

Bioenergy Production from Oil Mill Wastewater by Methanation Process: Treatment and Valorization

Satyaapir Sahu¹, Anil Kumar² and Beemkumar N³

¹Assistant Professor, Department of Ayurveda, Sanskriti University, Mathura, Uttar Pradesh, India, ²Assistant Professor, College Of Agriculture Science, Teerthanker Mahaveer University, Moradabad, Uttar Pradesh, India, ³Professor, Department of Mechanical Engineering, Faculty of Engineering and Technology, JAIN (Deemed-to-be University), Bangalore, India

Abstract

Olive mill wastewaters (OMW), which are produced in large quantities as a result of the olive oil extraction process and have a high concentration of organic phenolic chemicals, pose a serious environmental risk. These substances are known to be harmful and, if not adequately managed, can harm ecosystems. Physico chemically characterise raw OMW in great detail. Anaerobic digestion (AD) is used to treat the OMW in order to create Methane (CH₄) and remove hazardous substances. Analyse the digestate that is still in the digester to determine its agricultural quality. Collection of unprocessed OMW samples from the extraction of olive oil. Analysis of the OMW samples' physicochemical composition, which included measuring pH, determining the amount of suspended matter, and identifying volatile substances. Mesophilic temperature setup and operation of a semi-continuous anaerobic digester. CH₄ production is being watched over, and yield is being measured. Analysing the characteristics of the digestate produced by the AD process, including the presence of important agricultural nutrients as NH₄, P, K, and Mg. The physicochemical examination showed that the examined OMW samples had a pH value of 5.8, which indicated excessive acidity. Additionally, they had a sizable amount of suspended materials. The AD process was successful in converting organic components into CH₄ because the CH₄ production reached a value of 250 ml/g VS. There was also a buildup of volatile solids, indicating the presence of organic chemicals that had not yet completely broken down and Methanogenic potential (MP) reduces the load rises. The study showed that AD, which generates CH₄ and breaks down OMW's harmful components, can successfully treat OMW. The significant CH₄ production discovered suggests that this waste stream has the potential to produce electricity. The procedure' leftover digestate is useful for agriculture and can be used as fertiliser or an inoculum (IM) to start additional AD studies.

Keywords: Olive mill wastewaters (OMW), Methanogenic potential (MP), Methane (CH₄), Anaerobic digestion (AD), inoculum (IM)

Full length article *Corresponding Author, e-mail: satyaapir.samch@sanskriti.edu.in

1. Introduction

From a socioeconomic perspective, the olive oil sector was crucial, but it also produces a lot of waste, such as liquid effluent and solid waste in the form of olive mill wastewater and olive pomace [1]. The risks of contamination must be reduced through efficient treatment of these wastes. The amount of wastewater produced by an olive mill is normally approximated based on the extraction method utilised, with 1 to 1.5 litres of wastewater being produced per kilogramme of olives. Lipids and polyphenolic chemicals in olive mill effluent are responsible for its phytotoxicity. In order to solve this problem, anaerobic fermentation (AF) has become the principal method of treatment for lowering the level of OMW while likewise producing energy in the form of biogas. This biogas used to generate heat and energy [2]. However, due to the inhibiting nature of polyphenols and the acidity of olive mill Sahu et al., 2023

wastewater, which normally has a pH between 4.5 and 5, the AF process encounters difficulties. These elements have the result that the process is unstable. However, valuing organic waste through AD not only lessens environmental pollution but also has the ability to generate a sizable amount of renewable energy [3].

Olive mill wastewater's high concentration of organic components has made it challenging to implement AD. Furthermore, the effluent from olive mills is low in nitrogen (0.2 to 0.5 g/l) and acidic (pH = 5), as well as having low levels of "Alkalinity" (1 to 2 g CaCO₃/l) [4]. To solve these difficulties, a number of pre-treatment techniques have been used, including dilution pre-treatment employing aerobic culture. These pre-treatment methods work to lessen the inhibitory effects and improve the anaerobic digesting environment [5].

This study uses AD to process unprocessed raw OMW in order to measure the amount of CH₄ produced. The

product's ability to be utilised as fuel for cars or for the cogeneration of heat and electricity, which can lower greenhouse gas emissions, is the key advantage of this technology. For this firm, treating OMW is still a major challenge, thus AD has become a recognised and effective method.

