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Impact of Carbon-based Nanoparticles on Biogas Yield from Anaerobic Co-Digestion of Cow Dung and Yam Peels

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Abstract

The aim of this work is to evaluate the impact of carbon-based nanoparticles on biogas yield from anaerobic co-digestion of cow dung and yam peels. The biomass used for the work include sawdust, cow dung and yam peels. The sawdust was obtained from a saw mill and carbonized in a muffle furnace at 600°C. It was then ground into fine particles using hammer mill. The Nano-sized sample was chemically activated using 0.85 M phosphoric acid at a 1:1 (by weight) ratio of the sample to the dilute acid. The sample was washed with distilled water until a neutral pH of 7 was obtained. It was then dried in an electric oven at a temperature of 80°C for 3 hours to obtain the activated carbon-based nanoparticles (ACBNPs). The ACBNPs was characterized using DLS, SEM-EDS, FTIR, XRD and BET. The DLS show that it has a dominant particle size of 11.89 nm confirming that the sample fall within the nanoscale. The SEM results show that the sample has good morphology with contrasts on the surface while the EDS shows that the atomic concentration of carbon in the sample is 71.22, meaning that the carbonization process was successful. The BET results show it has a specific surface area of 250 m^2/g and mesoporous particles with good adsorptive properties. The cow dung was collected from a cattle market while the yam peels were collected from a restaurant, pretreated and put in a paste form. The substrates, cow dung and yam peels, were characterized. Microbial assay on the cow dung showed the presence of the microorganisms that are active in anaerobic digestion. Design of experiment (DOE) was generated (a total of 20 runs) using CCD of the design expert software to evaluate the individual and interactive effects of three factors - time, substrate ratio and nanoparticle concentration. Batch experiments were conducted and the daily biogas yields were measured by downward displacement of water in measuring cylinders. Experimental method was employed to evaluate the performance of the anaerobic digestion process while Response Surface Methodology (RSM) was utilized to model the digestion process. The DOE analysis using Response Surface Methodology (RSM) generated a quadratic model showing the individual and interactive effects of the independent factors on the biogas yield. The maximum value of the biogas yield, as predicted by RSM was 2649.5 ml, while the optimal factors were Time (12 days), Substrates mixing ratio (45% or CD: YP ratio of 9:11) and NPs concentration (164 mg/L). The results demonstrated that the addition of nanoparticles significantly improved biogas and methane yields compared to the control (without nanoparticles). Precisely, the biogas yield increased by 33.2% while the methane content increased by 4.1%. This improvement is attributed to enhanced microbial activity, improved electron transfer, and accelerated degradation of complex organic substrates.

Keywords: anaerobic digestion, cow dung, yam peels, nanoparticles, carbo

Introduction

The issue of global warming brought on by the use of fossil fuels has prompted a greater quest for renewable and ecologically suitable energy sources. In addition, the total volume of solid and organic waste produced is growing even more quickly, which could have detrimental effects on the environment if improperly handled (Abdelwahab, Mohanty, Sahoo, Behera and Fodah, 2021). The main feedstock for the production of bioenergy is solid waste, which includes municipal, dairy, and wastes from farms. (Obaideen *et al.*, 2022). Due to inadequate waste management methods, only livestock manure is utilized as fertilizer among all agricultural wastes in developing nations like Nigeria. Others are more or less allowed to putrefy, creating nuisance and unaesthetic environment to mankind. In Nigeria, for instance, cow dung are found at the cattle market and also littered along our major roads and farmlands where pastoral cattle herders graze their herds. A study of Anambra State's main slaughterhouses revealed that 15563 kg (15.6 tons) of fresh cow manure are produced per day (Umeghalu, Chukwuma, Okonkwo and Umeh, 2012).

Anaerobic digestion (AD) is the most important biological process that transforms organic materials into a kind of CH_4 by-product (Abdelsalam *et al.*, 2019). It is a set of biological processes where organic material is biodegraded by microorganisms in the absence of oxygen to produce final products like biogas (Abdelsalam and Samer, 2019). Anaerobic digestion is a promising renewable energy source for producing biogas, however process constraints frequently impede its conversion efficiency, process stability, product quality, and economic viability. Yam peels are one of the agricultural wastes that are mostly disposed carelessly without any attempt to harness the bioenergy in them. They contain protein, fibre as well as minerals and therefore, can be useful in biogas production especially co-digesting with cow dung to enhance C/N ratio. The simultaneous digestion of two or more substrates is known as co-digestion. It is typically done to improve the quality of the digestate, balance the nutrients due to their varying C/N ratio, increase the biogas yield, and support system stability. The stability of the anaerobic process may be improved by anaerobic co-digestion of various organic materials due to improved carbon to nitrogen (C/N) balance (Zhang, Xiao, Peng, Su and Tan, 2013).

