect between nutritive cations and anions (Monlau*et al*., 2015). The application of pyrochar to soil has been reported to minimize the dispersal of pesticides and the leaching of heavy metals with their subsequent accumulation in plants (Uchimiya*et al.*, 2010).

The environmental implications of using pyrochar from AD digestate in agricultural soils have been well-reported by many researchers. These include: pyrochar from digested dairy waste (DW) with BET surface area and total pore volume of 161.2 $m^2 g^{-1}$ and 0.147 cm3 g^{-1} while whole sugar beet (SB) was 48.6 $m^2 g^{-1}$ and 0.034 $cm^3 g^{-1}$ (Inyang*et al.*, 2012). Yao *et al*. (2011) reported that pyrochar obtained from anaerobically digested biomass has a higher accessible surface area (336 m^2 g^{-1}) than pyrochar produced from raw feedstock (2.6 m^2 g^{-1}). Pituello*et al.* (2014) reported that a pyrochar produced using a digestate from cattle manure and silage combo exhibited a higher phosphorus concentration than the initial feedstock, suggesting that they can be used for low nutrient release as phosphorus is an important element for plant growth. Therefore, the development of pyrochars with superlative properties using anaerobic digestate is an added advantage to the AD process.

Despite this potential of exploiting pyrochars produced from digestate as soil amenders, there are some aggressive components of the digestate which can have negative effects on the soil, crops, and even the environment. In view of this, characterization of digestates after the AD process as well as pyrochars produced from the digestate is necessary. Before application, these bioavailable compounds should be known and their potential negative effects isolated and carefully addressed.

4.3 Digestate Valorization: Emerging Opportunities for Activated Carbon Development

The adsorption capacity of pyrochars can be further improved by converting them into activated carbons (ACs). However, the activation process requires two further stages of pyrolysis of the carbon at a temperature below 800 ^{o}C, followed by a steam activation process (Ioannidou and Zabaniotou, 2007). The activation process promotes not only the stability of the material but also its porosity and surface area. In view of this, ACs from digestate residues have been found to exhibit a much higher accessible surface area and porosity than pyrochars (Monlau*et al*., 2015).

Specifically, activated carbons from the digestate residues of the AD process using dairy manure exhibited a high accessible surface area and pore volume of 1950 m^2 g^{-1} and.232 cm^3 g^{-1}, respectively(Yuan *et al.*, 2011). Presently, there is a greater demand for activated carbon due to the emerging non-biodegradable contaminants from industrial wastes constituting an environmental issue (vide supra). Activated carbon is the most efficient adsorbent that removes these contaminants, and its consumption has been reported to have dramatically increased recently (Monlau*et al*., 2015). Commercial activated carbons are produced from expensive but nonrenewable materials (coal, etc.), making the ACs expensive and much more expensive if not produced locally. AD digestate residue is suitable for the production of low-cost value-added ACs (Yuan *et al.*, 2011).

4.4 Digestate Valorization: Emerging Opportunities in Bioethanol Production

Anaerobic digestate residue has an edge over the conventional feedstock exploited for bioethanol production. Two types of anaerobic digesters (CSTR and PFR) as reported by Yue*et al.* (2011) were exploited for bioethanol production using AD digestates. It was found that 105 g kg^{-1} dry digestate and 85 g kg^{-} dry digestate of bioethanol were produced from CSTR and PFR, respectively (Yue*et al.*, 2011). The higher bioethanol production from the CSTR digestate was due to its higher cellulose content than in the PFR digestate (Yue*et al.*, 2011). Similar results were obtained during switchgrass bioethanol fermentation (Yue*et al.*, 2010). Digestate residue after co-digestion of corn stover and swine manure (40 and 60% fresh material, respectively), as reported by MacLellan*et al.* (2013), produced 152 g CH_4 and 50 g ethanol kg^{-1} DM via coupling AD and the bioethanol production process.

AD Digestates perform better than the conventional feedstock for bioethanol production as the digestate has been hydrolyzed during AD process (Yue*et al.*, 2011). At this stage, its lignin content is not only enriched with cellulose but also enhanced its accessibility (Monlau*et al*., 2015). AD process with low hydraulic retention time (HRT) has equally been exploited as a biological pretreatment process to alter the composition of the fibrous feedstock, making it suitable as a cellulosic feedstock for ethanol production (Yue*et al.*, 2011). This strategy is a relatively new concept and its role is still controversial in the literature (Vancov *et al.*, 2015).

However, in an attempt to advance this strategy, Yue*et al.* (2011) showed that anaerobically digested manure contains less hemicellulose (11%) and more cellulose (32%) than raw manure and it presents better enzymatic digestibility than switch-grass. This strategy needs to be probed further. Meanwhile, the conventional bioethanol production process involves mechanical fractionation and/or thermo-chemical pretreatment followed by enzymatic hydrolysis-ethanolic fermentation (Monlau*et al*., 2015). In a related study by Motte*et al*. (2015), it was found that anaerobic biological degradation prior to mechanical fractionation could significantly reduce the energy requirement of the milling step from 142 kWh t^{-1} for initial feedstock to 95 kWh t^{-1} for digestate.

5.0 CONCLUSION

Generally, effective utilization of any process output or effluent requires its full valorization into a product of higher value. Anaerobic digestion technology is not an exception, as the full valorization of its products (biogas and digestate streams) is a necessity. This paper concludes that digestate valorization beyond bio-fertilizer application is the way forward to prevent the Gregory effect confronting the AD process. Valorization of the AD digestate increases the economic prospects and curtails the impending environmental issue of an AD/biogas project. However, most of the digestate valorization processes reviewed are challenged as the technology is still in its nascent state. Intensive research in this direction is the way forward to ensure that AD technology is not a waste generation strategy but a waste disposal/treatment strategy.

Acknowledgement

The earlier version of this paper was presented during the 52nd Annual General Meeting, Conference and Exhibition of the Nigerian Society of Chemical Engineers (NSChE), Nov. 10-12, 2022, Ilorin, Nigeria.

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10.51975/24380103.som

Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

SYNTHESIS, CHARACTERIZATION AND EVALUATION OF THE CORROSION BEHAVIOUR OF CARBON STEEL C1020 IN *JATROPHA* OIL BIODIESEL

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ABSTRACT:

In this work, jatropha oil biodiesel (JOB) was synthesized and characterized using gas chromatography-mass spectroscopy (GC-MS), Fourier transform infrared (FTIR) spectroscopy and ultra violet-visible (UV-Vis) spectrophotometry. Its corrosion impact on C1020 carbon steel was investigated at room temperature (25 ℃) using extensive weight loss measurements complemented with UV-Vis, FTIR and optical surface camera imaging. The C_{18} *fatty acids were the most dominant components of JOB. Weight loss measurements revealed that the steel corrosion rate in the biodiesel increased with the immersion time and exhibited a corrosion rate of 0.0036 mpy after 56 days. UV-Vis analysis further confirmed the* C_{18} *fatty acid groups as the major acid responsible for the corrosion attack. The acid induce the corrosion by physically adsorbing on the steel surface using their C–O and C=O functional groups based on FTIR and UV-Vis results. Optical surface imaging affirmed the occurrence of localized pitting corrosion after prolonged exposure. The results of the investigations revealed that over time, diesel engine components made of steel would suffer corrosion-related surface degradation when biodiesel is incorporated in the fuel system resulting in diminished structural integrity and contamination. Thus, organic, non-reactive inhibitors could be investigated in mitigating corrosion tendencies associated with biodiesel.*

Keywords: *Biodiesel production; Chromatographic analysis; Weight loss; Spectroscopic investigations; Optical microscopic study.*

1.0 INTRODUCTION

Energy is an indispensable ingredient that sustains life in any society. Fossil fuel contributes more than 60 % of the global energy supply (Chandran 2020). However, the global deposit of crude oil and other fossil fuels is presently depleting and several global regulations have been promulgated to minimize the use of fossil fuels due to their negative environmental impact. Currently, researchers are intensifying efforts in promoting the replacement of conventional fossil-derived diesel with greener, cheaper and efficient bio-derived diesels (Kugelmeier *et al.*, 2021; Chandran 2020; Monteiro *et al.*, 2018). In recent times, biodiesel has become more attractive due to its numerous technical advantages which it has over conventional diesel fuel. According to (Adama 2021; Alves *et al.*, 2019; Fazal *et al.*, 2019; Ahmmad *et al.*, 2018), these advantages include inherent lubricity, higher cetane number and flash point, very low contents of aromatic and sulfur compounds, and less exhaust emissions.

Biodiesel is produced by the transesterification reaction of vegetable oil or animal fats in the presence of a homogeneous or heterogeneous catalyst. This reaction leads to the formation of glycerol (as by-product) along with corresponding fatty acid alkyl esters (which is the biodiesel) (Adama *et al.*, 2021; Bala *et al.*, 2017). Despite its numerous advantages, the chemical composition of biodiesel, and its other characteristics like high viscosity, auto-oxidation (oxidative instability) and high volatility, make biodiesel corrosive to automobile and industrial engine parts which run on diesel (Fazal *et al.*, 2019; Fernandes *et al.*, 2013). In recent times, non-edible oil such as *jatropha* oil biodiesel has shown to be a potential substitute for crude oil-derived diesel. Its corrosion impact on steel had been investigated by several researchers.

Kaul *et al.*, (2007) compared the corrosive characteristics of *jatropha curcas*, *Karanja*, *Mahua*, and *Salvadora* biodiesels with those of diesel fuel. They observed that *Jatropha curcas* and *Salvadora* biodiesels were more corrosive for both ferrous and non-ferrous alloys as compared to *Karanja* and *Mahua* biodiesels. In another work, Ahmmad *et al.*, (2018) also investigated the corrosion of copper, stainless steel and aluminum alloys in *jatropha* biodiesel using weight loss measurement and chemical analysis of biodiesel before and after immersion (to determine concentration of fatty acids in the diesel). The main fatty acids observed in the tested biodiesel were oleic, linoelic, palmitic, and stearic acids. Apart from linoelic acid, the compositions of all other acids were increased after the immersion test of 1600 h, and copper showed the highest corrosion rate while stainless steel showed the least corrosion rate. Furthermore, Dharma *et al.*, (2019) investigated the corrosion behaviour of mild steel immersed in fuel blends of *jatropha curcas* biodiesel and *ceiba pentandra* biodiesel for up to 800 h at ambient temperature using weight loss and SEM characterization. Akhabue and Nduka (2016) studied the corrosion behaviour of brass, galvanized steel and stainless steel in blends of *jatropha* biodiesel and diesel using weight loss method and also determination of the acid value, peroxide value, and iodine values of the fuels during the immersion. They visually investigated the degradation of the metal coupon surfaces and colour after the immersion test. The corrosion rates were observed to increase with increase in the volume of the diesel added. Akhabue *et al.,* (2014) also investigated the corrosion of mild carbon steel and aluminum on immersion in *jatropha curcas* biodiesel blended with different volumes of diesel fuel for 18 weeks at room temperature. Their findings showed that the corrosion rates increased with increases in immersion times at varying degrees for both mild carbon steel and aluminum. Chourasia *et al.*, (2020) investigated the behaviour of numerous biodiesel including *jatropha* biodiesel corresponding to corrosion when exposed to the surface of metal coupons. The investigation was conducted using static immersion tests, scanning electron microscope (SEM) and surface roughness analysis at ambient temperature conditions.

In all these studies, the authors have only focused on understanding the rate of corrosion of the alloys without clear understanding of the mechanism by which the corrosion phenomena occur. Such mechanisms could be understood by augmenting the weight loss measurements with surface and spectroscopic investigations over time. It will explain the mode of surface interaction between the organic molecules in the biodiesel and the metal atoms released from the alloy surface during the corrosion. Furthermore, augmenting these measurements with a spectroscopic technique will enable us to understand the nature of chemical reaction between the organic molecules in the biodiesel and the metal atoms from alloy surface. Thus, the justification for this study examine the corrosion effect of *jatropha* oil biodiesel on C1020 carbon steel by complementing weight loss measurements with time-evolution UV-Vis, and FTIR characterizations and surface morphology. The findings would make significant contributions to research on alternative source of fuel for the automotive industry.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials and reagents used in the research include *jatropha* oil, *jatropha* oil biodiesel, methanol, potassium hydroxide pellets, polishing papers (#400, #600 and #800 grit sizes), thread, carbon steel coupons (C1020), analytical grade acetone, ethanol, hydrochloric acid and diethyl ether. The equipment used in the research include vortex mixer, centrifuge, separating funnels and settling tanks, weighing balances, measuring cylinders, heating mantles, conical and flat bottom flasks, immersion bottles, Buchler torramet specimen dryer, Stabinger viscometer, JASCO770-UV–Vis spectrophotometer (200–650 nm), Cary 60 UV-Vis spectrometer, Cary 630 FTIR equipment, XRF (ThermoFisher Scientific Inc. Ecublens, SARL, Switzerland) ARL 9900 IntelliPower series 0699, x-ray fluorescence (XRF) spectrometer with integrated x-ray diffractometer (XRD) capacity), GC-MS/FID equipment (Agilent 7890 A, Agilent Technologies, USA), density meter, cloud and pour point tester, and flash point tester.

2.2. Methods

2.2.1. Production of *jatropha* oil biodiesel

Jatropha oil biodiesel production was performed using two-step reaction involving acid-catalyzed esterification and base-catalyzed transesterification. *Jatropha* oil was obtained from jatropha seed using mechanical extractor following procedures adopted in an earlier study (Adama, 2021). The produced *jatropha* oil had saponification value of 224.4 mg KOH/g, acid value of 46.7 mg KOH/g, free fatty acid (FFA) value of 23.35 mg KOH/g, oil limit 0.50 mg KOH/g. The FFA was first converted to esters in a pretreatment process with methanol using a molar ratio of oil to methanol of 1:12 at 60 ℃ and after 120 mins, as reported previously (Adama, 2021; El-Sherbing *et al.*, 2010; Sarin *et al.*, 2010). The base-catalyzed transesterification of the esterified *jatropha* oil was conducted using methanol as the alcohol and potassium hydroxide being the catalyst. The transesterification process was investigated at a molar ratio of 6:1 (for methanol : oil) using procedures reported by other authors (Adama, 2021; Adama *et al.*, 2021; El-Sherbing *et al.,* 2010; Sarin *et al.*, 2010; Verma *et al.*, 2016). On completion of reaction, the product was transferred into a separating funnel and allowed to settle for 24 h. Two distinctive layers of jatropha oil biodiesel and glycerol were formed and the glycerol (lower) layer was drained-off accordingly. Warm water was used to remove excess amount of methanol, glycerol and catalysts from the *jatropha* oil biodiesel produced. The produced biodiesel was then subjected to physicochemical analysis, fatty acid profiling and fuel property analysis.

2.2.2. Characterization of *jatropha* oil biodiesel

The *jatropha* oil biodiesel was subjected to various physicochemical, spectroscopic and fuel property analyses according to known standards such as AOAC, ASTM, and AOCS (Adama *et al.*, 2021). This was done to determine the various properties of the *jatropha* oil such as saponification value, iodine value, peroxide value, free fatty acid value, acid value, specific gravity value, refractive index, moisture content, density, ash content, smoke point, fire point, flash point, cloud point, FTIR and viscosity using appropriate standard test methods and apparatus as reported in an earlier study (Adama, 2021). GC-MS (Agilent 7890 A, GC-MS by Agilent Technologies, USA) was used to identify the different fatty acid molecules present in the *jatropha* biodiesel. Using a Cary 630 FTIR equipment, the functional groups present in the *jatropha* oil biodiesel samples were identified. UV-Vis spectroscopy was used to identify any complexion product in the *jatropha* oil biodiesel samples. This was carried out using a JASCO770-UV–Vis spectrophotometer in the range of 200–650 nm.

2.2.3. Elemental composition of carbon steel coupon

The elemental composition of the C1020 steel coupons was investigated using a Thermo Scientific (ThermoFisher Scientific Inc. Ecublens, SARL, Switzerland) ARL 9900 IntelliPower series 0699 x-ray fluorescence (XRF) spectrometer with integrated x-ray diffractometer (XRD) capacity. A QuantAS software for total elemental analysis of up to 86 elements was included with a 12-point sample charger and a goniometer capable of analyzing specific element quantitatively and qualitatively. It has an elemental range: Boron (B) – Americium (Am), LLD (Concentration): 0.1 ppm – 100 %, resolution: $(M_n - K_a)$ – 135 eV, and sample throughput of up to 240 per 8 hours day. The elemental composition of the C1020 steel coupons is as recorded in an earlier study (Adama and Onyeachu, 2023).

2.2.4. Weight loss measurement

The C1020 steel bars were machined into weight loss coupons with dimensions 3 cm × 3cm × 1 cm. The sample preparation involved abrading with emery papers of variable grades (400, 600, 800 and 1200 grades). The coupons were then washed thoroughly with distilled water, degreased in acetone and dried with warm air following procedures adopted in similar studies (Adama and Onyeachu, 2023; Samuel and Gulum, 2018). Weight loss measurements were conducted after 7, 14, 28, and 56 days immersion of duplicate coupons in 200 ml solution of the *jatropha* oil biodiesel at room temperature (25 ℃) and under static condition. After each immersion time, the coupons were retrieved, cleaned according to ASTM G109 standard procedure, and reweighed in order to determine weight loss (ΔW) based on Equation (1) and corrosion rates, CR (mpy) based on Equation (2) (Onyeachu *et al.*, 2022; Akhabue and Nduka, 2016).

$$\Delta \mathrm{W}\ (\mathrm{g}) = \mathrm{W}_{(\text{before immersion})} - \mathrm{W}_{(\text{after immersion})} \quad (1)$$

$$\mathrm{CR}\ (\mathrm{mpy}) = \frac{\Delta \mathrm{W} \times 3.45 \times 10^{6}}{\mathrm{A} \times \rho \times \mathrm{T}} \quad (2)$$

Where w = weight of coupon,
$\Delta\mathrm{w}$ = weight difference
A = total surface area of coupon,
ρ = density $(^{\mathrm{g}}/_{\mathrm{cm}^3})$
T = immersion time in days,
CR = Corrosion rate
mpy = millimeters per year

2.2.5. Post weight loss investigations

The ultra violet–visible (UV–Vis) experiments were conducted before coupon immersion in the *jatropha* oil biodiesel and after 28 and 56 days immersion in the *jatropha* oil biodiesel, with the aid of a JASCO770-UV–Vis spectrophotometer (200–650 nm) using a dual beam operated at a resolution of 1 nm with a scan rate of 200 nm min^{-1}. UV-Vis spectroscopy was used to identify any complexion product in the *jatropha* oil biodiesel samples. Fourier Transform Infra-red (FTIR) spectrophotometry was used to identify the functional groups present in the *jatropha* oil biodiesel samples. The FTIR analysis was conducted using a Cary 630, Agilent Technologies, USA, Fourier transform infra-red spectroscopy equipment. High resolution camera (ANDOR cameras series iDUS CCD) with 1024 x 256 pixels, 26 μm pixel size, peak QE of 95 % (Vis) made by Oxford Instruments, U.K was employed to determine the surface morphology of the corroded steel after 28 and 56 days immersion.

3.0 RESULTS AND DISCUSSION

3.1 *Jatropha* Oil Biodiesel Characterization

Table 1 shows the fatty acid methyl ester (FAME) standard components % composition of the *jatropha* oil biodiesel in terms of the fatty acid profile.

Table 1: ***Jatropha*** **oil biodiesel fatty acid profile (ppm) with corresponding % concentration**

Component	Retention time	Area	Height	External	% concentration
C_{12}	5.100	16379.841	303.839	2.920	5.720
C_{17}	10.070	7093.846	132.959	0.955	1.871
C_{14}	15.093	10170.810	190.425	5.043	9.879
C_{20}	20.310	11056.387	206.905	4.297	8.417
$C_{18:2}$	25.346	7836.259	146.816	18.01	35.281
$C_{18:3}$	30.073	3536.254	66.323	6.991	13.695
$C_{18:1}$	35.103	10246.983	191.342	6.660	13.046
$C_{16:2}$	40.060	5503.761	103.155	6.171	12.088
		71824.141		51.046	

The result revealed the prevalence of C_{18} fatty acid components in agreement with previous studies (Adama 2021; Mohammed-Dabo *et al.*, 2012; El-Sherbiny *et al.*, 2010). It can be observed that the C_{18} fatty acid is the most abundant component that can impact heavily on the oxidative stability and corrosivity of the biodiesel.

Table 2 presents a comparative analysis of the physicochemical properties of the produced biodiesel with similar studies and with different standards.

Table 2: Comparative analysis of physicochemical properties of investigated jatropha oil biodiesel and similar studies with international standards.

Investigated Fuel properties	This work	Rabe 2010	Mohammed-Dabo *et al.*, 2012	Akhabue and Nduka 2016	Adama 2021	ASTM D6751	EN 14214
Density@40 ℃ (Kg/m^3)	847	879.5	869	874	857	880	860-900
Acid value (mg KOH/g)	0.670	39.23	0.75	0.4165	0.693	Max. 0.80	Max. 0.80
Viscosity@ 40 ℃ (mm^2/s)	7.001	4.8	4.6	1.904	1.24	1.9-6.0	3.5-5.0
Iodine value (g/100g oil)	68.53	104	103.2	98.525	76.14	Max. 120	Max. 120
Flash point	115	135	-	136	125	Min. 100-170	>120
Cloud point	-5	2.7	-	-	-2	-3 to -12	-
Pour point	-1	0.4	-	-	0	-15 to +16	-

The results obtained in Table 2 were in agreement with earlier studies conducted (Adama, 2021; Akhabue and Nduka, 2016; Mohammed-Dabo *et al.*, 2012; Rabe, 2010). The result of the viscosity of the produced biodiesel compared favorably with those obtained by Sharma and Singh (2009) (5.65 mm^2/s); Rabe (2010); Mohammed *et al.*, (2012); Akhabue and Nduka (2016) and Adama (2021). The result of the flash point analysis shows that the produced biodiesel is extremely safe to handle and use. Agarwal (2006) presented a flash point of 191 ℃ while Sharma and Singh (2009) reported a flash point of 170 ℃ for *jatropha* oil biodiesel.

The results of the fatty acid composition of the *jatropha* oil biodiesel in comparison to similar studies is presented in Table 3.

Table 3: Percentage fatty acid composition of investigated jatropha oil biodiesel in comparism to different studies.

Fatty acid	This study	El-Sherbiny *et al.*, 2010	Mohammed-Dabo *et al.*, 2012	Adama 2021
$C_{12:0}$	5.720	-	0.12	0.12
$C_{14:0}$	9.879	-	2.83	0.17
$C_{16:1}$	-	-	-	2.83
$C_{16:0}$	-	18.22	20.01	20.19
$C_{16:2}$	12.088	-	-	-
$C_{17:0}$	1.871	-	-	-
$C_{18:0}$	-	5.14	11.51	11.51
$C_{18:1}$	13.046	28.46	17.86	17.86
$C_{18:1}$, OH		-	0.72	0.72
$C_{18:2}$	35.281	48.18	44.95	45.30
$C_{18:3}$	13.695	-	0.18	-
$C_{20:0}$	8.417	-	0.79	0.79
Others	-	-	1.03	0.5

High level of similarity in terms of property investigations were exhibited as shown in the table.

3.2. Weight Loss Analysis

The corrosion rate (in mpy) experienced by the C1020 carbon steel after different periods of exposure in the *jatropha* oil biodiesel is provided in Figure 1.

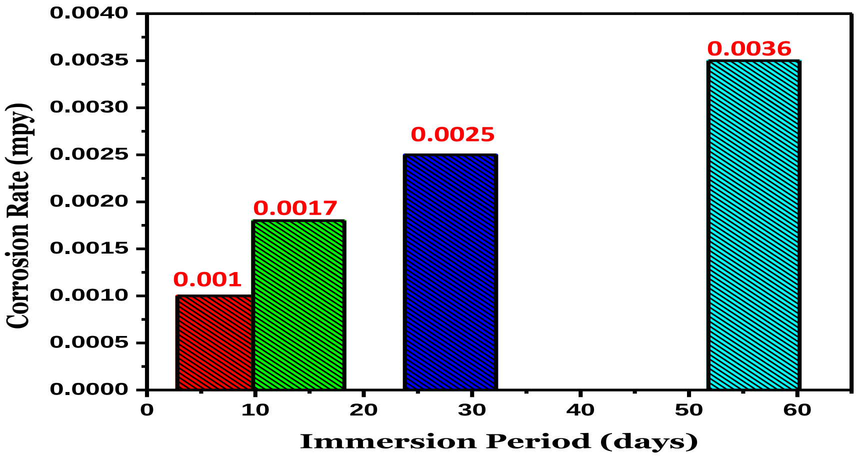

Figure 1: Corrosion rate experienced by C1020 carbon steel during 7, 14, 28 and 56 days immersion in *jatropha* oil biodiesel at ambient temperature.

Careful observation from Figure 1 shows that the steel corrosion rate increased with the immersion time. The loss of weight experienced by the alloy is attributed to the oxidation of its iron (Fe) atoms, during which the Fe atoms are transformed into Fe^{2+}ions at anodic sites on the steel surface, according to the reaction in Equation 3.

$$Fe = Fe^{2+} + 2e \quad (3)$$

This transformation leads to subtle detachment of the alloy elements and leads to the reduction in weight. This oxidation, and subsequent low in weight, is triggered by the adsorption of the organic components in the biodiesel which change the electrode potential of the steel and polarizes its active sites (David *et al.*, 2019). The loss in weight increases with immersion time, and implies that the steel surface is unable to form any surface corrosion product which can protect its surface from further attack by the biodiesel components (Fardilah *et al.*, 2022). These biodiesel components are, undoubtedly, the organic acids which were detected by GC-MS characterization. They can exist as negatively charged radicals which become attracted to the steel surface via electrostatic attraction to the Fe^{2+} cations generated due to dissolution. It is possible that these organic species could react with the

Fe^{2+} ions on the steel surface in order to form some complex products that can also be adsorb on the steel surface (Kugelmeier *et al.*, 2021; Hoang and Pham 2019). However, these products could be porous so it is impossible for them to block the active sites on steel surface from being continuously dissolved over time of exposure. Otherwise, it is possible that the complex products formed on the steel surface further activate the active sites on the steel surface to promote more Fe^{2+} release from the steel in order to continuously react with the organic species. This is why the corrosion rate of the steel increases with time. This finding is consistent with other observations in literature (Fazal *et al.*, 2019; Jin *et al.*, 2015).

3.3. UV-Vis, FTIR and Surface Morphology Characterizations

After 28 and 56 days of immersing the steel coupons in the *jatropha* oil biodiesel, the solution was analyzed with a UV-Vis spectrophotometer to unravel any chemical interaction between the biodiesel components and the steel surface, as shown in Figure 2.

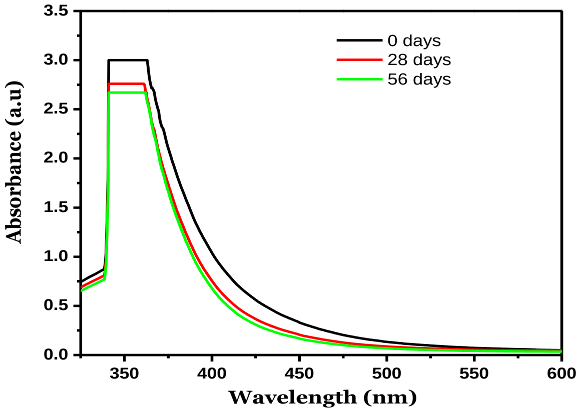

Figure 2: UV-Vis spectra of *jatropha* oil biodiesel before and after 28, 56 days immersion of C1020 carbon steel.

Figure 2 shows the UV-Vis spectral before immersion (0 day) and after 28 and 56 days of immersion in the *jatropha* oil biodiesel. The most visible peak is observed around 352 nm and is consistent with the signals due to poly-unsaturated components of the biodiesel. Such poly-unsaturated compounds include linoleic acid methyl ester containing C_{18} (Fardilah *et al.*, 2022; Chuck *et al.*, 2010). This results agrees with the GC-MS results which shows that the C_{18} are the most abundant components of jatropha oil biodiesel. It implies that the linoleic acid is majorly responsible for attacking the steel surface and inducing the biodiesel corrosion. Furthermore, the intensity of the C_{18} peak decreases over the time of immersion, as shown in Figure 2. This indicates that the components are continuously depleted in the biodiesel solution as a result of their adsorption on the steel surface (Kugelmeier *et al.*, 2021; Hoang and Pham, 2019; Fazal *et al.*, 2019). Furthermore, no new peaks are formed in the UV-Vis spectrum, which indicates that no chemical reaction occurs between the Fe atoms and the organic acid. This inferred that the organic acid was only adsorbed physically on the active sites.

The FTIR spectra in Figure 3 shows the profile of pure *jatropha* oil biodiesel and the corrosion product layer formed on the steel surface after 56 days of immersion in the biodiesel. The peak at 1739.2 cm^{-1} indicate the presence of C=O that is present in the triglycerides. The peak signal at 2925.1 cm^{-1} is attributed to anti-symmetric and symmetric stretching vibrations of C-H in CH_2 and CH_3 (Vinoth *et al.*, 2019; Rocabruno-Valdes *et al.*, 2018). The peaks at 1166.1 cm^{-1} indicate the stretching vibration of C-O ester. These peaks in the pure jatropha oil biodiesel are also present for the corrosion product scale formed on the steel surface after the longest immersion time (56 days). This indicates interaction between biodiesel with the steel surface based on these functional groups (especially the C-O and C=O groups) (Alves *et al.*, 2019). This finding support the results obtained from UV-Vis analysis and confirms that the biodiesel attacks the steel surface using its organic acids (most possibly the linoleic acids).

