CHEMICAL ENGINEERING TRANSACTIONS

VOL. 113, 2024

A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.cetjournal.it

Guest Editors: Jeng Shiun Lim, Bohong Wang, Guo Ren Mong, Petar S. Varbanov
Copyright © 2024, AIDIC Servizi S.r.l.
ISBN 979-12-81206-14-4; **ISSN** 2283-9216

DOI: 10.3303/CET24113006

Optimising Anaerobic Co-digestion of Sewage Sludge and Municipal Landfill Leachate with Sugarcane Bagasse derived Biochar using Response Surface Methodology

Denzil E. Estrice*, Manimagalay Chetty

Department of Chemical Engineering, Faculty of Engineering and the Built Environment, Durban University of Technology, 70 Steve Biko Rd, Musgrave, Berea 4001, South Africa.
21549710@dut4life.ac.za

Anaerobic co-digestion (ACD) is prominent as a technology that offers a sustainable alternative energy source in biogas while contributing to sustainable waste management. However, several issues linked to process stability and performance remain, including slow anaerobe acclimation and growth. This study aimed to optimise the anaerobic co-digestion of sewage sludge and municipal landfill leachate by establishing the optimal operating conditions for ACD with biochar addition. Response surface methodology (RSM) Box-Benken design (BBD) was utilised to investigate the impact of critical process parameters: temperature, co-substrate loading rate, and Biochar loading rate on biogas yield. The response model underwent analysis of variance (ANOVA) analysis to determine the relevant elements that were accurately incorporated into the models. The model was determined to have a confidence level of 95 %. The adep accuracy, which quantifies the ratio of the signal to the noise, met the acceptable criterion of being more than 4. The ratio for Biogas Yield achieved was 15.9195. The optimum operating conditions were observed to be co-substrate loading of 1:20 (leachate; total reactor volume), BC loading of (6.7 g/L), and a temperature of 54.99 °C, achieving desirability of 94.10 %. A mean biogas yield of 60.45 mL/gVS$_{added}$ was achieved under the optimum conditions.

1. Introduction

Addressing the challenges of waste management and ensuring access to clean energy are crucial steps in our collective journey towards a sustainable and fair future (Kay Lup et al., 2023). These barriers align with the United Nations' Sustainable Development Goals (SDGs), which were ratified in 2015. Oil, coal, and natural gas have long been the backbone of the modern industrial world, providing the energy needed to keep everything running smoothly. However, it is important to note that fossil fuels are becoming scarcer and are closely linked to the issue of global warming and its effects on the environment. Transitioning to other forms of renewable energy is crucial in order to address climate change and reduce our reliance on fossil fuels (Osman et al., 2023). The rapid industrialization of numerous developing countries has led to a rise in the production of industrial and municipal wastewater, which is known for its significant organic matter content. When properly processed, wastewater has the potential to be a valuable and environmentally friendly energy source. This can be achieved through the production of biogas using anaerobic digestion, as demonstrated by Rajeshwari et al. in 2000. Wastewater treatment works produce sewage sludge, which is commonly used as a feedstock for biogas generation. This is because sewage sludge has a higher biochemical methane potential in comparison to other feedstocks used for biogas production. According to Bachman (2015), it is believed to possess an energy capacity ten times greater than what is needed for its treatment. Sewage sludge, which is a key component in wastewater treatment works (WWTW), consists of both secondary and primary sludge. Davidsonn (2007) found that under ideal conditions, primary sludge has the potential to generate 300 – 400 (Nm^3/t organic dry matter) of biomethane. In addition, recent studies and analysis of SS have revealed that it possesses an energy content of approximately 19 MJ (5.2 kWh/kg) on a dry weight basis. This energy content is comparable to that of low-grade coal, as stated by Musvoto et al. (2018). Anaerobic co-digestion is widely recognized as a viable treatment method for both municipal and industrial wastewater. Similar to mono-substrate digestion, co-digestion also

