Conversion of Biogas from Municipal Solid Waste of Kaduna State to Bio-methane.

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Abstract
The uncontrolled hazardous wastes from industries mixing up with municipal wastes create potential risks to human health. A major environmental concern is gas released by decomposing garbage which increases the concentration of atmospheric greenhouse gases. This study was aimed to convert biogas to bio-methane from municipal solid wastes (MSW) in Kaduna State, Nigeria. The segregates of the wastes were immersed in sodium hydroxide (NaOH) solution for 5 days to reduce the lignocellulosic content and was further homogenized in a propeller mixer. Proximate composition, physical and chemical and properties were analyzed. The mean values for moisture, ash, pH, temperature, total solids, volatile solids, BOD and COD were 65.10%, 1.10%, 5.49, 23°C (initial), 6560mg/L, 84.53%, 205mg/L and 540mg/L respectively. The lignocellulosic content of the waste were of 34%, 33%, 33% for hemicellulose, lignin and celluloses, and nutrient values of 32:1 for carbon-to-nitrogen ratio, 0.48% and 1.16% for total sodium and phosphorous which indicate the MSW as potential substrates for biogas production. The waste fractions were assessed for a total hydraulic retention period of 35 days and ambient temperature (37°C) with neutral pH (7.35 to 8.0). The maximum gas yield was between 1070 and 1402 cm3 within 20-30 days retention time. There was substantial decrease in biogas yield that correlated with temperature fluctuation within 10-15 days retention time, obtaining a value from 868 to 589 cm3 at 33 to 31°C. The biogas produced were characterized as 68.57% CH4, 30.21% CO2, 1.31% H2S and 1.50% O2 (25-30 days), which significantly increased with regards to CH4 (87.52%) and significantly decreased to CO2, H2S and O2 (9.58, 0.38 and 0.01%) respectively. Municipal solid wastes (MSW) can be utilized for biogas production thereby solving the waste disposal problem.

Keywords: Biogas, Bio-methane, Municipal Solid Waste.

INTRODUCTION
The environmental risks of municipal solid wastes has been reduced by advances in waste reduction, recycling and composting techniques, but land filling is still the most common waste disposal option worldwide (Uzodima et al., 2007; Mouhoun-Chouaki et al., 2019).

Biogas is an output of a biological process from anaerobic digestion of organic waste including biomass-enrich waste water, municipal solid wastes, manure, sewage sludge and biodegradable feedstock (Shahidul et al., 2018; Leh-Togi Zobeashia et al., 2018).

Biogas is a colorless, combustible gas composed of 50-70 percent methane (CH4), 30-45 percent carbon dioxide (CO2), and trace amounts of nitrogen (N2), hydrogen sulphide (H2S), ammonia (NH3), and oxygen (O2). Because of the abundance of raw materials such as animal, human, agricultural, and industrial wastes, the economic prospects of biomass energy in Africa are excellent. In the generation of biogas, waste like as cow dung and human feces can be utilized as inocula (Nwabunwanne et al., 2020). Biogas production will not only function as a sewage treatment unit, but also as a renewable energy source of employment, because large sums spent on trash removal can be saved for other purposes (Omaliko, 2006; Manish et al., 2019; Nwabunwanne et al., 2020).

Purification and upgrading of the gas is necessary because purified biogas provides reductions in greenhouse gas emissions as well as several other environmental benefits when used as a vehicle fuel. Reducing CO2 and H2S
content will significantly improve the quality of Biogas upgrading is a gas separation task, finally yielding 95-97% methane-rich product gas stream with a certain specification depending on the raw biogas composition (Angelidaki et al., 2018). This separation task comprises the separation of carbon dioxide (and thus increasing the heating value and wobbe-index), the drying of the gas, the removal of trace substances like oxygen, nitrogen, hydrogen sulphide, ammonia or siloxanes as well as the compression to a pressure needed for the further gas utilization (Petersson and Wellinger, 2009; Angelidaki et al., 2018; Baena-Moreno et al., 2019).