In the article [1], solar energy was initially transformed into electricity, which warmed a mixture of POME and calf dung (CM), keeping the reactor at a mesophilic temperature. The analysis of the ACoD operation was conducted semi-continuously for POME and CM. In addition, the CH₄ composition has increased from 64.13% with no pre treatment to 72.4% with it. To measure both the final CH₄ output and the kinetic constants, a kinetic study of the prospective CH₄ production from POME was conducted. In the article[2] the CH₄ composition has increased from 64.13% with no pre treatment to 72.4% with it. To measure both the final CH₄ output and the kinetic constants, a kinetic study of the prospective generation of CH₄ from POME was conducted. In the study[3], an integrated bio refinery idea was presented that combines two biological platforms to valorize palm oil mill effluents and produce high-value products like microbial oils and bioenergy at the same time. The lipids from the aerobic fermentation of palm oil mill effluents were then produced. The goal of the research[4] was to scale up a biohythane manufacturing process from palm oil mill effluent using thermophilic mixed consortia and thermophilic methanogenic organisms at different HRT and OLRs. The yield and pace of biohythane production were tracked in order to determine the stability and usability of biohythane in a pilot size process. A brief description of how POME will effect the environment was provided in the paper[5]. Additionally, the advantages and therapeutic potential of microalgae for POME are assessed. Additionally, the POME's microalgal growth conditions were evaluated in order to determined the ideal environment for microalgae cultivation. Furthermore, the viability of experimental investigations on the properties and functionality of microalgae was being assessed. Article [6] explored how to maximise the value of crude glycerol obtained from biodiesel and its hydrolysis byproduct (ethanol) when added to palm oil mill effluent (POME) to increase biogas production efficiency under mesophilic AD. The research [7] used an anaerobic bacterial consortium of *Bacillus toyonensis* strain BCT-71120 and *Stenotrophomonas rhizophila* strain e-p10 to decompose the organic and inorganic components in POME, produce biogas, and lower the values of COD, BOD, and total suspended solid (TSS). On TSS, COD, BOD, and pH, the impact of fermentation time was examined. For the purpose of determining the ideal fermentation time, a specific growth rate and generation period were defined.

A significant amount of trash, notably palm oil mill effluent (POME), a type of wastewater that can seriously harm the environment, has been released while the palm oil sector has seen global uptrends. This has made it extremely difficult for palm oil mills to adhere to the rules of standard discharge limits, which has led to the creation of a number of bioremediation techniques and bioreactors, which are detailed in the paper[8]. In the article [9] employed an advanced oxidation processes (AOPs) to treat hazardous. These technologies, which are creative, have recently found

significant applicability in numerous bioenergy production processes. There was no thorough documentation on how biofuel production operations have increasingly incorporated these oxidation processes, despite the growing interest in the application of AOPs in boosting the production of bioenergy. The study [10] can serve as a springboard for discussing the viability of biogas generation in Malaysia in the context of efficient waste disposal and energy recovery. Due to their major impacts, this analysis places particular emphasis on land use change (LUC), and water consumption (WCP), global warming (GWP). The review [11] briefly discussed the issues involved in resource recovery from wastewater using algal growing, biomass harvesting, and various technologies used to convert algal biomass into bioenergy. The study [12] evaluated the enhancement of biohythane production from palm press fibre (PPF), decanter cake (DC), an oil palm trunk (OPT), empty fruit bunch (EFB) and an oil palm frond (OPF) through co-digestion with palm oil mill effluent (POME) the two-stage AF. As a low-cost pre-treatment technique to improve the biodegradability of solid waste, co-fermentation in the first stage may also be taken into consideration. The goal of the study [13] was to determined how a thermophilic continuous set up consisting of an upflow anaerobic sludge blanket - hollow centred packed bed (UASB-HCPB) bioreactor responds to thermal pre-treatment and solid loading change via dewatering. A more efficient transfer of mass and transit in the medium was made possible by the stable solids content, which is what contributed to the improved process performance. The primary goal of the study [14] was to evaluate the cultivation and biomass production of *Arthrospira platensis* Gomont 1892 (spirulina) using nutrients from dairy industry wastewater and their subsequent usage in anaerobic co-digestion with bovine dung with the notion of boosting CH₄ production. The experiment was conducted to ascertain the properties of both substrates prior to co-digestion. The main goal of the study [15] was to determine whether co-digestion of bovine manure and palm oil mill effluent in a solar-assisted bioreactor (SABr) may result in enhanced biogas production. The POME and CM combination was heated by the solar panel in order to maintain the requisite reactor temperature after solar radiation was initially transformed into energy.

