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The Potential of Recycling Used Engine Oil for Biogas Generation by Co-Digestion with Animals' Manure: Experimental and Kinetic Study

Hassan S. Jasim[1,*], Zainab Z. Ismail[2]

Department of Environmental Engineering, College of Engineering, Baghdad University, Baghdad, Iraq

hassan.jassim2011m@coeng.uobaghdad.edu.iq[1], dr.zainab.zead@coeng.uobaghdad.edu.iq[2]

ABSTRACT

This study investigates the potential of biogas recovery from used engine oil (UEO) by co-digestion with animals' manure, including cow dung (CD), poultry manure (PM), and cattle manure (CM). The experimental work was carried out in anaerobic biodigesters at mesophilic conditions (37°C). Two groups of biodigesters were prepared. Each group consisted of 4 digesters. UEO was the main component in the first group of biodigesters with and without inoculum, whereby a mix of UEO and petroleum refinery oily sludge (ROS) was the component in the second group of biodigesters. The results revealed that for UEO-based biodigesters, maximum biogas production was 0.98, 1.23, 1.93, and 0 ml/g VS from UEO±CD, UEO±CM, UEO±PM, and UEO, respectively, whereby, for the UEO=ROS-based biodigesters, maximum biogas production was 3.49, 2.47, 3.64 and 2.44 ml/g VS from UEO+ROS±CD, UEO+ROS±CM, UEO+ROS±PM, and UEO+ROS, respectively. These results indicated that UEO was not feasible and efficient for biogas recovery since biogas production was very low in the first group of biodigesters compared to its recovery in the second group. A modified Gompertz model was applied to study the kinetics of the bio-digestion process. Measured and predicted values of biogas generation were fitted well with determination coefficients higher than 0.92.

Keywords: Biogas, Anaerobic Co-Digestion, Used Engine Oil, Petroleum Oily Sludge.

*Corresponding author

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امكانية اعادة تدوير زيت المكائن المستخدم لإنتاج الغاز الحيوي بواسطة الهضم المشترك مع مخلفات الحيوانات: الدراسة التجريبية والحركية

حسن سعد جاسم[1]،* ، زينب زياد اسماعيل[2]
قسم الهندسة البيئية، كلية الهندسة، جامعة بغداد، بغداد، العراق

الخلاصة

تبحث هذه الدراسة إمكانية استرجاع الغاز الحيوي من زيت المحرك المستخدم (UEO) عن طريق الهضم المشترك اللاهوائي مع روث الحيوانات (البقرCD والغنم CM والدجاج PM). تم اجراء العمل التجريبي في مفاعلات هضم لاهوائي تحت درجة حرارة متوسطة 37 ^{o}م حيث تم تحضير مجموعتي هضم لاهوائي كل مجموعة تتكون من أربعة هواضم . كان UEO المكون الرئيسي في المجموعة الأولى مع او بدون اللقاح اما المجوعة الثانية تم خلط UEO مع الحمأة النفطية (ROS) ليشكل المكون الرئيسي. أظهرت النتائج أنه بالنسبة إلى الهواضم المعتمدة على UEO، كان الحد الأقصى لإنتاج الغاز الحيوي 0.98، 1.23، 1.93، وصفر مل/غم VS من UEO ± CD، UEO ± CM، UEO ± PM، UEO على التوالي، اما عند الخليط UEO = ROS كان الحد الأقصى لإنتاج الغاز الحيوي 3.49، 2.47، 3.64 و 2.44 مل/غم VS لكل من UEO + ROS ± CD و UEO + ROS ± CM و UEO + ROS ± PM و UEO + ROS ، على التوالي. أشارت هذه النتائج إلى أن UEO في حد ذاته لم يكن مجديًا وفعالًا لاستعادة الغاز الحيوي حيث كان إنتاج الغاز الحيوي منخفضًا جدًا في المجموعة الأولى من أجهزة التحلل الحيوي مقارنة باستعادته في المجموعة الثانية التي تشير. لدراسة الحركية تم تطبيق نموذج Gompertz المعدل لعملية الهضم الحيوي. تمت مطابقة البيانات المتوقعة والتجريبية لإنتاج الغاز الحيوي بشكل جيد مع معامل التحديد اعلى من 0.92.