Bio-methane is defined as methane produced from biomass, with properties close to natural gas; it is an interesting fuel to support the transition from fossil fuels to renewable energy and to achieve the greenhouse gas emission reduction targets in different ways (Shahidul et al., 2018). Bio-methane can be used for exactly the same applications as natural gas, if the final composition is in line with the different natural gas qualities on the market (Baena-Moreno et al., 2019). Therefore, it can be used as a substitute for transport fuels, to produce combined heat and power (CHP), heat alone or serve as feed stock for the chemical sector. It can be transported and stored in the facilities and infrastructure available for natural gas. Bio-methane can be produced by upgrading biogas or as so called bio-Synthetic Natural Gas (SNG) from thermo-chemical conversion of lignocellulosic biomass or other forms of biomasses (Seiffert and Ronsch, 2012).

**MATERIALS AND METHODS**

**Collection and Processing of Samples**

Fresh cow dung was collected from Zango-Abattoir in Tudun Wada Area of Kaduna State, Nigeria in clean polythene bags and transported to microbiology laboratory, Kaduna State University (KASU), Kaduna, Nigeria for further processing. Municipal Solid Waste (MSW) was obtained in clean air tight plastic buckets from Kaduna State University (KASU) market and transported to microbiology laboratory KASU, Nigeria. The non-biodegradable components of the MSW such as bones, eggshells, plastics and leather were removed (Rao et al., 2000).

**Pretreatment and Homogenization of the MSW**

In order to achieve a cleaned digester feed, the substrate (waste) was processed in the laboratory by shredding it into smaller parts and immersing it in 20g of sodium hydroxide (NaOH) solution for 5 days, then homogenized in a propeller mixer to prevent reactor

<table>
<thead>
<tr>
<th>Weight of Sample</th>
<th>W1</th>
<th>W2</th>
<th>W3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture (%)</td>
<td>W2 - W1 x 100</td>
<td>W2 - W1</td>
<td>W3</td>
</tr>
<tr>
<td>Ash (%)</td>
<td>W2 - W3 x 100</td>
<td>W2 - W1</td>
<td>W1</td>
</tr>
</tbody>
</table>

**Determination of moisture content**

A clean crucible was dried to a constant weight in a furnace; the crucible was then removed and cooled at room temperature; and the weight was measured as W1 (AOAC, 2016). The crucible were weighed with an analytical electronic balance (Sartorius, USA) and placed in a desiccator, with the weight recorded as W1. W2 was calculated by transferring two grammes of waste sample into the crucible and recording the weight. The sample with the crucible was therefore placed in a furnace at a temperature (Carbolite, UK) with the temperature adjusted to 5500 degrees Celsius. The percentage of Ash content was calculated using the formula:

\[ \text{% Ash} = \frac{W_2 - W_3}{W_1} \times 100 \]

**Determination of crude lipid**

The round bottom flask and oil were weighed after cooling (W2). The fatty acid profile was determined using the following formula (AOAC, 2016):

\[ \text{% Crude Lipid} = \frac{W_2 - W_1}{W_2} \times 100 \]

**Conclusion**

The processed sample was kept at room temperature in airtight container buckets for subsequent analysis (Akpor and Muchie 2011).
Determination of carbohydrate

It was calculated using the formula adopted from AOAC (2016): The total carbohydrate content was determined by difference. The sum of percentage moisture, ash, crude lipid, crude fiber and crude protein was subtracted from 100.

\[
\% \text{ Carbohydrate} = 100 - (\% \text{ moisture} + \% \text{ ash} + \% \text{ fat} + \% \text{ protein} + \% \text{ fiber})
\]

Physical and chemical composition of the Wastes

The techniques that were used in the analysis were in accordance with Standard Method for the Examination of Water and Waste Water (APHA, 2005) and Akpor and Muchie (2011).

Determination of Temperature and pH

The glass electrode pH meter (PHS-25, HELMREASINN) was turned on and placed in the MSW sample about two inches on the meter. The pH meter takes about 2 minutes to read and the value on the screen was recorded. The pH meter was neutralized using distilled water before taking another reading. A buffer solution was used to standardize the pH meter.

The temperature of MSW measurement was performed with a “mercury in glass” thermometer. The thermometer was held by the handle and the end of the thermometer was placed in the waste sample for two minutes before the rise of mercury in the thermometer was noted. This method was carried out in triplicate to ensure that the reading was accurate (APHA, 2005).

