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Analyses of Biogas and Digestate from Cattle Dung Anaerobic Digestion

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A B S T R A C T

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This paper analyzed biogas and the digestate from cattle dung after anaerobic digestion. Eight kilograms (8 kg) of sun-dried cattle dung mixed with equal volume of water was subjected to anaerobic digestion for 14 days in a digester made of mild steel. The digester had a diameter of 0.3 m, 0.6 m height and 2 mm thickness. The biogas resulting from this setup was daily tested and finally burnt with a blue flame on the 13th day of incubation. The biogas was refined through sodium hydroxide (NaOH) solution. In the chromatographic analysis, the raw biogas produced 56.20% CH₄, 39.51% CO₂, 1.92% CO, 1.84% H₂S and 0.53% NH₃. The refined one comprised 70.28% CH₄, 27.82% CO₂, 1.55% CO, 0.34% H₂S and 0.00% NH₃. The specific gas constant (R_i) of the biogas increased from 0.3027 kJ/kgK at the raw stage to 0.3459 kJ/kgK when refined. Similarly, the cv improved from 0.9082 kJ/kgK to 1.0376 kJ/kgK that of cp from 1.2080 kJ/kgK to 1.3803 kJ/kgK and the values for the ratio of the specific heats agreed with the standard values of perfect polyatomic and diatomic gases. The calorific values of the biogases in the same order were 19.15 MJ/m³ and 22.62 MJ/m³. The sun-dried cattle dung and the digestate analyses with atomic absorption spectrophotometer showed respectively 0.13 and 0.20 for Fe (%), 0.49 and 0.55 Ca (%), 0.14 and 0.32 K (%), 0.28 and 0.34 Na (%) and 0.59 and 1.46 P (%) while the values for Co and Mn remained at 0.0001% and 0.03% respectively. The Kjeldahl test results for N₂ (%) were 1.06 and 1.40 while the oil (%) Soxhlet test results were 2.48 and 0.16 respectively. It was therefore concluded that the digestate had higher values of nutrients than the undigested cattle dung.

1. Introduction

The concept of waste to wealth is a global phenomenon now. This refers to a situation where substances that are ordinarily considered waste materials are converted through value addition to useful substances that would benefit man. The conversion process of the substance, substrate or the input on ground would always depend on the path ways to-and-the end product. The path way may be physiochemical or biochemical. The physiochemical phenomenon involves subjecting a particular material to a process or processes as pathways to achieve a desired product that could be physically and chemically related to the waste. The recycling of plastic and rubber wastes and the melting of parts of broken down machinery and other irons and metals to produce new and useful products are common examples of this phenomenon. The biochemical waste to wealth approach is such that the substrate is

made to undergo series of processes in order to give rise to different products which can each exist on their own. Examples in this phenomenon may include fermentation of biodegradable substances to obtain products like alcohol and biogas.

Cattle dung, being a biodegradable waste undergoes the biochemical pathway through natural anaerobic digestion (AD) reaction to yield biogas and the sludge effluent. The biogas is usually combustible and the effluent a quality bio-fertilizer (Budiyono et al., 2010). The utilization of biogas produced through this process has the potential of reducing the emission of greenhouse gases (GHGs) and consequently contribute to climate change mitigation due to the controlled release of methane and nitrogen dioxide which have global warming potentials of 25 and 310 respectively (Ozor et al., 2014). Manonmani et al., (2017) reported that 18% of global greenhouse gas emission comes from the animal production sector. The conversion of cow dung into methane instead of allowing it to decompose could reduce global

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warming by 99 million tons (4%) (Ozor et al., 2014).

The effluent has the potential to replace the inorganic fertilizer with no undesirable consequence to the soil.

The AD is a biological process used to convert organic wastes to biogas and a stable product for land application without adverse effects to the environment (Budiyono et al., 2010). They also reported that the anaerobic fermentation of manure for biogas production does not reduce its value as a fertilizer supplement, as available nitrogen and other substances in the treated sludge. They equally reported that biogas production increases significantly between 10 – 50 days due to exponential growth of microorganisms' responsible for the fermentation. After 50 days biogas production tends to decrease and attain a stationary phase of microbial growth. AD was found to be a way of treating wastes with high organic load (Sae et al., 2009).