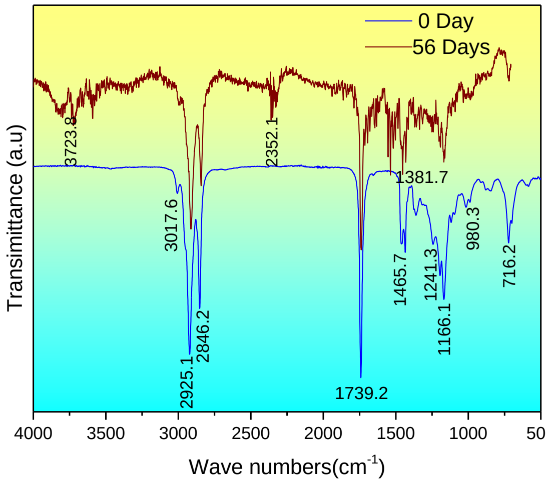

Figure 3: FTIR spectrum of jatropha oil biodiesel before and after 56 days immersion of C1020 carbon steel.

The surface morphological characterization using high resolution optical camera is shown in Figure 4.

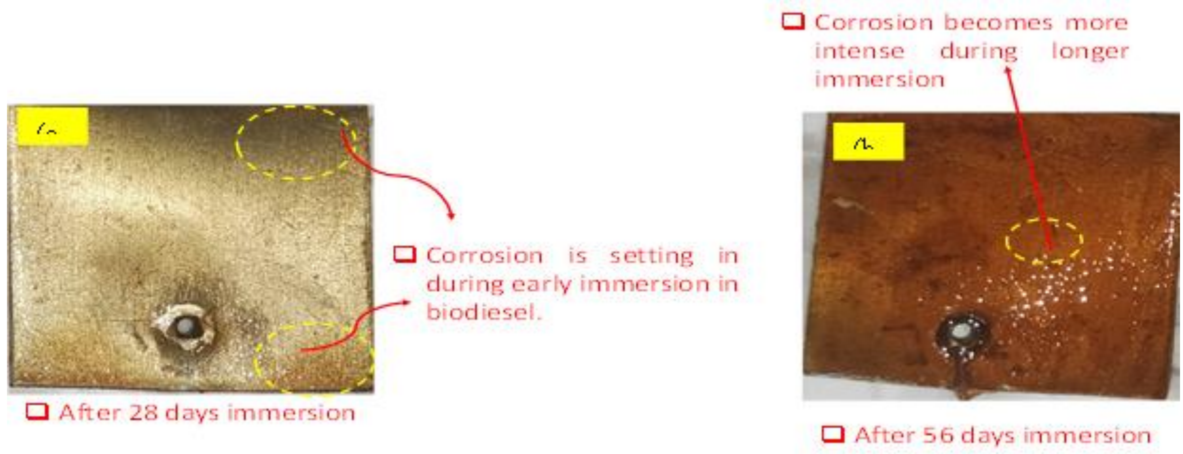

Figure :4: Optical camera images of steel coupon immersions in jatropha oil biodiesel after 28 and 56 days.

After 28 days of immersing the steel coupons in the jatropha oil biodiesel, the resultant surface feature is presented in Figure 4 (a). The regions where corrosion is initiated can be seen in the circled areas near the edges of the coupon. Some pits could also be seen on the coupons as indications of localized attack by the biodiesel components (Alves *et al.,* 2019). Pitting corrosion is usually attributed to local corrosion cells created by the scratches made on the sample during sample preparation by polishing (Fardilah *et al.*, 2022; Dharma *et al.,* 2019; Fazal *et al.*, 2019). Within these regions, it is believed that metal atoms possess higher energies that make them transport from the interior of the alloy matrix towards the alloy surface where they participate in the corrosion reactions (David *et al.*, 2019; Monteiro *et al.*, 2018). After 56 days Figure 4 (b), the surface of the steel has become highly corroded with somewhat brownish scale deposition on the steel surface. This implies that time increases the rate of attack of the steel surface by the biodiesel components (Chandran, 2020; Chourasia *et al.*, 2020; Alves *et al.*, 2019). From a another perspective, it could be assumed that time-lag also increases the extent of acidification within the biodiesel which causes more free fatty acids to be generated within the system and increase the extent of attack on the steel surface (Adama *et al.*, 2021; Adama, 2021). When the images in Figure 4 were compared, it can also be assumed that the attack on the steel surface by the biodiesel component seems uniform, since the entire steel surface is covered evenly with the corrosion product layer without patches (Ahmmad *et al.,* 2018). Meanwhile, the extent of localized pitting corrosion appears to be diminished, as a result of this uniform attack (Fardilah *et al.,* 2022; Akhabue and Nduka 2016). This result shows that over time, diesel engine parts made of steel would suffer corrosion-related surface degradation when biodiesel is incorporated in the fuel system and this would diminish the structural integrity of the steel material and also contaminate the prevailing fuel system.

CONCLUSION

Jatropha oil biodiesel fuel has been investigated via the process of production, characterization and analysis of its corrosive behaviour on C1020 carbon steel in solution for 7, 14, 28 and 56 days upon static immersion. The over-ridding fatty acid group influencing the corrosivity, oxidative instability and behaviour of the biodiesel was the C_{18} fatty acid group of molecules. The loss in weight increased with increase in immersion time which is attributed to the oxidation of the iron atoms at the anodic sites on the steel surface leading to subtle detachment of the alloy elements, hence reduction in weight. No chemical reaction occurred between the iron atoms in the steel and the organic molecules in the biodiesel indicating a physical adsorption on the steel surface. GCMS and UV-Vis corroborated this finding and further revealed that the attack on the steel surface which induced the corrosion was majorly from the C_{18} fatty acid groups identified via GCMS. UV-Vis analysis showed signals consistency with poly-unsaturated components of the biodiesel. Thus, the biodiesel interacted with the steel surface using C-O and C=O functional groups and also present in the corrosion scales formed on the steel surface after the longest immersion time which the FTIR analysis confirmed and in accordance with UV-Vis analysis. The rate or extent of attack on the steel surface by the biodiesel components were time-dependent. Hence, the extent of acidification within the biodiesel due to the time-lag caused more free fatty acids to be liberated in solution which further increased the extent of attack on the steel surface. This was corroborated by optical surface image analysis. The results of the investigations confirmed that over time, diesel engine components made from steel materials could suffer corrosion-related surface degradation when biodiesel is incorporated in the fuel system resulting in diminished structural integrity and contamination. However, organic, non-reactive eco-friendly inhibitors can be investigated and utilized in the mitigation of biodiesel-induced corrosion of jatropha oil biodiesel on the carbon steel.

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10.51975/24380104.som
Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

APPLICATION OF ARTIFICIAL NEURAL NETWORK (ANN) AND SENSITIVITY ANALYSIS TO ENHANCE BIOETHANOL YIELD IN A BUTADIENE PLANT

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ABSTRACT

The paper discusses the uses of bioethanol as a consumer product and its potential in butadiene production for vehicle tire manufacturing. The primary aim of this research is to improve bioethanol production by using sensitivity analysis in the extractive distillation process of a butadiene plant. The study specifically focuses on optimising essential variables in this process, such as the molar reflux ratio of the azeotropic column and the number of stages in both the azeotropic and extractive distillation columns using Aspen Plus V11. Given the complexity of bioethanol production through extractive distillation, precise modelling is crucial for increasing efficiency and yield. To address this challenge, the study introduces Artificial Neural Networks as a modelling approach. The optimised values for variables 1, 2, and 3 within their respective ranges were determined to be 4.62709, 17, and 20, respectively, using a simulated butadiene plant. The results obtained from training the ANN model showed an R-value of 0.99998 and a Mean Square Error of 2.25E-09, which is close to zero. This indicates that the model accurately represents the process. The effectiveness of the ANN approach in precisely predicting key process variables and optimising extractive distillation for bioethanol production was demonstrated.

Keywords: *Bioethanol, butadiene plant, sensitivity analysis, ANN, ethanol recovery.*

1.0 INTRODUCTION

Since the late 1990s, the rising demand for elastomers and polymers in Asia has resulted in an increased need for butadiene. China, being the largest producer and consumer of butadiene, primarily obtains it through naphtha cracking (Omofoyewa, 2023). Other production routes from hydrocarbons have been explored over the years, however, because of growing worries about the environment and economic considerations, there has been an increased emphasis on exploring alternative approaches to produce 1, 3-butadiene from sustainable sources (La-Salvia *et al.*, 2015).

Butadiene is a four-carbon element (C_4) necessary for the manufacture of rubber, mainly used in the making of tires for cars and Sports Utility Vehicles (SUVs). 1, 3-Butadiene ($H_2C=CH-CH=CH_2$) is a straightforward yet significant conjugated diene, which is a colourless and flammable gas at normal room temperature, accompanied by a sharp, fragrant smell (Omofoyewa, 2023). Butadiene, which is also known as C_4H_6, has a boiling point ranging from 23 to 24.8 °F (-5 to -4 ℃). While it is insoluble in water, it has a slight solubility in methanol and ethanol. It plays a vital role in the manufacturing of several elastomers, including acrylonitrile-butadiene-styrene, styrene-butadiene rubber, polybutadiene and diverse arrays (Ouhadi *et al.*, 2014) and is also used in the production of various synthetic rubbers, including nitrile-butadiene rubber (NBR), polybutadiene (PB) and styrene-butadiene rubber (SBR), which find widespread use in tire manufacturing. Since 2013, Genomatica and Braskem, a prominent butadiene producer situated in Sao Paulo, Brazil, have been working together to investigate the potential of manufacturing bio-based butadiene (Burk, 2016). One of which this way, is the production of butadiene using bioethanol as the feedstock.

In the last few decades, the significant focus of the scientific community has been on generating renewable biofuels from sustainable sources as they have the potential to provide energy, substitute fossil fuels and even serve as raw materials for some producer-goods. Among these biofuels, bioethanol stands out as particularly intriguing due to its versatile usage and its beneficial effects on the environment (Busic *et al.*, 2018). Bioethanol is mainly produced by yeast fermentation of carbohydrate sources (i.e., biomass) which could be rich in sugar, or starch and are lignocellulosic in nature (Mussato *et al.*, 2010).

Numerous physical factors like temperature, sugar concentration, pH, fermentation time, agitation rate, inoculum size etc affect bioethanol production (Zabed *et al.*, 2014). However, the ethanol recovery from the fermentation broth is a major parameter of concern as this in turn influences the butadiene production. Various techniques are employed for pure ethanol recovery, but the conventional techniques include azeotropic distillation, liquid-liquid extraction, and extractive distillation (Nitsche and Gbadamosi, 2017). Large-scale operations primarily utilize extractive distillation. The effectiveness of these techniques is also influenced by operational parameters.

This study utilizes sweet sorghum plant biomass as a raw material for the manufacturing of butadiene. Sweet sorghum is a crop that is known for its high biomass and sugar yield. The juice extracted from the stalks of sweet sorghum possesses a significant quantity of fermentable sugars, such as glucose, sucrose, and fructose. The high biomass and sugar yield of sweet sorghum as a feedstock render it an appealing option for bioprocessing applications, including the manufacturing of butadiene (Wu *et al.*, 2010). The juice is then fermented to produce bioethanol. Amidst several methods of producing butadiene, the production of butadiene from bioethanol is a successful and effective substitute that has been adopted in the industry before the advancement of naphtha cracking techniques. Processes for the conversion of ethanol to butadiene have been employed since 1920 and served as the primary production method until the conclusion of World War II (Chieregato *et al.*, 2016; Sun *et al.*, 2015).

To produce butadiene, the two-step production process (Ostromisslenski process) was considered. The overall process is divided into four distinct parts namely the extraction process, fermentation process, distillation process and butadiene separation process. Bioethanol has emerged as a sustainable alternative to fossil fuels, contributing to the reduction of greenhouse gas emissions and dependence on non-renewable resources. The extractive distillation process offers an effective means of bioethanol production by separating azeotropic mixtures. Accurate modelling of the extractive distillation process is essential for achieving optimal production efficiency and yield. Traditional modelling approaches often struggle to capture the intricate non-linear relationships present in the process. Artificial Neural Networks (ANNs) have shown promise in modelling complex systems by mimicking the human brain's ability to learn and adapt (Rathi *et al.*, 2023).

By studying the sensitivity analysis on some operational parameters like the temperature of the feedstock, number of distillation stages, and molar reflux ratio of the distillation column in a butadiene plant, this paper seeks to enhance the bioethanol recovery from the extractive distillation section. This study also explores the application of ANNs in modelling the extractive distillation process for bioethanol production.

2.0 METHODOLOGY

2.1 Process Simulation and Thermodynamic Model

The ethanol plant's process simulation was carried out using Aspen Plus V11 software with the non-random two-liquid (NRTL) thermodynamic model for determining activity coefficients in the liquid phase. Ideal behaviour assumptions were made for the vapour phase to define thermophysical properties (Omofoyewa *et al.*, 2023).

2.2 Process Description

The ethanol production process from sweet sorghum within a butadiene plant involves several distinct stages. The overall process was divided into four sections: juice extraction, fermentation, ethanol dehydration, and butadiene separation. The design flow diagram (Figure 1) illustrates these stages.

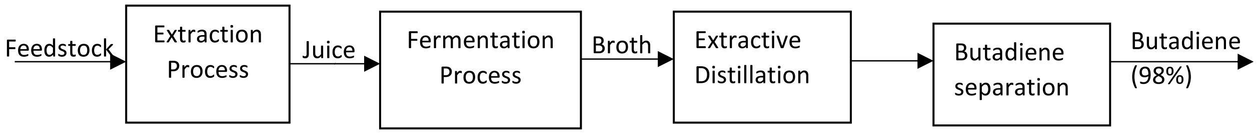


Figure 1: Overall Process Design Flow Diagram (Omofoyewa *et al.*, 2023)

Sweet sorghum juice was extracted to obtain its sugar content. Ethyl acetate was used as the solvent for extraction. Operating conditions and feedstock composition were defined (Table 1). The fermentation process involved the continuous bioconversion of sugars from the sweet sorghum juice into ethanol. Simplified stoichiometric equations (1) and (2) were used, assuming the juice's glucose and sucrose content. Ethanol concentration ranged from 5 to 12 wt%. Ethanol separation from the fermentation broth was achieved through extractive distillation using ethylene glycol as a solvent. This choice of solvent modified the relative volatility of the components for effective separation. Multiple fractional distillation columns were utilized to enhance ethanol purity.

Table 1: Feedstock Composition and Operating Conditions (Omofoyewa *et al.,* 2023)

Component Name	**Value**
Mass Fraction:	
Sucrose	0.074419
Glucose (as Dextrose)	0.45515
Fructose	0.470432
Vapour/Phase Fraction	0.00
Temperature (ºC)	25
Pressure (bar)	1
Mass Flow (kg/hr)	125000

$$C_{12}H_{22}O_{11} + H_2O \rightarrow 4C_2H_5OH + 4CO_2 \quad (1)$$

$$C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2 \quad (2)$$

Overall process flowsheet

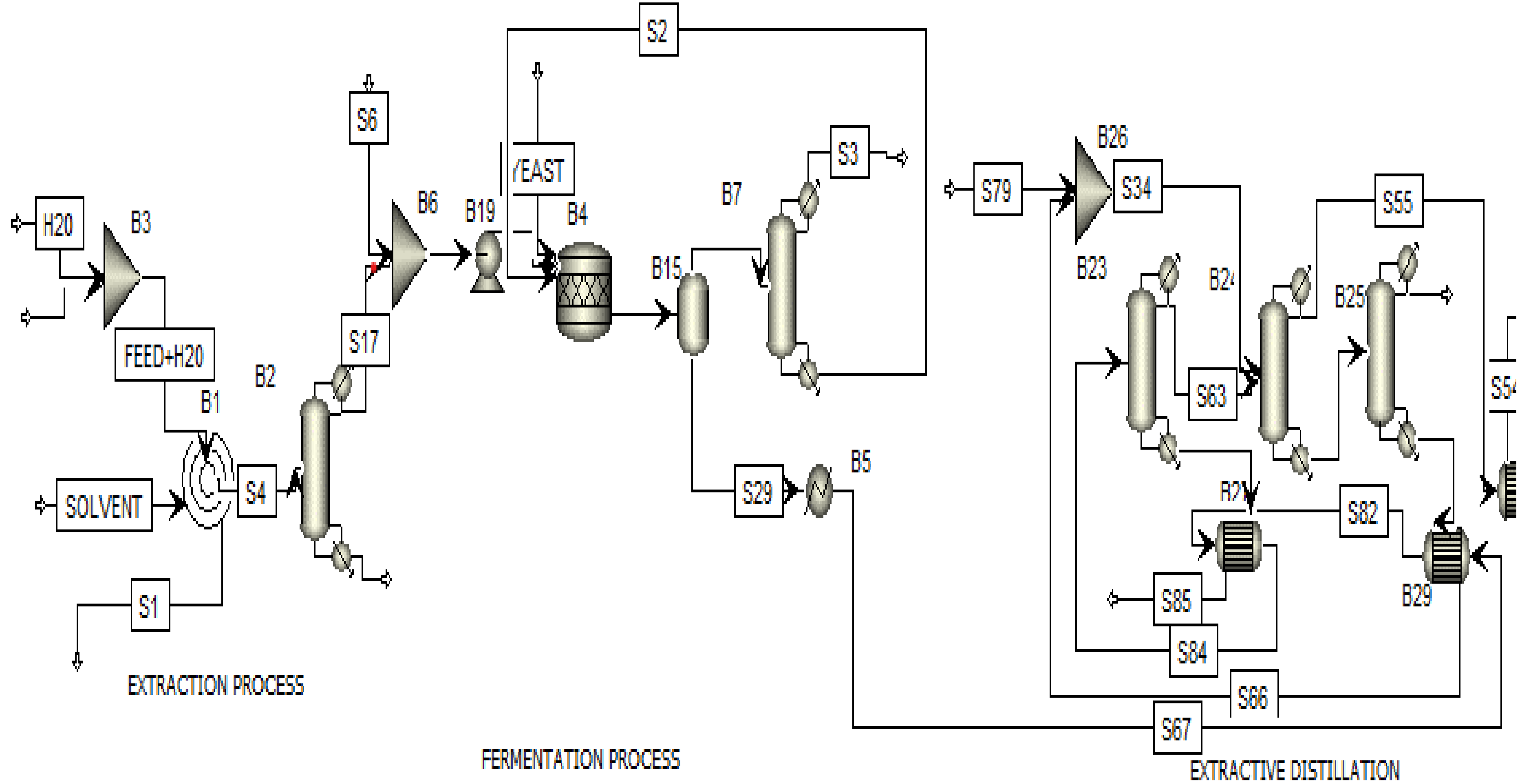


Figure 2: Process Flow Sheet for the Production of Bioethanol from Sweet Sorghum in a Butadiene Plant (Omofoyewa *et al*., 2023)

2.3 Sensitivity Analysis Study

For this analysis, a nested sensitivity analysis was carried out on five process variables that the system (butadiene plant) was sensitive to. The study was to see the effect of the variation on the ethanol recovery from the extractive distillation process. The parameters chosen (manipulated variables) include:

- Temperature of feed
- Vapour fraction of feed
- Molar reflux ratio of the azeotropic and extractive distillation column
- Number of stages of the azeotropic and extractive distillation column

After determining the variables to which the system was sensitive, such as the molar reflux ratio of the azeotropic distillation column (variable 1), the number of stages of the azeotropic distillation column (variable 2), and the number of stages of the extractive distillation column (variable 3), a nested sensitivity analysis was carried out to see the combined effect of varying these parameters to achieve optimum ethanol recovery. The results are discussed in the results section. The sensitivity analysis tool of Aspen Plus V11 was used to achieve this feat.

2.4 Artificial Neural Network

The tool used for the artificial neural network modelling of the extractive distillation process for the production of bioethanol in a butadiene plant was MATLAB R2022b. However, the data used for the modelling were generated via Aspen Plus V11. Sensitivity analysis demonstrated which input parameters have the most significant impact on the output variables, aiding in process optimization. Simulated data from the extractive distillation process, including the molar reflux ratio of the azeotropic distillation column (variable 1), number of stages of the azeotropic distillation column (variable 2), and number of stages of the extractive distillation column (variable 3), are collected and pre-processed for training and validation. A two-layer feedforward neural network with sigmoid hidden neurons and linear output neurons architecture was selected as shown in Figure 3. The input layer consists of process parameters, while the output layer represents target variables such as bioethanol recovery.

The dataset is divided into training and validation sets. Levenberg-Marquardt Back propagation algorithms were employed to adjust the weights and biases of the network to minimize the prediction error. The trained ANN model is evaluated using various metrics, including Mean Squared Error (MSE) and R-squared values.

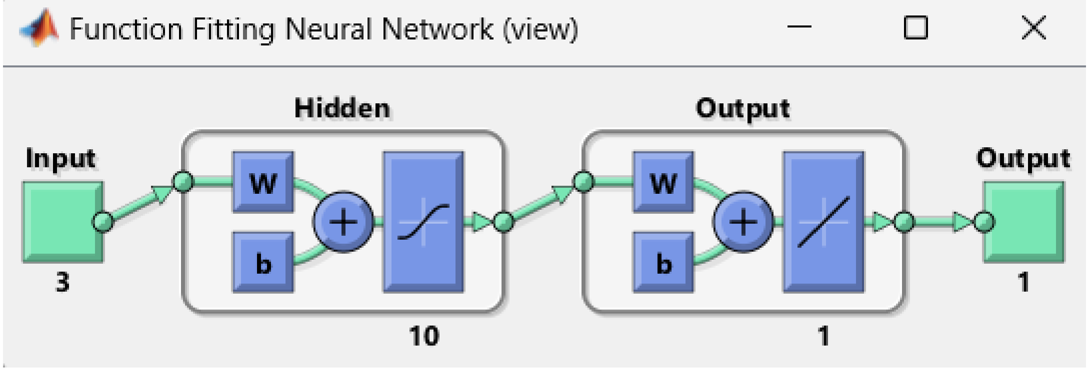


Figure 3: Feed Forward Neural Network of the Process

3.0 RESULTS AND DISCUSSION

3.1 Sensitivity Analysis and Optimization

Sensitivity Analysis was carried out in the process in a nested fashion. The parameters considered as the manipulated variables include the molar reflux ratio of the azeotropic distillation column (variable 1), the number of stages of the azeotropic distillation column (variable 2), and the number of stages of the extractive distillation column (variable 3). The range of inputs for the manipulated variables is shown in Table 2.

Table 2: Range of Inputs for Manipulated Variables

Variable	Start point	Endpoint
1	0.8	6
2	20	30
3	14	17

Figure 4 shows the output of the graphs that were optioned with variable 2 as the parametric variable.

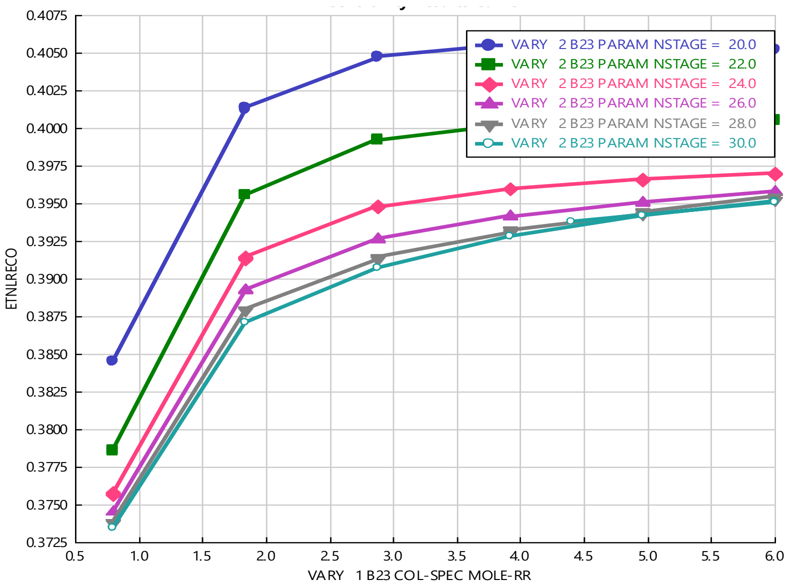
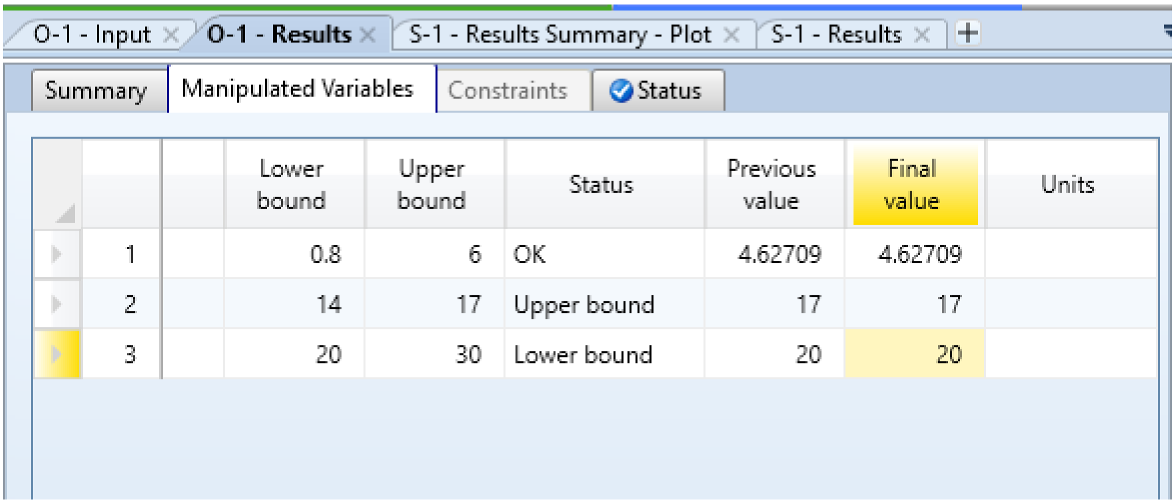


Figure 4: Graph showing the relationship between the number of stages and the reflux ratio

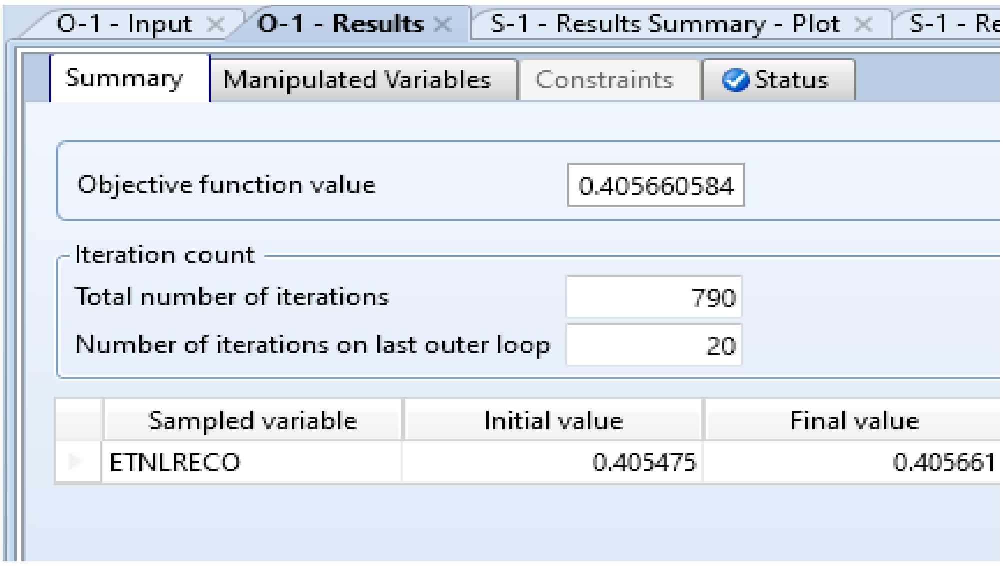
Figure 4 shows generally an increased ethanol recovery as the molar reflux ratio of the azeotropic distillation column with a reduced number of stages in that same distillation column. A better ethanol recovery of about 0.4055 was achieved with a minimal number of stages at 20 stages for the azeotropic distillation column (variable 2) with the maximum reflux ratio of the azeotropic distillation column (variable 1) at 6.

The optimization was carried out using the optimization tool on Aspen Plus V11 to see how much ethanol recovery could be enhanced (maximized) within the specified inputs for the manipulated variables used for the sensitivity analysis. Figures 4 and 5 show the optimized value of these parameters alongside the maximized ethanol recovery within the ranges of the manipulated variable.