2. Materials and methods

In 3L under mesophilic conditions (35–40°C), AD was conducted. The reactor is equipped with two suitable devices for sample collection for analysis and the production of biogas, and it is fully agitated using an electromagnetic stirrer that is connected to a motor. Between the digester and the gasometer, a bubbler with a solution of NaOH (6N) is installed, and it is used to collect CO₂ from the biogas. The digester was linked to a gasometer, which allowed for the calculation of CH₄ production. In this study, samples of olive mill wastewater (OMW) were taken from an extraction unit of an olive oil works that uses a press system and a three-phase method to extract olive oil. Figures 1 and 2 are provided the physicochemical components of the OMW samples, which are not accessible in the given context.

2.1 IM and Substratum

An IM was produced utilising a mesophilic anaerobic reactor from a Marrakech wastewater treatment facility to start the AD process. This IM was chosen because it has a high methanogenic activity, which indicates that it can create CH₄ effectively during the AD process. The introduction of the microbial consortia adapted to mild temperatures to the OMW treatment system is made possible by using a mesophilic anaerobic reactor as the source of the IM. This is necessary for the AD process to run smoothly and remain stable. The study seeks to improve the biogas output and overall efficiency of the AD process for treating the OMW samples taken from the industrial site by utilising the high methanogenic activity of the chosen IM.

2.2 Process and chemical evaluation

There are two stages to the digester startup procedure. The digester is first inoculated with an IM containing microorganisms that produce CH₄ at an organic loading rate of 8 gVS/L. This guarantees long-term biogas production and quickens the digester's startup. In the second stage, the treated substrate (OMW) is fed to the digester at various organic loading rates ranging from 0.5 to 2.5 gSV/L. A gasometer and the water displacement method are used to calculate the volume of CH₄ produced. Atomic absorption spectroscopy is used to examine the elements (Cu, BP, Zn and Fe) in the samples using a flame-based Analytik Jena NONAA350 instrument. By filtering on a cellulose filter and weighing the sample before and after it has dried in an oven at 105°C, suspended matter is identified. The Folin Ciocalteu method is used to gauge the levels of total phenols in the samples. Using a pH metre and a standard acid solution (sulfuric acid), "Alkalinity" is determined. Total solids are determined by weighing the residue after the sample has been dried for 24 hours at 105°C. The sample is dried for two hours at 550°C in a muffle furnace to produce mineral solids, and the difference between total solids and mineral solids is used to calculate volatile solids.

3. Results and Discussions

3.1 Productivity of CH₄

The correlation between CH₄ productivity and load addition in the digester is shown in Figure 3. The following CH₄ production volumes were noted: For the corresponding loading of 1, 1.5, 2, 2.5 and 3 g VS/L, the digester volumes were 110, 249, 323, 336, and 410 ml/L. The data clearly show that CH₄ productivity rises as the digester's load is increased. This suggests that the rate of organic loading and CH₄ production are positively correlated. The reported CH₄ productivity increases with increased load. The increased availability of organic substrates for methanogenic bacteria to convert into CH₄ is responsible for this trend. As more organic material is added to the digester, the microbial population has more substrate to break down, which increases the production of CH₄.

3.2 Digester stability

In this experiment, we tracked the changes in pH and "Alkalinity", which stand in for the stability control parameters of AD. In order to achieve the largest biogas yield, it is crucial to guarantee that the experiment is carried out under stable conditions. Several tests have demonstrated that there are numerous elements that influence the process of AD. Figure 4 depicts the changes in the digester's "Alkalinity". To work successfully, the "Alkalinity" brought on by calcium bicarbonate must be quite high. In a reactor that operates in good condition, it is required to have at least 1000 mg/L of "Alkalinity" (expressed in milligrammes of CaCO₃ per litre). It is noted that the average "Alkalinity" in the digester is 1400 mg/L. This value is ideal because AD requires an "Alkalinity" range of 1000 to 3000 mg/L. As a result, measuring "Alkalinity" allows us to evaluate the stability of AD and its resistance to acid attack. Additionally, when the digester has a high "Alkalinity", it can have a good buffer capacity, which is actually the neutralising effect. Figure 5 depicts the pH changes that occurred in the digester over the course of the AD process. Due to the fact that each of the microbial groups involved in the reaction has a specific pH range for optimum growth, the anaerobic degradation process is highly dependent on pH. The ideal pH range for mesophilic AD is between 6.5 and 7.5, depending for each species of bacterium.