A lot of work has been done in these areas of biogas generation and production of biofertilizer. For example, biogas is a product of anaerobic digestion (AD) of biodegradable materials. It is a natural process that converts a portion of organic carbon in manure into principally methane and carbon dioxide (Beddoes et al., 2007). Prasad (2012) reported that biogas comprised 50-70% methane, 30-40% carbon dioxide, 5-10% hydrogen, 1-2% nitrogen and traces of water vapour and hydrogen sulphide. Musa and Raji (2016) generated biogas from cow dung and that of goat and reported that the cow dung recorded a better yield. Yaru et al., (2013) found out that a mixture of cattle dung with plantain peels though with longer retention time, produced more biogas than only cattle dung. AD based on liquid manure is wet fermentation (Madlener et al., 2012).

Biogas obtained from AD process is a source of renewable energy with reasonable calorific values once the right percentage of the constituents is attained. For instance, Raja and Lee (2012) showed the calorific values of biogas of variable percentage of 56%, 62% and 70% methane to be respectively 20 MJ/m³, 22.1 MJ/m³ and 25.0 MJ/m³ while propane gas had 46.0 MJ/m³. Itodo, (2007) described biogas, with a calorific value of 22 MJ/m³, as a colourless, blue burning gas that can be used for cooking heating and lighting. He added further that the quality of biogas (methane content) would depend on factors as carbon-nitrogen ratio of the feed stock, pH, retention time, loading rate, temperature, total solids content and presence of toxic substances to the anaerobes in the digester.

The energy content of the biogas can be analysed from the point of its specific heat capacities and the specific gas constant as they are properties that vary with temperature. Since the gas is compressible, the specific heats vary with temperature and pressure and are determined experimentally for real gases. However, each specific heat is constant for perfect gas and it is independent of

temperature (Eastop and McConkey, 1996).

The waste digested slurry left after the biogas from AD is generated can be used for plants nutritional purposes in the form of biofertilizer. Lukehurst et al., (2010) in their work involving utilization of digestate from biogas plants categorized nutrients in plant and animal products in anaerobic digestion feedstock into macronutrients, micronutrients and heavy metals. They also reported that composition of animal waste depends mainly on the type of digestive system of the animal as high proportion of potassium, phosphorus and 55-95% of nitrogen in animal diets is excreted through faeces and urine. Yongabi et al., (2009) found out that anaerobically digested cow dung slurry yielded pathogen free manure, biofertilizer and had potentials to improve the antimicrobial activity of medicinal plants. Dahunsi et al., (2013) worked on evaluating the fertilizer from combined anaerobic breakdown of food wastes and human excreta which was limed with sodium hydroxide. They reported that it proved to be an excellent substrate for biofertilizer preparation.

According to Silva and Uchida (2000), plants require 16 essential elements for normal functioning life, growth and optimum production. These elements include nitrogen, potassium, phosphorus, magnesium and sulphur. Others are calcium, iron, zinc, manganese, copper, boron, molybdenum and chlorine. Deficiency of any of the elements can disrupt the normal growth of the plants. McCauley et al., (2009) therefore grouped symptoms due to the deficiency of any of these nutrients in to five: stunted growth; chlorosis, interveinal chlorosis, purplish – red colouring and necrosis. They also categorized these nutrients in to mobile and immobile elements. Mobile nutrients are the nutrients that can move from the older leaves to the younger parts of the plants when the supply is inadequate and so the deficiencies are observed in older or lower leaves such as nitrogen, phosphorus and. The immobile elements on the other hand are those nutrients that are not able to move from one plant part to another and therefore their deficiencies will occur on the younger or upper leaves such as boron, calcium and iron.