		Lower bound	Upper bound	Status	Previous value	Final value	Units
1		0.8	6	OK	4.62709	4.62709	
2		14	17	Upper bound	17	17	
3		20	30	Lower bound	20	20	

Figure 5: Optimized Values of the Chosen Variables



Objective function value: 0.405660584

Iteration count
Total number of iterations: 790
Number of iterations on last outer loop: 20

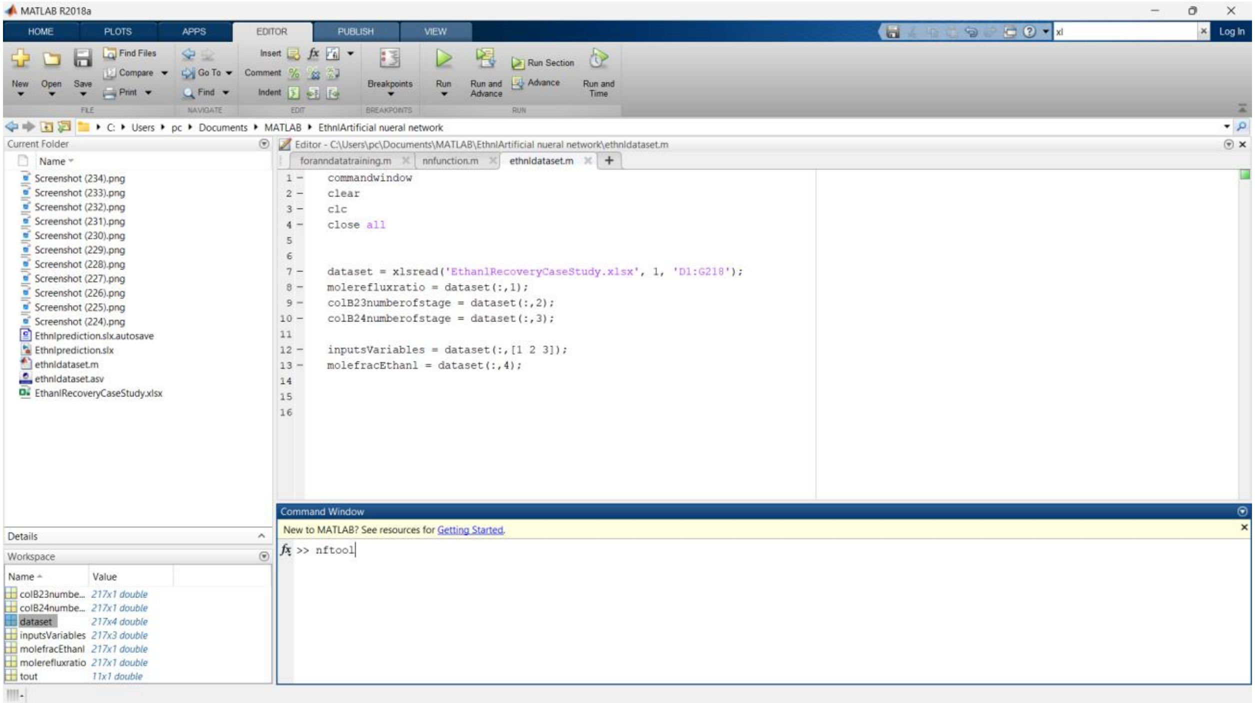
Sampled variable	Initial value	Final value
ETNLRECO	0.405475	0.405661

Figure 6: Obtainable Optimal Value of Ethanol Recovery

Figure 5 shows that the optimum values of the variables 1, 2 and 3 within the varied ranges were 4.62709, 17 and 20 respectively, while Figure 6 shows that the ethanol recovery was able to be optimized from a production rate of 0.405475 to 0.405661. While practically this may show as an infinitesimal increase, this goes to show that the chosen operational parameters within the chosen range may not pose any significant changes. While working out these ranges may not be economically feasible, other physical factors like temperature, sugar concentration, pH, fermentation time, agitation rate, inoculum size etc could be explored to see how this might further enhance the ethanol recovery (De Silva *et al*., 2022).

3.2 ANN

The results indicate that the ANN model outperforms conventional modelling techniques in capturing the complex relationships within the extractive distillation process. The ANN's ability to handle non-linear interactions and adapt to changing conditions allows for more accurate predictions of key process variables (Shen, 2018). Figure 7 shows the MATLAB mfile code for the process.



```
commandwindow
clear
clc
close all


dataset = xlsread('EthanlRecoveryCaseStudy.xlsx', 1, 'D1:G218');
molerefluxratio = dataset(:,1);
colB23numberofstage = dataset(:,2);
colB24numberofstage = dataset(:,3);

inputsVariables = dataset(:,[1 2 3]);
molefracEthanl = dataset(:,4);
```

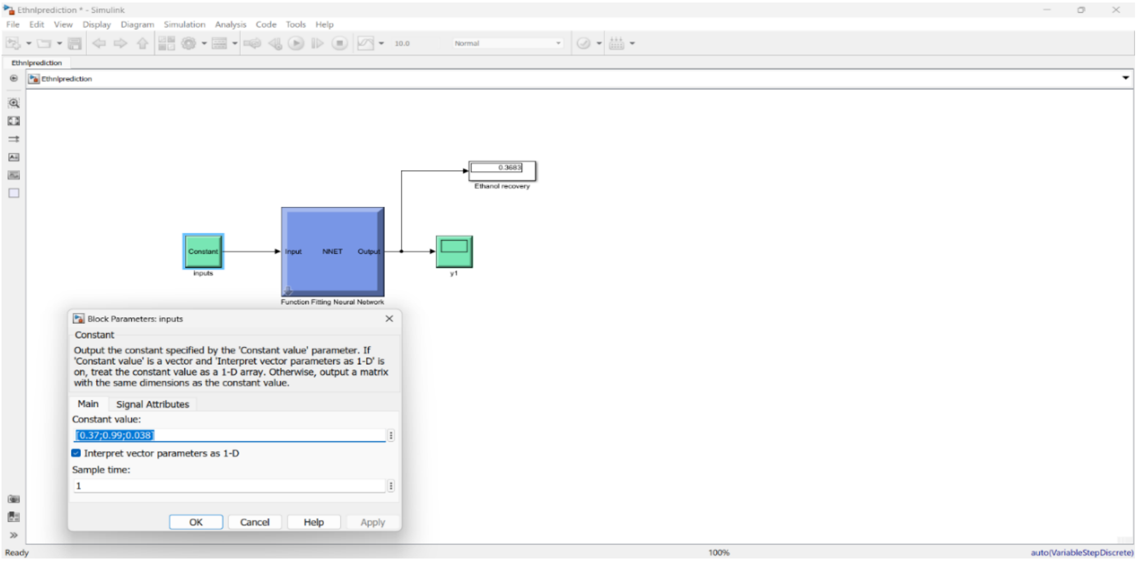
New to MATLAB? See resources for Getting Started.

```
>> nftool
```

Workspace

Name	Value
colB23numbe...	217x1 double
colB24numbe...	217x1 double
dataset	217x4 double
inputsVariables	217x3 double
molefracEthanl	217x1 double
molerefluxratio	217x1 double
tout	11x1 double

Figure 7: MATLAB *Mfile* Code for the Process



Block Parameters: inputs

Constant

Output the constant specified by the 'Constant value' parameter. If 'Constant value' is a vector and 'Interpret vector parameters as 1-D' is on, treat the constant value as a 1-D array. Otherwise, output a matrix with the same dimensions as the constant value.

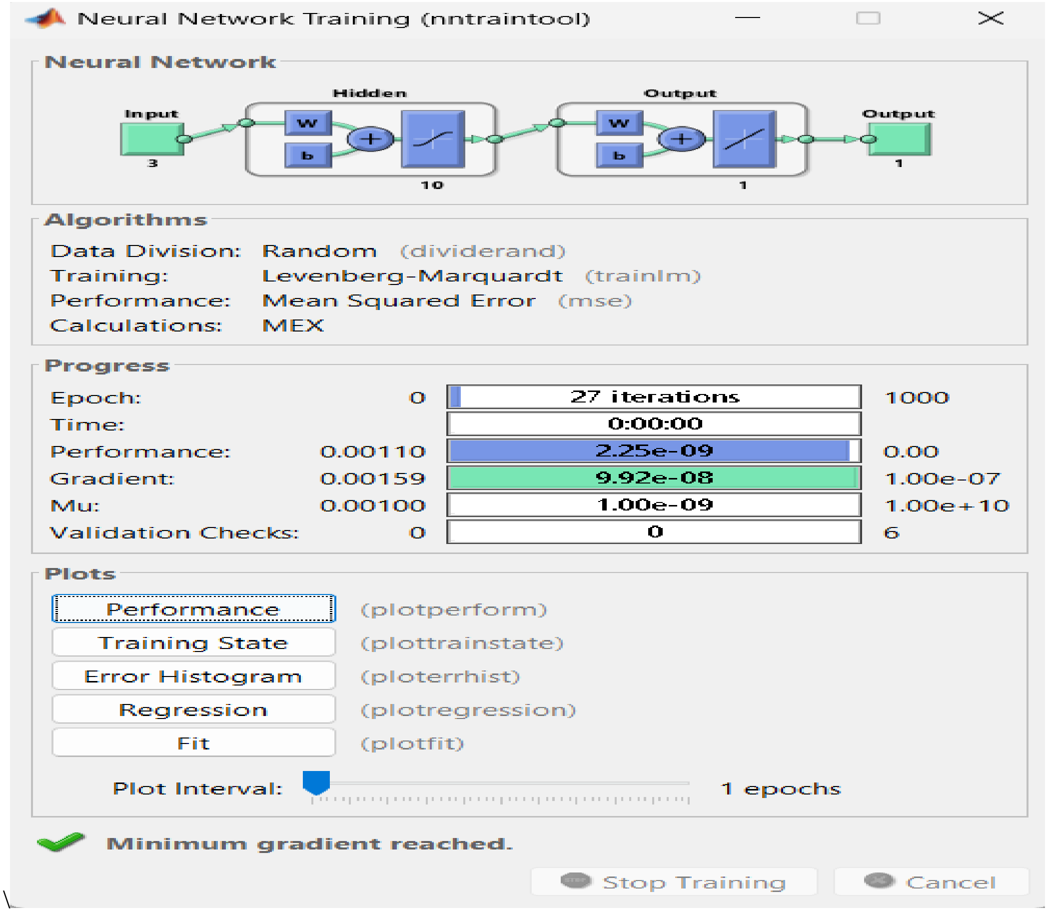
Main | Signal Attributes

Constant value: [0.37;0.99;0.038]

☑ Interpret vector parameters as 1-D

Sample time: 1

Figure 8: Simulink Result of the Process



Algorithms

Data Division: Random (dividerand)
Training: Levenberg-Marquardt (trainlm)
Performance: Mean Squared Error (mse)
Calculations: MEX

Progress

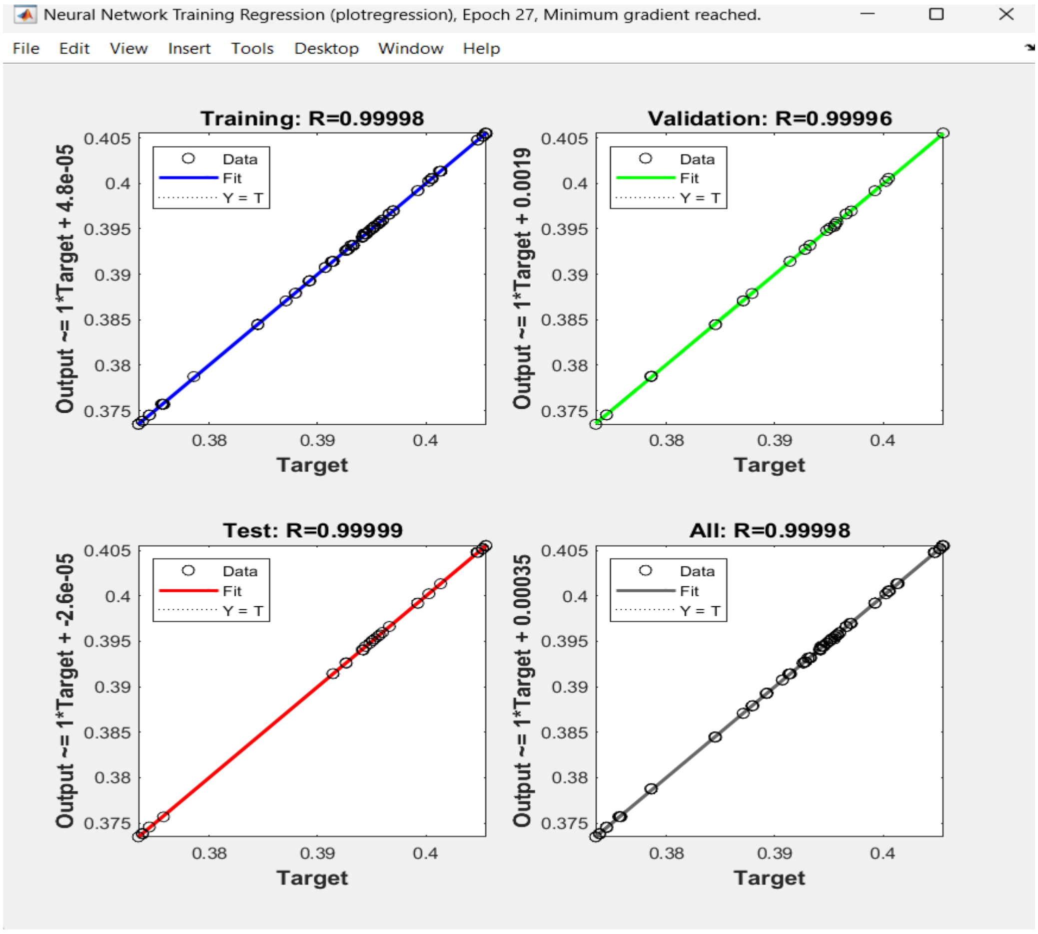
Epoch:	0	27 iterations	1000
Time:		0:00:00	
Performance:	0.00110	2.25e-09	0.00
Gradient:	0.00159	9.92e-08	1.00e-07
Mu:	0.00100	1.00e-09	1.00e+10
Validation Checks:	0	0	6

Plots

Plot Interval: 1 epochs

✓ Minimum gradient reached.

Figure 9: Neural Network Result



Neural Network Training Regression (plotregression), Epoch 27, Minimum gradient reached.

Figure 10: R-Value of the ANN Training

The developed ANN model can be integrated into process control systems to optimize bioethanol production. It provides real-time predictions and recommendations for adjusting process parameters to achieve desired product specifications. Moreover, the model can serve as a valuable tool for process engineers in designing new bioethanol production facilities and conducting feasibility studies.

5.0 CONCLUSION

In summary, this study focused on increasing bioethanol production by applying sensitivity analysis and a neural network model within the extractive distillation unit of a butadiene plant. By conducting a meticulous sensitivity analysis of three critical variables, namely the molar reflux ratio of the azeotropic distillation column (variable 1), the number of stages of the azeotropic distillation column (variable 2), and the number of stages of the extractive distillation column (variable 3), insight was obtained into the potential avenues for enhancing the bioethanol recovery. The results obtained from the sensitivity analysis led to the determination of the optimized values for the respective variables within their specified ranges. These optimized values, 4.62709 for variable 1, 17 for variable 2, and 20 for variable 3, signify the points at which the bioethanol yield can be maximized in the simulated butadiene plant. An infinitesimal increase of the bioethanol recovery from 0.405475 to 0.405661 was obtained. While this may not signify realistic changes, this goes to show the possibility of bioethanol recovery either outside the specified ranges or exploring other physical factors. The results also highlight the ANN's capability to accurately predict key process variables and optimize production efficiency. The application of ANNs in bioethanol production showcases the potential of machine learning techniques in enhancing sustainable energy processes.

ACKNOWLEDGEMENTS

Special thanks go to Aare Afe Babalola, LL.B, FFPA, FNIALS, FCIArb, LL.D, SAN, OFR, CON – The Founder and President, and the Management of Afe Babalola University, Ado-Ekiti, Ekiti State, Nigeria for providing a very conducive environment and the

necessary materials that enabled the accomplishment of this research work.

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OPTIMIZATION OF CATALYTICALLY INDUCED BIO-GASIFICATION OF ORGANIC WASTE USING A CYLINDRICAL BATCH DIGESTER

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ABSTRACT

This study was carried out to investigate the production capacity of biogas from mesophilic anaerobic digestion of cow dung (CD) with activated carbon as a catalyst. Laboratory scale digesters were constructed and set-up to digest cow dung. The experiment was a batch process and gas volume from the setup was collected every 5 days for 35 days. Total biogas volume obtained at the elapse of 35 days was 551 mL. Retention time of 15 days gave the maximum biogas volume of 170 mL, at pH 7 and catalyst dose of 3 g. The main effect plots demonstrated that each independent variable contributed significantly in biogas volume generation. The volume of biogas generated from the optimum predicted analysis was 188 mL at process conditions of pH 7.2, catalyst dose of 3.5 g and time of 15.5 days. The verification experiments agreed with the predicted values with standard error value of 1.06%. Overlay contour plot established optimum areas where the predicted response variable is in an acceptable range (≥ 80%) with respect to optimum conditions. Face-centred central composite design (FCCD) approach was suitable for optimizing the process giving higher biogas volume when compared to the main effect plots.

***KEYWORDS**: Biogas; anaerobic digestion; cow-dung; catalyst; response surface methodology.*

1. INTRODUCTION

Nigeria is a country with great fossil fuel deposits, but unfortunately, the supply chain of the end product after refining is fragmented. Presently, Nigeria is not only faced with the challenges of pollution and depletion of fossil fuels, but also the high cost of energy usage is a menace ravaging its productivity. To this end, there is a need for an intense search for alternative, cleaner, renewable and sustainable energy sources, among which is biogas (Kana et al., 2012; Zareei & Khodaei, 2017).

Biogas is a mixture of colourless, flammable gases obtained by the anaerobic digestion of animal/plant-based organic waste, agricultural waste, and industrial and domestic waste materials (Abubakar et al., 2021; Dahunsi et al., 2017; Okoye et al., 2020; Omotioma et al., 2021). It is a combustible gas consisting of methane, carbon dioxide, and small traces of hydrogen sulphide, ammonia, nitrogen, and hydrogen (Abubakar et al., 2021). The composition of biogas mainly depends on feed materials and biogas is generally composed of 55-65% methane, 35-45% carbon dioxide, 0-3% nitrogen, 0-1% hydrogen, and 0-1% hydrogen sulphide (Chun, et al., 2015). Biogas is generated when bacteria degrade biological material in the absence of oxygen, in a process known as anaerobic digestion (Abubakar et al., 2021).

Anaerobic digestion is a biochemical process that happens naturally in which anaerobic bacteria decompose organic matter in environments with little or no oxygen and produce biogas (Møller et al., 2004). It is widely used for the treatment and energy recovery from many kinds of biomass, usually agricultural products and agro-based industrial wastes (Dahunsi et al., 2017; Ukpai and Nnabuchi, 2012). It is an efficient method that employs diverse microbial groups for waste conversion into biogas.

In Nigeria, there are abundant animal wastes such as cow dung, goat pellets, camel, sheep, donkey and horse wastes whose potential is yet to be fully tapped for energy generation (Igoni et al., 2007; Olurunnisola., 2007). Biogas produced from animal waste is widely used as a renewable bio-fuel source. This source of energy is regarded as cheap and clean and is also known to produce a residue with a high fertilizer value for crop production (Møller et al., 2004; Owen, 1979). For efficient biogas production, the most appropriate raw material must be selected, but the direct use of some substrates is hampered by nutritional imbalances, lack of diversified micro-organisms, and the effects of operational factors (Achinas and Euverink, 2019; Ali et al., 2016). Therefore, it is often necessary to pre-treat and re-use biomass, and co-digestion is recommended to overcome nutritional deficiencies in the residues so as to improve biogas production in digesters (Nielfa et al., 2015).

Cow manure, also known as cow dung, is *mostly digested grass* and, depending on the cattle's diet, grain, fruits, or vegetables. Cow dung is one of the most important sources of bio-fertilizer but at the same time cow's urine, cow's horn, and carcass of a cow can be used for preparing effective bio-fertilizer (Raj et al., 2014).
Response Surface Methodology (RSM) was used to develop a mathematical correlation between the operating variables affecting the responded variable(s). In alternative terms, it is a mathematical technique applied in the progression of an appropriate functional relationship between the response and the related input variables (Abubakar et al., 2021). It analyzes the effects of several independent variables on the responses (Al Imam et al., 2013). The main idea of RSM is to use a sequence of designed experiments to obtain an optimal response (Montgomery and Myers, 2002). The main advantage of RSM is the economy in the number of experimental trials needed to evaluate multiple parameters and their interactions (Chun, et al., 2015). RSM is important in process design and optimization, as well as for improving the performance of the system (Al Imam et al., 2013; Ifegbo et al., 2022). The technique is very popular in physical and chemical experimental designs and optimizations for experimental cost reduction. Central Composite Design (CCD) is one of the most utilized optimization tool for the development of analytical procedures. Face-centered central composite design (FCCD) is a tool implemented in the CCD. A CCD is made FCCD when the value of α is unity (Obiora-Okafo and Onukwuli, 2021). The face-centered option ensures that the axial runs are in the range of the factorial portion (Onukwuli, et al., 2018). The overlaid contour plot (OCP) is used to visually identify an area where the predicted means of the response variable is in an acceptable range. A compromise among the optimum conditions for response is desirable. The desirability function approach, together with graphical optimization, was used to achieve this goal (Moghaddam et al, 2010).

The aim of this research was to evaluate the biogas production potential of cow dung with the aid of a catalyst via anaerobic digestion. The optimization of the process parameters was carried out to establish a bench-mark for cow dung's usage as a bioenergy option.

2. MATERIALS AND METHODS

2.1. Samples Collection

Fresh cow dung (CD) of approximately 40 kg was collected in 20 L buckets from Amansea abattoir, Anambra State. The sample was scrapped off the feed lanes this was the inoculum used for the study. The sample was immediately transported to the Chemical Reaction Laboratory of the Chemical Engineering Department, Nnamdi Azikiwe University, Awka, Anambra State, for further analysis. 1 kg activated carbon (AC) precursor used as a catalyst was purchased from the Enugu urban market, and it was stored in an air-tight container in line with the procedure employed by Salam et al. (2015).

2.2. Preparation of the Slurry and Laboratory Sized Bio-Digester Setup

The CD was mechanically pre-treated by reducing the sizes to the range of 2.0 – 4.0 mm using standard laboratory sieves. This was achieved by breaking the semi-solid mass into smaller chunks for ease of slurry formation and solubilization. 500 g of the mechanically pre-treated CD was weighed out for each batch of the analysis and then mixed with 1500 mL of water (including 500 mL buffer solution) in a plastic bowl so that the ratio of dung to water is 1:3 (Ukpai and Nnabuchi, 2012). To each digester, appropriate mixture quantity was added to obtain the desired slurry mix. Each digester was thoroughly stirred by mechanical shaking, to ensure the formation of a homogeneous mixture. A schematic set-up diagram of the bio-digester is shown in Figure 1 (Salam et al., 2015). A PVC pipe from the digester lid was connected into an inverted 100 cm^3 measuring cylinder, filled with brine water in the water trough; such that the outlet was directed upward in the cylinder. Another plastic pipe was used to take the displaced water from the displacement tank to the water collector, which is fitted air tight in the displacement tank to the water collector and inserted up to bottom part of it. The gas produced in the digester passed through the pipe to the displacement tank. The experiment was carried out in batch laboratory scale reactors (digesters) with capacities of 2.5 L. Digestion were done at mesophilic temperatures of 27^oC - 35^oC for 35 days at various catalyst doses and varying slurry pH. During the investigation, the volume of gas produced was measured with the help of the water displacement method (Al Imam et al., 2013; Salam et al., 2015) considering that the volume of the produced biogas was equivalent to the displaced water in the water collector. 0.2 M of NaOH and HCl were used to adjust the pH of the Buffer solution using a Digital pH meter (HANNA model).

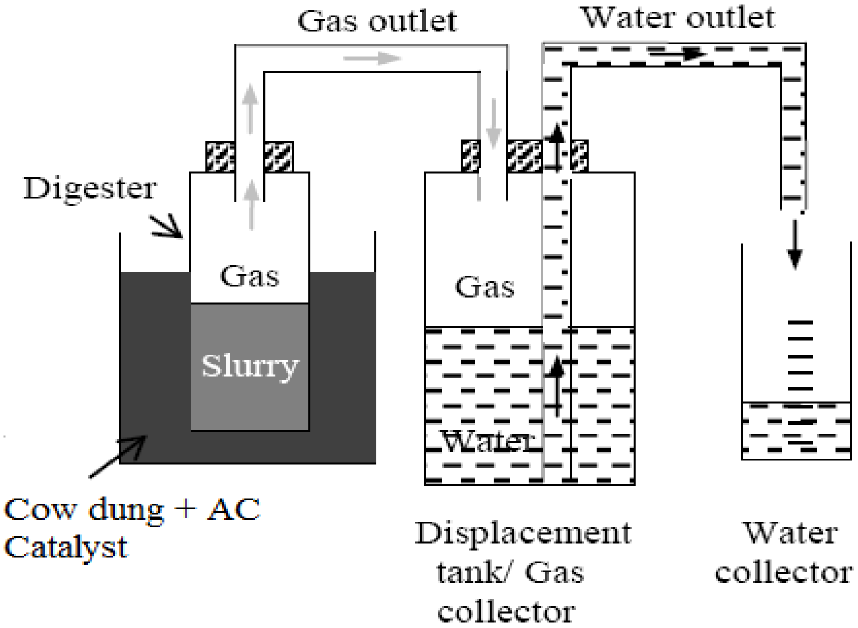

Figure 1: Schematic diagram of the experimental set-up for anaerobic digestion of cow dung (Salam et al., 2015).

2.3. Biogas Composition

Biogas compositions such as CH_4, CO_2, O_2, N_2, H_2 and H_2S were measured using a portable gas detector Geotechnical instrument (GA 45) (Vindis, et al., 2008). The measurements were done in duplicate, twice a week for each reactor. Biogas samples were taken using a 100 µL gas-tight syringe (Fisher Scientific, USA); from the reactors' head space after releasing the gas.

2.4. Design of Experiment and Optimization Studies

Minitab software, version 16, was used for the design of the experiment and optimization of variables: reaction pH, catalyst dose, and retention time for CD. Response Surface Methodology (RSM) was used to develop a mathematical correlation between the operating variables affecting biogas production (Montgomery and Myers, 2002; (Abubakar et al., 2021). It was the statistical method used for experimental modeling and analyzing the relationships between the input and response variables, while analysis of variance (ANOVA) was used for the analysis of regression coefficient, prediction equation, and case statistics. Main effects and 3D plots were obtained using the software to analyze the effects of variables individually and their interactions to determine their optimum level. The overlaid contour plot (OCP) was used to visually identify an area where the predicted means of the response variable is in an acceptable range specified.

Central composite design (CCD), a very efficient design tool for fitting second-order models, was used in the experimental design. The CCD was well suited for fitting quadratic surfaces, which usually works well for process optimization (Obiora-Okafo, et al., 2020). Face-centered central composite design (FCCD) was implemented in the CCD. A CCD was made face-centred by the choice of $\alpha = 1$ (Montgomery, 2001). FCCD is having the position of the star points at the face of the cube portion of the design (Montgomery and Myers, 2002). The face-centered option ensured that the axial runs would not be any more extreme than the factorial portion. The independent variables selected for this study were reaction pH (A), catalyst dose (B), and time (C). A 2^3 two-level factorial for three independent variables consisting of 8 cube factorial points coded to the usual ± notation, 6 axial points and 4 centre points in a cube and 2 centre points in axial were conducted, given a total of 20 experiments. Mathematically, Equation (1) was used to determine the total number of runs performed. The total number of experiments, N is:

$$N = 2^k + 2k + n \qquad (1)$$

Where k is the number of factors, and n is the centre points. The experimental design table is presented in Table 1. Minitab software version 16 was used to analyze the experimental data fitted to a second-order polynomial model to optimize the variables in the anaerobic process.

It also demonstrated the analysis of variance (ANOVA). The response was used to develop an empirical model that correlated the response to the variables using a second-degree polynomial equation as given by Equation (2):

$$Y = b_0 + \sum_{i=1}^{n} b_i X_i + \sum_{i=1}^{n} b_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} X_i X_j + \varepsilon \qquad (2)$$

Where Y is the predicted response, b_0 is the constant coefficient, b_i is the linear coefficient, b_{ii} is the quadratic coefficient, b_{ij} is the interaction coefficient, X_iX_j is the coded values of the variables, n is the number of independent test variables, and ε is the random error.

The adequacy of the proposed model was revealed using the diagnostic tool provided by analysis of variance (ANOVA). The quality of the model fit was expressed by the coefficient of determination (R^2). The R^2 value provides the extent of variability in the interaction between the response and the factors (Tengku et al., 2014). The analysis was done by means of Fisher's 'F' test and P-value (probability). Model terms were evaluated by the P-value using 95% confidence level (Montgomery and Myers, 2002). The optimum operating conditions of the biogas production were obtained by analyzing the main effects plots, 3D surface plots, contour plots, and overlaid contour plotting using the software.

Table 1: Levels and range of the variables tested in the CCD design

Variables	**Factors**	**Unit**	**Range and levels**				
			Lowest -α	**Low -1**	**Center 0**	**High +1**	**Highest +α**
pH	A	-	6.6	6.6	7.05	7.50	7.50
Catalyst Dose	B	(g)	1	1	3	3	3
Time	C	(days)	10	10	15	20	20

3. RESULTS AND DISCUSSION

3.1. Biogas Production

The volume of biogas and the cumulative biogas produced in the anaerobic OFAT process during 35 days are shown in Figure 2 and 3, respectively. In Figure 2, the maximum biogas produced was 170 mL and was achieved at 15 days of operation, at pH 7 and temperature 27 – 32°C. The cumulative biogas volume generated from the batch process was 551 mL as shown in Figure 3. From 0 – 10 days, 16.7% biogas was produced. Then, between 11 – 20 days, biogas production increased to about 55.2 %. Finally, from 21 – 35 days, biogas production reduced to about 27.9 % and ceases after 35 days. The production of biogas in the first five days for the cow dung was gradual. This can be drawn to the fact that most cows feed on fibrous materials and micro-organisms require a longer time to degrade fibrous materials. This is predicted because the biogas production rate in the batch condition is directly equal to specific growth of the methanogenic bacteria (Al Imam et al., 2013). The hydraulic retention time for the CD with catalyst is 35 days, and gas production measurement started on the 5th day with a biogas volume of 5 mL. This result and findings are similar to the works done by Al Iman et al. (2013) and Ozor et al., (2014). The result shows clearly that CD enhanced with AC catalyst is an effective feedstock for anaerobic digestion and could significantly enhance the cumulative biogas production.