Determination of Total Solids (TS) and Total Suspended Solids

Total solids (TS): An empty evaporating dish (A) was weighed. Aliquot of fifty milliliters (50ml) was placed on the evaporating dish. The waste sample was equally distributed around the bottom flask. A steam bath was used to evaporate the sample. The waste sample was therefore dried at 105°C for 12 hours before being weighed and chilled to balance the temperatures in desiccatrors. The residue was boiled for one hour before being cooled and measured. The total solid was then computed using the following formula adopted from APHA (2005) below:

\[
\text{Mg total solids/L} = (A-B) \times 1000
\]

Where: 
A= weight of filter + dried residue (mg) 
B= weight of filter (mg)

Total Suspended Solids (TSS): Fifty milliliter (50ml) of the waste sample was filtered through a pre-weighed filtered paper to strain off and retain the suspended solids therein. The filtered papers were then dried in an oven at 105°C for one hour. The filter paper and the suspended solids were finally taken to the desiccators to cool before recording the weight. TSS was calculated using the formula adopted from APHA (2005):

\[
\text{Mg total suspended solids/L} = (A-B) \times 1000
\]

Where: 
A= weight of filter + dried residue (mg) 
B= weight of filter (mg)

Total Nitrogen

The nitrogen level was evaluated using the Kjeldahl method published by Michalowski (2013). The sample was weighed and heated at 360°C-410°C with concentrated sulfuric acid (H₂SO₄), which decomposed the organic material by oxidation and liberated the reduced nitrogen as ammonium hydroxide. To speed up the digestion, a catalyst (Hg₂SO₄) was added. To raise the boiling point of H₂SO₄, Na₂SO₄ also was added. When the liquor clarifies due to the emission of fumes, digestion is complete. After that, the sample solution was distilled with a little standardized sodium hydroxide (NaOH). Ammonia was formed when sodium hydroxide (NaOH) interacted with ammonium (NH₄⁺), (NH₃), which evaporated the standard solution. Thus, the amount of hydrogen in the sample can be determined using a strong acid in a straight acid-base titration (H₂SO₄).

\[
\% \text{ Nitrogen} = \frac{[(\text{ml standard acid} \times N \text{ of acid}) - (\text{ml blank} \times N \text{ of base})]}{\text{Weight of sample in grams}}
\]

Total Carbon

The carbon-nitrogen ratio was calculated using the results obtained from Carbon and Nitrogen contents. The carbon content of the sample was determined by Walkley-Black titration method as described by Sahilemedhin and Bekele (2000) and Faina et al. (2011). Fifty (50g) grams of the waste sample was weighed and transferred into a 500ml conical flask. Five milliliter (5ml) of 0.4 N of potassium dichromate (K₂Cr₂O₇) solution and 10ml of concentrated sulfuric acid (H₂SO₄) was added. The mixture of the flask was swirled 3 times and was allowed to stand for 18h in a fume hood at room temperature for the reaction to complete. Two hundred milliliters (200ml) of distilled water was added to the flask to dilute the suspension. Ten milliliter (10ml) of orthophosphoric acid and 1ml of diphenylamine indicator was also added, a deep violet colour
Organic carbon (%) in waste = \((V_{\text{blank}} - V_{\text{sample}}) \times M_{fe} \times 0.003 \times 100 \times f \times m_{c}\)

Where: 
- \(V_{\text{blank}}\) = volume of titrant in blank, ml
- \(V_{\text{sample}}\) = volume of titrant in sample, ml
- 0.003 = carbon oxidized
- \(f\) = correction factor, 1.3
- \(W\) = weight of waste, g
- \(m_{c}\) = moisture correction factor

**Total Phosphorous**

A UV spectrophotometer was used to determine the phosphorus level. Prior to assessment, the waste information was obtained and filtered via Whatman filter paper. 1mL of ammonium molybdate and 0.4mL of hydrazine sulphate were added to 2mL of sample. It was prepared in a 10ml flask with distilled water and cooked for 30 minutes at room temperature kept at 600C. A color shift was noted Macias-Corral et al., (2008). The UV spectrophotometer was used to measure the absorbance of the solution in the collection tube (Nollet, 2007). Phosphorous content is determined by the formula:

\[ P (\text{mg/Kg}) = (\text{Reading} \times 0.61 \times \text{dilution factor}) / (\text{Atomic weight of phosphorous}). \]