The objective of this paper therefore is show that cattle dung is a ready renewable energy source and the digestate a quality bio-fertilizer.

2. Theory of biogas production

Biogas as a product of biochemical metabolism fall in to three stage processes during production: hydrolysis, acidogenesis and methanogenesis. It is usually formed when bacteria decompose a mass of organic material anaerobically. Vindin et al., (2008), reported that the bacteria can only act on the organic matter and produce the biogas if it is sufficiently bloated with water to at least 50%. The hydrolysis phase involves the use of enzymes

secreted by the facultative bacteria such as staphylococci type to breakdown the complex organic substances into simple soluble molecules (Yadvika et al., 2004). The acidogenesis process is the breakdown of the simple molecules to Volatile Fatty Acids (VFAs) such as propionic and butyric acids, carbon dioxide, ammonia and hydrogen while the methanogenesis phase converts the VFAs to methane, carbon dioxide, nitrogen, and hydrogen sulphide. Fusarium fungal and staphylococcus bacteria are involved in the anaerobic digestion process (Itodo, 2007).

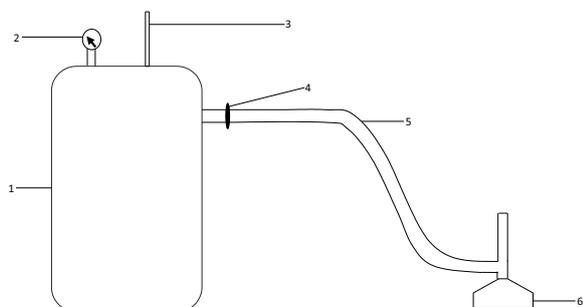
Enzymatic activities of micro-organisms responsible for the bioconversion of substrates to biogas are affected by temperature in ranges. Psychrophilic temperature range being below 20oC, mesophilic temperature range, 20-40oC and the thermophilic temperature range is 40-60oC. Thermophilic temperature range allows for shorter retention time and because of the high temperature of this phase there is little or no room for pathogens to thrive (Prasad, 2012).

3. Materials and Methods

3.1 Materials and biogas generation

The materials used for the biogas production are the digester made of mild steel containing gas valve, mercury-in-glass thermometer and pressure gauge, cattle dung, bagco sacs and water. Cattle dung was sourced from a local cattle ranch in Ilorin. This was crushed by beating it to pulp in bagco sacks made of synthetic fibres. Eight kilograms (8 kg) of the cattle dung was mixed with eight litres of water and the mixture charged to a digester up to three quarter of its volume. The digester was made of mild steel with a diameter of 0.3 m, 0.6 m height and 2 mm thickness.

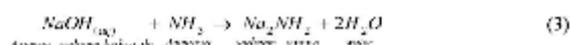
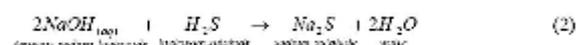
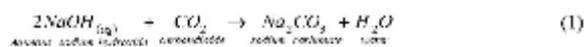
The mixture was stirred thoroughly to make uniform slurry before being closed tight and subjected to anaerobic digestion in the laboratory at the Kwara State Polytechnic, Ilorin. The schematic set up of biogas production in the digester is as shown in Figure 1. The gas was daily tested for combustion and it burnt with a blue flame on the day 13 of incubation.



1. Digester ; 2. Pressure gauge; 3. Thermometer; 4. Gas outlet valve; 5. Hose; 6. Burner

Figure 1: Schematic of the digester

The raw biogas was collected in a cylinder using a sooter. Thereafter the biogas from the digester was refined by passing it through aqueous sodium hydroxide to remove carbon dioxide, hydrogen sulphide and ammonia as shown in equations (1), (2) & (3) and this was equally collected in another cylinder. Both raw and the refined biogas were subjected to chromatographic analysis. Their calorific values were also determined.