Paper Received: 28 April 2024; Revised: 7 September 2024; Accepted: 22 October 2024
Please cite this article as: Estrice D.E., Chetty M., 2024, Optimising Anaerobic Co-digestion of Sewage Sludge and Municipal Landfill Leachate with Sugarcane Bagasse Derived Biochar Using Response Surface Methodology, Chemical Engineering Transactions, 113, 31-36 DOI:10.3303/CET24113006

holds promise for energy generation. Applying anaerobic co-digestion as a treatment method offers dual benefits. Firstly, it provides a sustainable solution for treating concentrated organic industrial wastewater. Secondly, it serves as a viable mechanism for producing sustainable energy, specifically green biogas. Despite the widespread recognition of the benefits of co-digestion, its implementation in wastewater treatment plants (WWTW) is still limited. There are several factors that can limit the effectiveness of biogas production, such as the absence of suitable co-substrates and the uncertainty surrounding their impact on the quality of biogas and biosolids output. According to Moretta (2023), enhancing biogas yield and quality can be achieved by implementing process optimization techniques, such as substrate pretreatment procedures and the utilization of additional materials (Obileke et al., 2024). Conductive carbonaceous compounds, such as Biochar (BC), have garnered significant interest as additives in anaerobic digestion due to their ability to enhance methane production (González et al., 2018). Biochars are carbonaceous materials with a porous structure that are created through the thermochemical conversion of biomass in an environment with low oxygen levels (Pan et al., 2019). In their study, Nanda et al. (2016) highlighted the numerous advantages of BC compared to other additives. They emphasized the ability to produce BC with a wide range of physicochemical properties through careful selection of feedstock, pyrolysis conditions, and activation processes. Research has confirmed the possibility of increasing methane production through the addition of BC. This has been supported by various potential mechanisms, such as enhancing buffering capacity, mitigating inhibitory effects, immobilizing biomass, facilitating syntrophic metabolisms, improving digestate quality, and upgrading biogas (Pecchi and Baratieri, 2019). Thus, the objective of this study is to enhance the anaerobic co-digestion of sewage sludge and municipal landfill leachate by determining the most favourable operating conditions for ACD with the inclusion of biochar. The optimization process was carried out utilizing design of experiment (DOE) techniques, specifically response surface methodology (RSM). The utilization of RSM served as a technique for optimization, as well as for the identification and quantification of the relationship between different variables and the corresponding outcomes.

2. Material and Methods

2.1 Biochar synthesis

The sugarcane bagasse was obtained from a nearby Tongaat Hullet sugar refinery in Kwa-Zulu Natal province. The bagasse underwent a thorough cleansing process to eliminate any traces of soil and other undesirable substances. It was then finely milled to achieve a size of 3 cm. Following the milling process, the bagasse was carefully dried in a specialized oven set to a precise temperature of 105 °C for a duration of 24 h. The sugarcane bagasse underwent pyrolysis at three distinct temperatures: 350 °C, 450 °C, and 550 °C. This process took place for a duration of 30 min within a muffle furnace, resulting in the production of biochar. The entire procedure was carried out in a 1 L lab-scale pyrolysis reactor. The biochar produced was ground into small particles measuring between 0.8 and 2.0 mm in size. The size of the particles was determined using particle size analysis.

2.2 Characterization of biochar

The synthesized biochars were characterized to validate the synthesis by examining the morphology, which was analyzed using scanning electron microscopy (SEM) techniques. The analysis was performed using the Nova Nano SEM, in conjunction with EDT and TLD detectors.

2.3 Design of Experiments

The Box-Behnken design was utilized to assess three process parameters using Design-Expert software (version 11.1.2.0). The variable being assessed was biogas yield, which is used to evaluate the performance of the process. The response variables underwent statistical analysis using RSM to evaluate the impact of interactions between the process components on the Biological Methane Potential (BMP) process. The experimental data obtained from the BBD runs were utilized to develop regression models for the process by fitting a generic model, as documented by Ghaleb et al. (2020). factors and coded levels. Table 1 presents the design of experiment.