Recent studies have demonstrated that the application of nanoparticles can significantly enhance biogas production. It has been observed that using nanoparticles, microelements, or substances smaller than 100 nm can increase the methane yield in AD by increasing the accessibility of anaerobic bacteria to the fiber content of harvested lignocellulosic waste (Ajayi-Banji, Pourhashem, Rahman and Feng, 2024). For instance, a meta-analysis by (Castro, Resende, Taveira, Enrich-Prast and Abreu, 2024) reported that nanoparticle addition can increase biogas yield up to several orders of magnitude under optimized conditions, although results may vary depending on nanoparticle type and operational parameters . This enhancement is largely attributed to improved microbial interactions, catalytic effects, and facilitation of direct interspecies electron transfer (DIET), a key mechanism in methanogenesis.

Among the various nanoparticle types, carbon-based materials (such as activated carbon and carbon nanotubes) have been shown to enhance electron conductivity within anaerobic digesters, thereby accelerating syntrophic microbial interactions and methane production (Ziganshina and Ziganshin, 2022) . In previous researches, there is a lack of integrated studies that simultaneously optimize key process parameters such as retention time, nanoparticle concentration, and substrate ratio, particularly in co-digestion systems involving locally available feedstocks such as yam peels. This study therefore, addresses this gap.

2.0 Materials and Methods

2.1 Materials

The ACBNPs was derived from sawdust obtained from a carpentry shop located at Enugu. The cow dung was collected from cattle market at Ugwuoba Garki, Enugu State while the yam peels were obtained from a restaurant at Ifite, Awka metropolis, Anambra State, Nigeria. The chemicals used were obtained from Bridge head market Onitsha and Chemical Engineering Laboratory, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria and were of analytical grade.

2.2 Methods

2.2.1 Preparation of the Substrates

Fresh wet cow dung and yam peels were used for this work. The substrate which served as a control was prepared by adding distilled water to the fresh cow dung at 1:1 volume ratio as suggested in the work by Farghali *et al.* (2020). On the other hand, the yam peels were first washed with distilled water to remove the dirts in them. They were then ground with a mechanical grinding machine to obtain a homogeneous liquid ready for characterization and anaerobic digestion.

2.2.2 Preparation of ACBNPs

The sawdust used in the experimental studies was obtained from a timbre shed located at Enugu. It was first washed with distilled water and then dried in an oven at 80ºC to remove moisture. The dried sample was placed in a tray and heated in a muffle furnace for carbonization at the temperature of 600ºC for 2 hours. The carbonized sample was then ground into fine particles using hammer mill. The nano-sized sample was chemically activated using 0.85 M phosphoric acid at a 1:1 ratio (by weight), of the sample and dilute acid. The sample was washed with distilled water until a neutral pH of 7 was obtained. It was then dried in an electrical oven at a temperature of 80°C for 3 hours and stored for use as nanoparticles for enhancement of biogas production.

2.2.3 Characterization of ACBNPs and Substrates

The physicochemical properties of the substrates were determined using AOAC methods. The morphology of the ACBNPs were examined using Scanning Electron Microscope (SEM) while XRF analysis was carried out for elemental composition. In addition, BET was carried out to obtain the specific surface area, DLS for the particle size distribution, FTIR for the functional groups present and XRD, to determine the internal atomic structure of ACBNPs.

2.3 Batch Anaerobic digestion experiment

Central Composite Design (CCD) of the Design Expert software was used to generate the Design of Experiment (DOE) (Table 1) which involves 20 digesters. The factors are A-time (days), B-NPs concentrations and C-Substrates ratio while the response is the biogas yield (Y). Table shows the upper and lower limits that were set for the factors. It shows a range of 3 factors used in the experiment. These factors with their selected range were used to predict the response which is the biogas yield and to determine the model that best fits the data. To obtain the substrate volume of 1.6 L, fresh cow dung (CD) and yam peels (YP) mixed in different volume ratios to obtain 800 ml of substrate. It was mixed with 800 ml of distilled water to obtain a slurry. The slurry was introduced into a 3-litre biodigester made of plastic with working volume of 1.6 litres and a headspace of 1.4 litres. The daily biogas yield was measured using the method of downward displacement of water in a measuring cylinder. The set-up was replicated for the 20 runs. An additional digester comprising of 400 ml of CD and 400 ml of YP was equally set up as a blank or control (without nanoparticles).