3.2 Gas Chromatography (GC)

The chromatography system is composed of the gas chromatography equipment, detector and a recorder for plotting chromatographs. The experiment was conducted with thermal conductivity detector (TCD) at Multi- Environmental Management Consultants Ltd, Igbe, Ikorodu, Lagos, Nigeria. The equipment model is HP6890 with HP ChemStation and Rev. A09.01 (1206) software. The carrier gas was helium at 20 ml/min flow rate with the inlet temperature of 145 °C while the inflow of the carrier gas was 26 ml/min in the column with dimensions and type of $30\text{ m} \times \frac{1}{8}\text{ mm} \times 0.85\ \mu\text{m}$ and Heysep DB 100/120; Deerfield, Illinois respectively. The oven temperature was programmed at 140°C in 6 min and ramped at 50 oC/min to and maintained at 175 °C.

3.3 Determination of thermophysical properties of the raw and refined biogas

The thermophysical properties of the biogases were determined using equations (4) to (14) adopted from Joe (1996) and Rogers and Mayhew (1988):

Number of moles, n

$$n = \frac{m}{M} \quad (4)$$

$$\text{Mass fraction, } m_a = \frac{m_i}{m} \quad (5)$$

Mole fraction,

$$x_i = \frac{n_i}{\sum_{i=1}^n n_i} = \frac{n_i}{n} = \frac{m_i / M_i}{n} \quad (6)$$

Specific biogas constant, R

$$R = \sum_{i=1}^n R_i = \sum_{i=1}^n \frac{m_i}{m} \frac{R_o}{M_i} \quad (7)$$

Specific heats at constant pressure, cp, and at constant volume, cv of bio

$$c_p = \sum_{i=1}^n \frac{m_i}{m} C_{pi} \tag{8}$$

$$c_v = \sum_{i=1}^n \frac{m_i}{m} C_{vi} \tag{9}$$

For diatomic gases

$$C_{vi} = \frac{5}{2} R_i \tag{10}$$

$$C_{pi} = \frac{7}{2} R_i \tag{11}$$

For polyatomic gases

$$C_{vi} = 3R_i \tag{12}$$

$$C_{pi} = 4R_i \tag{13}$$

$$\gamma = \frac{c_p}{c_v} \tag{14}$$

where,

Ro is the Universal gas constant;

Ri is the specific gas constant of a component gas in the biogas mixture;

mi is the mass of a component gas in the mixture;

Cpi and Cvi are the specific heat capacities of a constituent gas of biogas at constant pressure and volume respectively;

and

γ is the ratio of cp to cv

3.4 Determination of calorific values of biogas

The determination of the calorific values of the biogas samples was done with the open flame calorimeter. The biogas was burnt in an open flame by way of the gas calorimeter Junkers. The heat of combustion released by the burner was transferred to a heat absorbing air through a heat exchanger which resulted in the temperature rise of the fluid. This temperature increase was used to determine the calorific value of the gas.

3.5 Determination of some plant elemental nutrients of cattle dung and the digestate

3.5.1 Mineral analysis

The mineral analysis was done using UNICAM 929 London atomic absorption spectrophotometer (AAS) powered by SOLAAR software.

The weight of 0.2 g ashed sample (cattle dung and the digestate) was individually introduced into a pre cleaned borosilicate 250 ml beaker for digestion and 30 ml nitric acid was added to it. The set up was then placed on a hot plate for digestion in a fume cupboard.

The beaker and its content were allowed to cool after the digestion. Once more, 20 ml nitric acid was added to the beaker and the substance was further digested in the same fume cupboard. The mixture was allowed to cool at room temperature and it was filtered. The filtrate was now mixed with de-ionized water before analyzing the digested samples with the AAS with a detection limit of 0.0001mg/l. The particular cathode rays for calcium, potassium and sodium were used for the detection of the elements.

3.5.2 Detection of phosphorus

The weight of 250 mg of the cattle dung and digestate were each introduced into Schrödinger flask and were exposed to excess oxygen. The resulting product was digested with nitric acid. This was boiled for a minute in so as to convert phosphorus pentoxide to orthophosphate. The solution was passed through a 10 cm long resin column and the filtrate collected. A colour agent (2 ml) was added for the absorbance reading at 650 nm in a 10 ml Pyrex tube.