**Feeding Substrate and Anaerobic Bio-Digestion**

Anaerobic digestion of the substrate carried out on laboratory scale using a 20litres dispenser bottle that served as a bio-digester. Seven liter (7L) of water was combined with 2g of MSW, and 1g inoculum was added to the digester. The inoculum (cow dung) was diluted with water in a 2:1 ratio, resulting in a slurry, and fibers were screened using a sieve with a mesh size of (0.5cm x 0.5cm) mesh size (Baba-shehu and Nasir, 2012). The laboratory scale bio-digester was connected to a rubber tube via a gas outlet pipe, which functioned as a storage tank for the biogas generated (Ikpi et al., 2018). The methane content was also tested by lighting flame on the Bunsen burner connected to the digester. The gas that came out of the digester was checked whether it burns the colour of the flame and the odour were also checked. The percentage of gases produced was also characterized using the 4-in-1 multi-gas analyzer before and purification.

**Biogas conversion to Bio-Methane**

For the purpose of this research, three methods were employed (Angelidaki et al., 2018).

i. **The Iron- Sponge Scrubber Technique (ISST):** The ISST was meant for absorbing or removal of hydrogen sulphide (H\(_2\)S). A 500mL Erlenmeyer flask (filtration flask) was used as the scrubber; it was filled with iron and zinc sponge, a bottle cork was used to close the top of the flask (Plate II).

ii. **The Water Scrubbing Technique (WST):** An Erlenmeyer flask was also used with the same perforated bottle cork that was attached to the outlet pipe of ISST. This allowed the gas to pass into the flask, where the water absorb CO\(_2\) and biogas bubbled up and was passed through the side opening of the flask to the outlet pipe attached to it that served as an inlet pipe to the next purification flask.

iii. **Absorption Technique (AT):** Absorption technique is usually used to absorb or remove water vapor or moisture that has been saturated during anaerobic digestion and CO\(_2\) purification process, thus water vapor may result in corrosion and clogging issues to equipment.
Statistical analysis.
Statistical analysis of the data acquired from the various treatments was performed using that analysis of variance (ANOVA) and Student T-test. The means separated using the Fishers Least Significance (LSD) at 95% significant level using Statistical Package for Social Science (SPSS) version 22 16th edition. All data were interpreted as means ± Standard deviation of duplicate trials.

RESULTS AND DISCUSSION
Proximate Composition of Municipal Solid Wastes (MSW)
The proximate composition of the municipal solid wastes (MSW) is interpreted in Fig. 1. The results revealed that the MSW had high mean
value of moisture (65.10%), crude protein (16.2%) and fiber (14.34%). The carbohydrate and nitrogen free extract was (10.90%) while Generally, organic matter consists of moisture, ash and volatile solids. During anaerobic digestion, microorganisms action indicated that the relative moisture, ash and volatile solids in a given MSW influences the amount of biogas produced (Abdulrahim et al., 2015). However, Abdulrahim et al. (2015) reported moisture content of 83% and ash of 1.5% which are slightly higher than values for moisture (65.10%) and ash content (1.10%) observed in the present study, this might be due to reduction in total solids which connotes substrates digestion by anaerobic microorganism and depicts system stability. Also the study is similar to the findings of Osibote et al. (2019) who reported the value for moisture (65.5%) and ash (1.08%). Moisture content ranged from 61% to 87% will provide greater activity of water as soluble enzymes and coenzymes needed for metabolic activities will help the growth of microorganism (Osibote et al., 2019).

Fig. 1. Proximate Composition of Municipal Solid Wastes (MSW)

Physical, chemical and Nutrient Properties of Municipal Solid Waste (MSW)

The physical and chemical properties of the wastes are presented in Table 1. Municipal solid wastes was found to be slightly acidic having the initial pH of (5.49) at temperature of (23°C initial), the study also revealed that MSW had a high mean value of volatile solids (84.53%), Total Solids (6560mg/L), Total Suspended Solids (3360mg/L), Total Dissolved Solids (3299mg/L). MSW shows an acceptable mean value of chemical oxygen demand and biological oxygen demand account for about 540 and 205mg/L respectively (Table 1).