Table 1: Parameters and their Levels for the Biogas Process from CCD

Factor	Name	Units	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	Time	Days	3.95	14.05	-1 ↔ 6.00	+1 ↔ 12.00	9.00	2.54
B	NPs conc.	mg/L	65.91	234.09	-1 ↔ 100.00	+1 ↔ 200.00	150.00	42.39
C	Substr. ratio	%	7.96	92.04	-1 ↔ 25.00	+1 ↔ 75.00	50.00	21.20

Table 2: Design of Experiment (DOE) for the batch experiments

Run	A:Time (Days)	B:Nano.Conc. (mg/L)	C:Substr.Ratio (%)	Biogas yield (ml)
1	12	200	75	
2	12	100	75	
3	6	200	75	
4	9	150	50	
5	6	200	25	
6	12	100	25	
7	6	100	75	
8	9	150	50	
9	9	150	8	
10	9	150	50	
11	6	100	25	
12	4	150	50	
13	9	66	50	
14	12	200	25	
15	9	150	50	
16	9	150	50	
17	9	150	92	
18	14	150	50	
19	9	234	50	
20	9	150	50	

3.0 Results and Discussion

3.1 Characterization Results of ACBNPs

3.1.1 FTIR of ACNPs

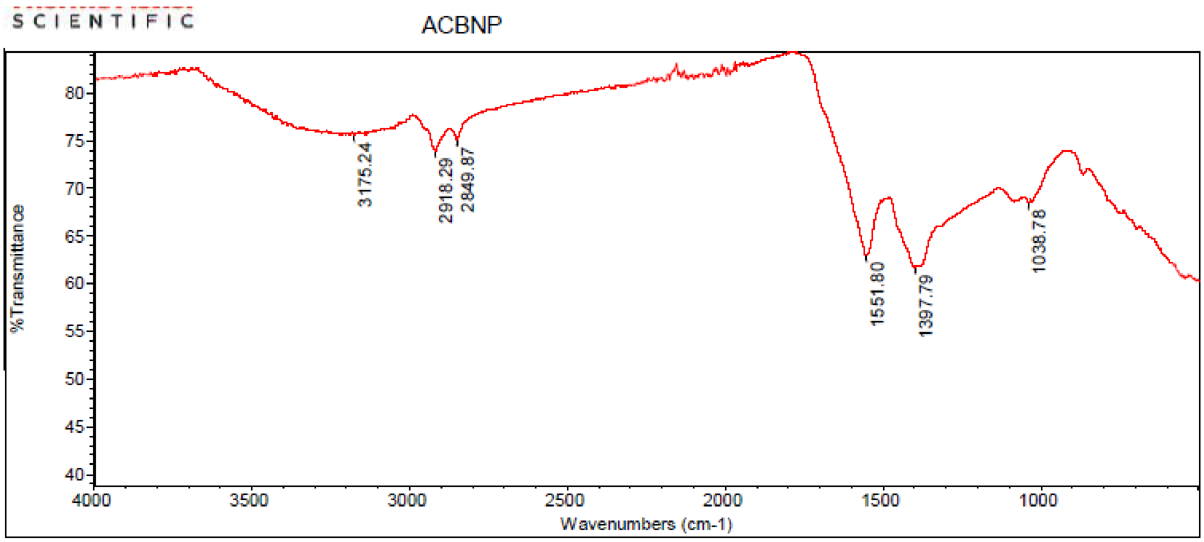

Figure 1: FTIR analysis of ACBNPs

Table 3: Peak, intensity and Assignments of FTIR analysis of ACBNPs

Peak (cm-1)	Intensity (Absorbance)	Assignment	Functional group identification
3175.24	6-medium	N-H stretching (amine/amide) or O-H stretching(hydrogen bonded hydroxyl)	Cellulose, hemicellulose, adsorbed moisture
2918.29	4-medium	C-H asymmetric stretching (aliphatic –CH2- groups))	Cellulose backbone chains and lignin side chains
2849.87	3-medium	C-H symmetric stretching (aliphatic –CH2- groups)	Cellulose backbone chains and lignin side chains
1551.80	7-strong	C=C aromatic stretching or N-H bending (amide) or COO- asymmetric stretching or C-N stretching	Phenylpropane units in lignin
1397.79	8-very strong	C-H bending or COO- symmetric stretching or C-N stretching	Cellulose and hemicellulose
1038.78	9-very strong	C-O stretching	Ethers, alcohols or phenolic groups