Table 1: DOE factors and coded levels

Process parameter	Factor code	Coded level		
		-1	0	+1
Temperature	A	25	37	55
ISR	B	1:2	1:1	1.5:1
Biochar loading (g/L)	C	2.5	5	10

Eq(1) below presents the generic multi-varied regression model equation:

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \alpha_3 C + \beta_4 D + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta 44 D^2 s + \beta_{12} AB + \varepsilon \quad (1)$$

3. Results and Discussion

3.1 Biochar Characterization

Figure 1a-f displays SEM micrographs (50 and 20 kx) of biochars produced at different temperatures. The BC350 image indicated that the biomass had undergone a series of changes, resulting in the formation of a unified mass of vesicles through softening, melting, and merging Figure 1a. The vesicles are a result of the release of volatile gases from the biomass. As temperatures increased, the biomass released more volatile gases. Subsequently, the vesicles located on the surface of BC450 experienced rupture when subjected to a decrease in temperature. As a result, the morphology of BC450 exhibited a range of pore configurations. Displayed in Figure 1f. The SEM images clearly illustrate that the biochar materials have a flake-like morphology, with sizes varying in the micrometer range. On the other hand, the breakdown of the porous structure was observed at higher temperatures and is clearly visible in the BC550 sample. This phenomenon can be explained by the intensified and accelerated process of carbonization (Niju et al., 2019). BC450 displayed the highest concentration of micropores on its surface when observed at a field view of 4.13 μm, making it the optimal choice for the anaerobic digestion process.

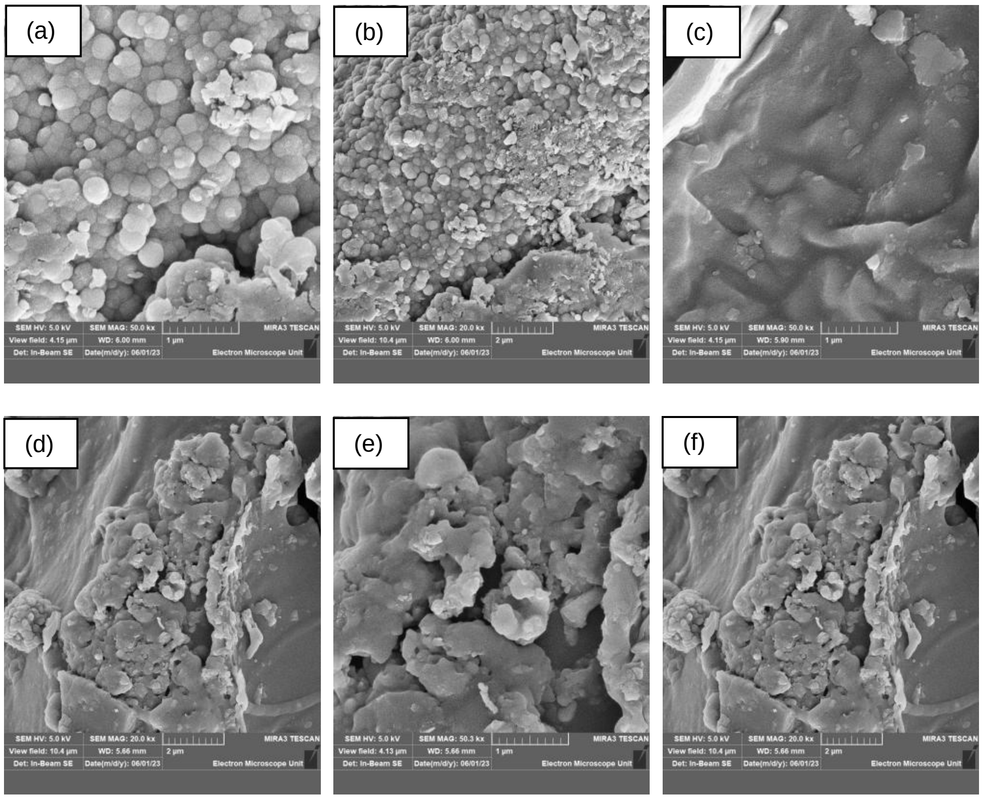

Figure 1: SEM images of (a) BC 350; (b) BC 350; (c) BC 550; (d) BC 550; (e) BC 450; (f) BC 450