الكلمات الرئيسية: الغاز الحيوي، الهضم اللاهوائي المشترك، زيت المحركات المستخدم، الحمأة النفطية.

1. INTRODUCTION

Energy is an essential factor in modern-day societies and industries. Worldwide energy demand is expected to increase by 5-fold by 2100. Fulfillment of these energy huge quantity cannot be solely met by fossil fuels because of their adverse environmental effects such as the increase in the emission of greenhouse gases and air pollution **(Tabatabaei et al., 2020; Abdullah et al., 2020)**. Energy is important in domestic, commercial, and industrial businesses. Therefore, it is necessary to find new environmentally friendly renewable energy sources as alternatives to fossil fuels. Biogas recovery from waste materials could be considered a sustainable source of renewable energy sources and for reducing the large accumulated amounts of solid waste **(Montañés et al., 2015; Ellacuriaga et al., 2021)**. The process of anaerobic digestion is a potential technology for carbon recovery. It is a process of biological decomposition in which bacteria are used in the absence of oxygen to digest and decompose complex organic matter into simple organic substances through four stages, including hydrolysis, acidogenesis, acetogenesis, and methanogenesis, where CH_4 is the main target in gas production. Since microorganisms carry out the process, it is sensitive to changes in temperature, acidity, and percentage C/N ratio **(Hassan et al., 2016; Choong et**

al., 2016; Rashama et al., 2019). Biogas can be created from the anaerobic digestion of different waste materials, such as agricultural waste, energy crops, and waste from particular industries. Anaerobic co-digestion for biogas production utilizes a combination of different feedstocks to improve the biogenic methane content of the biogas **(Hanif et al., 2022)**. Petroleum industries include various processes, including exploration, oil production, transportation, and oil derivates production. During these processes, different types of wastes are generated, such as drilling fluids, petroleum wastewater, sludge from treatment plants, and the bottom tank sludge **(Abid et al., 2018)**. As an example, in Iraq, the North Refineries Company (NRC) in Baiji produced about 3000 – 3500 m^3/year of petroleum refinery oily sludge (ROS) **(Abdulqader et al., 2022)**. On the other hand, used engine oil (UEO) is another type of hydrocarbon toxic waste that is generated in huge quantities. Several studies are available on biogas production from different waste materials such as agro-industrial wastewater **(Olvera and Lopez, 2012)**, sewage sludge **(Rivero et al., 2013)**, marine macroalgae **(McKennedy and Sherlock, 2015)**, medical cotton industry **(Ismail and Talib, 2016)**, municipal solid waste and fruit and vegetable **(Pavi et al., 2017)**, waste alcoholic beverage **(Montes and Rico, 2020)**, olive pomace **(Ayadi et al., 2020)**, sugarcane vinasse **(Kiani et al., 2022)**, leaf litter of neem **(Muhammad and Chandrab, 2021)**, potato crop residues **(Soltaninejad et al., 2022)**, and mixed food waste **(Perman et al., 2022)**.
However, limited studies are available on biogas production from oily sludge by co-digestion. (**Yang et al., 2020)** investigated the biogas production from co-digestion of corn stover and oil sludge. (**Sampson, 2020)** studied the recovery of biogas from the digestion of petroleum sludge inoculated with methanogenic bacteria (*Methanobrevibacter*) as a single type of anaerobic bacteria isolated from a cow's intestine at mesophilic conditions. (**Ghaleb et al., 2021)** assessed the co-digestion of oily sludge with sugarcane bagasse as a co-substrate of high content of C/N to improve the biogas yield at mesophilic conditions. (**Shi et al., 2022)** studied the effect of biochar dose on the anaerobic digestion of naphthalene contained-oily sludge at mesophilic conditions. To the authors' knowledge, the previously reported studies have dealt with assessing the biogas production from used engine oil as a hazardous waste material of no economic value.
This study aimed to investigate the potential of biogas production from the used engine oil by itself and mixed with petroleum refinery oily sludge by anaerobic co-digestion with three types of animals manure, including cow dung, cattle manure, and poultry manure at mesophilic conditions. In addition, the Modified Gompertz Model was applied to study the kinetics of the co-digestion process.