The FTIR spectrum which is in the transmittance mode shows corresponding absorbtion peaks. Table shows the major peaks were separated and allotted based on the standard Infra Red correlation tables for functional groups. The relative numerical intensity, numbered from 1 to 10, describes how strong or weak an absorption peak appears compared to others in the same spectrum. It is based on the depth of the transmittance dip relative to the baseline. The classification is as follows: High intensity is represented by 8-10; medium or moderate intensity, 4-7 and lower intensity, 2-4. Table 3 shows that the spectrum is consistent with a material containing aliphatic chains, aromatic/unsaturated groups, and oxygen/nitrogen-containing functionalities (typical for functionalized carbon nanoparticles).

3.1.2 SEM-EDS

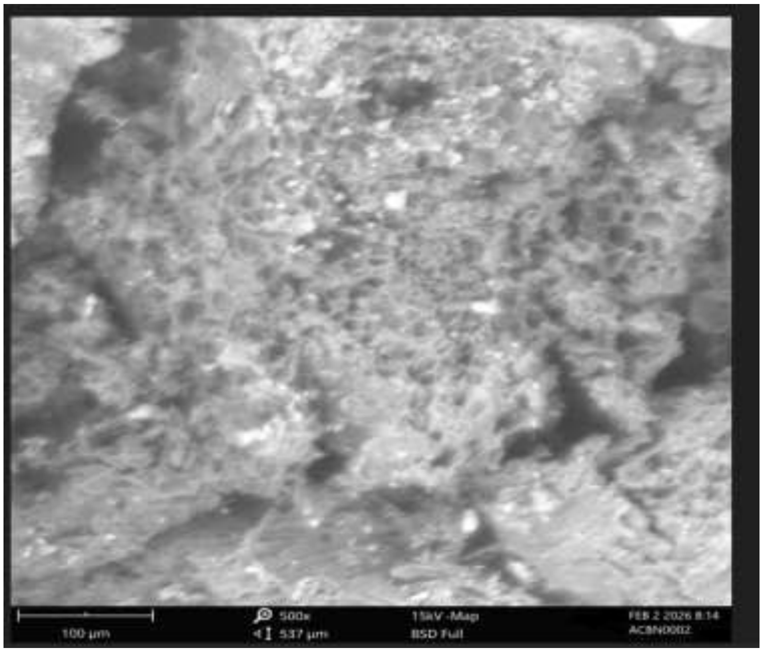

Plate 1: SEM micrograph of ACBNPs

The SEM micrograph of ACBN at 500× magnification reveals a highly porous, irregular surface morphology characteristic activated biomass carbon. The extensive macropore network (5–50 µm) with interconnected channels facilitates mass transport to the internal micropore structure. This morphology, combined with the high carbon content (69.41 atomic%) and substantial residual potassium (12.24 atomic%), confirms successful development of a porous activated carbon structure while highlighting the need for optimized washing protocols to remove inorganic residues.