3.5.3 Determination of nitrogen

The nitrogen content of each sample was determined using the Kjeldahl procedure. One gram (1g) of the sample was put into a conical flask and 20 ml of 1.25% sulphuric acid was added to it. This was allowed to boil gently for thirty minutes. It was thereafter filtered with buckner funnel and rinsed with de-ionized water. The residue was washed thoroughly with hot distilled water and then mixed with 1.25% sodium hydroxide. This was boiled gently for thirty minutes and then a cooling pipette was used to maintain a constant volume of the filtrate as it was filtered through the buckner funnel. Thereafter the residue was washed with hot distilled water, rinsed with 10% hydrochloric acid and with industrial methylated spirit. This was finally dried in an oven and afterward ashed to 550 °C in a furnace.

3.5.4 Oil Content

The soxhlet method of oil extraction was used in the analysis of the oil of the cattle dung and the digestate. During the analysis, 0.25 g of each sample was weighed in a porous thimble and then placed in a well cleaned 250 ml extracting flask which had earlier been weighed. Petroleum ether (200 ml) was also added to the flask and the setup was placed in the soxhlet extractor. The extraction was done for five hours.

The porous thimble was cautiously removed and the petroleum ether in the tube was collected so that it could be recycled for use again. The extraction flask with the content left was removed from the heating mantle arrangement when it was free of petroleum ether and then dried in an oven at 105 oC for one hour. Thereafter the flask with dried content was cooled in a desiccator and

the weight measured. The weight of the ether to the weight of the sample expressed as a percentage gives the total percentage of crude fat in the sample.

4. Results and Discussion

The results for raw and refined biogas are shown in Table 1. In the second column of the Tables, the mole of CH₄ increased while the moles for the rest gases decreased. This was because the gases were absorbed by the NaOH(aq) as the biogas was passed through it such that NH₄ was completely absorbed leading to its zero value. This was also responsible for the decrease in mass of each gas in the mixture from 27.19 kg to 24.04 kg in the 4th column of the Tables. The energy content per volume of the biogas as seen with the specific gas constant, the specific heat at constant volume and that at constant pressure in columns 6, 7 and 8 increased as a result increase in the percentage of CH₄.

Table 1: Percentage composition of the raw and NaOH(aq) refined biogas

Constituent	Percentage of raw biogas	Percentage of refined biogas
Methane (CH ₄)	56.20	70.28
Carbon dioxide (CO ₂)	38.51	27.82
Carbon monoxide (CO)	1.92	1.55
Ammonia (NH ₃)	0.53	0.00
Hydrogen sulphide (H ₂ S)	1.84	0.34
Total	99.00	99.99

Table 2: Thermophysical properties of raw cattle dung biogas

Constituents	Mole	Molar mass (kg)	Mass in mixture (kg)	Mass fraction	Specific gas Constant, R _i (kJ/kgK)	Constant volume specific heat, c _v (kJ/kgK)	Constant pressure specific heat, c _p (kJ/kgK)	Ratio of specific heats (γ)
CH ₄	0.5620	16	8.9920	0.3307	0.1718	0.5155	0.6873	1.3333
CO ₂	0.3851	44	16.9444	0.6231	0.1177	0.3532	0.4710	1.3333
H ₂ S	0.0184	34	0.6256	0.0230	0.0056	0.0169	0.0225	1.3333
CO	0.0192	28	0.5376	0.0198	0.0059	0.0147	0.0205	1.4000
NH ₃	0.0055	17	0.0935	0.0034	0.0017	0.0050	0.0067	1.3333
TOTAL	0.9902		27.1931	1	0.3027	0.9082	1.2080	1.3301