2. MATERIALS AND TECHNIQUES

2.1 Used Engine Oil (UEO)

The used engine oil (UEO) is a black hydrocarbon residue of a sticky texture. Samples were collected from local vehicle maintenance garages. Characteristics of UEO are given in **Table 1**. The average concentrations of the major constituents of UEO samples were (in mg/L); 40548 ± 10000, 50000 ± 15000, 259400 ± 40000, and 10.7 ± 1.6 for total suspended solids (TSS), volatile suspended solids (VSS), Chemical oxygen demand (COD), and pb^{+2}, respectively at a pH range of 5.5-6.0.

2.2 Petroleum Refinery Oily Sludge

Actual samples of petroleum refinery oily sludge (ROS) were collected from the final collection tank of ROS at Al-Daura Refinery, Mid-Land Refineries Company (MRC) in Baghdad. Normally, ROS is a mixture of different residues from various sources, including; primary and secondary API separators tanks, oily scum from the dissolved air flotation tank, oily wastewater, activated sludge and wasted sludge from the secondary clarifier, and the oily flocs from coagulation and flocculation unit. Characteristics of ROS are given in **Table 1**.

Table 1. Quality and characterization of the real samples of ROS

Constituents	Units	Average concentration
COD	mg/l	30,000 ± 500
Total dissolved solids (TDS)	mg/l	2,965 ±150
Total suspended solids (TSS)	mg/l	3,270 ± 200
Total volatile solids (TVS)	mg/l	11976 ± 100
Oil & grease	mg/l	2,310 ± 100
Total petroleum hydrocarbons (TPH)	mg/l	980 ± 80
pH	-	7.3 ± 0.1
Sulfate (SO_4^{-2})	mg/l	1,484 ± 150
Nitrate (NO_3^-)	mg/l	500 ± 40
Phosphate (PO_4^{-3})	mg/l	900 ± 100
Bulk density	kg/m^3	1,060
Heavy metals		
Pb^{+2}	mg/l	6.35 ± 0.3
Zn^{+2}	mg/l	0.18 ±0.02
Cu^{+2}	mg/l	2.40 ±0.05
Cd^{+2}	mg/l	Nil
Total Cr	mg/l	Nil
V^{+2}	mg/l	Nil

2.3 Inoculum

Cattle manure (CM), poultry manure (PM), and cow dung (CD) were individually used to inoculate the UEO and the mixture of ROS and UEO as co-substrates to fructify the content of bacteria, improve the efficiency of composting, and boost the anaerobic co-digestion process in the bio-digesters. The animals' manure samples were collected from the livestock sheds and live poultry shops. The microbial analysis of manure samples indicated that the dominant bacterial cells were *Escherichia Coli*, *Serratia fonticola*, and *Escherichia Coli* in the CM, PM, and CD, respectively, in addition to the presence of methanogens in the 3 types of inoculums.

2.4 Digesters Setup

Eight bench-scale biodigesters were set up in duplicate and operated in a batch mode under strict anaerobic mesophilic conditions. Each biodigester was a 500-ml Pyrex borosilicate heatproof code glass bottle. The total volume of the contents in each reactor was 400 ml. The details of the contents in each digester are given in **Table 2**.

Table 2. Details of bio-digester contents

	Digester No.	Symbols	Experimental conditions	Volumes of materials in the digesters
Group 1	1	UEO	Uninoculated UEO	400 ml
	2	UEO+CD	UEO inoculated with cow dung	320ml+80ml
	3	UEO+CM	UEO inoculated with cattle manure	320ml+80ml
	4	UEO+PM	UEO inoculated with poultry manure	320ml+80ml
Group 2	5	UEO+ROS	UEO with ROS (Uninoculated)	200ml+200ml
	6	UEO+ROS+CD	UEO with ROS inoculated with cow dung	160ml+160ml+80ml
	7	UEO+ROS+CM	UEO with ROS inoculated with cattle manure	160ml+160ml+80ml
	8	UEO+ROS+PM	UEO with ROS inoculated with poultry manure	160ml+160ml+80ml