3.13 XRD of ACNPs

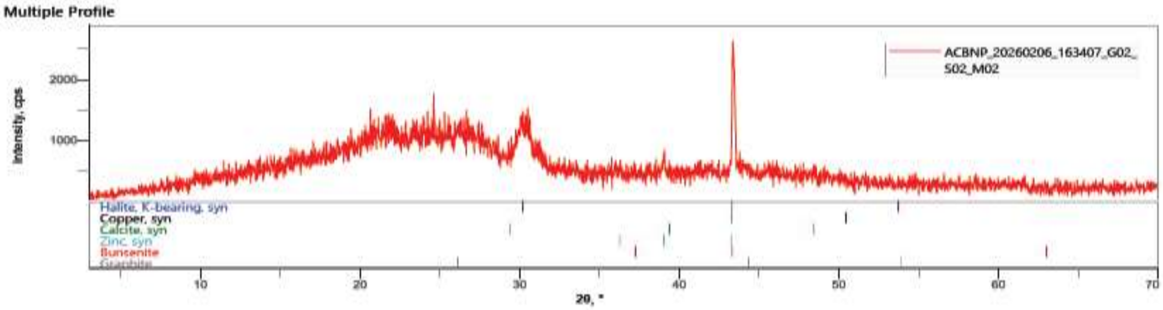

Figure 2: XRD analysis of ACNPs

The diffraction pattern of the ACBNP sample reveals a wide, diffuse hump between about 15 and 35° (2θ). This suggests that the structure is mostly amorphous carbon structure expected for activated carbon possibly made up of disordered silica and leftover organic-mineral phases. It is structurally reactive and contains a few and moderate-intensity crystalline peaks on top of this amorphous background. This suggests that there are small amounts of crystalline phases, like quartz (SiO_2) and calcium-based minerals (e.g., calcite). Previous studies reveals that materials with a higher amorphous content, such as sample ACBNPs, typically exhibit: greater surface area and porosity, enhanced adsorption capacity and improved microbial attachment. These properties facilitate enzyme accessibility and substrate breakdown, thereby accelerating hydrolysis and early-stage digestion.

3.1.4 Dynamic Light Scattering (DLS) of ACBNPs

Table 4: Dynamic Light Scattering (DLS) results

Particle size distribution by intensity				Particle size distribution by volume		
Sample	Z-average Particle size (nm)	Dominat particle size (nm)	Polydisperity index (PDI)	Z-average Particle size (nm)	Dominat particle size (nm)	Polydisperity index (PDI)
ACNP	55.92	15.00	0.234	55.92	11.89	0.234

The DLS reports (Table 4) shows that ACBNPs weighted mean particle size of 55.92 nm. On the other hand which shows that the sample is a nanoparticle since the particle size is less than 100 nm. Predominant particle size is 15 nm by intensity and 11.89 nm by volume distribution with minor aggregation. The PDI is 0.234 which lies in the range of 0.1 – 0.3, showing that there is moderately narrow size distribution and the acceptable uniformity.

3.1.4 Characterization results of the substrates

Table 5: Physicochemical properties of the substrates

Parameters	**Yam peel**	**Cow dung**
Moisture content (%)	56.217	66.154
Ash content (%)	3.869	1.113
pH	4.85	7.25
Total solids (%)	43.783	33.846
Hemicellulose (%)	20.765	11.855
Cellulose (%)	18.541	11.656
COD (mg/L)	480	320
Total Nitrogen (%)	1.232	3.192
Total organic carbon (%) blend	19.711	25.321
Total volatile solid (%)	36.399	15.278

Table 5 shows the characterization of the YP and CD on wet basis.

3.1.5 Microbial Assay

Table 6: Microbial Assay results

Bacteria	**Status**	**Participatory stage**	**Min.Duration (days)**
Archaea spp	Present	Methanogenesis	5-10
Escherichia coli (E.coli)	Present	Acidogenesis	1-2
Salmonella enteric	Absent		
Staphylococcus aureus	Absent		
Bacillus spp	Present	Hydrolysis	1-2
Mycobacterium tuberculosis	Absent		
Pseudomonas aeruginosa	Present	Hydrolysis, acetogenesis	1-2, 2-3
Vibrio cholera	Present	Acidogenesis	1-2
Streptococcus pneumonia	Absent		

Table 6 shows the bacteria present and those absent in a cow dung sample analyzed. Bacillus spp. and Pseudomonas aeruginosa break complex organic polymers into simple soluble compounds. They secrete enzymes that break down cellulose, proteins, and fats into sugars, amino acids and fatty acids respectively. Vibrio cholera and E. coli are the acidogenic bacteria found in the sample. Both convert the sugars (by fermentation) and amino acids to volatile fatty acids, hydrogen and carbon dioxide. Pseudomonas aeruginosa also play a role in the acetogenesis step. They convert the organic acids into acetic acid, hydrogen and carbon dioxide. Finally, Archaea spp, the methanogens present, convert the acetic acid into methane and carbon dioxide.