3.2 Response surface methodology

An analysis of variance, also known as ANOVA, was conducted to evaluate the statistical significance of the model. An optimization technique was utilized to optimize the desired objectives, such as maximizing the yield of Biogas (measured in mL/gVS_{added}), COD, and VS removal (measured as a percentage). Ultimately, model validation was carried out. The fit of the model was evaluated using various statistical measures, including the adjusted R2, coefficient of determination (R2), coefficient of variance (CV), standard deviation (SD), and appropriate precision (AP). In addition, the significance of the model equations for each output parameter was assessed using Fisher's F-values and corresponding p-values at a 95 % confidence level. The results can be

located in Table 2. The experimental results were analyzed using a second-order polynomial equation to create a model for the biogas system. The Design Expert software presented a quadratic model for response 1, which was expressed as a function of the temperature, ISR, and biochar loading. In addition, the model incorporated the total of the constants, three linear effects (A, B, C), three quadratic effects (A^2, B^2, C^2), and three interaction effects (AB, AC, BC). Based on the analysis, it has been determined that the most effective sequence of interacting factors for maximizing Biogas yield is (AC > BC > AB).
The model equations were derived using coded values for Biogas Yield, as shown in Eq (2).

$$\text{Biogas yield} = 3.529 + 18.7166A - 5.37982B + 7.53929C - 0.837095\,AB + 9.01348AC + 0.725124BC + 3.63431A^2 + 20.6419B^2 + 15.0254C^2 \quad (2)$$

Table 2: ANOVA regression model (quadratic) for biogas yield (mL/gVS$_{added}$)

Source	Sum of Squares	df	Mean Square	F-values	p-values	
Model						
A-Temperature	2802.50	1	2802.50	153.94	< 0.0001	
B-Cosubstrate loading ratio	231.54	1	231.54	12.72	0.0161	
C-Biochar loading rate	454.73	1	454.73	24.98	0.0041	
AB	2.80	1	2.80	0.1540	0.7109	
AC	324.97	1	324.97	17.85	0.0083	
BC	2.10	1	2.10	0.1155	0.7477	
A^2	48.77	1	48.77	2.68	0.1626	
B^2	1573.25	1	1573.25	86.42	0.0002	
C^2	833.59	1	833.59	45.79	0.0011	
Residual	91.03	5	18.21			
Lack of Fit	71.75	3	23.92	2.48	0.3001	not significant
Pure Error	19.27	2	9.64			
Cor Total	6158.02	14				
Std. Dev.	4.27	-	-	-	-	-
Mean	24.49	-	-	-	-	-
C.V. %	17.42	-	-	-	-	-
R^2	0.9852	-	-	-	-	-
Adjusted R^2	0.9586	-	-	-	-	-
Predicted R^2	0.8065	-	-	-	-	-
Adeq Precision	15.9195	-	-	-	-	-

3.3 Interactive influence of input factors on biogas yield.

The 3D and contour plots in Figure 2 are graphical representations of the impact of each input factor on biogas yield. The temperature and BC loading were shown to be the most sensitive parameters, with co-substrate loading being the next most influential.
Figure 2 illustrates the correlation between Temperature, Co-substrate, and Biochar loading rate on biogas yield, as depicted in the graphical representation. The biogas yield demonstrates a positive correlation with temperature, reaching its highest point at 55 °C (coded factor 1). Studies have demonstrated that thermophilic digestion is highly effective in destroying viruses and pathogens while also maximizing biogas production. Additionally, it has been observed that thermophilic digestion leads to higher concentrations of volatile fatty acids (VFAs), which can impact the stability of the process and potentially result in process failure (Al-Sulaimi et al., 2022). A notable correlation was found between the biochar loading and the biogas yield, reaching its maximum at a loading of 10 g/L. Studies have shown that biochar can accelerate the breakdown of complex organic matter, which may result in higher methane production by boosting the performance of certain enzymes. This, in turn, could lead to an increase in soluble organic compounds in anaerobic digestion systems used for sludge treatment (Khalid et al., 2021).