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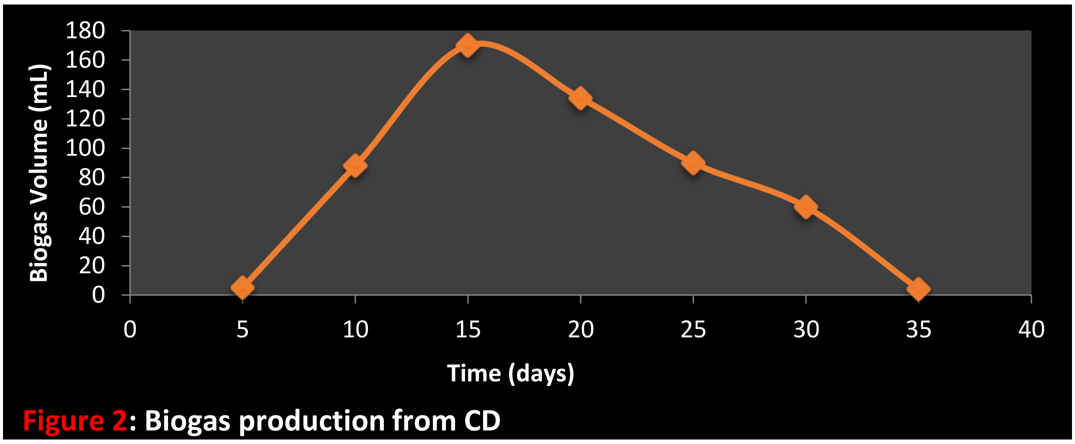


Figure 2: Biogas production from CD

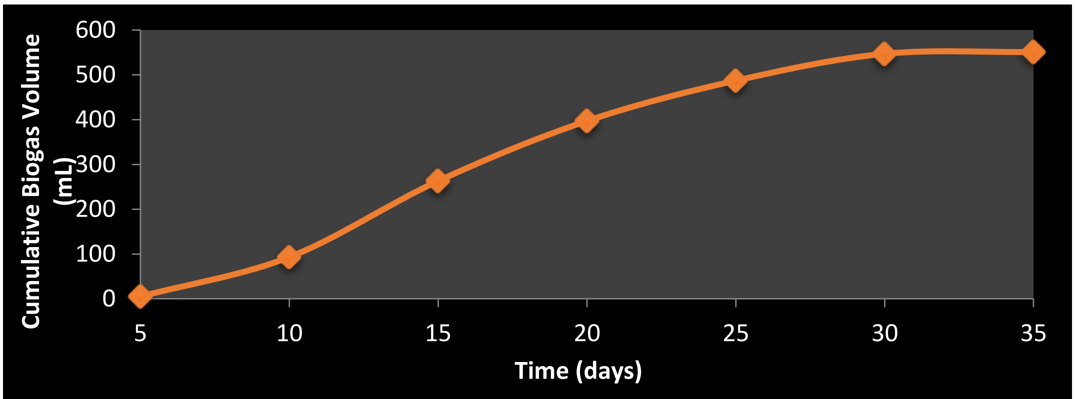


Figure 3: Cumulative biogas produced in days

3.2. Biogas Quality

The quality of the biogas generated was analyzed at 10-day intervals, that is, 3 times for 35 days of digestion. Table 2 shows the average percentage of the gases produced with an average CH_4 content of 62.3%. The low oxygen content obtained signifies that digestion was anaerobic (Vindis, et al., 2008).

Table 2: Components of biogas produced

Gases	10 days	20 days	30 days	Average Gases Produced
CH_4 (%)	62.3	62.5	62.0	62.3
CO_2 (%)	29.5	30.5	32.0	30.7
O_2 (%)	0.8	0.9	0.6	0.8
N_2 (%)	1.8	2	0.9	1.6
H_2 (%)	0.9	1	1.2	1.0
H_2S (%)	0.05	0.1	0.1	0.1

3.3. Factor Optimization

To study the effect of factor interactions, experiments were performed for different combinations of the factors. The responses to the combinations were obtained. The experimental design matrix showing the coded and uncoded factor combinations together with the experimental (exp) and predicted (pre) are shown in Table 3.

Table 3: CCD Model for interactions in coded/uncoded form and response result for biogas volume

Std Order	Run Order	A: pH		B: Catalyst Dose (g)		C: Time (days)		Biogas Volume (mL)
		Coded	Uncoded	Coded	Uncoded	Coded	Uncoded	Response
18	1	0	7.05	0	3	1	20	139
19	2	0	7.05	0	3	0	15	168
17	3	0	7.05	0	3	-1	10	141
14	4	1	7.50	0	3	0	15	168
16	5	0	7.05	1	5	0	15	186
20	6	0	7.05	0	3	0	15	168
13	7	-1	6.60	0	3	0	15	136
15	8	0	7.05	-1	1	0	15	147
7	9	-1	6.60	1	5	1	20	149
1	10	-1	6.60	-1	1	-1	10	89
11	11	0	7.05	0	3	0	15	168
12	12	0	7.05	0	3	0	15	168
10	13	0	7.05	0	3	0	15	168
9	14	0	7.05	0	3	0	15	168
4	15	1	7.50	1	5	-1	10	122
8	16	1	7.50	1	5	1	20	138
6	17	1	7.50	-1	1	1	20	95
3	18	-1	6.60	1	5	-1	10	111
5	19	-1	6.60	-1	1	1	20	94
2	20	1	7.50	-1	1	-1	10	90

The adequacy of the model was justified through ANOVA as shown in Table 4. The quadratic regression analysis shows the model was significant at a 95% confidence level by Fisher's testing. This was confirmed having obtained the F-value of 8.33. The P-value result of less than 0.05 (P-values of regression ≤ 0.05) showed a statistically significant model. The models did not exhibit lack-of-fit indicating insignificant lack-of-fit. The coefficient of determination (R^2) measures the model's overall performance. A high R^2 value, close to 1, is desirable and ensures a satisfactory adjustment of the quadratic model to the experimental data. The R^2 value of 97.0% indicates the model's total variation is 3.0% which is in an acceptable range (Obiora-Okafo, et al., 2020). The difference between the value of predicted R^2 and adjusted R^2 was 0.029, which is less than 0.2, indicating the accuracy of the model. It was also observed from Table 4 that the linear terms and square terms were significant to the response, whereas the interaction terms were insignificant to the response.

Table 4: Analysis of Variance for Biogas Volume

Source	DF	Seq SS	Adj SS	Adj MS	F Value	p-value	
Blocks	1	800.8	504.85	504.85	8.33	< 0.018	Significant
Regression	9	17102.6	17102.6	1900.29	31.35	< 0.0001	
Linear	3	5737.8	4812.7	1604.22	26.47	< 0.0001	
Square	3	11345.8	11345.8	3781.94	62.39	< 0.0001	
Interaction	3	19.0	19.0	6.33	0.10	0.955	
Residual Error	9	545.5	545.5	60.61			
Lack of Fit	5	545.5	545.5	109.11			
Pure Error	4	0.0	0.0	0.00			
Cor Total	19	18449.0					
R - Squared							97.0%
Adjusted R - Squared							93.8%

Second-order quadratic Equation (3) shows the final empirical equations in terms of actual factors after excluding the insignificant terms. Positive signs in front of the equations indicate an interactive positive effect among the factors. In conclusion, the quadratic model for the response measured is significant and adequate.

The regression equation is

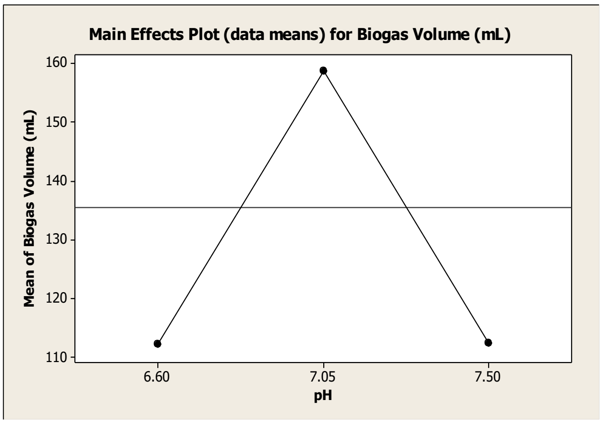
$$Biogas\ Volume\ mL = +9625.92 + 2721.55^{*}pH + 5.05^{*}Catalyst\ dose + 21.88^{*}Time - 193.42^{*}pH^{*}pH - 0.77^{*}Time^{*}Time \qquad (3)$$

3.4 Evaluation of Operation Parameters

3.4.1. Main effects plot for Biogas Volume (mL)

The main effect plots for the factors affecting the biogas production are shown in Figure 4. Main effects indicate the individual effects of the variables studied on biogas yield. The pH has the highest effect on the process. There was a higher biogas volume of 158 mL at a pH value of 7.05, as observed in Figure 4a. Higher biogas yield has been reported at pH ranges of 6.8 – 7.5 (Ubwa et al., 2013). Figure 4b indicates the effect of catalyst dosage on biogas volume. The highest volume generated was achieved at 3 g, with about 150 mL of biogas generated. The effect of retention time on biogas volume is shown in Figure 4c. The plot indicated that the effect on the biogas volume was highest at 15 days with volume of 155 mL. Anaerobic process performance is mostly evaluated through a time-dependent increase in the volume of biogas generated.

a



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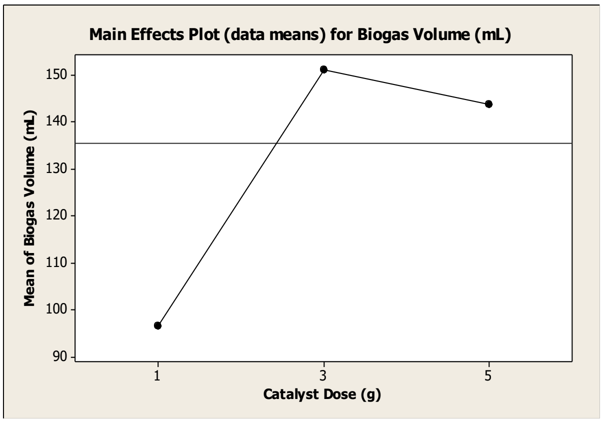

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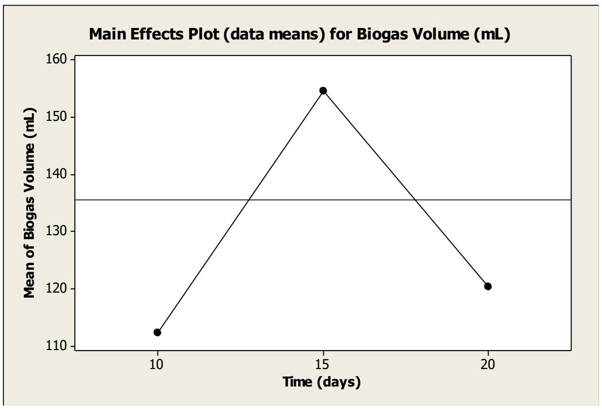
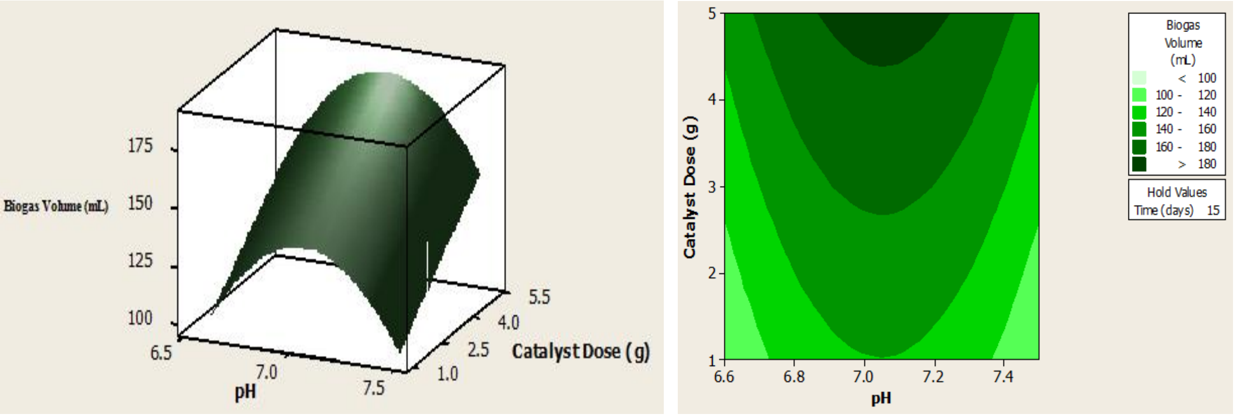


Figure 4: Main effects plot for biogas volume (mL): a) Effect of pH; b) Effect of catalyst dose; and c) Effect of time

3.4.2. Response surface and contour factor interactions

The 3D surface and its corresponding contour plots are represented in Figure 5 for better interacting factor effects on efficient biogas generation by the anaerobic process. The plots show that the maximum biogas volume was experienced in the pH range of 6.9 – 7.2, catalyst doses from 3 – 5 g, and retention time from 12 - 18 days. The response surface plots gave an indication that the maximum biogas volumes generated are located inside the design boundary (Obiora-Okafo and Onukwuli, 2021). From the optimization study, the pH must be controlled in order to establish optimum conditions in the process. The effectiveness of the biogas yield is significantly dependent on pH as shown in Figure 5a. The anaerobic process demonstrated higher gas yield at neutral pH values. The result also indicated that the volume of biogas generation increased more at higher catalyst dosages as shown in Figure 5b. This shows that catalyst addition enhances biogas production during the anaerobic digestion process of cow dung. Figure 5c finally showed that the production of biogas was at its maximum around 15 days. Similar results have been obtained in the use of RSM for the prediction of biogas and biodiesel productions (Abubakar et al., 2021).

a



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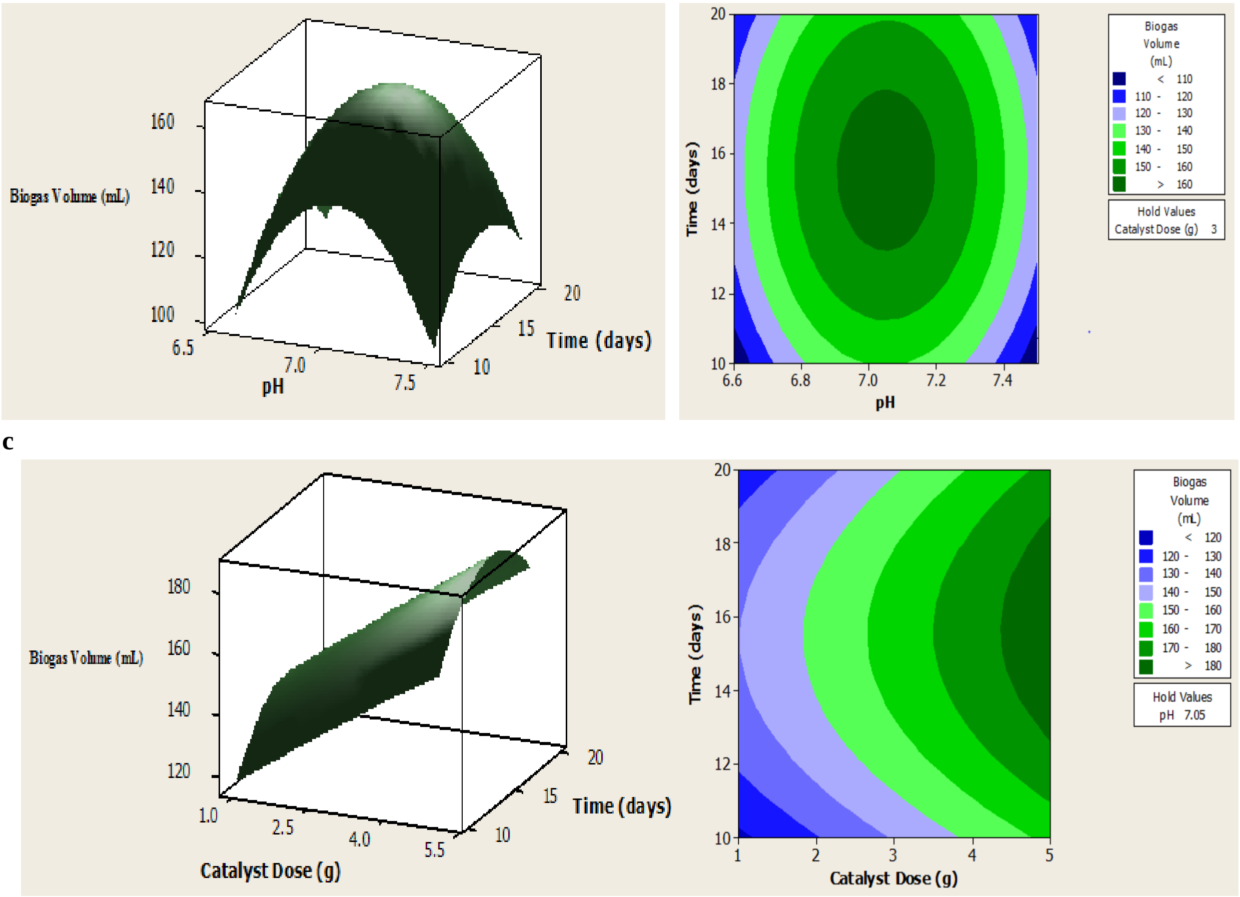

Figure 5: 3D Surface and its corresponding contour plots for biogas volume as a function of a) pH and catalyst dose at 15 days; b) pH and time at 3 g dosage; and c) catalyst dose and time at pH of 7.05.

3.5. Optimization Analysis

Minitab 16 was used to optimize the biogas volume generation process. Process optimization searches for a combination of factor levels that simultaneously satisfy the criteria placed on each response and factors. Numerical optimization was employed, and the desired maximum goal was set for each factor and response (Obiora-Okafo, et al., 2020). These goals were combined into an overall desirability function; for effective maximization of the function. Optimal conditions and the optimization results are shown in Table 5.

3.5.1. Model validation and confirmation experiments

The optimum predicted values were further validated by carrying out the experiment at the optimal predicted conditions, and the results of the experimental values were also shown in Table 5. The experimental data confirm good agreement with RSM results. The verification experiments demonstrated a good agreement between the experimental and predicted values, indicating that the RSM approach adopted was appropriate for optimizing the anaerobic process. The standard error between the predicted and the experimental value was obtained as 1.06%. The value was less than 4%, indicating a very good prediction by the model. The adequacy of the model was once again verified effectively by the experimental data validation (Obiora-Okafo, et al., 2020).

Table 5: Confirmation of the model result using optimum predicted values

Variables	Units	Optimum Predicted Values
Reaction pH	-	7.2
Catalyst Doses	g	3.5
Retention Time	days	15.5
Biogas Volume (predicted)	mL	188
Biogas Volume (experimental)	mL	186

Standard Error	%	1.06

3.5.2. Overlaid contour plots (OCP)

By defining the desired limits, the optimum condition was visualized graphically by superimposing the factors in an OCP, as shown in Figure 6. The white shades are the optimum predicted regions showing the areas that satisfy the response goal of greater than or equal to 80% biogas yield for the factor interactions at mid hold values. Areas that did not fit the optimization criteria were shaded gray (Anyaene et al., 2023; Obiora-Okafo and Onukwuli, 2021). OCP is mostly applied when there is an emergency because it reduces preparation time and experimental costs. It shows clearly the highly efficient region of the contour (Montgomery and Myers, 2002).

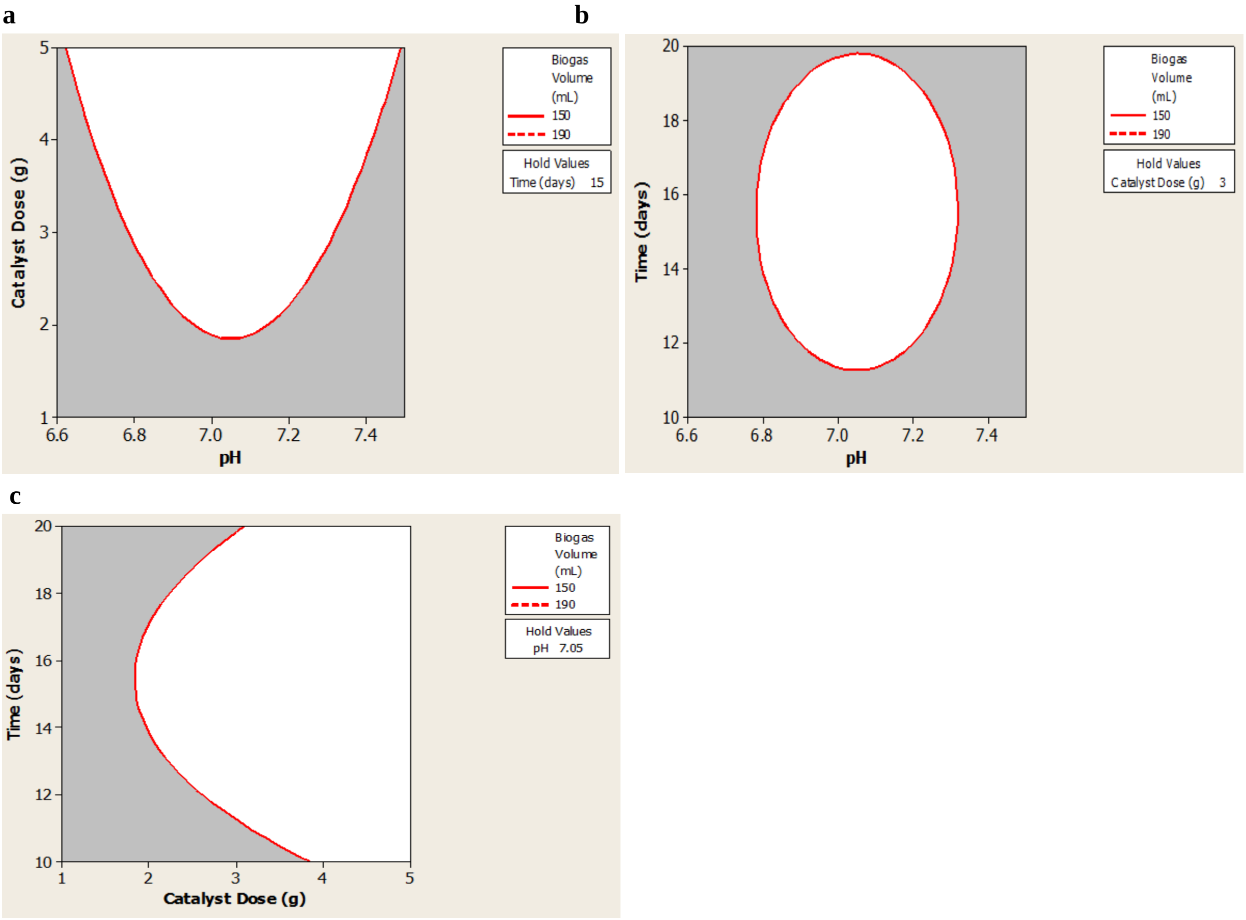

Figure 6: Overlaid Contour Plots for Biogas volume (mL): (a) pH and catalyst dose at 15 days; (b) pH and time at 3 g dosage; and (c) catalyst dose and time at pH of 7.05

CONCLUSION

This study revealed that cow dung has great potentials for the generation of biogas and its usage should be encouraged due to its low time yield and reasonable volume of biogas production. From the study, activated carbon catalyst is a potential catalyst for anaerobic mesophilic digestion of CD using batch digesters. The digesters and slurry were operated under atmospheric pressure and mesophilic temperature range of 27 - 35ºC. Reaction pH, catalyst dose, and retention time influenced the volume of the biogas generation, and their optimum experimented and predicted conditions were identified. The optimal process conditions from the main effects plot partly agrees with the OFAT study. The 3D surface/contour and overlaid contour plots produced the region of maximum biogas yields for the factors in the ranges of 6.9 – 7.2 for pH, catalyst doses from 3 – 5 g, and retention time from 12 - 18 days. Response surface methodology result agreed with one factor at a time (OFAT), giving a higher biogas volume of 186 mL. Further confirmation experiments demonstrated a good agreement with the predicted values, indicating that FCCD was an effective optimization tool for the anaerobic process. Further usage of cow dung for biogas purposes is encouraged due to its abundance and high energy yield.

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10.51975/24380106.som

Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

CHARACTERIZATION OF SELECTED PLANT AND VEGETABLE SOURCE-BASED STRAIGHT-RUN OILS FOR POTENTIAL TRANSFORMER OIL

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ABSTRACT

Mineral oil has been the sole insulation fluid used in electrical transformer due to its availability and high dielectric strength. However, this oil had been reported as non-environmentally friendly and it is associated with other health related issues, posing threats to users and the environment at large. The search for alternative and environmentally benign oil has led to investigation on the potentials of oil from plant and vegetable sources which are biodegradable as transformer oil is desired. In this work, oils were extracted from Soybeans, Moringa oleifera, Jatropha curcas and Parkia biglobosa using n-hexane via soxhlet extraction process. The straight-run oils extracted were characterized. Physicochemical properties and breakdown voltage of the oils extracted based on ASTM standard without being transesterified revealed: pH values ranging from 6.1 to 6.6, flash points ranging from 253℃ to 331℃, and densities ranging from 0.78 to 0.91 g/ml respectively. Saponification values for Moringa oleifera and Jatropha curcas oils ranged between 150 and 244 mg KOH/g fell within the standard range. The iodine values of 71.06 and 123.09 mg KOH/g were also within the ASTM standard. The breakdown voltage ranged between 20 and 48 kV for all the oils extracted and met the ASTM standard of 11- 40 kV for transformer oil application. Thus, the oils had high potential for use as an alternative to mineral oils for transformers and breakers cooling system. It is therefore recommended that the cultivation of jatropha curcas should be explored for sustainable production of non-edible oil as alternative to mineral oil for transformer application.

Keywords: *Breakdown voltage, Breakers, Environmental benign, Extraction, Threats.*

1.0 INTRODUCTION

A transformer is a device that is used to transport electric energy from one alternating-current circuit to one or more of her circuits, either increasing (stepping up) or reducing (stepping down) the voltage. This equipment is used in the power sector from generation to transmission and distribution. Miadonye et al. (2023) stated that “the average age of a transformer on the grid is about 35 years, while they are expected to operate for 30–40 years under ideal conditions, with industrial transformers having a life expectancy of just 20–25 years”. For transformer to perform efficiently during its lifespan, it must satisfy some basic requirement to prevent faults when insulation paper is immersed which is considered as the lifeline of the transformer (Li et al., 2017; Kouassi et al., 2018).

The General Electric produced the first transformer as far back as 1892 with the commercial production in 1899 using mineral oil as its coolant. Oil is required for cooling and insulating parts of transformer, to enhance heat dissipation process as transformer core and coils heat up when in circuit, and to provide indication for monitoring the condition of transformer in-use (Chairul et al., 2020). Zaharaddeen et al. (2013) also reported that transformer oil forms a very significant part of the transformer insulation system and has the important functions of acting as an electrical insulation as well as coolant to dissipate heat losses. Over a century, mineral oil has been used as heat transfer medium and insulating liquid in the power transformers due to its high dielectric strength (Deraman et al., 2020). Nevertheless, the mineral oil has its drawbacks as regards low fire point, not environmentally friendly, low biodegradability (García et al., 2021). Most often, the presence of moisture, acid and the polymerization occurring in transformer oil is due to condensation from storage tanks (Lelekakis et al., 2014; Li et al., 2017; Kouassi et al., 2018), thereby aiding transformer aging while the condensed moisture acts as catalyst for the aging, thereby leading to contamination and oxidization of the oil (El-Sayed et al., 2009). Furthermore, the mineral oil is non-renewable and could fail to meet demand in the event of global crude oil depletion (Miadonye et al., 2023). Other challenges associated with mineral oil includes its high cost of remediation in event of spillage, hazardous to human (its droplets on the skin gets dry and subsequently lead to cracking; when ingested can cause diarrhea and vomiting, and is highly flammable when spilled over). In view of the numerous challenges associated with mineral-base oil used in transformer, the Cooper power system engineers and manufacturer of bio-based dielectric coolant (fluid) began research and development of natural esters in the early 1990s, the result was an edible seed oil-based dielectric coolant called environment FR3 that is used in the company's new line of environtran transformer U.S Patent (September 28, 1999; Rafiq et al., 2015). Thus, the research on the use of esters vegetable-based transformer oil became subject of intense research globally.