3.2 ANOVA Study

Table 7: Biogas yield of the anaerobic digestion involving ACBNPs using RSM

Source	**Sum of Squares**	**df**	**Mean Square**	**Estimated coeffs,**	**F-value**	**p-value**	
Model	1.535E+07	9	1.706E+06	2461.58	246.88	< 0.0001	significant
A-Day	1.335E+05	1	1.335E+05	98.88	19.32	0.0013	
B-Nano. conc	42419.83	1	42419.83	55.73	6.14	0.0327	
C-Substr. ratio	4.095E+05	1	4.095E+05	173.17	59.26	< 0.0001	
AB	24200.00	1	24200.00	55.00	3.50	0.0908	
AC	99012.50	1	99012.50	-111.25	14.33	0.0036	
BC	6.938E+06	1	6.938E+06	-931.25	1003.92	< 0.0001	
A^2	89827.97	1	89827.97	78.95	13.00	0.0048	
B^2	3.502E+06	1	3.502E+06	-492.92	506.68	< 0.0001	
C^2	4.478E+06	1	4.478E+06	-557.45	648.01	< 0.0001	
Residual	69107.48	10	6910.75				
Lack of Fit	69107.48	5	13821.50				
Pure Error	0.0000	5	0.0000				
Cor Total	1.542E+07	19					

3.3 Model Validity

The ANOVA study presented in Table 7 shows that the whole model is valid and adequate to represent the data. This is validated by the fact that the p-value is significant ($p<0.05$), lack-of-fit p-value is insignificant, R^2 and predicted R^2 are close to 1. Also, the coefficient of variation is low showing that the data is accurate and can be reproduced.

3.4 RSM Model Equation and Interpretation of the Coefficients Involving ACNPs

A model equation was developed by RSM for the response variable (Y) as a function of three independent variables as shown in Table 7. The model equation generated is quadratic showing the individual or interactive effects of the factors. The model equation generated is given by:

$$Y = 2461.58 + 98.88A + 55.73B + 173.17C - 111.25AC - 931.25BC + 78.95A^2 - 492.92B^2 - 557.45C^2 \quad (1)$$

Where,
Y = biogas yield
A = time
B = nanoparticle concentration
C = substrate ratio

Positive signs on the coefficients in equation (1) show that the associated factors have positive effect on the yield while negative signs show the opposite. For example, -111.25AC implies that increasing both time and the substrates ratio at constant NPs concentration will decrease the biogas yield. The magnitude of the coefficients shows factors with dominant impact on the response. Also, the interaction terms (BC, AC etc) show the interdependence of the two factors that are placed together.

Table 8: Model Summary Statistics

Std. Dev.	83.13	R²	0.9955
Mean	1798.25	Adjusted R²	0.9915
C.V. %	4.62	Predicted R²	0.9634
		Adeq Precision	45.5292

The Predicted R² of 0.9634 is in reasonable agreement with the Adjusted R² of 0.9915; since the difference between them is less than 0.2. In addition, Adeq Precision of ratio 45.529 indicates an adequate signal. This model can be used to navigate the design space.

3.5 Cumulative Biogas Yields

Table 9: Comparison of the Experimental and RSM predicted Biogas Yields of ACBNPs

Run	:Time (Days)	B:Nano. Conc. (mg/L)	C:Substr. Ratio (%)	Biogas Yield (ml)	RSM Predicted
1	12	200	75	840	830.45
2	12	100	75	2595	2471.48
3	6	200	75	835	745.18
4	9	150	50	2465	2461.58
5	6	200	25	2000	2038.83
6	12	100	25	480	485.14
7	6	100	75	2580	2606.21
8	9	150	50	2465	2461.58
9	9	150	8	550	593.64
10	9	150	50	2465	2461.58

11	6	100	25	250	174.87
12	4	150	50	2500	2518.58
13	9	66	50	915	973.65
14	12	200	25	2680	2569.10
15	9	150	50	2465	2461.58
16	9	150	50	2465	2461.58
17	9	150	92	1100	1176.12
18	14	150	50	2750	2851.18
19	9	234	50	1100	1161.11
20	9	150	50	2465	2461.58

Table 9 shows the cumulative biogas yield of 20 runs used in the experiment. Run 18, containing 150mg/L of ACBNPs the highest yield, a cumulative biogas yield of 2750 ml in 14 days. The results show that ACBNPs improved biogas production, This agrees with the findings by (Hassanpourmoghadam *et al.*, 2023) However, excessive NPs concentration led to reduced yield, consistent with the inhibitory effects reported by (Kumar *et al.*, 2021). Also, the RSM predicted values is consistent with the experimental values, showing that RSM is a suitable tool for modelling or predicting anaerobic digestion processes.