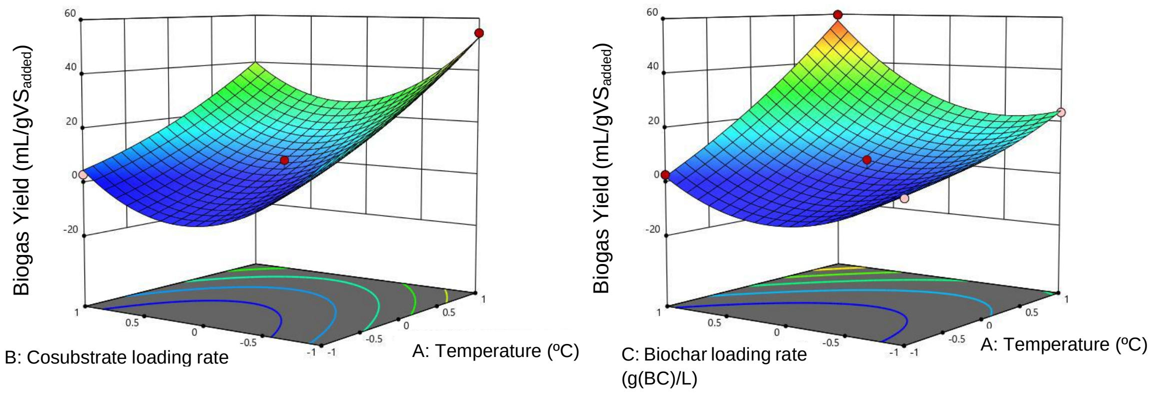

Figure 2: Impact of input factors on biogas yield

3.4 Process optimisation and model validation.

The numerical optimization tool in RSM was utilized to optimize the three input variables. Table 3 illustrates that all process responses were optimized, while the input variables (the Co-substrate loading, temperature, and BC loading) remained within the specified range. Figure 3 displays the ramp graphs illustrating the optimal operating conditions and the gained desirability. It was found that by maintaining a temperature of 54.99 ºC, at a co-substrate loading ratio of 1:20, and a BC loading rate of 6.7 g/L, a biogas yield of 59.75 mL/gVS $_{(added)}$ was achieved, with a performance rating/desirability of 94.00 %.

Table 3: Factor parameters

Factor	Goal	Lower limit	Upper limit
A: temperature	Is in range	25	55
B: Co-substrate loading rate	Is in range	1:20	1:5
C: Biochar loading rate (gBC/L)	Is in range	2.5	10

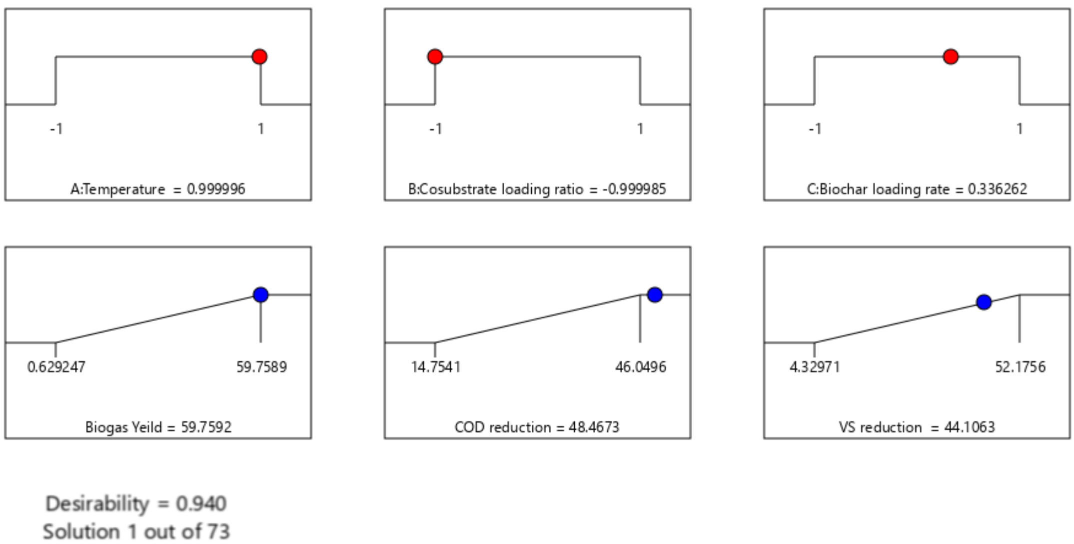

Figure 3: Ramp plot for optimum operating conditions

Table 4 displays the three separate runs of experiments for each process that were conducted under optimal operating conditions to assess the suitability and validity of the suggested models. The experiment resulted in an average biogas yield of 60.45 mL/gVS%. The observed value closely aligns with the anticipated value, exhibiting a discrepancy of less than 5 %. This indicates that the models possess the capability to effectively depict the relationship between observed and projected outcomes. The observed results fall within the predicted interval values (PI), encompassing both the low and high ends. Hence, the model for biogas yield exhibited a commendable level of predictive accuracy.