Some researches on the use of vegetable oil-based transformer oils include Garba et al. (2013), which produced and characterized bio-based transformer oil from *jatropha curcas* seed, and concluded based on chemical and electrical tests that the values were in agreement with the ASTM specifications, but recommended for further work. Egbuna et al. (2016) produced transformer oil from castor seed as an alternative to conventional transformer oil, and recommended the oil as a potential feedstock for the production of transformer oil. Abdulmumin et al. (2017) characterized the physicochemical properties of *jatropha curcas, moringa oleifera* and castor seed oils for industrial usage as transformer oil via soxhlet extractor by heating at 40 to 60 oz and cold press method; their findings recommended the oil for usage provided that it is refined to required standards. Yu et al. (2017), synthesized vegetable oils produced from soyabean, canola and peanut as renewable low-viscosity dielectrics potentials, the finding showed that canola oil extracted via methyl ester was the best candidate as low viscosity vegetable oil-based dielectric. Deraman et al. (2020), carried out the experimental study on the potential of waste cooking oil as a new transformer insulating oil based on the chemical modification reaction, and concluded that the oil almost fulfilled the requirement as a transformer insulating oil and recommended further treatment before usage as transformer oil. Oyelaran et al. (2020) produced bio-based transformer oil from calabash seed oil and recommended the oil as a replacement for conventional transformer oil as it had met the ASTM standard. Das et al. (2023), carried out an experimental investigation on breakdown performance of coconut oil for high voltage application and concluded that the coconut oil is the most encouraging for attaining high breakdown voltage. All these efforts were made to find environmentally friendly and biodegradable transformer oil from vegetable source which could serve as a partial replacement for conventional mineral oil from hydrocarbon as transformer oil. Miadonye et al. (2023) reported using soxhlet extraction as the most suitable extraction methods using four different solvents: acetone, chloroform, ethyl acetate and petroleum ether on the physicochemical properties of tomato seed oil. Thus, in this study, further work on determination of the potentials of the produced seeds oils (straight-run) for use as transformer oil as recommended by Garba et al. (2013), refining of the oil recommended by Abdulmumin et al. (2017) were carried out without being transesterified, by using normal hexane for extraction as against acetone, ethanol and methanol at a set temperature of 60^{o}C to maintain steady process to avoid solvent escape (Egbuna et al., 2016).

2.0 MATERIALS AND METHODS

2.1 Materials

Raw materials used in this work are *Jatropha curcas* (common Nigerian name in Hausa is Bini da Zugu, in Igbo is called Olulu Idu, Owulu Idu and Yoruba is called Botuje, Lapalapa funfun); *Moringa oleifera* (common Nigerian name in Hausa is Barambo, Zoggale, in Igbo is Odudu Oyibo'in and in Yoruba is called Ewele); *Parkia biglobosa* (common Nigerian name in Hausa is DawaDawa, in Igbo is called Ogiri and in Yoruba is called Iru) and Soya beans seeds. All the seeds were sourced from Bauchi State, North Eastern Nigeria.

The reagents used are of analytical grade purchased from Laboratory reagents dealers in Bauchi Metropolis and Chemistry Laboratory, Abubakar Tafawa Balewa University, Bauchi such as N-hexane, hydrochloric acid (HCl), sodium hydroxide (NaOH) solution, potassium hydroxide (KOH) solution, potassium iodide solution, sodium thiosulphate ($\mathrm{Na_2\,S_2O_3}$), phenolphthalein, magnesium sulphate ($\mathrm{Mg_2SO_4}$), starch and distilled water.

The equipment used are soxhlet extractor kits (250 mL), condenser, mortar and pestle, heating mantle, round bottom flask, measuring cylinders, thimble, digital weighing balance (0.00 g accuracy), burette, pipet, laboratory oven, crucibles, stopwatch, viscometer (1048 Technico size D), thermometer, pH meter 3510 Jenway model, Megger oil test set (OTS 60 PB), density bottle of 50 mL, separating funnel, retort stand with clamps among others.

2.2 Methods

2.2.1 Seeds conditioning

The seeds obtained from various locations of Bauchi State North Eastern Nigeria were conditioned via manual screening of foreign material and separated from the shells by dehulling. The desired seeds were dried in the laboratory for 5 days at environmental temperature being natural as an alternative to using oven drying; thereafter the seeds were crushed with mortar and pestle, and sieved into a known particle size of 1 mm and weighed for further usage.

2.2.2 Oil extraction using soxhlet extractor

The oil extraction was carried out using n-hexane solvent contacted with the weighted average amount of 153g of soya bean, 375.1g of *moringa oleifera*, 108.25g of *jatropha curcas* and 139g of *parkia biglobosa* and gave the exact value of 28.49g of soy bean oil, 149.9g of *moringa oleifera* oil, 43.80g of *jatropha curcas* oil and 27.17g of *parkia biglobosa* oil of crushed seeds of 1 mm sizes in a thimble made of white cotton cloth, which was inserted into the soxhlet extractor. The soxhlet extractor was equipped with condenser connected to the flask containing the solvent (n-hexane). The soxhlet extractor with the content was then heated steadily at 60℃ based on the established procedure (Egbuna et al., 2016) to prevent solvent escape. As it was boiling, the vapour from the distillation arm condenses back to the chamber containing the crushed seeds in the thimble, this was to ensure that solvent vapour condensed and drips back down into the chamber containing the crushed seeds. While the extract seeps through the micro pores of the thimble and fills the siphon tube. The extraction process was carried out every hourly, while the extract was then removed from the thimble, dried in the oven, cooled in the desiccators and weighed again to determine the amount of oil extracted (Egbuna et al., 2016). At the end of the extraction, the resulting mixture containing the oil was heated to recover solvent from the oil. This was followed by refining which is the washing of the oil with warm distilled water and magnesium sulphate (Garba et al., 2013).

2.2.3 Moisture content

The produced oils were dehydrated after extraction and degassed in an oven at 80℃ for 24 hours and followed by drying to reduce the presence of moisture content at 105℃ for 1 hour (Akpan et al., 2006; Mukasa-Tebandeke et al., 2014; Adekunle and Oparanti, 2023). To determine the moisture, the oil sample was weighed before drying (W_1) using crucible, after 1 hour drying, it was removed from the oven and placed in a dissector to cool for 30 minute and reweighed (W_2). The percentage moisture was then calculated using Equation 1.

$$\mathrm{M.C} = \frac{\mathrm{W_1} - \mathrm{W_2}}{\mathrm{W_1}} \times 100\% \qquad (1)$$

where; M. C is the moisture content (%), W_1 is the weight of oil sample before drying (g) and W_2 is the weight of oil sample after drying (g).

The percentage yield was determined using Equation 2, known quantity of the sample was taken before extraction as (W_b), after the oil extraction, solvent recovery and refining, it was weighed after extraction (W_a).

$$\mathrm{Yield} = \frac{\mathrm{W_b} - \mathrm{W_a}}{\mathrm{W_b}} \times 100\% \qquad (2)$$

2.3 Characterization of the Produced Oils

The produced oils were characterized for both physical, chemical and tested for dielectric strength using the relevant standard procedure (Garba et al., 2013; Egbuna et al., 2016; Adekoya and Adejumobi, 2017; Abdulmumin et al., 2017; Das et al., 2023) in triplicate.

2.3.1 Determination of pH

For each of the test, 50 ml of oil was taken in 250 ml beaker, a pH meter electrode standardized with buffer solution was immersed into the oil to read the value which was read and recorded (Akpan et al., 2006).

2.3.2 Determination of flash point

The flash point (FP) was determined based on procedures of Garba et al. (2013) and Egbuna et al. (2016), via an improvised method. For each oil, 20 mL was taken in a test tube which was heated over a hot plate steadily until test flame causes the vapour above oil sample ignite. The value of thermometer at the point of ignition was recorded, and the average of 3 tests results was taken as the FP of each oil.

2.3.4 Determination of boiling point

The boiling point was determined by taking 50 ml of oil in a 100 ml conical flask with thermometer inserted, and then heat was applied until the first bubble was observed and value recorded (Garba et al., 2013).

2.3.5 Determination of melting point

The melting point was determined by taking 60 ml of the oil in 100 ml beaker, and allowed to solidify over the night in the refrigerator. The solidified oil was then removed from the refrigerator, followed by insertion of a thermometer into the oil, and then heat was applied to the solid oil. The first point when the oil began to melt was recorded as the melting point.

2.3.6 Determination of density

The density was measured by taking 5 ml of oil in a 50 ml density bottle against the mass of the oil (g) and was calculated using Equation 3 (Akpan et al, 2006).

$$\rho \ = \frac{\mathrm{g}}{\mathrm{ml}} \qquad (3)$$

where ρ is the density g/ml, g is the mass of the oil and ml is the volume of the oil.

2.3.7 Determination of viscosity

The viscosity of the oil was determined using viscometer 1048 model Technico size "D". Oil sample was taken at temperature of 28 ℃ until it filled to a standardized mark of the viscometer; and a stopwatch was set to determine the time it took the measured oil to flow until the oil passes through to the mark of the viscometer (t_1). Same process was repeated for water at time (t_o). The (t_o) is the time it took the water to pass through the viscometer and (t_1), the time it tookthe oil to pass through the viscometer. This was followed by calculation of absolute viscosity using Equation (4) (Association of Analytical Chemist, 1975).

$$\text{Absolute viscosity} = \frac{t_1 - t_0}{t_0} \qquad (4)$$

where; t_1= oil sample time (seconds), t_o = time for water (H_2O) seconds))

The kinematic viscosity was calculated using the value of absolute viscosity in Equation (4) and the density was determined using Equation (1) and calculated using Equation (5).

$$\text{Kinematic viscosity} = \frac{\text{absolute viscosity}}{\text{density}} \qquad (5)$$

2.3.8 Determination of specific gravity

A clean dry bottle of 25 ml capacity was weighed and filled with oil and reweighed to give weight of oil. The oil was substituted with water in the bottle after cleaning and dried. The bottle containing oil was reweighed to give the weight of water. Specific gravity was obtained from the expression of mass of substance by mass of equal volume of water using Equation (6) (Association of Analytical Chemist, 1975).

$$\mathrm{S.G} = \frac{(\text{weight oil+container}) - \text{ weight of empty container}}{\text{weight of equal volume of water}} \qquad (6)$$

where; S.G is the specific gravity

2.3.9 Determination of free fatty acid (FFA)

Free fatty acid was determined based on the procedure which was adopted from British Standard Institute (BSI, 1995). The oil was placed in conical flask and warm steadily, followed by addition of 2 to 3 drops of phenolphthalein indicator and titrates with potassium hydroxide solution while shaking vigorously until change in colour became stable. The end point was recorded and percentage free fatty acid (%FFA) was calculated using Equation (7).

$$\%\mathrm{FFA}\, \frac{\text{Titre x N x 28.05}}{\mathrm{W}} \qquad (7)$$

where; N is the Molarity of the base potassium hydroxide (KOH) and W is the weight of the oil sample (g).

2.3.10 Determination of acid value

For each of the samples, 10 ml of the oil was weighed and added to 25 ml of neutral ethyl alcohol in a 250 ml conical flask. Heat was applied to the mixture for thirty (30) minutes over water bath and allowed to cool at room temperature. After cooling, 2-3 drops of phenolphthalein indicator were added to the mixture and titrated with 0.1M (KOH) potassium hydroxide to the end point with constant shaking of constituent until a faint permanent pink colour appears, the value was read and recorded using Equation (8) (BSI, 1995).

$$\text{Acid Value(Av)} = \%\text{FFA x } 1.99 \qquad (8)$$

where; AV = acid value and 1.99 is a factor for the calculation.

2.3.11 Determination of saponification value

The process was carried out in two (2) stages, the blank (B) and using oil (S) tests based on standard procedure (Lab Oils and Fats, 2012) and ASTM D464.

i. **Blank test (B) was without oil against dilute HCl acid (0.5M HCl):** for each of the tests, 0.5 mol/l of potassium hydroxide ethanol was taken into the flat bottom flask followed by addition of 2-3 drops of phenolphthalein indicator and was titrated with dilute HCl acid (0.5M HCl) to obtain the titre value.

ii. **Using oil (S) against dilute HCl acid (0.5M HCl)**

For each of the tests, 2 g of oil was weighed into 250 ml conical flask, followed by pipetting 0.5 mol/l potassium hydroxide ethanol solution into the conical flask and thoroughly mixed to form a homogenous phase. Thereafter, 2-3 drops of phenolphthalein indicator were added to the mixture and titrated against dilute hydrochloric acid (0.5 M HCl) solution to the end point until the pink colour of the indicator disappeared. The saponification value (S.V) was calculated using Equation (9).

$$\mathrm{S.V} = \frac{56.1(\mathrm{B} - \mathrm{S})\mathrm{N}}{\mathrm{W}} \quad (9)$$

where; B is the volume of standard HCl required for the blank test (ml), N is the molarity of the HCl used acid (0.5M), S is the volume of standard HCl required for the oil (ml), W is the weight of oil sample (g) and S.V is the saponification value.

2.3.12 Determination of iodine value

5 g of oil sample was placed in 250 ml conical flask; 20 ml of carbon tetrachloride was added to dissolve the oil in the flask and the mixture was stirred and kept in dark place for 30 minutes. Later on, 20 ml of 15% potassium iodide and 100 ml of distilled water were added using a measuring cylinder. 2 drops of starch indicator were added to the sample and titrated with 0.1 M sodium thiosulphate solution until the yellow colour changed to blue, the value was read, recorded, and calculated using Equation (10) (Pardeshi, 2020).

The same experiment was repeated with blank without oil.

$$\mathrm{I.V} = \frac{\mathrm{N(B{-}S)} \times 12.69}{\mathrm{W}} \quad (10)$$

where; I.V is the iodine value, B is the blank titre value, N is the normality of sodium thiosulphate, S is the titre value and 12.69 is the factor for iodine value.

2.3.13 Determination of dielectric strength

Megger oil test set (OTS 60 PB) equipment was used for the dielectric strength commonly referred to as breakdown voltage. The oil tester is an automatic operating device which records the data after several runs. The instrument was set to 2.5 mm^2 electrode gap based on ASTM D877 (Adekoya and Adejumobi, 2017) and ASTM D1816 (2013), standard and was plugged to power until steady state. About 200 ml of oil were taken and put in the container followed by insertion into the OTS and switched on to stir for five (5) minutes. After stirring for five minutes, the OTS started reading until it breakdown at the peak and the reading was recorded. Re-run for two minutes each was conducted five times until breakdown after each re-run and the values were recorded repeatedly up to five times before it gave results as passed or failed.

3.0 RESULTS AND DISCUSSION

This section presents the results of the produced and characterized (*Jatropha curcas, Moringa oleifera, Parkia biglobosa* and Soyabean) seeds oils as a transformer insulation fluid. Three (3) tests were carried out on each of the oil samples for accuracy and precision. Table 1, present the characterized physicochemical properties and dielectric strength (breakdown voltage) of oils produced.

Table 1: Typical Physicochemical Properties and Dielectric Strength of Vegetable Seeds Oil

Properties	Vegetable Seeds Oil Type				Standard ASTM/BSI
	Soyabean	*Moringa oleifera*	*Jatropha curcas*	*Parkia biglobosa*	
Moisture Content (%)	7.34	4.03	1.75	6.15	< 10
pH	6.1	6.4	6.6	6.4	5.5 – 8.2
Flash Point (℃)	327	253	331	298	140 - 155
Boiling Point (℃)	136	150	167	138	120 - 230
Melting Point (℃)	-9	-10.1	-7.9	-12	
Density (g/ml)	0.91	0.78	0.80	0.86	0.55 – 0.89
Viscosity @29℃ (mm^2/s)	26.33	43.85	33.42	45.20	9.2 - 27
Specific gravity	0.86	0.80	0.91	0.85	0.89 – 0.91
%FFA (mg KOH/g)	11.78	16.89	22.78	14.53	0.01 – 0.08
Acid Value (mg KOH/g)	23.44	33.61	45.33	28.92	0.01 – 0.03
Saponification value (mg KOH/g)	95.48	224.40	178.11	57.50	150 - 244
Iodine Value (mg KOH/g)	118.02	71.06	110.40	123.09	55 – 120
%Yields	18.62	40.93	44.38	19.68	
Dielectric strength (kV)	29	48	20	23	11– 40

The determination of moisture content (M.C) of the produce oils was based on Equation 1. It is as an essential property which indicates the quantity of dissolved water in the oil. The percentage moisture content and the yields were determined as presented in Table 1 for the oils. The M. C content of all the produced oils was less than 10%. According to Das et al. (2023) analysis, if "M. C of oil is less than 10%, such oil is considered as clean oil which will also help to prevent oil breakdown strength under Quasi-uniform electric field and it will remain unaffected." In this work, the 10% M. C of all the oils satisfied the recommended oil requirement for use as coolant in transformer and breaker. While the oil yields were determined based on Equation 2 to know how much of the oil is available. Moringa oleifera and Jatropha curcas had high oil yields compared to soyabean and parkia biglobosa seeds. This could be due to the nature of seeds which are hard and consume greater amount of solvent to penetrate into the particle sizes of the soyabean and parkia during extraction process. While *Moringa oleifera and Jatropha curcas* seeds are soft such that the solvent could easily penetrate during extraction process to produce more oil.

The results of the pH values for the produced oils as presented in Table 1 showed that, pH values are slightly acidic but weak acid as the pH values are similar to the one reported in the literature as 5.5 to 7.5 (Emil et al., 2009) which fell within the range values of 5.5 to 8.2 of ASTM 2013 standard for mineral oil used as transformer oil.

The flash point (FP) is the minimum temperature at which the oils can ignite when the vapour phase mixes with air. It also indicates when the risk of fire can be prevented in handling the oil during transportation and storage (Egbuna et al, 2016). The FP at which the produced oils ignited is presented in Table 1 for the oil's soya oil (327℃),*moringa oleifera oil* (253 ℃), jatropha curcas oil (331℃) and *parkia biglobosa oil* (298℃) respectively. This FP surpassed the recommended ASTM standard range of 140℃ – 155℃ for mineral oil used as transformer oil and minimum of 250℃ for natural esters oil according to IEC62770 standard (Das et al, 2020). High FP oil implies that, the produced oils can be used even in areas where there is high temperature to prevent automatic ignition (AI) and fire hazards during transportation, storage and are considered safe. It also indicates that high FP in insulation oil shows low tendency of fire outbreak which is advantageous for high voltage transformer (Adekunle and Oparanti 2023).

Density of the oil which is the mass of the oil per unit volume of the oil was determined to show the capacity of the oil to yield high energy that would be available for work output. The densities of the produced seeds oil were found to be within the ASTM standard ranged values 0.55 – 0.89 g/cm^3 except that of soybean oil which is 0.91 g/cm^3.These values are high compared to the density obtained by (Garba et al., 2013) for jatropha curcas oil of 0.725 g/cm^3.

Viscosity of the produced oils: viscosity is a fundamental factor which determines the performance of a cooling insulation of a transformer, thus it becomes very significant that the transformer oil is expected to have low viscosity to enable it circulate for ease heat transfer. High viscosity implies poor heat transfer capability that could result into oil aging with continuous air supply (Das et al., 2023). The resulting viscosity from the produced soybean, *moringa oleifera, jatropha curcas and parkia biglobosa* oils were 26.33, 43.85, 34.05 and 45.20 mm^2/s respectively. Obviously, the results show that only soybean oils had low viscosity which fell within the ASTM standard range of 9.2 to 27 mm^2/s at 29 ℃. Although Adekunle and Oparanti (2023) reported the standard viscosity requirement for new ester oil is 35 mm^2/s at 40 ℃, but it varies based on the temperature at which the viscosity was tested. The values obtained from this work at 28 ℃ fell within these ranged values.

The percentage free fatty acid results as presented in Table 1, showed that the refined oils had increase in %FFA which is out of the recommended ASTM values of 0. 01 to 0.08 for the mineral oils used in transformer. According to Oyelaran et al., (2020), most seed oils contain phosphatides in the form of lecithin causing gum or waxes in oils, this could be the reason that had contributed to the oils having high %FFA.

Saponification value was determined for the produced oils and the results show that the values are within the ASTM range values of 150 to 244 mg KOH/g after degumming. Except for soya bean oil and *parkia biglobosa* oil even after refining (degumming) of the oils the values still remain below the threshold of 150 mg KOH/g as presented in Table 1, while *moringa oleifera and jatropha curcas* oil improved from 67.32 to 224.40 mg KOH/g and from 50.49 to 178.11 mg KOH/g respectively. High saponification values indicate greater portion of low molecular weight of fatty acid with possibility of oxidization of the oil which can cause sludge and corrosion in the system when in operation. The values obtained from this work could be compared to those obtained by (Egbuna et al., 2016) from castor oil; (Abdulmumin et al., 2017) from jatropha, moringa and castor oils.

Table 1 presents the characterized properties of the produced oils. The iodine values of 118.02, 71.06, 110.40 and 123.09 mg KOH/g was determined for soya bean, *moringa oleifera, jatropha curcas* and *parkia biglobosa* oils. High iodine values imply the oil is saturated with fatty acid which is prone to fast oil degradation catalyzing the oil reactivity such as oxidization and polymerization reaction, most especially when the temperature is high along with the dissolve oxygen gas present in the air (Garba et al, 2013; Egbuna et al, 2016). Suitable oil for use as transformer oil should have iodine value within the range of 55 – 120 mg KOH/g based on the ASTM standard. Thus; the oils produced from this work were within the ranged values except for the *parkia biglobosa* which has 123.09 mg KOH/g.

Dielectric strength: the measurement of dielectric strength (DES) which is known as the breakdown voltage (BDV) of the produced oils were carried out using automatic oil testing equipment based on the ASTM D877 standard, this test is the most common test administered on transformer oil. Table 1, presents the results of tests carried out on oils. The results show that the unrefined oils had low breakdown voltage ranging from 3 kV – 12 kV for moringa oleifera to Parkia biglobosa as the lowest and highest, while others are in between the values. The oil tester showed that only parkia biglobosa oil which breaks at 12 kV met the 11 kV transformer oil requirements and classified passed, while other oils fail the test. The low BDV was as results of impurities and other contaminants which could affect the performance of the oils this is supported by Falatah and El-Naggar (2018) report on quality of transformer oil and possible impurities that can affect the dielectric strength. When the oils were refined, the BDV improved tremendously from 7 – 29 kV for soybean oil, 3 – 48 kV for moringa oleifera oil, 3 – 20 kV for jatropha curcas oil and 12 – 23 kV for parkia biglobosa oil respectively as tested at 2.5 mm gap setting based on the standard as the tester showed oil passed after seven (7) runs. The oil refining had great impact which resulted into the improvement of the BDV of the produced oils as demonstrated. These results, had met the requirement for 11 – 33 kV transformer oil currently in-use as insulation and cooling oil which satisfy the ASTM 2013 standard of 11 – 40 kV range for mineral oil.

4.0 CONCLUSION

In this work, some selected plants (*Jatropha curcas and Parkia biglobosa*) and vegetables (*Moringa oleifera* and soyabean) seeds oils were extracted using normal hexane via soxhlet extractor and characterized for potential use as a transformer oil and breaker coolants. All the straight-run tested oils physicochemical properties met the ASTM standard for transformer oil. Nevertheless, the use of soyabean oil, *moringa oleifera oil and parkia biglobosa* oil as transformer oil may result in cost being edible oils for human consumption, as they are used as food supplement and of high nutritional value. The only viable oil which is non-edible is the *Jatropha curcas* oil and also had the highest yields of 44.38%, with the lowest M. C of 1.70%, Flash point of 331 ºC and the breakdown voltage of 20 kV. It is recommended to key into cultivation of more *Jatropha curcas* for mass production of the oil which is not only limited for potential use as transformer oil, but also in circuit breakers, cables, capacitors, and tap changers among others in the electricity industry other than cosmetics.

ACKNOWLEDGEMENT

The authors greatly acknowledged Prof. Samson Duna, the Director General/Chief Executive Officer of the Nigerian Building and Road Research Institute for his financial assistance for sponsoring this paper, Bauchi Business Unit of Jos Electricity Distribution Company, and the Departments of Chemical Engineering and Chemistry of the Abubakar Tafawa Balewa University Bauchi, Nigeria for using their Laboratory facilities to do the research.

CONFLICT OF INTEREST

The authors declared that there is no conflict of interest in this research, as it has not received any grant to do this work.

ETHICS

There are no ethical issues with the publication of this manuscript.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article, while raw data that support this study is available from the corresponding author upon request.

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Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

ASSESSMENT OF WASTE SEGREGATION AWARENESS IN LAGOS STATE UNIVERSITY, EPE, LAGOS STATE, NIGERIA

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ABSTRACT

Proper solid waste management is very crucial for environmental sustainability and waste segregation is a vital component of solid waste management. The importance of waste segregation awareness cannot be overemphasized towards achieving sustainable environment. The aim of this work is to assess waste segregation awareness in Lagos State University (LASU), Epe campus with a view to provide valuable information for appropriate strategies formulation to promote and encourage waste segregation on campus. A questionnaire was designed and administered to respondents in LASU, Epe campus. The designed questionnaire was divided into sections A – G. Section A focused on the demographics characteristics of the respondents, section B beamed on attitude and environment (A and E), section C evaluated the social norms (SN) of the respondents, section D assessed the recycling habit (RH) while section E measured the perceived convenient and effort (PC and E) of the respondents. Other sections were section F which analysed the moral norms (MN) and section G which tested the state of knowledge, information and system trust (KI and ST). The data generated through the questionnaires were subjected to statistical analysis with the aid of an in-built solver tool in Microsoft excel version 2010. The statistical analysis carried out included minimum, maximum, mean, standard deviation (SD), standard error (SE) and coefficient of variance (CV). Others are median, mode and skewness. The results revealed that the mean values which varied between 4.13 and 4.79 implied that majority of respondents were aware of waste segregation. SD values ranged between 0.59 and 0.96 indicated that all values were clustered around the mean which showed the respondents were aware of waste segregation. SE values varied between 0.05 and 0.07 which implied the mean values obtained in this study were very close to true mean of people in LASE, Epe campus which also corroborated that the respondents were aware of waste segregation. The skewness values ranged between – 3.56 and – 1.15 which revealed the data obtained in this work fell towards the higher side of the scale and this is a pointer that the respondents' sampled can be taken as the true representation of people in LASU, Epe campus. Based on the statistical analysis conducted, it was concluded that the people in LASU, Epe campus were aware of waste segregation.

Keywords: Assessment, awareness, compliance, questionnaire, Lagos State University and waste segregation

1. INTRODUCTION

Solid waste management involves the control of generation, collection, storage, transport and transfer, energy recovery and disposal of solid waste in a way that the public health and environment are not negatively affected (Lema *et al*., 2019 and Popoola *et al*., 2023). Lack of proper management of solid waste has led to environmental, ecological and health problems (Salami and Susu, 2011; Salami and Susu,2013; Salami *et al*., 2015; Salami and Susu, 2019 and Adeyemo and Salami, 2022). Waste segregation is a very vital component of solid waste management (Jacob *et al*., 2021). It is the process of identifying, classifying, dividing and sorting of waste and waste products to make reuse, recycling and recovery possible to realize. The practice of waste segregation in developing countries (Nigeria inclusive) is very poor (Salami *et al*., 2015; Salami*et al*., 2020 and Salami *et al*., 2021) due to lack of awareness, certain attitudes and perception towards environmental concerns

(Jesson *et al*., 2009; Guerin *et al*., 2001 and Martin *et al*., 2006).

Scholars have carried out several works on assessment of waste segregation (Soran *et al*., 2015; Lin *et al*., 2017; Elemile *et al*., 2017; Yerbol *et al*., 2019; Yi, 2020; Soya *et al*., 2020; Fukda *et al*., 2018; Cheng, 2020; Otitoju and Seng, 2014; Krushna *et al*., 2022 and Deng *et al*., 2017). Soran *et al*. (2015) investigated the participation in source separation of municipal solid waste based on the theory of planned behaviour. The investigation revealed that the Persian version of the theory of planned behaviour is very suitable, reliable and very important in administrative activities for increasing participation in municipal solid waste source separation. Liu *et al*. (2017) worked on understanding household waste separation behaviour. The work suggested insightful future policies that will provide convenience for waste separation and collection facilities encourage recycling, promote community campaigns and education with a view of aiding people to form favourable habit for protecting the environment.

Elemile *et al*. (2017) studied the effects and provision of collection bins on source separation of solid waste among workers of a tertiary institution in Nigeria. The study showed that training and provision of collection bin was very suitable and effective in promoting source separation practice. It further recommended advocacy, training and provision of refuse bins to encourage source separation in the institution. Yerbol *et al*. (2019) carried out a survey on household solid waste sorting at source in developing economies using Nur – Sultan city in Kazakhstan as a case study. The survey indicated that 24 percent of respondents were practicing waste separation in the absence of/waste separation system by the local authorities. The survey equally revealed that waste separation could be practised if the local authorities provide recycling bins in proximity to residential buildings. Yi (2020)developed waste separation practice index among the community of tertiary institution in Malaysia. The developed index displayed that students are more committed to practicing waste separation compared to administrative and academic staff.

Soysa *et al.* (2022) analysed factors affecting waste generation and segregation behaviour using data from education communities in Western and North – Western provinces of Sri lanka. The results of the analysis could be very useful for developing influential policies at the local authority to improve and strengthen the solid waste management. It also provides valuable information needed to formulate appropriate and encourage waste segregation at source. It is evident from available literature that the assessment of waste segregation awareness in LASU, Epe, Lagos State, Nigeria has not been given adequate attention. Therefore, the aim of this work is to assess the waste segregation awareness in LASU, Epecampus. This work will provide valuable information require for appropriate strategies formulation to encourage and promote waste segregation at source in LASU, Epe campus which justifies this work. It will also give an insight on development of innovative and new approaches to waste management in LASU, Epe campus which further justifies the work.

2. METHODOLOGY

2.1 Study Area

LASU, Epe campus was a military barrack before 1996 but became a full fledge academic campus in 1996. The campus is a bid tract of land which runs into thousands of acreage, is located on 6.588ºN and 3.9896ºE (Salami and Folami, 2021). The campus houses the faculty of engineering which comprises the following department: mechanical, civil, chemical, aerospace and industrial engineering. The campus also accommodates school of agriculture and the newly established faculty of environmental sciences. Pre – degree studies and part time programmes for management courses. The campus is surrounded by coast valley landscape of Epe land use by the villagers for agricultural purposes (LASU Students' Handbook, 2015). There are accommodation on campus for staff and students of the university.