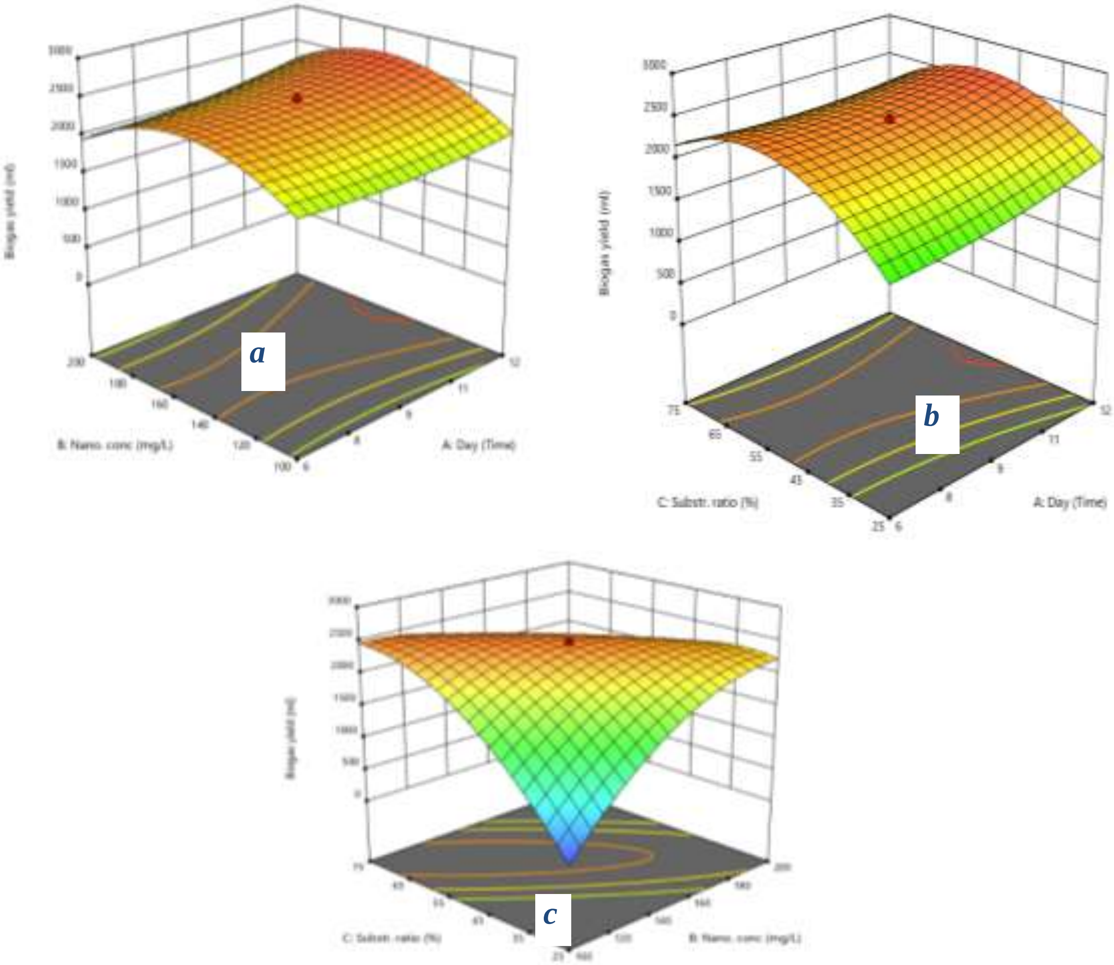

Figure 3(a-c): 3D interaction of the various factors that affect the biogas yield

Figure 3(a) shows that at constant substrate ratio of 1:1, increasing both time and NPs concentration leads to increase in biogas yield up to a maximum yield of about 2700 ml. Afterwards, there was decrease in yield. The NPs concentration that gave this yield is 150 mg/L substrates in about 10 days. On the other hand, Figure 3(b) shows the combined effect of both time and substrates ratio at constant NPs concentration of 150 mg/L. Initially, there was increase in biogas yield up to a maximum yield of about 2700 ml, afterwards, there was decrease in the yield. The substrate ratio that gave this yield is 50% (1:1) in about 11 days. Figure 3(c) shows that there is a strong interaction between the NPs concentration and substrates ratio. Both must be maintained at a particular range for optimal yield at constant substrate time.

3.6 Optimization studies for RSM

The optimization process was run generating about 35 iterations. The optimum value of the biogas yield was 2649.5 ml, while the optimal factors were Time (12 days), Substrates mixing ratio (45% or CD: YP ratio of 9:11) and NPs concentration (164 mg/L).