Every single digester was plugged tightly using a rubber stopper containing 2 punctures, each of 0.4 cm diameter, through which a small piece of a glass pipe was inserted, and the other terminus of the glass pipe was connected with a rubber tube for the generated biogas transport to the biogas measurement section. Parafilm was used for wrapping tightly the rubber stoppers to prevent any escape from the generated biogas. Flushing them with nitrogen for 10 min was performed to maintain strict anaerobic conditions in the biodigesters. Thermostatic water bath for keeping the digesters at the desired mesophilic conditions at a constant temperature (37°C). Shaking of digesters was daily performed to allow close contact between the bacteria and substrate in the mixture. For pH adjustment, Sodium bicarbonate ($NaHCO_3$) was used **(Al-mashhadani et al., 2015; Sevillano et al., 2020).** The water that overflowed to the displacement bottle was colored by food grade red color as shown in **Fig. 1.**

2.5 Methods of Analysis

Concentrations of total volatile solids (TVS), total suspended solids (TSS), and total dissolved solids (TDS) were carried out following the procedures in the Standard Methods **(APHA, 2005)**.

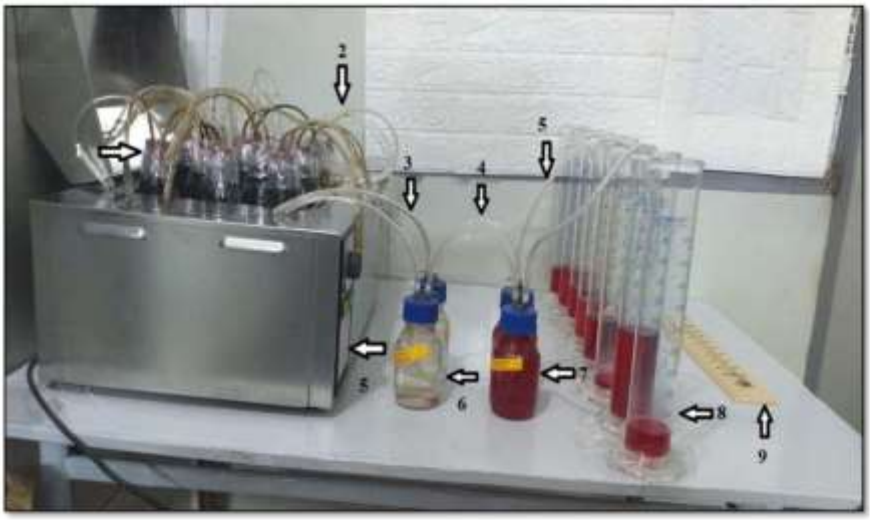

Figure 1. Photo of the experimental system set-up ((1) Heatproof code glass bottle as digesters with Rubber stopper, (2) Rubber tube, (3) Biogas input before alkaline solution, (4) Methane before water bottle, (5) Water bath, (6) Alkaline solution for CO_2 scrubbing, (7) Colored water in the displacement bottle, (8) Graduated cylinder, and (9) Mercury thermometer)

A COD analyzer (Type: Lovibond COD/RD/125) was used for measuring the concentrations of chemical oxygen demand (COD). Heavy metal concentrations were detected using atomic absorption spectroscopy (Model: GBC A.C.N. 005 472 686, Australia). Concentrations of other constituents, including NO^{-3}, PO_4^{-3}, SO_4^{-2}, and Cl^- were performed based on the procedures outlined in the Standard Methods **(APHA, 2005)**.

The water displacement method was applied for measurements of biogas production by passing the biogas through a bottle containing 1M (NaOH) to remove the (CO_2) from the biogas. The alkaline solution was periodically replaced to avoid the saturation of the alkaline solution with CO_2. Then the remaining CH_4 passed to another glass punnet, displaced the water, which overflowed into a volumetric measuring cylinder. The amount or volume of the displaced colored water was equal to the yield of CH_4. All measurements were conducted at room temperature and atmospheric pressure **(Almukhtar et al., 2012; Borowski et al., 2015; Ware and Power, 2018)**. According to (**Dechrugsa et al., 2013)**, the volumes of the produced biogas were recalculated for standard temperature (273 K) and pressure (1atm). For cross-checking measurements, the Gasmet DX4040 analyzer was utilized to assess the main constituents of the biogas produced as the end product of the anaerobic co-digestion process.