2.2 Design of Questionnaire

The questionnaire used for waste segregation awareness in this study was designed based on previous works of scholars. The complete questionnaire adopted is depicted in Appendix A. Section A of the questionnaire considered the demographics characteristics of the respondents which include gender, age, education level, status and department/unit/division in LASU Epe campus. Section B looked into the attitude and environment of the respondents by measuring 6 items to indicate value judgment on waste separation. For instance, waste separation aids protection and conservation of our environment and resources. The 5 – point scale between 1 and 5 represents strongly disagree, disagree, not necessary, agree and strongly agree respectively. The scale was used to rate the response of the respondents in each item. High score shows a more positive attitude towards waste and environment. Section C focused on social norms by evaluating 4 items reflecting support from peers and LASU management. For example, does LASU Epe campus management support you to perform waste separation? Each question was rated using 5 - point scale ranging between strongly unsupportive to strongly supportive. High score indicated more positive towards social norms.

Section D assessed the recycling habit by using 4 items. For instance, recycling of waste is a way of reducing waste to be disposed in landfill. Each item was rated on a 5 point scale from strongly disagree to strongly agree. High scorerepresentsa high affinity for recycling. Section E measured the perceived convenient and efforts with 6 items. For example, is there enough time for you to carry out waste separation? Each item was evaluated using the 5 - point scale ranging between totally no time to always have time. High score reflected a more positive towards perceived convenient and efforts for waste segregation.

Section F beamed on moral norms by measuring 4 items to reflect moral judgment for waste separation. For instance, do you belief you have the duty to perform waste separation? Each item was also rated on the 5 – point scales varying from strongly disagree to strongly agree. High score showed a more positive towards moral judgment for waste segregation. Section G tested the state of knowledge, information and system trust with the aid of 4 items. For example, LASU, Epe campus management places importance on waste separation. Each item was evaluated by applying the 5 – point scales ranging between strongly disagree to strongly agree. High score revealed a more positive towards state of knowledge, information and system trust.

2.3 Data Analysis

Thedata generated through the designed questionnaire were subjected to statistical analysis using the inbuilt solver tool in Microsoft excel version 2010. The statistical analysis carried out included minimum, maximum, mean, SD, SE and CV. Others are median, mode and skewness.

3. RESULTS AND DISCUSSION

200 designed questionnaires were administered to respondents out of which 181 were returned. 8 questionnaires among the returned ones were declared invalid due to ticking of more than one option in a particular question and as a result of incomplete answering of questions explicitly stated in the questionnaires. Hence 173 questionnaires were treated as valid questionnaire which were then used as a true representation of the population size of LASU, Epe campus. The demographics profile of respondents is presented in Table 1. Female and male have 56.07 and 43.93 percent respectively.

Table 1. Demographic profiles of respondents

Demographics	Number of respodents	% of respodents
Gender		
Male	76	43.93
Female	97	56.07
Age		
$\prec$ 20	31	17.92
21 – 30	76	43.93
31 – 40	34	19.65
41 -50	21	12.14
51 – 60	8	4.62
$\succ$ 60	3	1.74
Educational level		
No education		
Primary	4	2.31
Secondary	30	17.34
Undergraduate	94	54.34
Graduate	45	26.01
Status		
Staff	37	21.39
Non staff	23	13.29
Students	113	65.32
Staff cadre		
Senior	28	75.68
Junior	9	24.32

Those in the age between 21 and 30 yrs responded the most, having 43.93 percent and in term of educational level, undergraduates has 54.34 percent. Among the respondents, students have 65.32 percent while staff has 21.39 percent out of which 75.68 and 21.39 percent represent senior and junior categories respectively. Pakpour *et al*. (2014) revealed that only gender and age were significant factors that affect waste separation. Previous studies of Ekere *et al*. (2015), Padilla and Trujillo (2018) and Babaei *et al*. (2015) indicated that gender plays a vital role in waste separation at the source as female seemed to be more active than male in waste separation. In this work, 56.07 percent of the respondents declared valid were female. This also corroborates previous works of scholars pointing out the female are more dispose to waste separation than male.

The statistical analysis of variables influencing waste separation considered in this study is shown in Table 2. The mean is the average value and it is also referred to as expected value. The mean of all variables influencing waste segregation varied between 4.13 and 4.79. This is very high, which implied the majority of the respondents were aware of waste segregation. In the study of Liu *et al*. (2017), the mean of variables influencing waste segregation ranged between 3.23 and 4.41 on a scale of 5 and the study concluded the respondents have positive responses towards waste separation. The values of mean obtained in this present study were higher than values obtained in the work of Liu *et al* (2017). Hence it can be said that the respondents in LASU, Epe campus were aware of waste segregation.

Table 2. Statistical analysis

	A and E	SN	RH	P and CE	MN	KI and ST
Minimum	1	1	1	1	1	1
Maximum	5	5	5	5	5	5
Mean	4.27	4.43	4.13	4.39	4.79	4.43
Median	4	5	4	5	5	5
Mode	5	5	5	5	5	5
SE	0.06	0.06	0.07	0.07	0.05	0.06
SD	0.80	0.75	0.96	0.86	0.59	0.73
CV	0.39	0.35	0.47	0.41	0.27	0.35
Skewness	- 1.26	- 1.42	- 1- 15	- 1.79	- 3.56	- 1.76
Count	173	173	173	173	173	173

The SD values of variables influencing waste separation varied between 0.59 and 0.96. Standard deviation values less than 1 indicate all variables are relatively clustered around the mean (Ben, 2011). This implied that all variables used in this work concentrated around the mean which was an indication that the respondents in LASU, Epe campus were aware of waste separation. Moreover a relatively small SE is a pointer that the mean is reasonably close to true mean of the entire sample (Lin *et al*., 2017). The SE values in this work ranged between 0.05 and 0.07 which was very small. The SE values showed that the mean values obtained in this work were very close to true mean of the LASU, Epe campus mean which was an indication that there was awareness about waste segregation in LASU, Epe campus.

The skewness values varied between – 3.56 and – 1.15. All skewness values were negatives which implied all variables explained an asymmetrical distribution that the data obtained in this study fell towards the higher side of the scale which means the respondents sampled can be taken as a true representation of people in LASU, Epe campus. Based on the statistical analysis carried out in this work, it can be said that the people of LASU, Epe campus were aware of waste segregation. However, LASU, Epe campus management stills need to encourage and promote waste segregation at source for effective and proper solid waste management on campus.

4. CONCLUSION

Waste segregation awareness in LASU, Epe campus has been assessed. 200 questionnaires were administered, 181 were collected and 173 were declared valid. The demographics characteristics of the respondents showed that 56.07 percent of female took part in the study. The age between 21 and 30 accounted for 43.93 percent while respondents in undergraduate programme represented 54.34 percent. The mean values varied between 4.13 and 4.79 which implied majority of the respondents were aware of waste segregation. The SD values of all variables considered in this work ranged between 0.59 and 0.96 which were less than 1. This indicated that the variables were concentrated around the mean pointing that the respondents in LASU, Epe campus were aware of waste segregation. The SE values were very small, varied between 0.05 and 0.07 which indicated the mean values obtained in this study were very close to true mean value. The skewness values ranged between – 3.56 and – 1.15 which showed the data obt5ained in this work fell towards higher side of the scale. This implied the respondents sampled can be taken to be the true representation of people in LASU, Epe campus. Based on the statistical analysis conducted, it can be concluded the people in LASU, Epe campus were aware of waste segregation.

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Appendix A: The various sections used in the questionnaire for this study.

Section A: Demographics

Gender: Male / Female
Age: (a) Below 20 (b) 21 – 30 (c) 31 – 40 (d) 41 – 50 (e) 51 – 60 (f) above 60
Educational Level: (a) No education (b) Primary (c) Secondary (d) Undergraduate (e) Graduate
Status: (a) Staff (b) Non staff (c) Student
For staff:
Type: Academic / Non academic
Cadre: Senior / Junior
Department/Unit/Division: ……………………………………………………………………..
For Student:
Department: …………………………………………………… Level: ………………………

Section B: Attitude and Environment

(1) Practising waste separation gives happiness, so you should partake in it.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(2) Waste separation is a sign of personal hygiene.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(3) Waste separation aids protection and conservation of our environment and resources.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(4) Revenue can be generated by selling separated recyclable waste.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(5) By carrying out waste separation, you are showing example to others to be involved in waste separation.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(6) Waste separation helps to minimize cost of pollution control.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

Section C: Social norms

(1) Does LASU Epe campus management support you to perform waste separation?
(a) Strongly unsupportive (b) Unsupportive (c) Not sure (d) Supportive (e) Strongly supportive
(2) Are you supported by your friends or colleagues in LASU Epe campus to carry out waste separation?

(a) Strongly unsupportive (b) Unsupportive (c) Not sure (d) Supportive (e) Strongly supportive
(3) Would you like to be a member of any environmental organization in LASU, Epe campus?
(a) Very unwilling (b) Willing (c) Not necessary (d) Willing (e) Very willing
(4) Would you like to listen to environmental protection organization news, press calls and programs to perform waste separation?
(a) Very unwilling (b) Willing (c) Not necessary (d) Willing (e) Very willing

Section D: Recycling habit

(1) Recycling of waste is a way of reducing waste to be disposed in landfill.
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(2) Do you belief you should be involved in waste separation for easy recycling?
(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree
(3) How many days in a week do you belief waste separation should be carried out for easy of waste recycling?
(a) 0 (b) 1 – 2 days (c) 3 – 4 days (d) 5 – 6 days (e) everyday
(4) Are you ready to be involved in waste recycling business?
(a) Very unwilling (b) Willing (c) Not necessary (d) Willing (e) Very willing

Section E: Perceived convenient and Effort

(1) Is there enough time for you to carry out waste separation?
(a) Totally no time (b) Do not really have time (c) Depend on the situation (d) Often have time

(e) Always have time

(2) If there is no enough time, will you still carry out waste separation?

(a) Definitely will not (b) Will not (c) Depend on the situation (d) will (e) Definitely will

(3) Does LASU Epe campus have sufficient space for storage of separated waste?

(a) Totally no space (b) Do not really have space (c) Depend on the situation (d) Often have space (e) Always have space

(4) If LASU Epe campus does not have enough space for storage of separated waste, will you still carry out waste separation?

(a) Definitely will not (b) Will not (c) Depend on the situation (d) will (e) Definitely will

(5) Does management of LASU Epe campus provide facilities for waste separation such as different bags and bins?

(a) No facilities at all (b) little facilities (c) Not sure (d) Almost complete facilities (e) complete facilities

(6) Have you been involved in waste separation in LASU since you joined LASU Epe community?

(a) Completely I have not (b) I have not (c) Not sure (d) I have (e) Completely I have

Section F: Moral norms

Do you belief you have the duty to perform waste separation?

(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

(2) Do you feel comfortable when wastes are thrown in the waste bins without separation?

(a) Very comfortable (b) Comfortable (c) Less concern (d) Uncomfortable (e) Very uncomfortable

(3) Do you belief is a bad behavior not to be involved in waste separation?

(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

(4) Are you ready to change your behavior if you have not been carrying out waste separation?

(a) Very unwilling (b) Willing (c) Not necessary (d) Willing (e) Very willing

Section G: State of knowledge, information and system trust

(1) LASU Epe campus management places importance on waste separation.

(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

(2) LASU Epe campus management has been advocating for waste separation with a view to getting staff and non-staff to be involved in everyday waste separation.

(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

(3) Waste separation which is a way of solid waste management should be taught in the school.

(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

(4) People on LASU Epe campus know the different types of waste being generated on campus.

(a) Strongly disagree (b) Disagree (c) Not necessary (d) Agree (e) Strongly agree

Thank you for your contributions

10.51975/24380108.som
Journal of the Nigerian Society of Chemical Engineers, 39(1), 2024

OPTIMIZATION OF THERMOPHILIC ANAEROBIC LIQUID WASTES TREATMENT FOR THE PRODUCTION OF SUSTAINABLE ENERGY IN NIGERIA

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ABSTRACT

Energy Production from biogas could enhance realization of Nigeria's quest for zero carbon emissions by the year 2060, as biogas has less carbon.A biogas turbine power plant with thermophilic anaerobic liquid wastes treatment plant yielded 6,785.5953 kJ/s of heat energy. This heat helped turn a shaft at the speed of 23.565 ms^{-1} in a magnetic field to produce electricity. In accordance with the second thermodynamics law, that all the heat cannot be converted to work 6,781.152826 kilowatts of work was produced from that heat. Study reveals that 4,424 grid-tied biogas turbine power plants of 6,781.152826 kilowatts capacity can generate the required 30,000 Megawatts of energy. The number of plants could be reduced by scaling - up the design but small plants are easier to maintain. Economic analysis gave a net annual profit of ₦600,405,586.40, a rate of return of 32.61 % and a pay-back period of 3 years, 3 weeks. This shows that a biogas turbine power plant with a thermophilic anerobic liquid wastes treatment plant if well managed could be economically viable in Nigeria. The Nigerian Government and the private investors are therefore encouraged to set-up the plant as much profit could be made from the investment.

***Keywords**:*
Biogas, Liquid wastes (wastewater); Electricity; Pay-back period; Rate of Return on Investment.

1.0. INTRODUCTION

Sampson (2022) stated that thermophilic anaerobic treatment of liquid wastes produces biogas, a renewable energy source, useful for electric power generation and heating.

The use of non-renewable energy sources has resulted in high carbon emissions. These emissions deplete the ozone layer causing green house effect. The accumulation of untreated liquid wastes (wastewater as it is often mixed with water) causes environmental disaster. These have adverse effect on sustainable development. Sampson (2022) stated that reaction at thermophilic temperature (55 °C -75 °C) enhances higher reaction rates, increases death rate of pathogens and hence more liquid wastes could be treated and more biogas produced in a shorter time.

International Trade Administration (I.T.A.) (2020) stated that in 2006, the Nigerian Government initiated the Renewable Energy Master Plan (REMP) which sought to increase the supply of renewable electricity from 13 % of total electricity generation in 2015 to 23 % in 2025 and 36 % by 2030. Renewable electricity would then account for 10% of Nigerian total energy consumption by 2025. This was a workable plan if adequate technology had been adopted to realize this. It is stated in Premium Times (2022, August 25) that on August 24, 2022 the Federal Government of Nigeria launched the Energy Transition Plan (ETP) designed to simultaneously tackle the challenges of energy poverty and climate change, lifting hundreds of millions of Nigerians out of poverty. At the 2021 United Nations (UN) climate change conference, Nigeria committed itself to achieving zero gas flaring by the year 2060.

Sampson (2014) stated that Nigeria had earlier pledged to achieve zero gas flaring by the year 2020, 40 more years, yet no technology is adopted to realize this. An end to incineration of liquid wastes in burn pits but treating them in thermophilic anaerobic reactors could enhance realization of this goal.

Optimization: This technical paper studies the optimization of thermophilic anaerobic liquid wastes treatment for the production of sustainable energy in Nigeria using appropriate design equations and kinetic models solved using MAT LAB software to:

i. Examine the optimum retention time, reactor diameter, reactor volume, biogas production and reactor performance based on parameters such as biochemical oxygen demand and total hydrocarbon content.

ii. Predict the optimum conditions for thermophilic anaerobic liquid wastes treatment and the optimum amount of energy produced from the biogas-fed turbine power plant and how this energy could make-up for the Nigerian energy deficit. Costing and economic evaluation is key to the determination of the economic viability of the process.

2.0. MATERIALS AND METHODS

2.1. Materials

Liquid wastes sample was collected from the reserve pit of Ebocha Oil Field, Agip Oil Company, Omoku, and Rivers State, Nigeria. *Bacillus Stearothermophilus*, thermophilic bacteria was isolated from the liquid wastes and stored in glycerine. Oxoid Anaero Gen TM AN 0035A gas pack was used in a Labtech anaerobic Jar to create anaerobic condition.

2.2. Methods

2.2.1. Microbial Digestion

A tenfold serial dilutionof 1 mL of the liquid wastes sample was carried out using 9 mL of 0.85 % physiological saline after which 0.1 mL of the diluent streaked with 1.5 % nutrient agar was inoculated or cultured in culture plates for growth of micro-organism in anaerobic jar with gas pack and catalyst. This was incubated for 3 days at 75 ºC. Spore test, Gram staining and Sugar fermentation tests were carried out to identify *Bacillus Stearothermophilus*.as in American Public Health Association (APHA) (2001) standard methods.

The *Bacilus Steamothermophilus* was isolated followed by enumeration of colonies on plates. The isolated *Bacilus stearothermophilus* was used to digest the liquid wastes at 75 ºC.

2.2.2. Measurement of Heat Evolved from the Combustion Chamber

The heat of combustion of a sample mixture of hot recycled biogas turbine exhaust gas, air and biogas at 1350 ºC from the combustion chamber of a biogas turbine power plant was measured with the help of a bomb calorimeter. The number of moles was calculated as mass of the hot gas per average molecular mass of the components. The product of the number of moles and the heat of combustion gave the heat evolved from the combustion chamber.

2.2.3 Measurement of Total Hydrocarbon Content and Biochemical Oxygen Demand of the Liquid Wastes.

The Total Hydrocarbon Content and Biochemical Oxygen Demand of the liquid wastes were measured using American Public Health Association (APHA) Standard Methods As in APHA (2001).

2.3. Process Description of the Thermophilic Anaerobiic Liquid Wastes Treatment and Biogas Turbine Power Plant

2.3.1. Thermophilic Anaerobic Liquid Wastes Treatment Plant

The Liquid wastes pretreated in the preliminary and primary treatment unit of the thermophilic anaerobic liquid wastes treatment plant was digested with *Bacillus Stearothermophilus* in an anaerobic reactor to produce biogas in a process involving four basic steps: Hydrolysis, Acidogenesis, Acetogenesis and Methanogenesis. Among these, Hydrolysis was the rate limiting step.

2.3.2. Biogas Upgradation

Impurities in the biogas were removed by Pressure Swing Adsorption (PSA) on activated carbon. Sampson (2019) stated that since adsorption takes place at high temperature and pressure, desorption is achieved by depressurizing. Moisture was removed from the biogas by drying. The active site of the adsorbent retains water vapour and other pollutants thus decreasing adsorbent life, hence desorption is frequently carried out by depressurizing. Moreover, siloxanes are difficult to desorb from the adsorbent beds so adsorbent beds should be replaced regularly e.g. weekly.

The biogas was dried, compressed and sent to the combustion chamber of the biogas turbine power plant via a compressor.

2.3.4. The Biogas Turbine Power Plant: with optimization using Heat Recovery Steam Generator.

The biogas is used in the biogas turbine power plant instead of natural gas as shown in figure 1 below. The hot exhaust gas from the turbine was recycled to the inlet air to the combustion chamber. The mixture of the inlet air and the recycled exhaust gas was ignited. This ignition produced a hot gas which turned a shaft in a 3 - stage gas turbine. Besides Exhaust Gas Recirculation (EGR), economics of heat recovery was enhanced by using some of the exhaust gas in a Heat Recovery Steam Generator (HRSG) to produce steam for a steam turbine. This is called the combined cycle. The combined cycle combines the Rankine cycle (steam Turbine) and the Brayton cycle (Gas Turbine) thermodynamic cycles. The biogas turbine works as a heat engine converting heat energy from hot gas to work energy for turning a shaft in a magnetic field. This is in accordance with Lenz Law which states that an Electromotive Force (EMF) is induced whenever there is a change in magnetic flux linking a circuit. The electricity produced is stepped up by a step up transformer and sent to the various electric substations for onward transmission to various equipment requiring any particular voltage. The biogas turbine power plant is illustrated in figure 1 below.

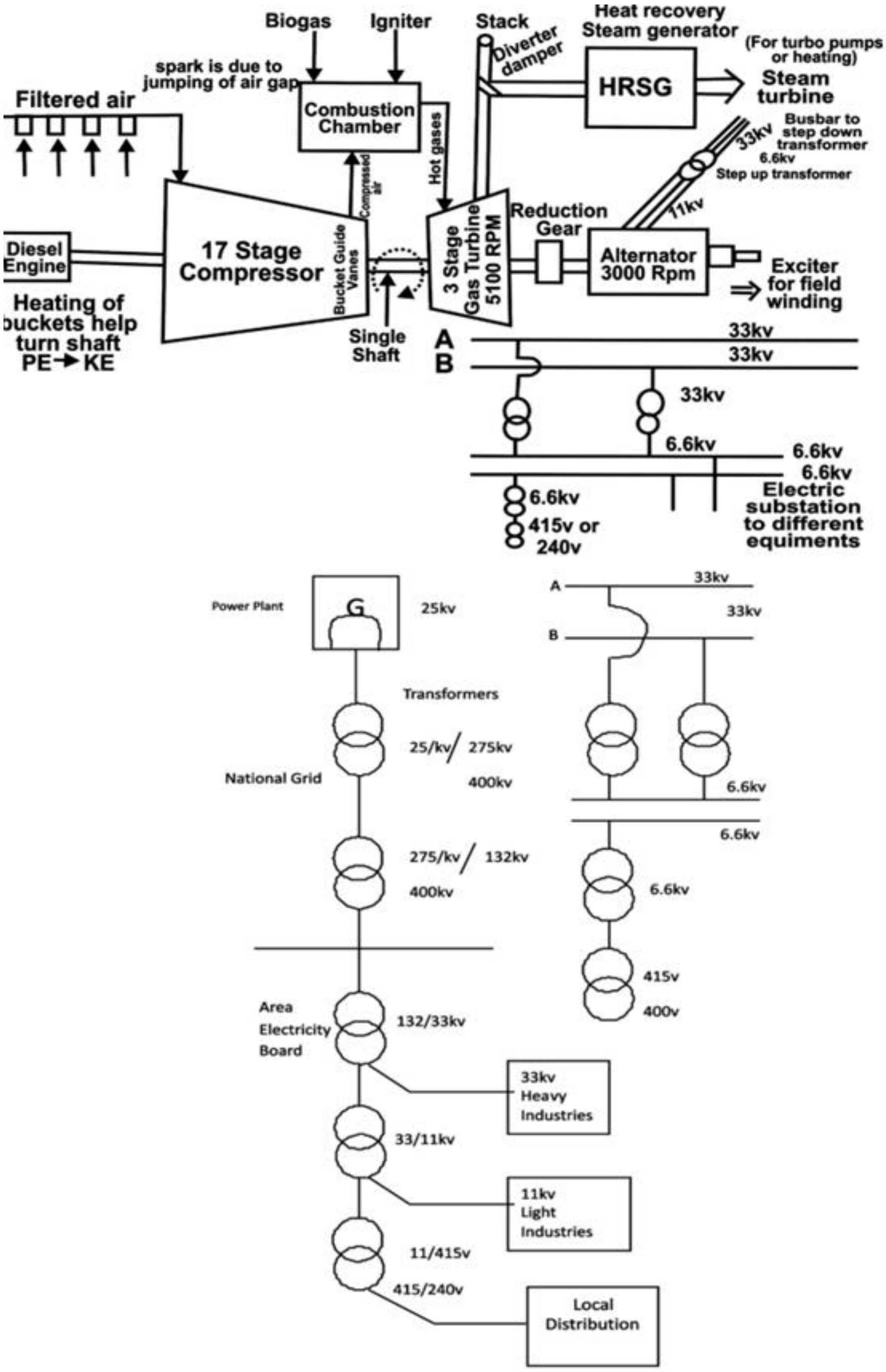

Fig. 1: Electric Power Generation, Transmission and Distribution
Source: Sampson (2018)

.4. Kinetic Models

The following kinetic models were derived in Sampson *et al.* (2021)

a. For the Liquid Wastes:

$$\frac{dC_2}{dt} = \frac{V.(C_{2,0} - C_2)}{V_R} - \frac{\mu_m C_2 C_1}{Y(K_m + C_2)} \qquad (1)$$

b. For the Biomass

$$\frac{dC_1}{dt} = \frac{V}{V_R}\left(C_{1,0} - C_1\right) + \frac{\mu_m C_1 C_2}{K_m + C_2} - k_d C_1 \qquad (2)$$

c. For the volume of the Anaerobic Reactor

$$\mathrm{d}\frac{V_R}{dt} = V_e \times A \qquad (3)$$

These kinetic models integrated using a MATLAB program gave the optimum reactor volume and values of other parameters in the kinetic models where C_2 is the final concentration of the Liquid wastes, $C_{1,}$ the final concentration of the biomass. $C_2,_0$ and $C_1,_0$ the initial concentration of the Liquid wastes and biomass respectively, V, the volumetric feed rate, $V_{R,}$ the reactor volume, Y, the yield coefficient $K_{m,}$ the Monods constant or substrate saturation constant $k_{d,,}$ the coefficient of endogenous respiration or specific maintenance rate $\mu_{m,}$ the maximum specific growth rate or half the maximal velocity concentration $V_{e,,}$ the velocity of the Liquid wastes. A, the cross sectional area of the reactor and t the time.

Volume of biogas produced was obtained from equations in Tchobanoglous *et al.* (2004) as follows:

$$V_{CH4} = (0.35)\left[(s_o - s)(Q)\left(\frac{10^3 g}{kg}\right)^{-1} - 1.42P_x\right] \qquad (4)$$

$$P_x = \frac{\left[YQ(s_0 - s)\left(10^3\ g/kg\right)^{-1}\right]}{[1 + k_d(HRT)]} \qquad (5)$$

Where V_{CH4} is the volume of biogas, S_o and S are the biochemical oxygen demand of the influent and effluent liquid wastes respectively. P_x is the net mass of cell tissue produced per day Q is the flow rate of biogas and HRT is the Hydraulic Retention Time.

Total Hydrocarbon Content (THC) of the Liquid Wastes

$$\begin{bmatrix} Gradient\ of a \\ Standard\ (1:1) \\ Bonny\ Light/Medium \\ Petroluem\ crude\ oil\ versus \\ its\ absorbance \end{bmatrix} \times \begin{bmatrix} Absorbance\ of \\ Liquid\ Wastes \\ sample) \end{bmatrix} \times \begin{bmatrix} Dilution \\ factor \end{bmatrix} \times \frac{\begin{bmatrix} Volume\ of\ Chloroform \\ used \\ for\ extration\ (mL) \end{bmatrix}}{[Volume\ of\ Extract\ (mL)]} \qquad (6)$$

Biochemical Oxygen Demand (BOD) of the Liquid Wastes

$$BOD_5 = \frac{DO_{initial} - DO_{Final}}{DilutionFactor} \qquad (7)$$

Audu (1999) gave equation for finding the heat evolved from a combustion chamber.

$$Heat\ Evolved = [Number\ of\ Moles] \times [Heat\ of\ Combustion] \qquad (8)$$

$$Quantity\ of\ Heat = Average\ Mass\ of\ components \times Average\ specific\ heat\ capacity\ of\ components \times rise\ in\ temperature \qquad (9)$$

$$Number\ of\ Moles = \frac{Mass}{Molar\ Mass} \qquad (10)$$

Therefore, Heat evolved from the combustion chamber

$$\Delta H = [Number\ of\ Moles\ of\ the\ hot\ combustion\ gas] \times [Heat\ of\ combustion\ of\ the\ hot\ combustion\ gas] \qquad (11)$$

$$Volume\ of\ Reactor = Volumetric\ Feed\ Rate \times The\ Hydraulic\ Retention\ Time\ (HRT) \qquad (12)$$

CHECK: The volume of reactor from this equation will be the same as that obtained from the kinetic models.

Validation of Kinetic Models. Using Root Mean Square Error

$$\frac{\sum(C_2,Exp - C_2,model)^2}{n} \qquad (13)$$

Where $\sum$ is summation, Exp, experimental and n the number of values.