3.3 Potential for Methanogens and Biodegradability

The highest amount of CH₄ that can be produced in an AD reactor per litre of effluent or per kilogramme of trash is known as the MP. The amount of dry matter in the substrate has an effect on the MP, which varies based on the substrate being treated and the pre-treatment used. In this experiment, the MP was determined to be 218, 249, 215, 170.9, and 165 ml/g VS for various loading of 1, 1.5, 2, 2.5, and 3 g VS/L (volatile solids per litre) shown in figure 6. These numbers show how much CH₄ is produced for each load per gramme of volatile solids in the substrate. It is important to note that the MP in this study reduces as the load rises. The biodegradability in the digester is shown in Figure 7 and demonstrates the degree of organic matter decomposition in relation to the amount added. The evolution of volatile solids removed in comparison to volatile solids introduced is shown. According to this study's biodegradability estimate of 64%. This biodegradability number sheds light on how efficiently organic matter is broken down during the AD process. A higher biodegradability suggests a more effective transformation of organic matter into biogas (including CH₄ and carbon dioxide) during the process of AD, since it shows an increased degradation rate of the waste.

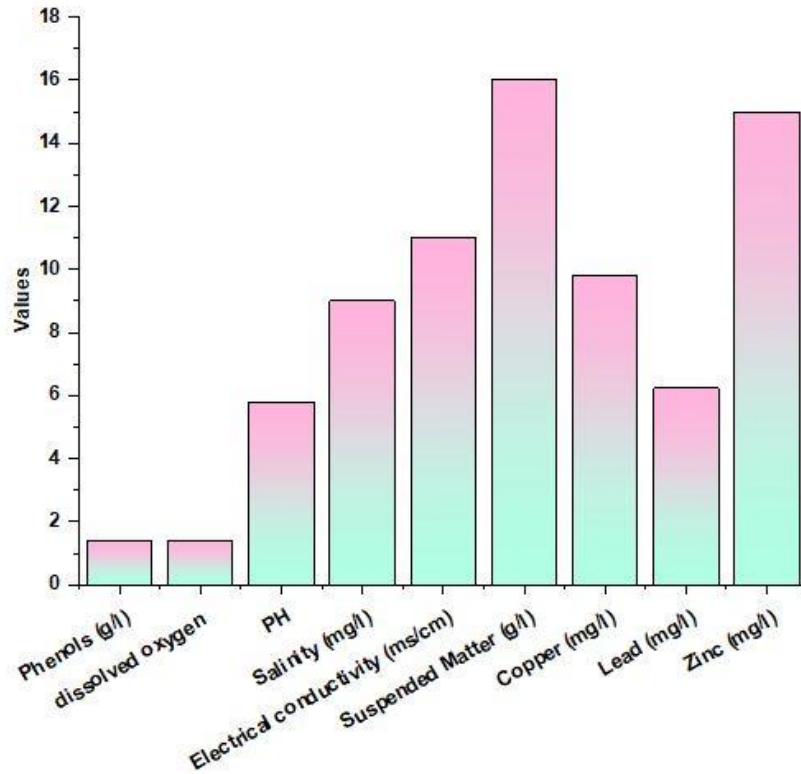


Figure 1: The characteristics of OMW for (0-18) values