3. RESULTS AND DISCUSSION

3.1 Influence of Inoculum on Biogas Production

Fig. 2 presents the plots of biogas production and methane yield from UEO during the 30 days of operation, representing the total period for biogas production. After 30 days, no biogas generation was observed. Anyway, volumes of the produced biogas and methane yield were significantly low in all digesters of UEO even in the inoculated UEO. This observation could be justified that UEO normally contains a relatively high concentration of lead leached from certain corroded parts of the engines. Lead is one of the most potential reasons for bacterial poisoning, which is based on inhibiting the activity of bacteria and disrupting the anaerobic digestion process. **(Alrawashdeh et al., 2020)** investigated the effect of heavy metals on the anaerobic digestion process and suggested that heavy metals such as Iron, Nickel, Lead, Zinc, Copper, and Chromium may inhibit the bacteria and decrease the efficiency of biogas generation and methane yield. Also, the presence of heavy metals decreases the removal efficiency of volatile solids (VS), COD, and the organic acid load, which causes the decrease of pH and inhibition of methanogenesis.

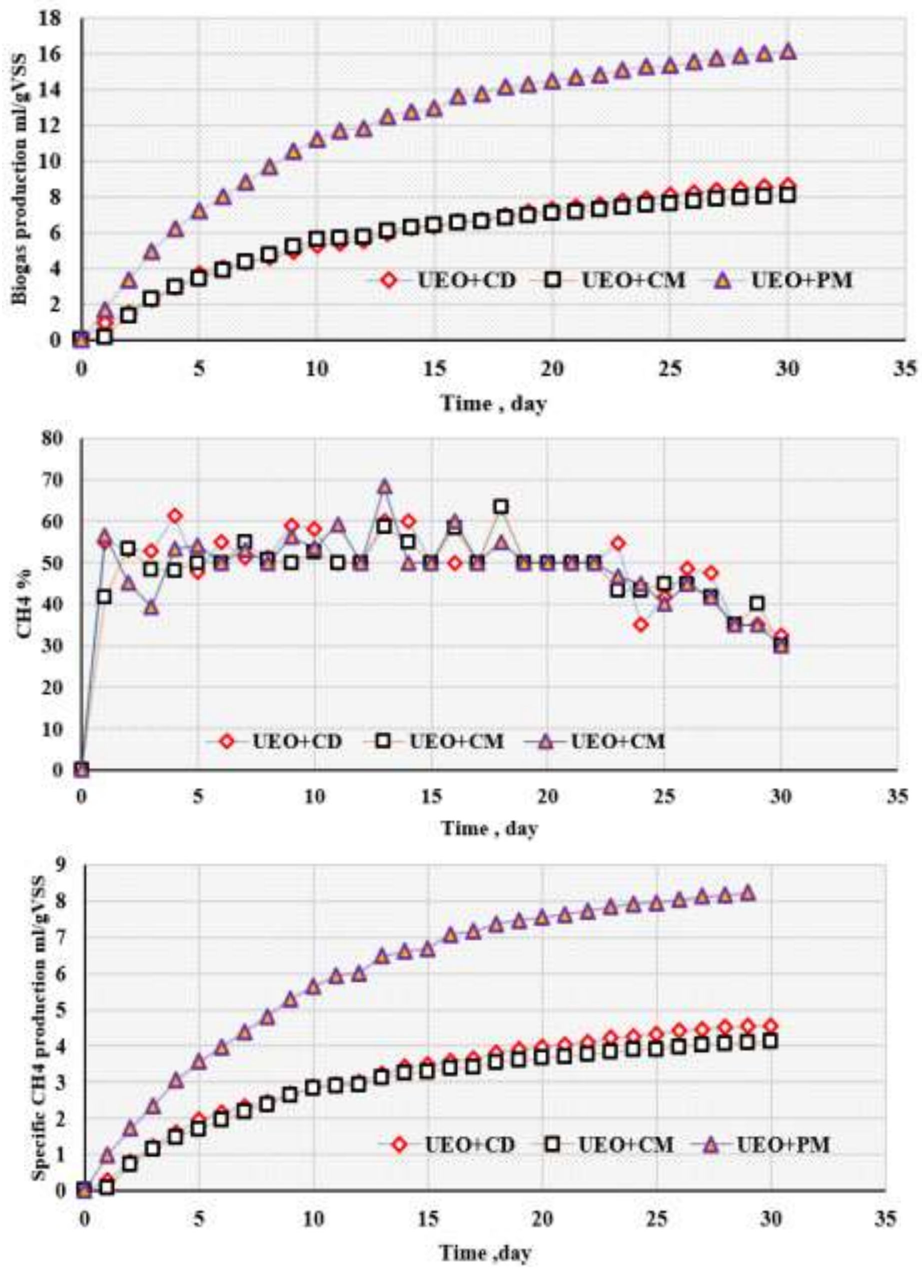

Figure 2. Profiles of biogas recovery and CH_4 yield from UEO with and without inoculums

Fig. 3 illustrates the plots of biogas generation and methane yield from mixed UEO and ROS. As shown in this figure, the duration of biogas production in this group of digesters was extended to 90 days compared to the first group of digesters. Also, the volumes of biogas production were greater when using mixes of UEO and ROS. It is worth mentioning that the addition of ROS to the UEO enhanced and increased the biogas recovery, which could be attributed to the assumption that crude oil is one of the main components of ROS in which; according to (**Sherry et al., 2020)** the crude oil is rich of methanogenesis bacteria. On the other hand, both inoculated UEO and ROS+UEO mix exhibited higher biogas production, as illustrated in **Table 3.**