Wikipedia (2023) gave equation for conversion of revolutions per minute (rpm) of turbine shaft rotation to metres per second as follows:

$$\frac{m}{s} = (rpm \times 2\pi \times radius)/60 \qquad (14)$$

Internal Energy (u)

$$KE + PE\ in\ the\ molecules\ of\ the\ hot\ gas \qquad (15)$$

$$PE = mgh \qquad (16)$$

$$Work\ done\ (W) = \Delta H - U \qquad (17)$$

$$KE = \frac{1}{2}mV^2 \qquad (18)$$

Number of Power Plants Required

$$\frac{\text{WorkRequired}}{\text{WorkProduced}} \qquad (19)$$

$$Purchased\ Equipment\ Cost\ (PEC) = Market\ price + procurement\ cost \qquad (20)$$

$$Physical\ Plant\ Cost = PEC\ (1 + f_1 + \ldots. + f_9) \qquad (21)$$

$$Fixed\ Capital\ (FC) = PPC\ (1 + f_{10} + f_{11} + f_{12}) \qquad (22)$$

Where

$f_1\ to\ f_{12}\ are\ cost\ factors, MF, Materials\ Factor, PF, Pressure\ Factor.$

$$Working\ Capital\ (WC) = 10\ \%\ of\ Fixed\ Capital \qquad (23)$$

$$\boldsymbol{Number\ of\ kilowatthours}(kwh) = \frac{W\ (Watts)}{1000} \times Number\ of\ hours\ of\ usage \qquad (24)$$

$$\boldsymbol{Sales\ of\ Electric\ Power} = Number\ of\ kilowatts\ \times Number\ of\ hours\ of\ Usage\ \times Cost\ of\ one\ kilowatthour\ \times Number\ of\ people\ that\ paid \qquad (25)$$

$\boldsymbol{Cost\ of\ one\ kilowatthour} =$ ₦59.64 (Nigerian Electric Regulatory Commission (NERC) (2023)) (26)

Net Annual Profit (NAP)

$$\boldsymbol{S}ales\ of\ Electric\ Power - Cost\ of\ Capital - Operating\ cost\ of\ the\ power\ plant - Operating\ cost\ of\ the\ Treatment\ Plant - Fixed\ capital\ of\ the\ Power\ Plant - Fixed\ Capital\ of\ the\ Treatment\ Plant - Sales\ of\ primary\ and\ secondary\ products\ and\ services\ from\ the\ Treatment\ Plant\ that\ was\ used\ to\ operate\ and\ manage\ the\ power\ plant. \qquad (27)$$

Cost of Capital

$$\boldsymbol{Cost\ of\ sourcing\ for\ capital} + the\ bank\ charges\ paid + the\ bank\ interest\ paid\ on\ the\ principal\ capital\ that\ was\ borrowed \qquad (28)$$

$\boldsymbol{Total\ Investment\ (TI)}$

$$Fixed\ Capital\ of\ the\ power\ plant + Fixed\ Capital\ of\ the\ Treatment\ plant + Working\ capital\ of\ the\ power\ plant + Working\ capital\ of\ the\ Treatment\ plant \qquad (29)$$

$$Working\ Capital\ (WC)\ 10\ \%\ \ of\ Fixed\ Capital \qquad (30)$$

$\boldsymbol{Operating\ Cost\ (OPC)}$

$$Fixed\ Cost + Variable\ Cost \qquad (31)$$

$\boldsymbol{Direct\ Production\ Cost}$

$$[Operating\ cost\ of\ the\ power\ plant + Operating\ cost\ of\ the\ Treatment\ plant] + 20\ \%\ of\ [\ the\ operating\ cost\ of\ the\ power\ plant + Operating\ cost\ of\ the\ Treatment\ plant] \qquad (32)$$

$\boldsymbol{Total\ Cost\ (TC)}$

$$Total\ Investment + Direct\ Production\ Cost \qquad (33)$$

Economic Analysis

$Net\ Present\ Value$ (NPV)

$$\frac{NAP \times N}{(1 + r)^n} \qquad (34)$$

$$\boldsymbol{Viability\ Test: NPV - TI}\ > 0$$

$\boldsymbol{Pay - Back\ Period\ (PBP)}$

$$\frac{Total\ Investment}{Net\ Annual\ \ Profit} \qquad (35)$$

$\boldsymbol{Rate\ of\ Return\ on\ Investment\ (ROI)}$

$$\frac{\boldsymbol{Net\ Annual\ Profit}}{\boldsymbol{Total\ Investment}} \times \frac{\boldsymbol{100}}{\boldsymbol{1}} \qquad (36)$$

3.0. RESULTS AND DISCUSSION

3.1. The Plant Capacity

Sampson (2022) stated that the plant capacity of the thermophilic anaerobic liquid wastes treatment plant was chosen to be 5 m^3 per day of liquid wastes based on information from industries that the thermophilic bacteria, *Bacilus Stearo thermophilus* can digest 5 m^3 of liquid wastes in one day.

3.2. Biochemical Test Results for *Bacilus Stearothermophilus*

Endospores stained green on spore test. Purple stain formed on grain staining. Acid was produced and colour changed from red to yellow on sugar fermentation test. Gas was produced in space in the durhan tubes.

3.3. Federal Environmental Protection Agency (FEPA) Discharge Limits

Optimization of liquid wastes treatment was enhanced by thermophilic temperature (75 ^{O}C) and optimum hydraulic retention time of 5 days, the time taken to attain FEPA discharge limits as illustrated in table 1 below.

Table 1: Initial Values and Final Values of Biochemical Oxygen Demand and Total Hydrocarbon Content
FEPA discharge limits taken from: Nigerian Federal Environmental Protection Agency (2002)
FEPA discharge limits taken from: Nigerian Federal Environmental Protection Agency (2002)

Parameter	Initial Value mg/L	Final Value mg/L	FEPA Discharge Limitsmg/L	Remarks
Biochemical Oxygen Demand	2,784.0	10. 0	10.0	Thermophilic anaerobic digestion of the liquid wastes destroyed the Biochemical Oxygen Demand (BOD) thus making more oxygen available for life in the aquatic environment.
Total Hydrocarbon Content	23.53	10. 0	10.0	Thermophilic anaerobic digestion of the liquid wastes removed hydrocarbons which makes the waste toxic to aquatic life and carcinogenic to man to an acceptable limit.

The liquid wastes were treated to Nigerian Environmental Protection Agency (FEPA) discharge limits. The results show that reaction at thermophilic temperature enhances higher reaction rates hence more liquid wastes were treated to FEPA discharge limits in a short period of 5 days.

3.4. Optimum Diameter of the Anaerobic reactor

Table 2 shows the diameter of the anaerobic reactor as obtained by optimization at the purchased equipment cost of the reactor.

Table 2: Optimisation of Anaerobic Reactor Diameter

Diameter (m)	Purchased Equipment Cost In British Pounds (£)
00	
0.5	200551988
1.0	896.63872
1.5	5649.902996
2.0	7573.42196
2.5	9383.5404

PEC of the anaerobic reactor in the 2023 = £ 8,345.466426

Interpolation for the diameter of the anaerobic reactor at £ 8,345.466426 gave 2.20 meters ≅ 220 centimeters. This is the optimum diameter of the anaerobic reactor.

3.5. Volume of the Anaerobic Reactor

Table 3 shows the optimum volume of the anaerobic reactor by optimization at the time taken to attain the Federal Environmental Protection Agency (FEPA) discharge limits.

Table 3: Volume of Anaerobic Reactor, Concentration of Liquid Wastes and Concentration of Biomass using MATLAB Software Program

Time (t) (days)	Volume of Liquid Wastes filled into the Anaerobic Reactor (V_R) m^3	Concentration of Liquid Wastes (C_2) (mg/L)	Concentration of Biomass (C_1) (mg/L)
0	0.0015	1.000	0.000015
1	5.0015	0.0014	0.00141
2	10.0015	0.0008	0.00135
3	15.0015	0.0005	0.00130
4	20.0015	0.0004	0.00125
5	25.0015	0.0003	0.00121

Integration of the Kinetic Models using a MATLAB program gave 25 m^3 as the optimum Volume of the anaerobic reactor. This was checked manually:

$$Volume\ of\ the\ Anaerobic\ Reactor = Volumetric\ feed\ rate\ \times Time$$

$$5\ m^3/day\ \times 5\ days = 25\ m^3$$

This is the volume of the reactor at day 5 when FEPA discharge limits were attained

3.6. Values of other Parameters as obtained from the MATLAB Integration

Experimental values of other parameters fed into the MATLAB Program gave the following results:

$C_{1,0}$ = 0.000015 mg/L	C_1= 0.00121 mg/L
$C_{2,0}$ =1.0000 mg/L	C_2= 0.0003 mg/L

3.7. Validation of Kinetic Models

A Root Mean Square Error (RMSE) of 0.85 shows that 85 % of the variability in the data set was explained by the Kinetics models.

3.8. Volume of Biogas

Substantial volume of biogas was produced from thermophilic anaerobic treatment of the Liquid Wastes as illustrated on Table 4 below.

Table 4: Volume of Biogas Produced

Parameter	Value
Influent BOD	2,784.0 mg/L
Effluent BOD	10.0 mg/L
Flowrate of Biogas	5.0 m^3/day
Net mass of cell tissue produced per day	0.24 kg/day
Volume of Biogas Produced	4,513.7 Litres/day (4.5137 m^3/day)

3.9. Generation of Energy

Energy substantial enough to make – up for the Nigerian energy deficit was produced from the biogas turbine power plant as illustrated on Table 5 below.

Table 5: Energy Generation

Parameter	Values
Heat evolved (ΔH)	6785.5953 kJ
Shaft speed	23.565 ms^{-1}
Kinetic Energy	4.442498 kJ
Work done	6,781,152.826 Watts Or 6,781.152826 kilowatts
Energy Requirement	30,000 MW
Number of Power Plants Required	4,424

The number of power plants can be substantially reduced by scaling-up the design but small plants are easier to maintain than big ones. 4,424 grid-tied biogas turbine power plants of 6,781.15286 kilowatts capacity evenly distributed in Nigeria can generate the 30,000 Megawatts of electric power required in Nigeria as estimated by ANED & DisCos (2022)

3.10. Costing

Cost analysis to enable the Nigerian Government and the private investors to determine whether the investment is a profitable one so as to source for capital and set – up the plant is illustrated on Table 6 below.

Table 6: Cost Analysis

Parameter	Values in Nigerian Naira(₦)
Purchased Equipment Cost	1,448,883,582
Physical Plant Cost	1,579,283,104.00
Fixed Capital	1,674,040,090.00
Working Capital	167,404,009.00
Number of kilowatt hours	20,343,458.48
Sales of electric power	606,641,931,800.00
Net Annual Profit	600,405,586.40
Cost of Capital	3,294,180,694.00
Total Investment	1,841,444,099.00
Fixed Cost	422,719,909.70
Variable Cost	845,439,819.30
Operating Cost	1,268,159,729.00
Direct Production Cost	1,521,791,675.00
Total Cost	3,363,235,774.00

High Profitability rate is expected as the sales of electric power ₦606, 641,931,800.00) is higher than the total cost of the plant (₦3, 363,235,774.00). A net annual profit of ₦600, 405,586.40 shows that high profit could be made from the investment. A total investment of ₦1, 841,444,099 is quite low.

3.11. Economic Evaluation

Economic analysis to enable the Nigerian Government and the Private investors determine whether the investment is economically viable so as to source for capital and set – up the plant is illustrated on Table 7 below.

Table 7: Economic Analysis

Parameter	Value
Net Present Value	₦6,648,600,317.00
Total Investment	₦1,841,444,099.00
NPV-TI	₦4,807,156,218.00
Pay-Back Period	3 years, 3 weeks
Rate of Return on Investment	32.61%

The plant is economically viable as the difference between the Net Present Value (NPV) and the Total Investment (TI) is greater than zero as in Anyanwu (1994). A pay-back period of 3 years, 3 weeks is low enough. This shows that the loan taken to build the plant could be repaid in a short period of 3 years and 3 weeks. A rate of return on investment of 32.61 % is quite reasonable.

4.0. CONCLUSION

Thermophilic anaerobic Liquid wastes treatment helps avoid accumulation of liquid wastes which causes environmental disaster as thermophilic temperature enhances higher reaction rates, increases death rate of pathogens and hence more liquid wastes can be treated in a shorter time. The biogas produced has less carbon hence carbon emissions could be eliminated by optimization of the biogas turbine power plant and thermophilic anaerobic liquid wastes treatment plant. This could help Nigeria realize her quest for zero carbon emissions by the year 2060. A biogas turbine power plant with a thermophilic anaerobic liquid wastes treatment plant having a pay-back period of 3 years, 3 weeks and a rate of return of 32.61 % and a net annual profit of ₦600, 405,586.00 if well managed, could be economically viable in Nigeria. The Nigerian Government and the private investors are therefore encouraged to source for capital and set-up the plant as much profit could be made from the investment.

5.0. RECOMMENDATIONS TO THE NIGERIAN GOVERNMENT

The following recommendations are made:

i. The Nigerian Government should set-up the thermophilic anaerobic Liquid wastes treatment plants with the biogas turbine power plants evenly distributed in Nigeria to help generate energy which shall make-up for the Nigerian energy deficit.
ii. The Nigerian government should promulgate legislative act against incineration of liquid wastes in Nigerian industries as treating the liquid wastes in thermophilic anaerobic liquid wastes treatment plants could enable Nigeria achieve zero gas flaring by the year 2060.
iii. The Nigerian Government should maximize and upgrade the production of biogas so that biogas can substitute natural gas as national and domestic fuel source being a renewable energy source with less carbon, natural gas reserved solely for exports.

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MODELLING AND SIMULATION OF A CYCLIC STEAM STIMULATION SYSTEM

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ABSTRACT

Cyclic steam stimulation (CSS) is a common thermal recovery technology used to remove heavy oil and bitumen from oil reservoirs. This research aimed to simulate and model an enhanced oil recovery method, particularly the thermal method of enhanced oil recovery using cyclic steam stimulation, and evaluate its effectiveness in enhancing oil recovery. To improve recovery efficiency, the study emphasized the importance of precise reservoir characterization, optimal steam injection procedures, and reservoir management techniques. The findings help to progress CSS technology by allowing operators to make more informed decisions and increase the economic viability of heavy oil and bitumen production. This research focused on the simulation and modelling of cyclic steam stimulation with Computer modelling group (CMG) software, a strong reservoir engineering tool widely used in the oil and gas sector. The base case has a cumulative oil value of 9,000 m^3 and an oil recovery factor of 5.1 SCTR or 42.5%, while the cyclic steam has a cumulative oil value of 20,500 m^3 and an oil recovery factor of 11.8 SCTR or 98.3%. CMG for thermal enhanced oil recovery, like cyclic steam simulation, saves cost and time, providing pre-process replicas, and helping oil companies plan optimal recovery methods.

Keywords: Cyclic steam, simulation, CMG, thermal recovery, reservoir.

1.0 INTRODUCTION

Enhanced oil recovery (EOR), also called tertiary recovery, is the extraction of crude oil from an oil field that cannot be extracted otherwise. EOR can extract 30% to 60% or more of a reservoir's oil compared to 20% to 40% using primary and secondary recovery (Walsh and Lake, 2003). Carbon dioxide and water are injected along with one of three EOR techniques: thermal injection, gas injection, and chemical injection. More advanced, speculative EOR techniques are sometimes called quaternary recovery (Wang *et al*., 2015).

Conventional recovery methods (primary and secondary) typically extract approximately one-third of the original oil in place in a reservoir. Estimates (made in the late 1970s) of worldwide oil-in-place range up to 1.5 trillion barrels; using that figure, it is estimated that the oil remaining as a residual oil saturation after conventional recovery would be approximately 1.0 trillion barrels. Several enhanced oil recovery (EOR) techniques generally grouped as tertiary production schemes have targeted this huge unexploited reserve.

The oil reservoir's recovery process involves three phases: primary, secondary and tertiary recovery. During the primary recovery phase, oil is produced due to natural reservoir energy (compaction drive, solution gas drive, water drive, gas cap drive, gravity drive) with recovery factors of 10-30%. Secondary recovery methods are water or gas injection for reservoir pressure maintenance and/or production increase. The additional recovery factor due to the application of these methods is 10-20%. The average recovery factor of currently producing oil reservoirs in the world is about 35-40% (Bonder, 2010).

There are three methods of enhanced oil recovery, which are: Gas injection, thermal injection and chemical injection. This research work focused mainly on the thermal injection method. In this approach, various methods are used to heat the crude oil in the formation to reduce its viscosity and/or vaporize part of the oil and thus decrease the mobility ratio. The increased heat reduces the surface tension and increases the permeability of the oil. The heated oil may also vaporize and then condense forming improved oil. Methods include cyclic steam injection, steam flooding and combustion. These methods improve the sweep efficiency and the displacement efficiency (Wu *et al.,* 2019). Steam injection, an enhanced oil recovery (EOR) method since the 1960s, is used for extracting heavy crude oil. It includes cyclic steam stimulation and steam flooding as key approaches (Zerkalov, 2015).

Sun and Ertekin (2015) reviewed the development of artificial neural network (ANN)-based proxy models for studying steam-assisted gravity drainage (SAGD) and cyclic steam stimulation (CSS) procedures, considering rock and fluid properties, aiming to predict oil flow rate, cumulative production, and the number of CSS cycles, thus offering a fast estimation tool for recovery in heavy oil reservoirs and potentially assisting in optimization and sensitivity analyses. Sun and Ertekin (2017) focused on developing a robust artificial neural network (ANN) surrogate model for cyclic steam stimulation (CSS). The ANN model, incorporating rock-fluid properties and cycle-switching schemes, was trained using an optimized network topology workflow to understand complex data structures in CSS processes, enabling its use as a classification and regression tool for process design and optimization. The model's validation against high-fidelity simulations showcased its potential as a powerful screening and design tool for global CSS process optimization.

Ersahin and Ertekin (2020) introduced three artificial neural network (ANN) models designed to efficiently evaluate cyclic steam injection (CSI) performance for enhanced oil recovery (EOR) in heavy oil reservoirs, to provide accurate and rapid results compared to time-consuming and costly commercial software. The ANN models, trained with various topologies and transfer functions using a dataset of 1,428 cases, are capable of mimicking commonly used numerical models and predicting performance indicators, viscosity contours, CSI design parameters, and reservoir properties while offering results within seconds as opposed to the commercial software's extended computational time. The user-friendly graphical user interface (GUI) of the ANN-based models further reduces the time required for software learning and execution.

Sun and Ertekin concentrated on precise ANN predictions for heavy oil recovery, while Ersahin and Ertekin prioritized speed, efficiency, and user-friendly interfaces for a comprehensive evaluation of cyclic steam injection (CSI). This study centres on simulating and modelling thermal enhanced oil recovery through cyclic steam stimulation and assessing its impact on improving oil recovery.

2.0 METHODOLOGY

2.1 Process Software

Computer Modelling Group (CMG) is a software business that develops reservoir modelling software for the oil and gas sector. CMG remains focused on the development and delivery of reservoir simulation technologies to assist oil and gas companies in determining reservoir capacities and maximizing potential recovery. Figure 1 shows the components of CMG.

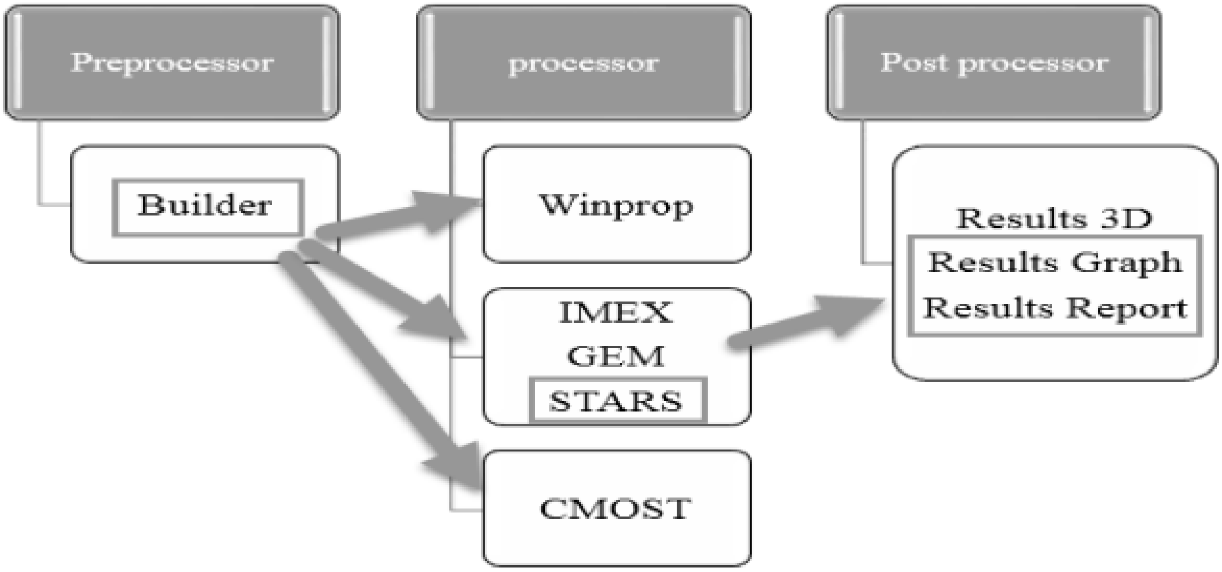


Figure 1: CMG Components (CMG template)

a) STARS (thermal process reservoir simulator)
STARS is a reservoir simulation tool equipped with thermal, k-value (KV), compositional, chemical reaction, and geomechanical capabilities, making it suitable for advanced modelling of recovery processes, such as steam injection, solvent injection, air injection, and chemical treatments. It stands out as the most comprehensive and versatile reservoir modelling solution available, thanks to its robust response kinetics and geomechanical functionalities (Maurya *et al*., 2017).

2.2 Workflow of the Research

Figure 2 shows the workflow of the research carried out in the CMG software.

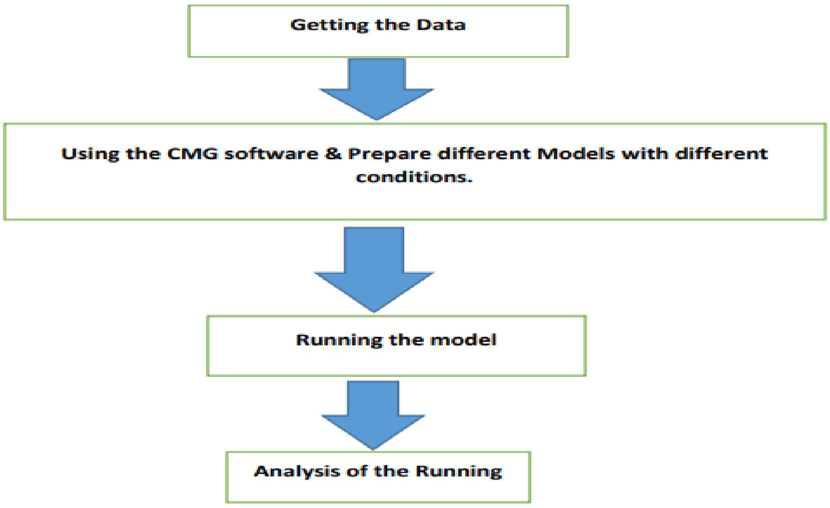
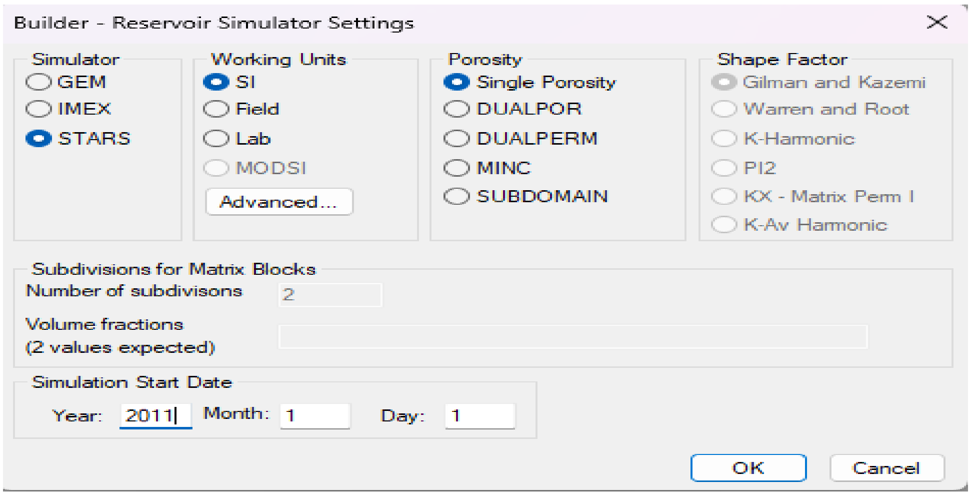


Figure 2: Methodology Flow Diagram

2.3 Simulation Process

- **Selection of simulator**

Figure 3 shows the reservoir simulator settings. The simulator selected was STARS, with a single porosity.


Builder - Reservoir Simulator Settings

Simulator: ○ GEM ○ IMEX ● STARS

Working Units: ● SI ○ Field ○ Lab ○ MODSI

Porosity: ● Single Porosity ○ DUALPOR ○ DUALPERM ○ MINC ○ SUBDOMAIN

Shape Factor: ● Gilman and Kazemi ○ Warren and Root ○ K-Harmonic ○ PI2 ○ KX - Matrix Perm I ○ K-Av Harmonic

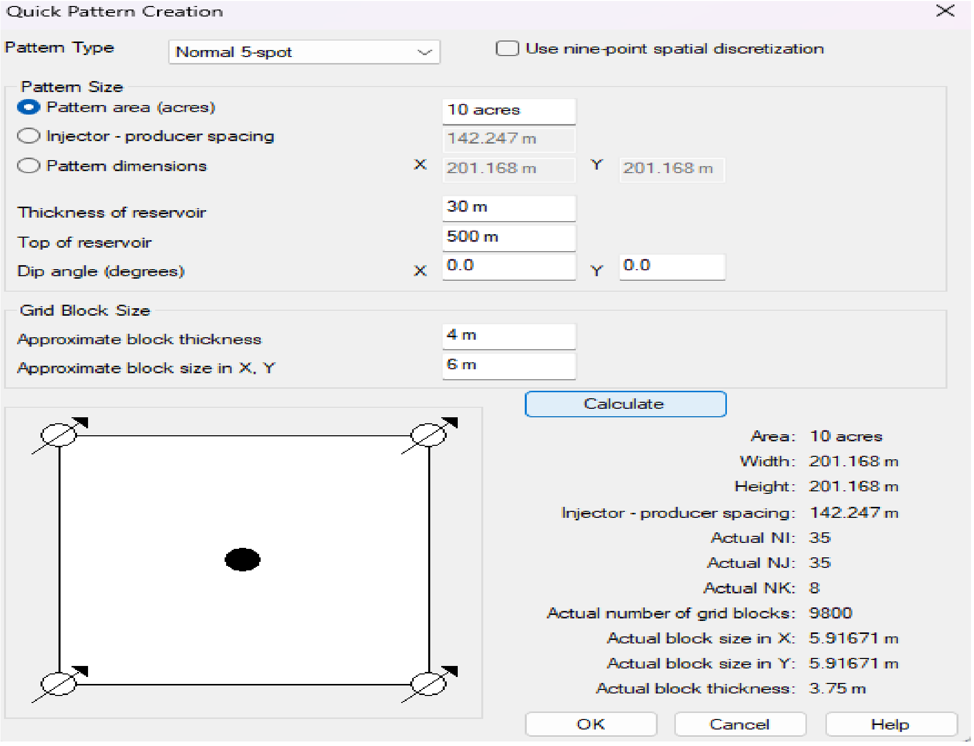
Subdivisions for Matrix Blocks
Number of subdivisons: 2
Volume fractions (2 values expected):

Simulation Start Date
Year: 2011 Month: 1 Day: 1

Figure 3: Simulator Settings

- **Grid creation**

Figure 4 presents the grid creation values. The pattern size, thickness of the reservoir, and pattern type are selected.



Quick Pattern Creation

Pattern Type: Normal 5-spot
☐ Use nine-point spatial discretization

Pattern Size
● Pattern area (acres): 10 acres
○ Injector - producer spacing: 142.247 m
○ Pattern dimensions: X 201.168 m Y 201.168 m

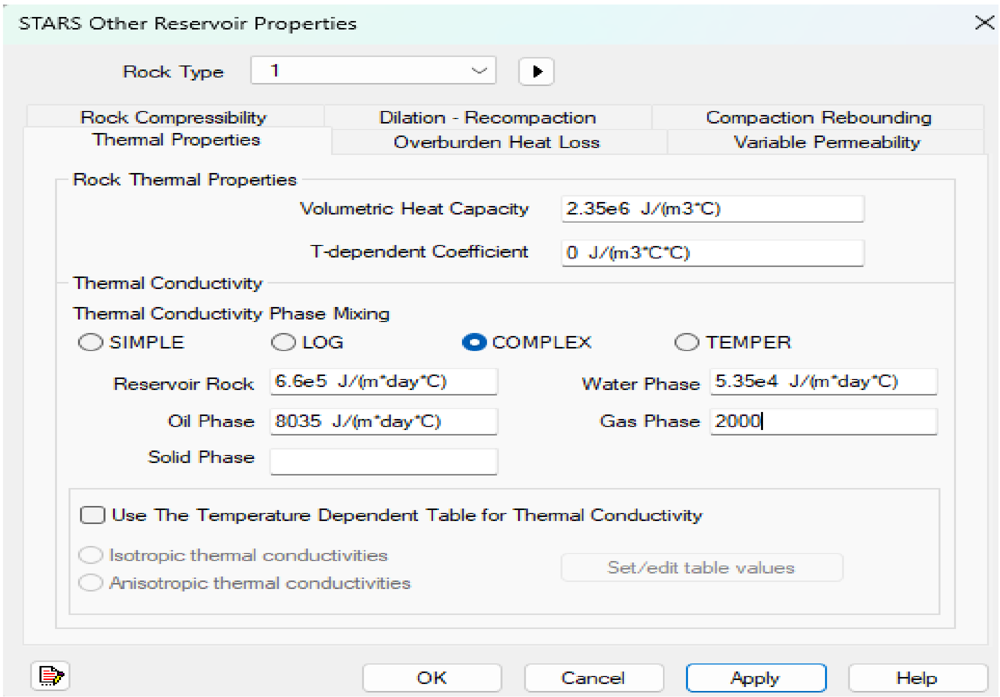
Thickness of reservoir: 30 m
Top of reservoir: 500 m
Dip angle (degrees): X 0.0 Y 0.0

Grid Block Size
Approximate block thickness: 4 m
Approximate block size in X, Y: 6 m

Area: 10 acres
Width: 201.168 m
Height: 201.168 m
Injector - producer spacing: 142.247 m
Actual NI: 35
Actual NJ: 35
Actual NK: 8
Actual number of grid blocks: 9800
Actual block size in X: 5.91671 m
Actual block size in Y: 5.91671 m
Actual block thickness: 3.75 m

Figure 4: Grid Creation Values

Other reservoir properties are selected as shown in figure 5. The rock type, thermal properties and thermal conductivity were selected.