In addition, the nutrient Content of the waste such as Nitrogen-Carbon ratio (32:1), sodium (0.48%) and phosphorus (1.16%) mean values were recorded respectively on Table 1. This is within the recommended range of 25:1 to 30:1 (Fricke et al., 2007) and also in agreement to the work of Tadesse et al. (2014). A suitable C/N ratio plays an important role for the proper proliferation of the bacteria for the degradation process. Furthermore, domestic sewage, animal and poultry wastes are examples of N-rich materials that provide nutrients for the growth and multiplication of the anaerobic organisms. On the other hand, N-poor materials like agricultural waste, green grass, etc. are rich in carbohydrates, substances that are essential for gas production. Excess of nitrogen leads to the formation of ammonia (NH₃), the concentration of which inhibits further growth of methanogens. Also, the values from the phosphorus content is in agreement with that of Marufa et al. (2021) which shows the promising implication of MSW compost as excellent fertilizer for agricultural purpose.
The lignocellulosic content of the waste is also presented in Table 1. The study revealed that the waste had a high mean value of hemicelluloses content (33%), cellulose (33%) and lignin (34%) respectively (Table 1).

### Table 1. Mean of Physical, Chemical and Nutrient Properties of Municipal Solid Waste (MSW)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Mean Values</th>
<th>Standard Deviation (±)</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>5.49</td>
<td>0.01414</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>23</td>
<td>0.56569</td>
</tr>
<tr>
<td>Volatile solids (%)</td>
<td>84.53</td>
<td>1.41421</td>
</tr>
<tr>
<td>Total solids (mg/L)</td>
<td>6560</td>
<td>8.48528</td>
</tr>
<tr>
<td>Total dissolved solids (mg/L)</td>
<td>3299</td>
<td>41.01219</td>
</tr>
<tr>
<td>Hemicellulose (%)</td>
<td>33</td>
<td>0.70711</td>
</tr>
<tr>
<td>Cellulose (%)</td>
<td>33</td>
<td>0.70711</td>
</tr>
<tr>
<td>Lignin (%)</td>
<td>34</td>
<td>0.70711</td>
</tr>
<tr>
<td>Carbon-Nitrogen ratio</td>
<td>32:1</td>
<td>0.07071</td>
</tr>
<tr>
<td>Total sodium (%)</td>
<td>0.48</td>
<td>0.01061</td>
</tr>
<tr>
<td>Total phosphorus (%)</td>
<td>1.16</td>
<td>0.01414</td>
</tr>
</tbody>
</table>

The initial temperature of the substrate was 23°C. Gradually, temperature within the digester increased between 27°C and 35°C compared to the ambient (temperature before digestion). The observed temperature of this study was within the range of what is considered to be suitable for anaerobic digestion as cited by El-Mashad et al. (2004), that microorganisms exhibit optimal growth and metabolic rates within a well defined range of temperatures (20-35°C).

This result also tallied with that of Aremu and Agarry (2012) and Asikong et al. (2016) on similar studies. pH is an important factor of biogas production. Efficient digestion occurs at a pH near neutrality, within a range of 6.0-8.0. The pH of the MSW was acidic with the value of 5.49 but after pre-treatment of the waste with sodium hydroxide solution, pH reached 7.2-7.8. The results on total solids and volatile solids showed that the samples possessed suitable properties for their use in anaerobic digestion. The total solids (TS) of the feedstock (MSW) was 6560mg/L, this can be categorized as having a high solid content regarding the report of Budiyono et al. (2010) stating that, the optimum solid content obtained for biogas production is in the range of 7-9%.

### Biogas production, Purification and Upgrade

**Biogas production:**

Figure 2, Shows the volume of biogas produced during anaerobic digestion. The yield varied significantly (P<0.000) between the retention time (days). It was observed that gas production started after about 6days of experimentation and increased with time 16days when the production rate declined due to temperature fluctuation. Highest biogas yield was obtained within 26 days of digestion with the volume of biogas between 1070 and 1402cm³. Considerably low amount of gas was produced as from 30th day of experimental setup where between 199 and 201cm³ were produced (Fig. 2).

**Biogas Purification and Upgrade:**

The results obtained from gas purification and upgrade is interpreted in Fig. 3 and 4. These figures illustrate the characteristics of biogas produced before and after purification.