Table 3. Influence of inoculum addition on biogas production

	Digester No.	Mixture symbol	Maximum specific biogas production (mL/g VSS)	Maximum specific CH_4 production (mL/g VSS)
Group 1	1	UEO	0	0
	2	UEO+CD	0.98	0.019
	3	UEO+CM	1.23	0.028
	4	UEO+PM	1.93	0.029
Group 2	5	UEO+ROS	2.45	1.890
	6	UEO+ROS+CD	3.49	2.620
	7	UEO+ROS+CM	2.48	1.450
	8	UEO+ROS+PM	3.65	2.450

This observation could be justified that the animals' dung is rich in methanogens and traces of metals. **(Wandera et al., 2018)** suggested that the presence of trace metals in animal manure results in an increment in methanogenic activity and accelerates the formation of methane. In this investigation, qualitative analysis of the animals' dung demonstrated that the poultry manure had higher phosphorous and nitrogen concentrations than cattle manure and cow dung. This fact may explain and justify the higher production of biogas from mixes inoculated with PM since poultry manure has a higher phosphorous and nitrogen content. **(Kafle and Chen, 2016)** reported an increase in biogas due to the increased percentage of nitrogen and phosphorous in poultry manure compared with cattle manure. Also, C/N is another important parameter known to be effective in the co-digestion process. The C/N ratio in the range of 20-30 represents the optimum acceptable range for biogas production by co-digestion process.

3.2 Kinetic Model

The biogas generation rate at batch mode condition is related to the specific growth rate of methanogenesis in the biodigester. Accordingly, the Modified Gompertz Model can be applied the predicting the rate of biogas generation **(Ejimofor et al., 2020)** as follows:

$$C_{(t)}= C_0 . \exp\{- \exp [(R_{max}\times 2.7183/C_0)\ (\lambda-t) +1]\} \quad (1)$$

where:
$C_{(t)}$ is the cumulative biogas generation at the digestion time (ml/g VS)
C_0 is the biogas potential of the substrate (ml/g VSS)
R_{max} is the maximum CH_4 yield rate (ml/g VSS.d)
λ is the lag phase (day)
t is the time (day)