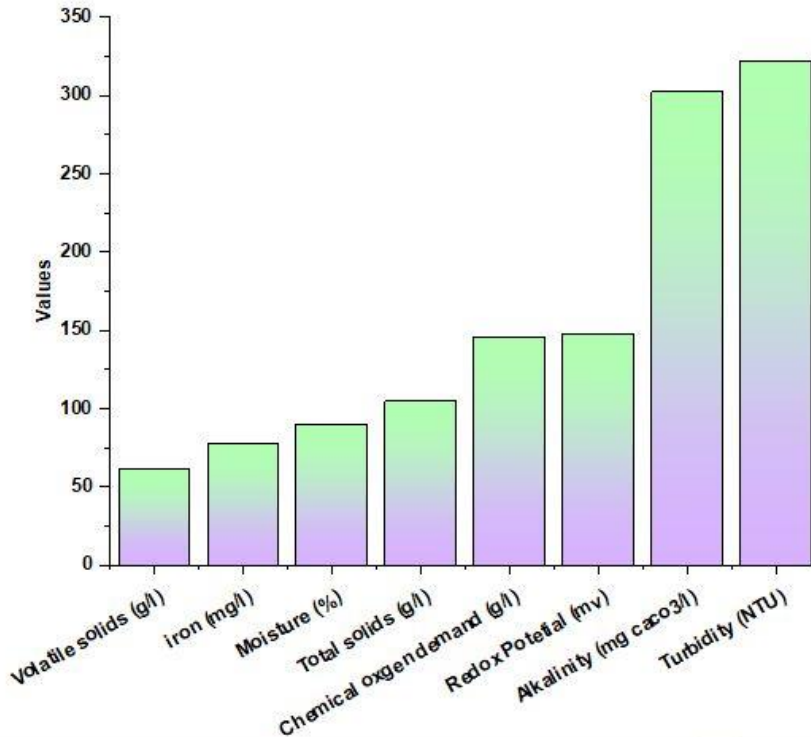


Figure 2: The characteristics of OMW (0-350) values

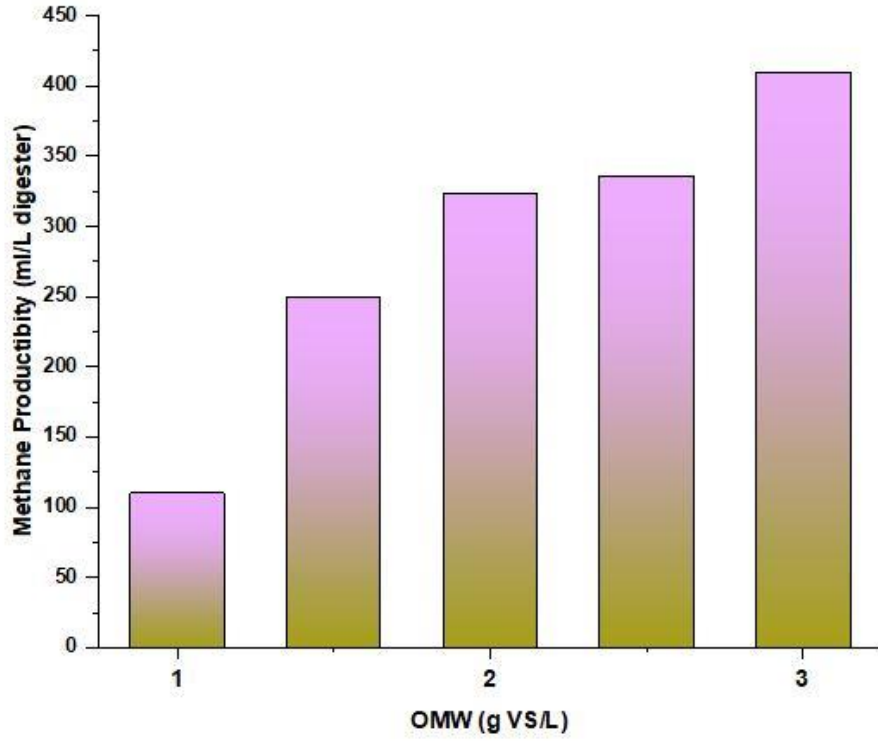


Figure 3: The correlation between CH₄ productivity and load addition

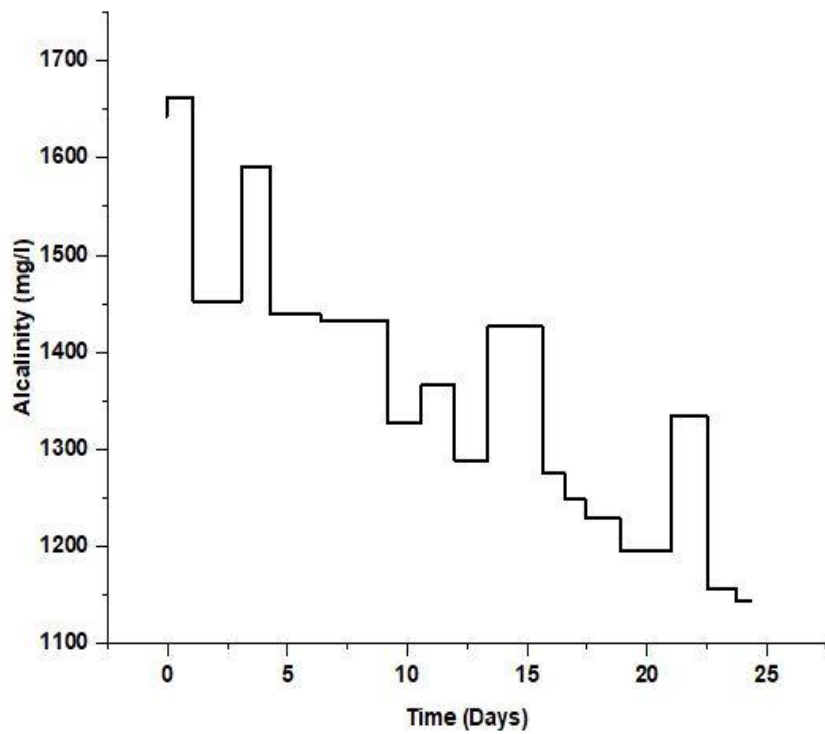


Figure 4: Variation of alkalinity in the digester

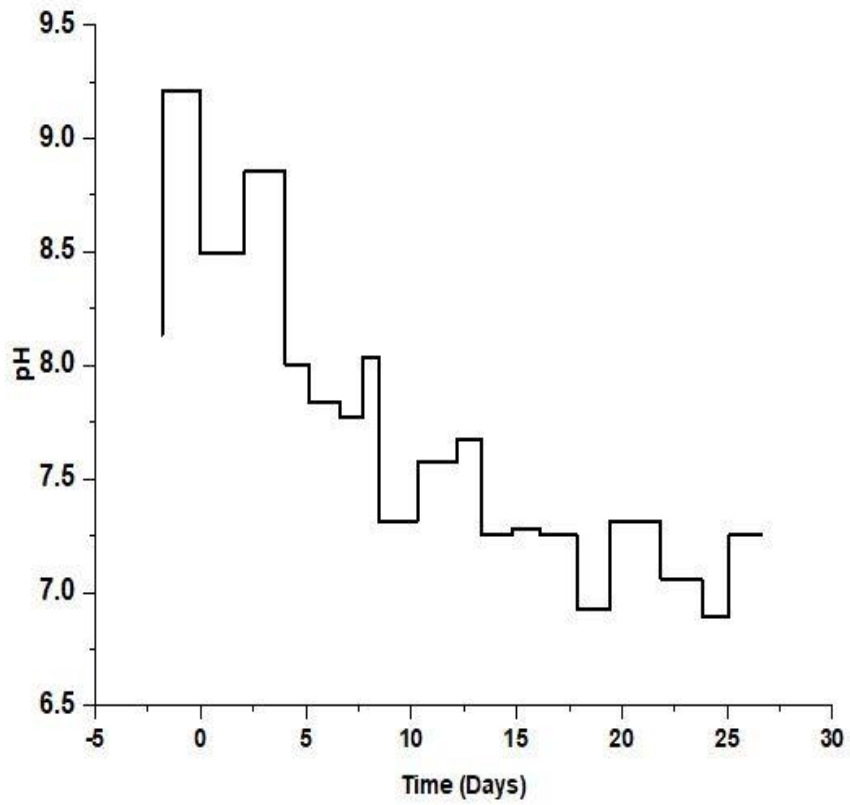


Figure5: Evolution of pH during anaerobic digestion of OMW

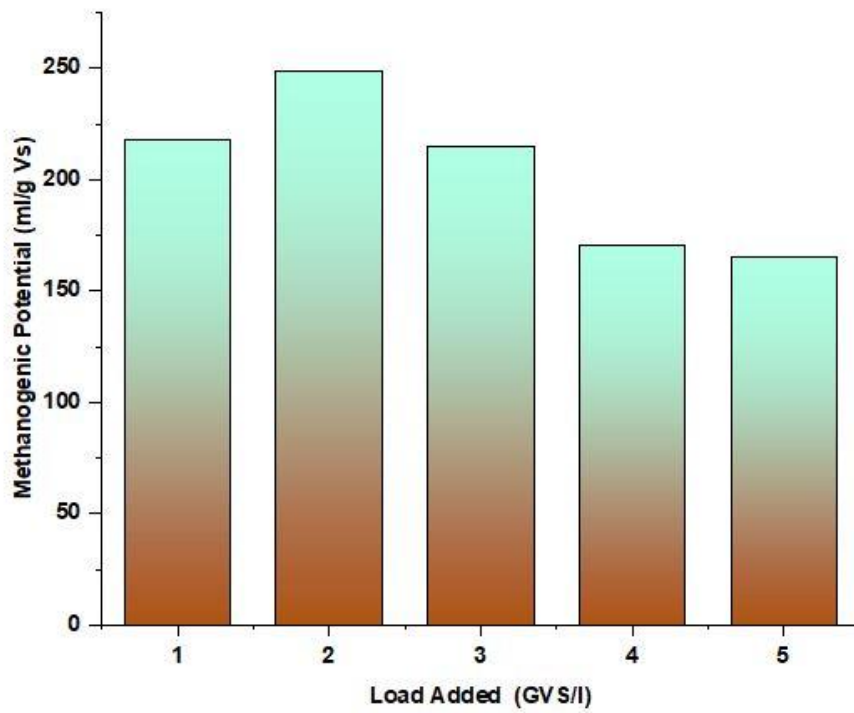


Figure 6: Methanogenic potential of OMW for different loads

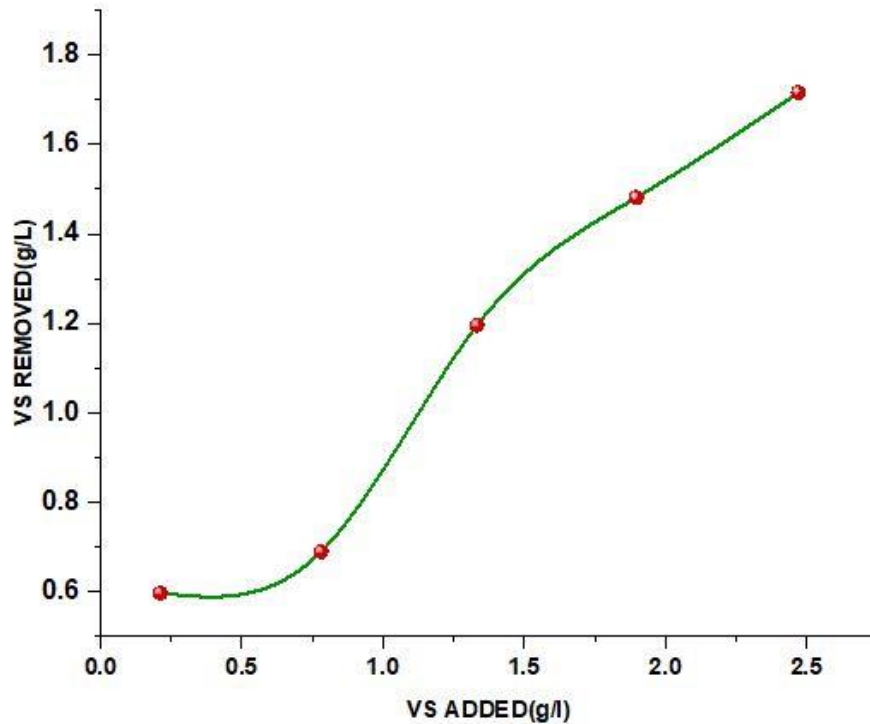


Figure 7: The relation between VS added and VS removed

4. Conclusions

This study emphasizes the significance of AF as a method of treating olive mill effluent. Due to its potential to lessen the negative environmental effects of this waste. AF can efficiently break down toxic organic matter that is present in the wastewater thanks to the activity of bacteria in the biomass. This technique also produces biogas, which ups its energy and environmental advantages. Furthermore, the digestate, which is the waste product left over after AF in the digester, has important qualities that can be applied in various contexts. As the digestate is rich in nutrients that can improve soil fertility and crop growth, one such application is as a fertiliser in agriculture. Additionally, the digestate can act as an IM to help other AD investigations get started by adding an active microbial mass.