3.7 GC Analysis of Biogas from ACBNPs-Enhanced Process

Figure 4: GC analysis of ACBNPs

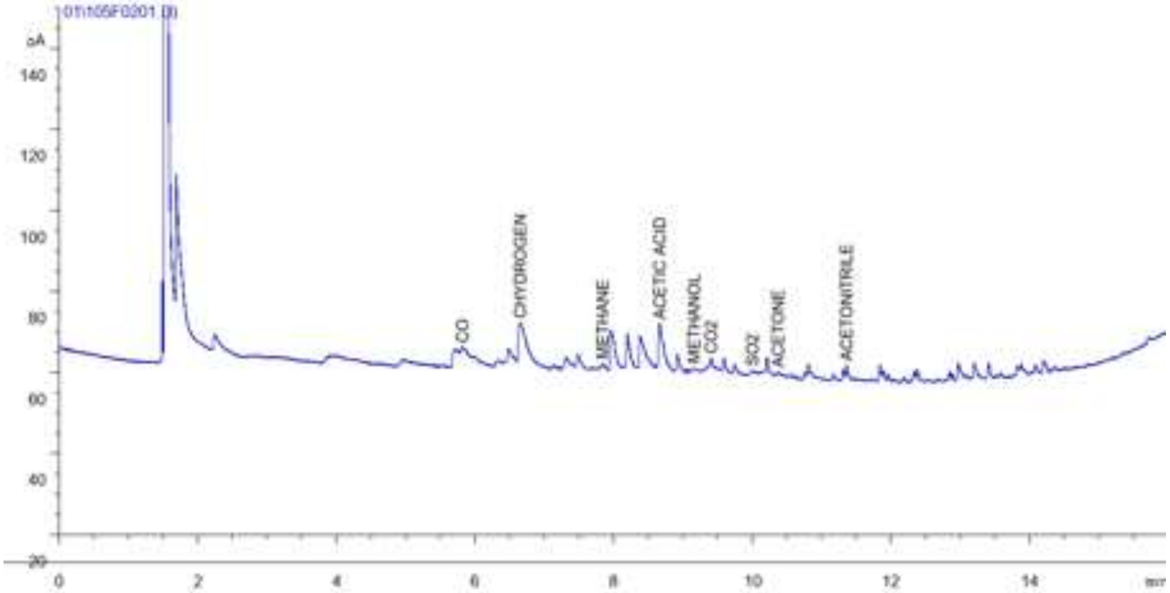

Table 10: GC results

Sample	Methane (%)	CO2 (%)	CO (%)	Acetic acid	Hydrogen (%)	Ethyl acetate (%)	SO2 (%)	Acetone (%)	O2 (%)	Acetonitrile (%)	Methanol (%)
Blank	77.21	11.76	0.01	0.09	7.59	-	1.18	1.06	‘-	1.10	0.09
ACNPs	80.35	9.02	0.211	0.02	7.81	-	0.66	0.78	-	0.42	0.73

Table 10 shows that the methane content from the ACBNPs-enhanced processes are higher than the one obtained in the blank. This is possibly due to that the ACBNPs adsorb some of the inhibitor present in the system and consequently promote the methane yield of the entire process in agreement with the report in the literature.

4.0 Conclusion

This study investigated the impact of carbon-based nanoparticles on biogas yield from anaerobic co-digestion of cow dung and yam peels. The research employed experimental anaerobic digestion and Response Surface Methodology (RSM). The results demonstrated that the addition of nanoparticles significantly improved biogas and methane yields compared to the control (without nanoparticles). This improvement is attributed to enhanced microbial activity, improved electron transfer, and accelerated degradation of complex organic substrates. Among the tested conditions, an optimum range of substrate composition, retention time, and nanoparticle dosage was identified through RSM optimization, leading to maximum methane production efficiency. The optimum value of the biogas yield was 2649.5 ml, while the optimal factors were Time (12 days), Substrates mixing ratio (45% or CD: YP ratio of 9:11) and NPs concentration (164 mg/L). Experimental results equally revealed that there was 33.2 % increase in biogas yield and 4.1 % increase in the methane content when ACBNPs was employed in the co-digestion of CD and YP compared when there is no addition of NPs.

5.0 Recommendation

Further research should investigate other nano-additives or hybrid nanomaterials to compare performance and identify more cost-effective alternatives. Real-time monitoring and control systems. Implementation of AI-based real-time monitoring systems is recommended to enhance process stability and improve operational control in large-scale digesters.

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Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used ChatGPT in order to interprete some results. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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