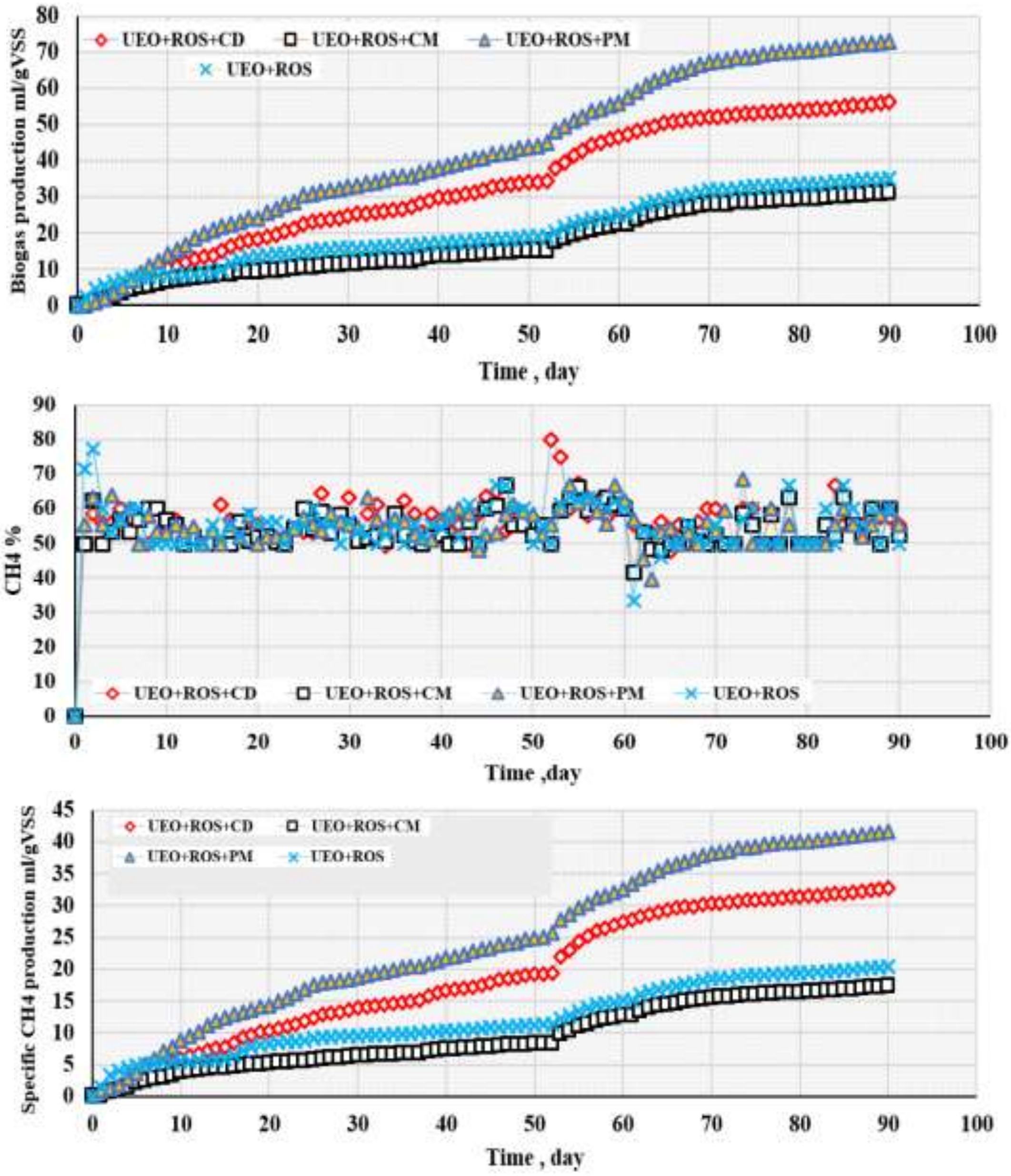

Figure 3. The profiles of biogas recovery and CH_4 yield from UEO+ROS with and without inoculums

To determine λ, R_{max}, generated biogas, and CH_4 yield, nonlinear least-square regression analysis was applied using SPSS **(IBM SPSS statistics V26, 2019).** The results are given in **Table 4.**

Table 4. Results of kinetic study at mesophilic conditions (37°C)