Table 3: Thermophysical properties of NaOH(aq) refined biogas

Constituents	Mole	Molar mass (kg)	Mass in mixture (kg)	Mass fraction	Specific gas constant, R _i (kJ/kgK)	Constant volume specific heat, c _v (kJ/kgK)	Constant pressure specific heat, c _p (kJ/kgK)	Ratio of specific heats (γ)
CH ₄	0.7028	16	11.2448	0.4678	0.2431	0.7293	0.9724	1.333333333
CO ₂	0.2782	44	12.2408	0.5093	0.0962	0.2887	0.3849	1.333333333
H ₂ S	0.0034	34	0.1156	0.0048	0.0012	0.0035	0.0047	1.333333333
CO	0.0155	28	0.4340	0.0181	0.0054	0.0134	0.0188	1.4
NH ₃	0	17	0	0	0	0	0	
TOTAL	0.9999		24.0352	1	0.3459	1.0376	1.3808	1.330749742

Similarly, results for the calorific values of the raw and refined biogases are as shown in Table 4. The ratio of the specific heats remained at 1.3333 for polyatomic gases and 1.4 for diatomic gas. This agreed with the submission of Rajput (2011), Joe (1996) and Rogers and Mayhew (1988).

The calorific values increased from 19.15 MJ/m³ of the raw biogas to 22.62 MJ/m³ when it was refined due to the improvement in the percentage of the CH₄ component as shown in Table 2. These calorific values were close to the net calorific values of 20 MJ/m³ 25 MJ/m³ for biogases containing 56% and 70% CH₄ respectively (Raja and Lee, 2012). However the variations could be as a result of the presence of the non combustible gases in the biogas mixture.

The results of the analyses of the plant nutrients in the cattle dung before and after anaerobic digestion are shown in Table 5.

The mineral analyses showed that elemental contents of the digestate were more than those of raw cattle dung for the same mass or quantity as Fe increased from 0.13% to 0.2%, Ca 0.49 to 0.55%, K 0.14 to 0.32%, Na 0.28 to 0.34% and P 0.59 to 1.46% while N₂ increased from 1.06 to 1.40% using the Kjeldahl method. The values for Co and Mn remained unchanged at 0.0001% and 0.03 % respectively. This showed that the digestate is a better bio-fertilizer than the raw undigested cattle dung. The nutrients are in agreement with the requirement for corn, soybeans and wheat (Mckenzie, 1998 and Landis, et al. 2005). However the soxhlet analysis showed that the percentage of oil decreased from 2.48% in the raw cattle dung to 0.16% in the digestate.

Specific heats at constant pressure, cp, and at constant volume, cv of biogas

Table 4: Calorific values of the raw and refined biogas

Sample	Calorific value (MJ/m ³)
Raw biogas	19.15
Refined biogas	22.62

Table 5: Some plant nutrients contained in the cattle dung and the digestate

Component (%)	Raw cattle dung	Cattle dung digestate
Iron, Fe	0.13	0.20
Cobalt, Co	0.0001	0.0001
Manganese, Mn	0.03	0.03
Calcium, Ca	0.49	0.55
Potassium, K	0.14	0.32
Sodium, Na	0.28	0.34
Phosphorus, P	0.59	1.46
Nitrogen, N ₂	1.06	1.40
Oil	2.48	0.16

5. Conclusion

The analyses of biogas and digestate from cattle dung through anaerobic digestion were carried out. The biogas ignited on day 13 of incubation. The refining of the biogas improved the CH₄ and the energy content in terms of the specific gas constant, specific heat at constant volume and that at constant pressure also increased. Its calorific value increased from 19.15 MJ/m³ to 22.62 MJ/m³. This showed that cattle biogas is a source of energy. The nutritional plant values increased from those of the raw cattle dung in the percentages of Fe with 0.13, Ca with 0.49, K 0.13, Na 0.28, P 0.59 and N₂ 1.06 to those of the digestate with percentages in the order as 0.20, 0.55, 0.32, 0.34, 1.46 and 1.40. The values for Co and Mn remained unchanged for both the raw cattle dung and the digestate at 0.0001% and 0.03% respectively. This showed that the digestate had higher elemental content than the raw cattle and could therefore be a better biofertilizer.