Figure 3, Presents the mean characteristics of the gas produced in percentages using the 4-in-1 gas analyzer. The result showed no gas production during the first week (0-5days). The maximum percentage CH₄ characterized was between 66.00 and 68.57% (30-35 and 25-30days). Percentage CO₂ was 37.32 and 39.50% (15-20 and 10-15days). Percentage H₂S was 0.81 and 1.89% (15-20 and 5-10days) and 2% for O₂ (25-30days). The least percentage CH₄ produced was at the second week obtaining a mean value of 10.50%, CO₂ (30.21) at 25-30days respectively (Fig. 3). The biogas produced was flammable, combustible with a blue flame and had a “rotten egg” odour.

Figure 4, Shows the percentages of biogas upgraded to bio-methane after a step wise purification process with iron sponge, water and silica gel. The purification varied significantly (P<0.000) while some showed positive correlation others are negatively correlated according to the statistical analysis (T-test). The maximum upgraded gas was achieved at 25-30days obtaining a mean value of 88% CH₄, 9.58% CO₂ with less values approximately 0% of other gases respectively (Fig.4).

Of the biogas produced during the hydraulic retention period (HRT) of 30 days (7 weeks). During the first week of digestion there was no gas production, this is similar to the findings of Al-moustapha et al. (2009) that reported gas production after day 7. This may be attributed...
to the accumulation of high volatile fatty acid (fats and oils) from the food residue which acted as inhibitors to biogas-producing organisms and could lead to scum formation (Egbere et al., 2010). The spiked increase in the volume of biogas in the third week (10-15 days) and its peak in the fifth week (20-25 days) indicated the acclimatization of the biogas producing microorganisms after the hydrolysis stage by the hydrolyzing organisms. This result corroborate with Rabah et al. (2010).

The action of methanogenesis for biogas production begins to decline at the sixth week (25-30). These may be due to several factors such as deposition of microbial metabolites, increase in temperature, decrease in pH, gradual exhaustion of nutrient from the substrate and replacement by organisms that tend to utilize some of the products of their action. This probably explained the continued decline in the volume of biogas in the seventh week (30-35days). This observation is in conformity with that of Asikong et al. (2016). Evidence of methane production was observed between the third (10-15days) and seventh week (30-35days) of digestion.

This study was set out to investigate the purification and upgrading processes of biogas using iron sponge, water scrubber and silica gel. The results obtained showed that hydrogen sulfide (H$_2$S) that can be involved in acidic rain if not properly removed from biogas was removed using iron sponge scrubber. This finding corroborates with that of Ebunilo et al. (2016). Also, the results proved that water scrubber had the ability to absorb or dissolve carbon (IV) oxide from the biogas produced, but was not able to absorb some escaped H$_2$S from the iron sponge scrubber as there were percentages of H$_2$S present in the bio-methane being purified. This is in contrast to the report of Ryckebosch (2011); that H$_2$S and CO$_2$ could dissolve in a water scrubber.

Silica gel has a great potential in absorbing water vapour or moisture from the gases being produced. The biogas produced from anaerobic digestion is usually saturated with water. Drying of the gas is most often necessary since some upgrading processes need relatively dry gas. Water vapour can be problematic. For example, when the water vapour passes from high to lower pressure it might condense into ice and be corrosive. The other problem is that it clogs pressure regulator in the distribution system’.

Fig. 2: Biogas produced from MSW
CONCLUSION

Organic fractions of municipal solid wastes (MSW) can be utilized as a sustainable energy source for biogas production and could eliminate wastes disposal problems by keeping the environment clean and could be important for saving in economic terms. Conversion of biogas to bio-methane has been achieved in this study; therefore, Nigeria needs to harness this process to boost its energy production and utilization. Also, the results showed that biogas can be purified easily using available local materials as scrubbers, the CO₂ and H₂S composition generated in the biogas was successively reduced to a minimal content, obtaining a high content of methane. Such purified biogas can be used to effect climate change by cleaner green energy production and creating a pollution-free atmosphere and thereby reducing the output of greenhouse gases (GHG). ‘If we can use locally available biomass for biogas production and purify the produced gas, we can make our rural areas self dependent for energy requirement’.
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