Table 4: Model validation

Analysis	Predicted mean	Standard deviation	n	SE Pred	95 % PI low	Data mean	95 % PI high
Biogas yield (mL/gVS$_{added}$)	52.7388	4.26676	3	4.44099	41.3229	60.4501	64.1547

4. Conclusions

The optimal operating parameters for the BC system were determined using RSM. A numerical optimisation technique was employed to optimise the desired goals, including maximum Biogas yield (ml/gVSadded), COD, and VS removal (%). It was discovered that with a temperature of 54.99 ºC, co-substrate loading ratio of 1:20 and a BC loading rate of 6.7 g/L at a desirability performance of 94.00 %, an optimal biogas yield of 59.75 mL/gVSadded was achieved. Although the acquired results show promise and are applicable, it is recommended that the study be scaled up to more accurately mimic actual conditions research, which would aid in identifying full-scale applicability.

References

Al-Sulaimi I.N., Nayak J.K., Alhimali H., Sana A., Al-Mamun A., 2022, Effect of volatile fatty acids accumulation on biogas production by sludge-feeding thermophilic anaerobic digester and predicting process parameters, Fermentation, 8(4), 184.

Bachman N., 2015, Sustainable biogas production in municipal wastewater treatment plants, IEA Bioenergy, Johannesburg, South Africa.

Kay Lup A.N., Soni V., Keenan B., Son J., Taghartapeh M.R., Morato, M.M., Poya Y., Montañés R.M., 2023, Sustainable Energy Technologies for the global south: Challenges and solutions toward achieving SDG 7, Environmental Science: Advances, 2(4), 570–585.

Khalid Z.B., Siddique Md.N., Nayee A., Adyel T.M., Ismail S.B., Ibrahim, M.Z., 2021, Biochar application as sustainable precursors for enhanced anaerobic digestion; A systematic review, Journal of Environmental Chemical Engineering, 9(4), 105489.

Manga M., Aragón-Briceño C., Boutikos P., Semiyaga S., Olabinjo O., and Muoghalu C.C., 2023, Biochar and its potential application for the improvement of the Anaerobic Digestion Process: A Critical Review, Energies, 16(10), 4051.

Moretta F., Goracci A., Manenti F., Bozzano G., 2022, Anaerobic Co-digestion Feedstock Blending Optimization, Chemical Engineering Transactions, 96, 295-300.

Musvoto E., Mgwenya N., Mangashena H., Mackintosh A., 2018, Energy Recovery from Wastewater Sludge – A Review of Appropriate Emerging and Established Technologies for the South African Industry, Water Research Commission, Report no TT 752/18, Pretoria, South Africa.

Niju S., Ajieth Kanna S.K., Ramalingam V., Satheesh Kumar M., Balajii, M., 2019, Sugarcane bagasse derived biochar – a potential heterogeneous catalyst for transesterification process. Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 45(4), 9815–9826.

Obileke K., Makaka G., Tangwe S., Mukumba P., 2024, Improvement of biogas yields in an anaerobic digestion process via optimization technique, Environment, Development and Sustainability

Osman A.I., Chen L., Yang M., 2023, Cost, environmental impact, and resilience of renewable energy under a changing climate: a review, Environ Chem Lett 21, 741–764.

Pan J., 2019, Achievements of biochar application for enhanced Anaerobic Digestion: A Review', Bioresource Technology, 292, 122058.

Pecchi M., Baratieri, M., 2019, Coupling anaerobic digestion with gasification, pyrolysis or hydrothermal carbonization: A review, Renewable and Sustainable Energy Reviews, 105, 462-475.