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Journal of Engineering and Engineering Technology

Contents

Volume 11 Issue 1, 2017

- | | | |
|----|---|--|
| 1 | Ajibade, F. O. and Adewumi, J. R. | Performance Evaluation of Aquatic Macrophytes in a Constructed Wetland for Municipal Wastewater Treatment |
| 12 | Fadeyibi, A., Osunde, Z. D., Yisa, M.G. Okunola A. A. | Investigation into Properties of Starch-based nanocomposite materials for fruits and vegetables packaging - A review |
| 18 | Melodi, M.M. and Agboola, D.A. | Labour Productivity Assessment of Dredging Operations in Some Selected Companies in Lagos State, Nigeria |
| 23 | Olubanjo, O. O. | Effects of Wastewaters From an Abattoir on Surface Water Quality in Akure, Ondo State, Nigeria. |
| 29 | Jabar, J. M., Lajide, L., Adetuyi, A. O., Owolabi, B. J. and Ayoade G. W. | Chemical modification of coir, empty fruit bunch and palm kernel fibres for polymer reinforcement |
| 37 | Awopetu O.O. | Effect of Rake Face Reinforcement on Cutting Tool Life of Cemented Carbide Tools during Turning of a-Titanium Alloy |
| 43 | Famurewa, J.A.V. and Faboya, E.T. | The Effects of Drying Temperature on the Physicochemical Properties of Ogbono (Irvingia Gabonensis) |
| 48 | Olutomilola E. O. and Ogedengbe T. I. | Evaluation of the Effect of Constituents on Insulation Quality of Selected Biomass Composites |
| 56 | Mogaji, P.B., Abioye, T.E., Ohuruogun, N.O. | Development of Epoxy Resin Composite Reinforced With Cow Horn and Cassava Particles for Car Dashboard |
| 62 | Akingbade, K. F. and Apena, W. O. | Modelling of Electric Power Subsystem for a Weather Balloon Microsatellite |
| 68 | Daniyan I.A., Bello E.I., Ogedengbe T.I., Lajide L. and Mogaji P.B. | Development of a Model For Predicting the Yield of Biodiesel During Biodiesel Production |

Volume 11 Issue 2, 2017

- | | | |
|-----|---|--|
| 73 | Ayodeji S. P., Adeyeri M. K. and Oni O. O. | Conceptual Design of Battery Powered Vehicle for Children Amusement Park Using Nigerian Anthropometric Parameters. |
| 81 | Owolabi, H. A., Omidiji, B. V., Oke, A. O. Morakinyo, A. T. | Development of a Small-Scale Motorized Dough Kneading Machine |
| 89 | Akingbade K. F. | Development of Chemical Proportion Formulation Model for Quality Control in Flexible Polyurethane Foam Production. |
| 96 | Isa J. and Aderotoye M. A. | Effect of moisture content on the physical and aerodynamics properties of cashew nut |
| 103 | Abegunde, A. J., ¹ Omidiji B. V. and Ajewole P. O. | Characterization of Ado-Ekiti River Sand for foundry applications |
| 112 | Yaru S. S. and Adegun I. K. | Analyses of Biogas and Digestate from Cattle Dung Anaerobic Digestion |
| 119 | Awopetu O. O., Farayibi P. K., Ismail K. A. | Optimization of Major Process Parameters for Surface Roughness of ASTM 25 Grey Cast Iron using Taguchi method |
| 126 | Afu D. J., Ifelola E. O., Ayodele S. G., Ekun A. and Itunu A. | Survey of the Effects of Operational Noise on Quarry Workers in Akoko-Edo Local Government Area of Edo State |
| 133 | Melodi, M. M. and Onipede, T. G. | Economic Appraisal of Some Selected Quarries Operation in Ondo State, Nigeria |
| 139 | Ojediran J.O., Fasinmirin J.T., Owa O.P. | Development and Performance Evaluation of an Indigenous Fish Feed Palletizing Machine |

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