STARS Other Reservoir Properties

Rock Type: 1

Rock Thermal Properties
Volumetric Heat Capacity: 2.35e6 J/(m3*C)
T-dependent Coefficient: 0 J/(m3*C*C)

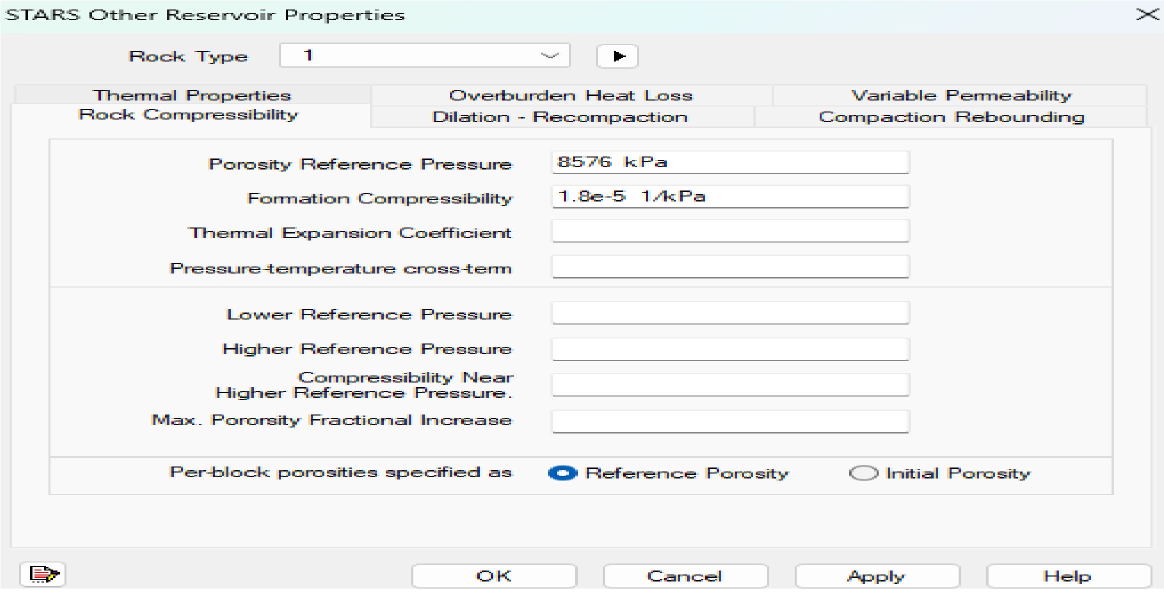
Thermal Conductivity
Thermal Conductivity Phase Mixing
○ SIMPLE ○ LOG ● COMPLEX ○ TEMPER

Reservoir Rock: 6.6e5 J/(m*day*C)
Water Phase: 5.35e4 J/(m*day*C)
Oil Phase: 8035 J/(m*day*C)
Gas Phase: 2000
Solid Phase:

☐ Use The Temperature Dependent Table for Thermal Conductivity
○ Isotropic thermal conductivities
○ Anisotropic thermal conductivities

Figure 5: Thermal Properties

Figure 6 shows the rock compressibility properties selected.



Rock Type: 1

Porosity Reference Pressure: 8576 kPa

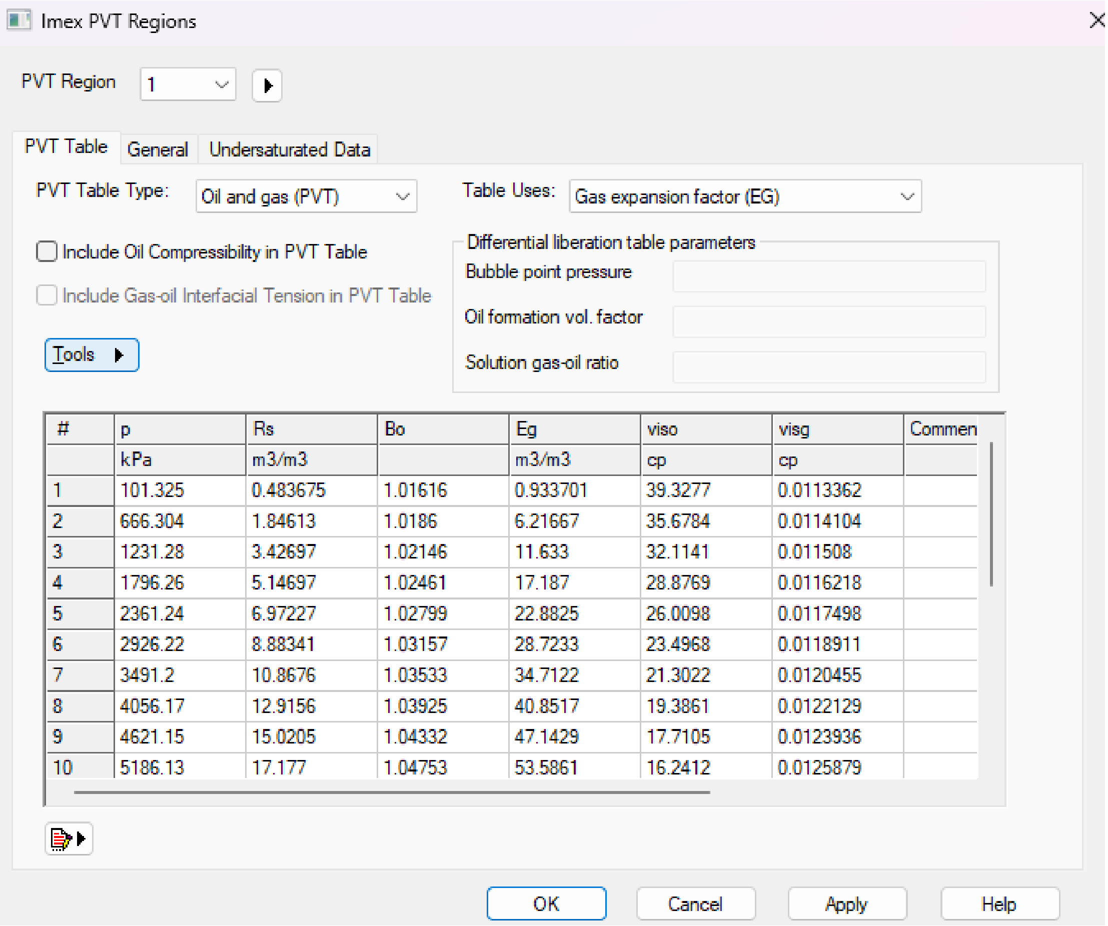
Formation Compressibility: 1.8e-5 1/kPa

Per-block porosities specified as ● Reference Porosity ○ Initial Porosity

Figure 6: Rock Compressibility

- **PVT table**

The Imex PVT regions are presented in Figure 7. The PVT table Type chosen was oil and gas (PVT).



PVT Region: 1

PVT Table Type: Oil and gas (PVT)

Table Uses: Gas expansion factor (EG)

☐ Include Oil Compressibility in PVT Table

☐ Include Gas-oil Interfacial Tension in PVT Table

#	p	Rs	Bo	Eg	viso	visg	Commen
	kPa	m3/m3		m3/m3	cp	cp	
1	101.325	0.483675	1.01616	0.933701	39.3277	0.0113362	
2	666.304	1.84613	1.0186	6.21667	35.6784	0.0114104	
3	1231.28	3.42697	1.02146	11.633	32.1141	0.011508	
4	1796.26	5.14697	1.02461	17.187	28.8769	0.0116218	
5	2361.24	6.97227	1.02799	22.8825	26.0098	0.0117498	
6	2926.22	8.88341	1.03157	28.7233	23.4968	0.0118911	
7	3491.2	10.8676	1.03533	34.7122	21.3022	0.0120455	
8	4056.17	12.9156	1.03925	40.8517	19.3861	0.0122129	
9	4621.15	15.0205	1.04332	47.1429	17.7105	0.0123936	
10	5186.13	17.177	1.04753	53.5861	16.2412	0.0125879	

Figure 7: PVT Table

3.0 RESULTS AND DISCUSSION

3.1 Base Case Analysis

Higher injection pressure and rate are needed to achieve a better recovery. Furthermore, the passage of time exerts a substantial influence on both oil production and the recovery factor in cyclic steam injection processes, underscoring the need for optimization. The production profile can be segmented into three primary phases. In the initial stage, oil production experiences a rapid surge as a result of the swift heating of the oil through the sudden injection of steam, a decrease in oil viscosity, and a partial infiltration of steam (Kamari *et al.*, 2015).

Figure 8 shows the relationship between the oil-water relative permeability of the model at its lowest temperature (37.7778 ⁰C) and its highest temperature (325 ⁰C). The graph illustrates the relationship between water-oil relative permeability and water saturation in a reservoir rock. Relative permeability measures fluid flow ease relative to water, which serves as a reference. As water saturation increases, oil's relative permeability decreases, while water's relative permeability rises, albeit at a slower rate. A critical point exists at 100% water saturation, where oil's relative permeability becomes zero, signifying no oil flow.

Understanding this critical saturation is vital in reservoir engineering for estimating recoverable oil volume. The graph also shows that oil has consistently higher relative permeability than water, attributed to oil's stronger attraction to pore surfaces as the non-wetting phase. This information aids in predicting oil and water flow rates within the reservoir. Notably, oil's relative permeability decreases faster than water as water saturation increases, especially when transitioning from larger to smaller pores. Additionally, water's relative permeability plateaus around 60% water saturation, indicating its maximum flow rate at that level.

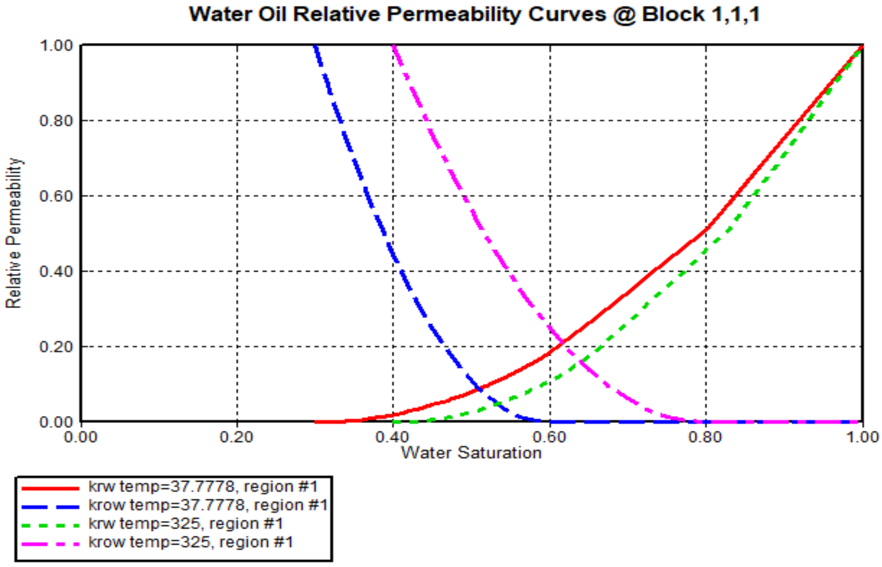


Figure 8: Oil-Water Relative Permeability of the Model

Figure 9 shows the relationship between the gas-oil relative permeability of the model at its lowest temperature (37.7778 ⁰C) and its highest temperature (325 ⁰C). The graph provided illustrates the relative permeability of gas and oil concerning liquid saturation. The red line represents gas permeability (k_{rg}), while the blue line represents oil permeability (k_{ro}).

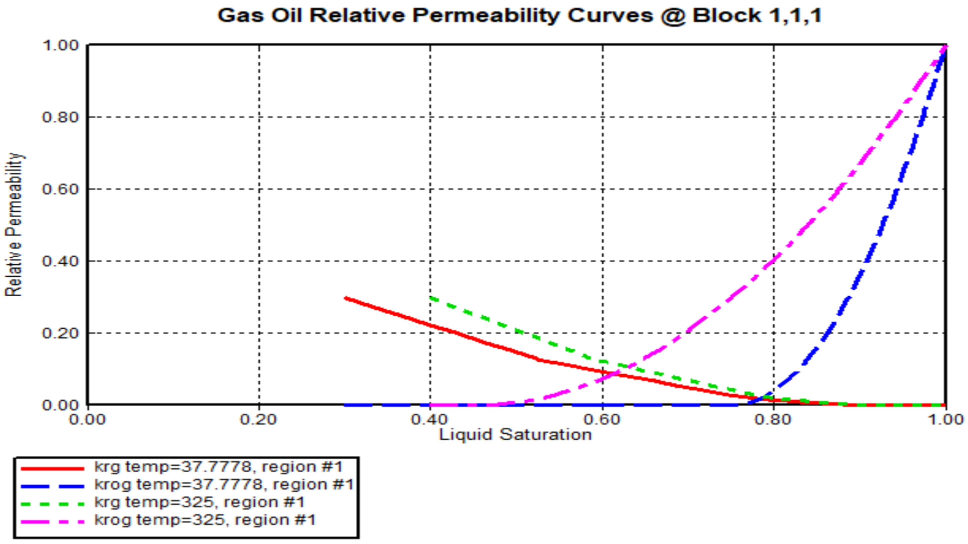


Figure 9: Gas-Oil Relative Permeability of the Model

Gas permeability increases as liquid saturation decreases, whereas oil permeability decreases with declining liquid saturation due to competition for pore space in the rock. Gas relative permeability increases with liquid saturation, displacing oil from larger pores, while oil relative permeability decreases. Gas relative permeability reaches its maximum at a liquid saturation of approximately 0.6 when it displaces oil from larger pores and flows through smaller pores with lower permeability.

Gas permeability is consistently lower than oil permeability at the same liquid saturation because oil has a stronger affinity for the rock surface. Specific observations from the graph include a residual oil saturation to gas (S_{or}) of around 40%, a critical gas saturation (S_{gc}) of about 10%, and relative permeability of gas at the critical gas saturation (k_{rogc}) of approximately 0.1. Conversely, oil relative permeability reaches its minimum at a liquid saturation of approximately 0.4, as it gets displaced from larger pores and flows through smaller pores. At a liquid saturation of 1 (complete liquid), gas relative permeability is zero, and oil relative permeability is one because gas cannot flow in fully saturated porous media. Temperature variation (37.7778 °C and 325 °C) suggests that the relative permeability curves may change with reservoir temperature.

This graph can be used to predict gas and oil flow patterns in a reservoir, calculate gas-oil ratios (GOR) at wellheads, production rates of gas and oil, and design gas injection projects to enhance oil recovery. The graph also highlights that gas dominates at high gas saturations due to its lower viscosity and lower susceptibility to capillary forces, whereas oil dominates at low gas saturations. The wettability of the reservoir affects the shape of the relative permeability curves, with oil as the wetting phase in a water-wet reservoir, causing it to coat rock surfaces

while gas flows through larger pores (Baban *et al.*, 2022).

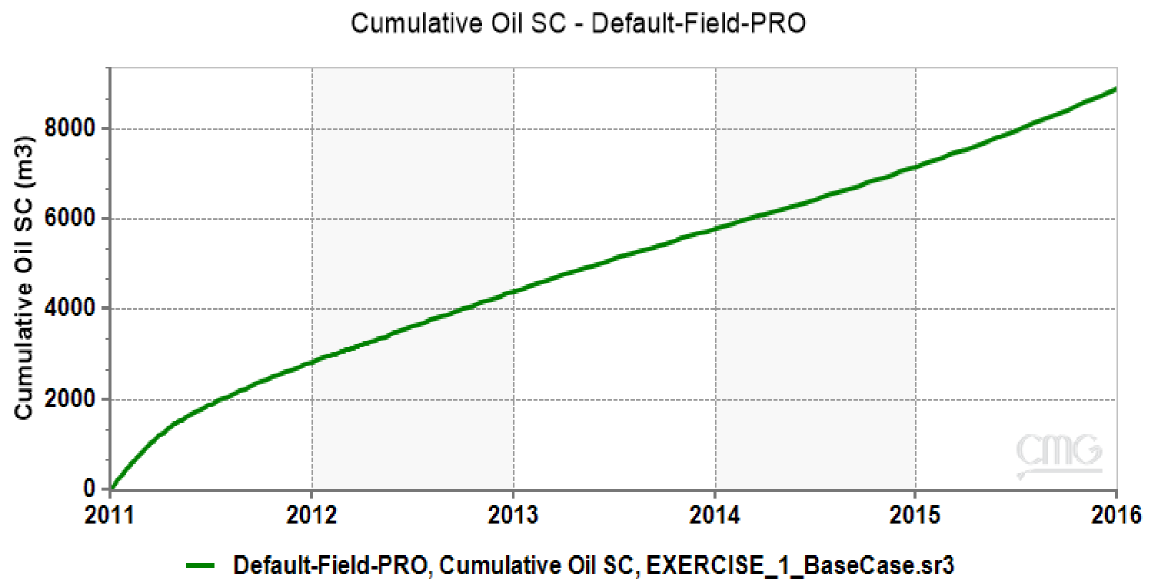


Figure 10: Cumulative Oil Curve

Figure 10 shows the cumulative oil recovery of the reservoir over time from 2011 to 2016. The cumulative oil volume increases proportionally with an increase in time. With the maximum value being slightly over 8500 m^3 over 5 years. The graph illustrates a consistent and upward trend in cumulative oil production over the years, indicating that the oil field is actively producing oil. Each year, the oil field produces more oil than the previous year, suggesting sustained growth in production. The cumulative oil production is still relatively low, indicating that the oil field is likely in its early production stages. The steady and increasing trend implies the oil field has the potential to yield a significant amount of oil in the future.

It is crucial to note that the graph does not predict future production levels, as it depends on factors such as reservoir size, characteristics, and production methods. The increase in cumulative oil production from 2011 to 2016 was around 6500 m^3. On average, the annual increase in cumulative oil production from 2011 to 2016 was approximately 1000 m^3.

In summary, the graph illustrates a positive trend in cumulative oil production for the given field, indicating potential for future oil extraction. Overall, the cumulative oil SC graph shows that the oil field is producing oil and that the cumulative oil production is increasing at a steady rate. This suggests that the oil field has the potential to produce a significant amount of oil in the future. However, the rate of increase is expected to slow down as production progresses.

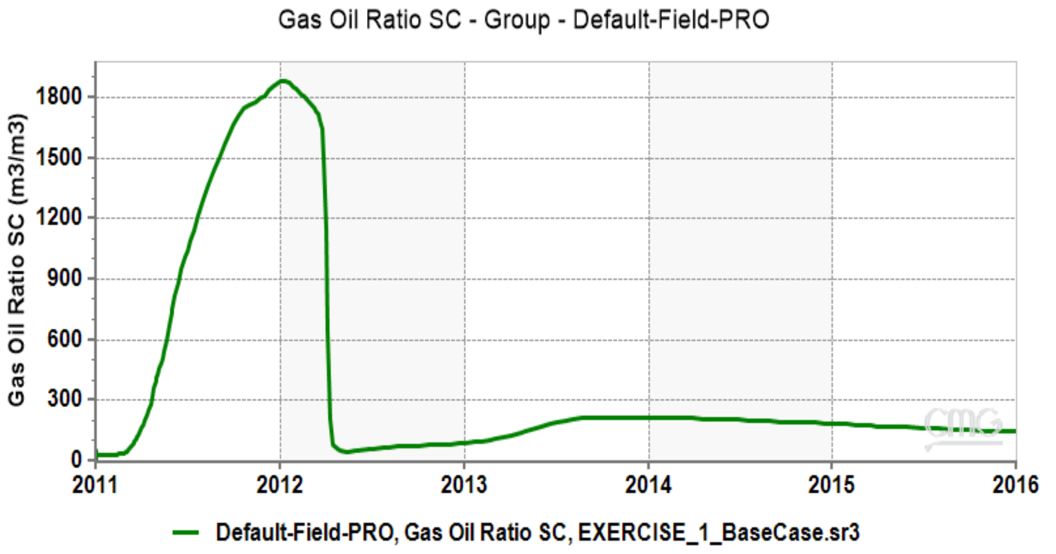


Figure 11: Gas Oil Ratio Curve

Figure 11 displays average Gas-Oil Ratio (GOR) data from 2011 to 2016, measuring the gas volume produced per unit of oil. GOR has steadily increased, suggesting either rising gas concentration in the reservoir or oil depletion. Specifically, GOR declined from about 1800 m^3/m^3 in 2012 to around 200 m^3/m^3 in 2016, an 86.67% decrease. The current GOR is below the global average, indicating greater depletion. The declining GOR implies reduced gas production relative to oil, potentially lowering revenue and complicating oil extraction due to reduced gas pressure assistance.

To address this decline, the operator may need Enhanced Oil Recovery (EOR) methods like waterflooding or gas injection. The increase in GOR may result from natural gas content, water infiltration, or gas injection. From the graph, it was deduced that the highest GOR in 2016 indicates an accelerating gas concentration increase. The lowest GOR in 2011 signifies a slower gas content rise. GOR remained relatively constant from 2012 to 2014, indicating a steady gas content increase.

In summary, the graph portrays a gradual GOR rise with annual variations. This data has critical implications for reservoir management, impacting decisions like optimal well shutdown timing and secondary recovery strategies.

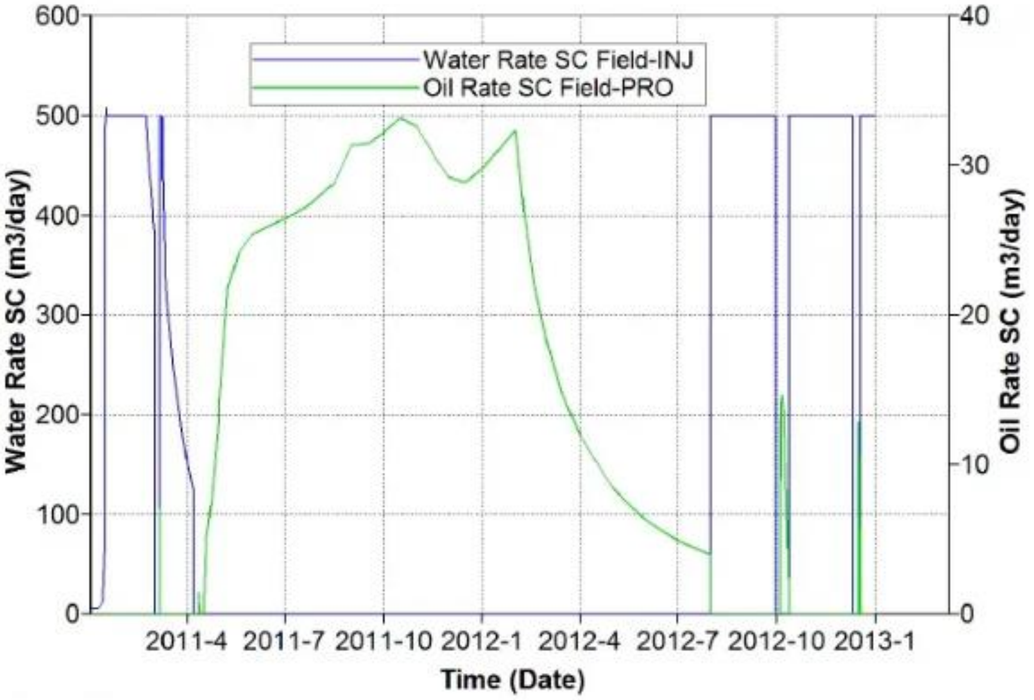


Figure 12: Water Rate Curve Vs. Oil Rate Curve

The figure illustrates water and oil rates for the SC field-NV and SC field-PRO, with water rate in blue and oil rate in red on the y-axis against time on the x-axis. Figure 12 shows the relationship between the water cut of the injection wells and the oil rate of the producer wells of the model for the first 2 years. It is observed how the datasets start with the injection for some time, soak, then produced either for the given 730 days (2 years) or until the rate drops below the minimum oil rate for switching which was set (for Groups 1 - 4; this is 0.5 m^3/day).

Generally, water rates are higher than oil rates, indicating a correlation between the two, likely due to water injection for pressure maintenance and production enhancement. However, both oil rates have declined over time due to reservoir depletion, while water rates have increased due to increased injection. Exceptions to the correlation exist, like in 2012-7 when water rates surged without a corresponding oil rate increase. Additionally, SC field-NV generally exhibits higher water and oil rates than SC field-PRO, possibly due to reservoir pressure or production strategy changes.

In summary, the graph shows mature oil fields experiencing a decline, but water injection sustains production. Water rates are rising, and oil rates are falling, indicating increasing water (water-to-oil ratio). Possible causes include water encroachment, gas injection, or a naturally water-rich reservoir. Recommendations include close monitoring, identifying the cause, and implementing mitigation strategies. Water injection may be used to sustain production, and the reservoir appears to be reaching the end of its productive phase.

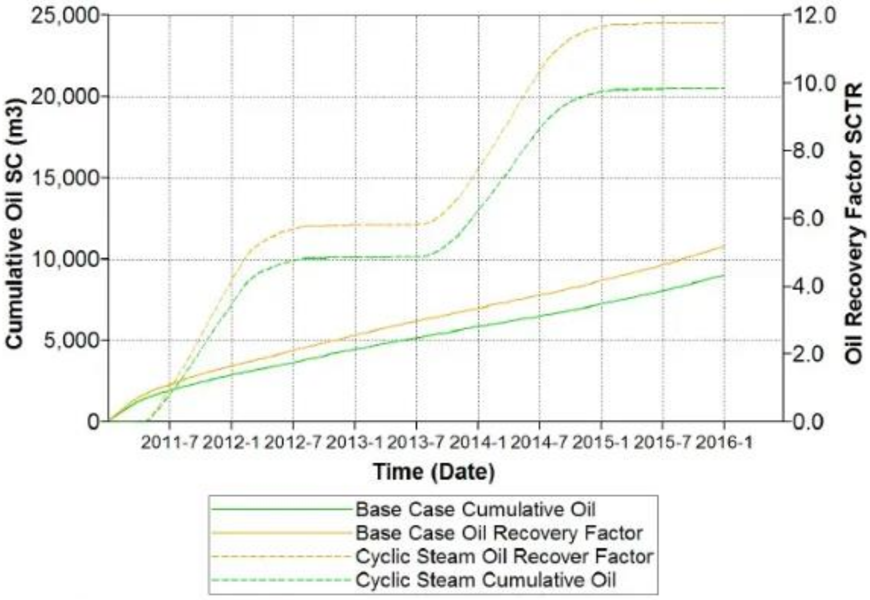


Figure 13: Comparison of Base Case and Cyclic Steam Results

Figure 13 shows the comparison of both cases: Base Case and Cyclic Steam (i.e. with and without the Enhanced Oil Recovery). The Base Case has a Cumulative Oil value of 9,000 m^3 and an Oil Recovery Factor of 5.1 SCTR or 42.5%, while the Cyclic Steam has a Cumulative Oil value of 20,500 m^3 and an Oil Recovery Factor of 11.8 SCTR or 98.3%. The graph depicts the oil recovery factor of cyclic steam, an enhanced oil recovery (EOR) method involving steam injection into heavy oil reservoirs. The graph shows the oil recovery factor as a function of time. The oil recovery factor is the percentage of the original oil in place (OOIP) that has been produced.

The oil recovery factor increases with the number of cycles due to reduced oil viscosity, making extraction easier. However, the rate of increase diminishes as the reservoir depletes with each cycle. Over time, the oil recovery factor rises, albeit at a decreasing rate, indicating reduced effectiveness of steam injection as the reservoir depletes. Higher reservoir temperatures result in higher oil recovery factors, as elevated temperatures reduce oil viscosity, facilitating production.

The graph also displays cumulative oil production from the reservoir. Cumulative oil production increases with time at an increasing rate, aligning with the rising oil recovery factor. This aligns with the rising oil recovery factor, as the reservoir volume remains constant. Overall, the graph demonstrates the potential for recovering significant amounts of oil from heavy oil reservoirs through steam injection cycles. Factors influencing the oil recovery factor include reservoir temperature, oil viscosity, and the number of cycles. Although it is effective in increasing the oil recovery factor, the rate of oil production decreases over time as the reservoir depletes. The efficacy depends on reservoir characteristics, necessitating reservoir modelling and pilot tests for optimization before commercial-scale implementation.

4.0 CONCLUSION

The use of CMG to simulate an enhanced oil recovery method (thermal) and to be exact, cyclic steam simulation method has many advantages such as saving cost and time. It aids in getting a replica of the expectations before the process is carried out physically. Oil companies use this software to carry out different methods of enhanced oil recovery to know their set expectations and to plan by selecting the best method of enhanced recovery to be used. The Base Case has a Cumulative Oil value of 9,000 m^3 and an Oil Recovery Factor of 5.1 SCTR or 42.5%, while the Cyclic Steam has a Cumulative Oil value of 20,500 m^3 and an Oil Recovery Factor of 11.8 SCTR or 98.3%. Overall, CMG software simulation and modelling have shown to be excellent tools for analyzing, optimizing, and managing CSS processes. The findings of the study give a framework for reservoir engineers to improve the economic viability of CSS and hence maximize the recovery of heavy oil and bitumen reserves. Future improvements in simulation technology and modelling methodologies will surely increase the efficiency and effectiveness of CSS procedures.

ACKNOWLEDGEMENTS

Special thanks go to Aare Afe Babalola, LL.B, FFPA, FNIALS, FCIArb, LL.D, SAN, OFR, CON – The Founder and President, and the Management of Afe Babalola University, Ado-Ekiti, Ekiti State, Nigeria. Through your vision, dedication, and commitment to education, you have created an institution that has become a beacon of knowledge and a nurturing environment for students like myself. Your unwavering support and investment in the development of our school have allowed us to receive a high-quality education,

equipped with the resources, facilities, and opportunities that have contributed to our growth and success. The passion and enthusiasm that you have instilled in the staff and faculty have made a significant impact on our educational experience.

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