References

- [1] B.K. Zaied, M. Nasrullah, M.N.I. Siddique, A.W. Zularisam, L. Singh, and S. Krishnan. (2020). Enhanced bioenergy production from palm oil mill effluent by co-digestion in solar assisted bioreactor: Effects of hydrogen peroxide pre-treatment. *Journal of Environmental Chemical Engineering*, 8(2): 103551.
- [2] K. Sani, P. Kongjan, C. Pakhathirathien, B. Cheirsilp, O. Sompong, M. Raketh, R. Kana, and R. Jariyaboon. (2021). Effectiveness of using two-stage AD to recover bio-energy from high strength palm oil mill effluents with simultaneous treatment. *Journal of Water process engineering*, 39: 101661.
- [3] Y. Louhasakul, L. Treu, P.G. Kougias, S. Campanaro, B. Cheirsilp, and I. Angelidaki. (2021). Valorization of palm oil mill wastewater for integrated production of microbial oil and biogas in a biorefinery approach. *Journal of Cleaner Production*, 296: 126606.
- [4] J. Seengenyong, C. Mamimin, P. Prasertsan, and O. Sompong. (2019). Pilot-scale of biohythane production from palm oil mill effluent by two-stage thermophilic AF. *International Journal of Hydrogen Energy*, 44(6), pp.3347-3355.
- [5] S.S. Low, K.X. Bong, M. Mubashir, C.K. Cheng, M.K. Lam, J.W. Lim, Y.C. Ho, K.T. Lee, H.S.H. Munawaroh, and P.L. Show. (2021). Microalgae cultivation in palm oil mill effluent (POME) treatment and biofuel production. *Sustainability*, 13(6): 3247.
- [6] P. Prasertsan, C. Leamdum, S. Chantong, C. Mamimin, P. Kongjan, and O. Sompong. (2021). Enhanced biogas production by co-digestion of crude glycerol and ethanol with palm oil mill effluent and microbial community analysis. *Biomass and Bioenergy*, 148: 106037.
- [7] M. Said, A.S. Sitanggang, R. Julianda, S.P. Estuningsih, and A. Fudholi. (2021). Production of CH₄ as bio-fuel from palm oil mill effluent using

- anaerobic consortium bacteria. *Journal of Cleaner Production*, 282: 124424.
- [8] W.Y. Chia, Y.Y. Chong, K.W. Chew, E. Vimali, M. Jayaram, A. Selvarajoo, K.S. Muthuvelu, P. Varalakshmi, P.L. Show, and S.K. Arumugasamy. (2020). Outlook on biorefinery potential of palm oil mill effluent for resource recovery. *Journal of Environmental Chemical Engineering*, 8(6): 104519.
- [9] M.M. M'Arimi, C.A. Mecha, A.K. Kiprop, and R. Ramkat. (2020). Recent trends in applications of advanced oxidation processes (AOPs) in bioenergy production. *Renewable and Sustainable Energy Reviews*, 121: 109669.
- [10] N.I.H.A. Aziz and M.M. Hanafiah. (2020). Life cycle analysis of biogas production from AD of palm oil mill effluent. *Renewable Energy*, 145, pp.847-857.
- [11] S.K. Bhatia, S. Mehariya, R.K. Bhatia, M. Kumar, A. Pugazhendhi, M.K. Awasthi, A.E. Atabani, G. Kumar, W. Kim, S.O. Seo, and Y.H. Yang. (2021). Wastewater based microalgal biorefinery for bioenergy production: Progress and challenges. *Science of the Total Environment*, 751: 141599.
- [12] C. Mamimin, P. Kongjan, O. Sompong, and P. Prasertsan. (2019). Enhancement of biohythane production from solid waste by co-digestion with palm oil mill effluent in two-stage thermophilic fermentation. *International Journal of Hydrogen Energy*, 44(32), pp.17224-17237.
- [13] S.N. Khadaroo, P. Grassia, D. Gouwanda, J. He, and P.E. Poh. (2021). Enhancing the biogas production and the treated effluent quality via an alternative palm oil mill effluent (POME) treatment process: Integration of thermal pre-treatment and dewatering. *Biomass and Bioenergy*, 151: 106167.
- [14] X. Álvarez, O. Arévalo, M. Salvador, I. Mercado, and B. Velázquez-Martí. (2020). Cyanobacterial biomass produced in the wastewater of the dairy industry and its evaluation in anaerobic co-digestion with cattle manure for enhanced CH₄ production. *Processes*, 8(10): 1290.
- [15] Z.B. Khalid, M.N.I. Siddique, M. Nasrullah, L. Singh, Z.B.A. Wahid, and M.F. Ahmad. (2019). Application of solar assisted bioreactor for biogas production from palm oil mill effluent co-digested with cattle manure. *Environmental Technology & Innovation*, 16: 100446.