	Mixture symbol	$C_{(t)exp.}$ (mL CH_4/g VS)	Gompertz model parameters				R^2
			λ (day)	$R_{max.}$ (mL CH_4/g VS)	C_0 (mL CH_4/g VS)	$C_{(t)\ predicted}$ (mL CH_4/g VS)	
For UEO, after 30	UEO+CD	4.56	2.768	0.412	8.62	8.29	0.982
	UEO+CM	4.12	1.880	0.456	8.12	7.95	0.975

	UEO+PM	8.25	2.218	0.934	16.13	15.85	0.990
UEO+ROS after 90 days	UEO+ROS-CD	32.69	2.012	0.882	56.21	52.74	0.969
	UEO+ROS-CM	17.33	2.011	0.421	31.15	27.98	0.930
	UEO+ROS-PM	41.59	0.277	1.083	73.11	67.93	0.963
	UEO+ROS	65.47	5.468	0.438	34.99	31.48	0.929

The profiles of measured and predicted biogas generation are shown in **Figs. 4 and 5.** It is noticeable that the predicted values of biogas generation by the Modified Gompertz Model fitted very with the experimentally measured values. These outcomes agreed well with the previously outlined findings.

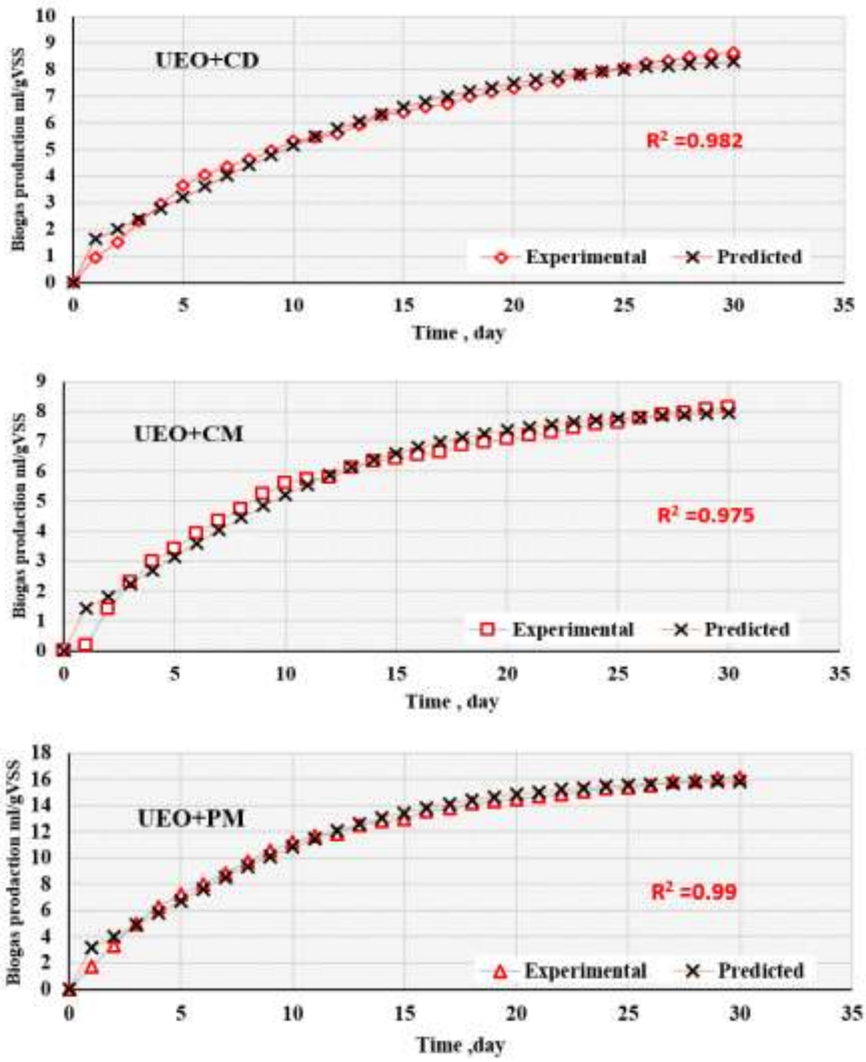

Figure 4. Predicted and experimental data for biogas production for UEO

(Shi et al., 2022) reported that the experimental results of biogas generated from the digestion of oily sludge waste were relatively congruent with the predicted values using the Modified Gompertz Model.

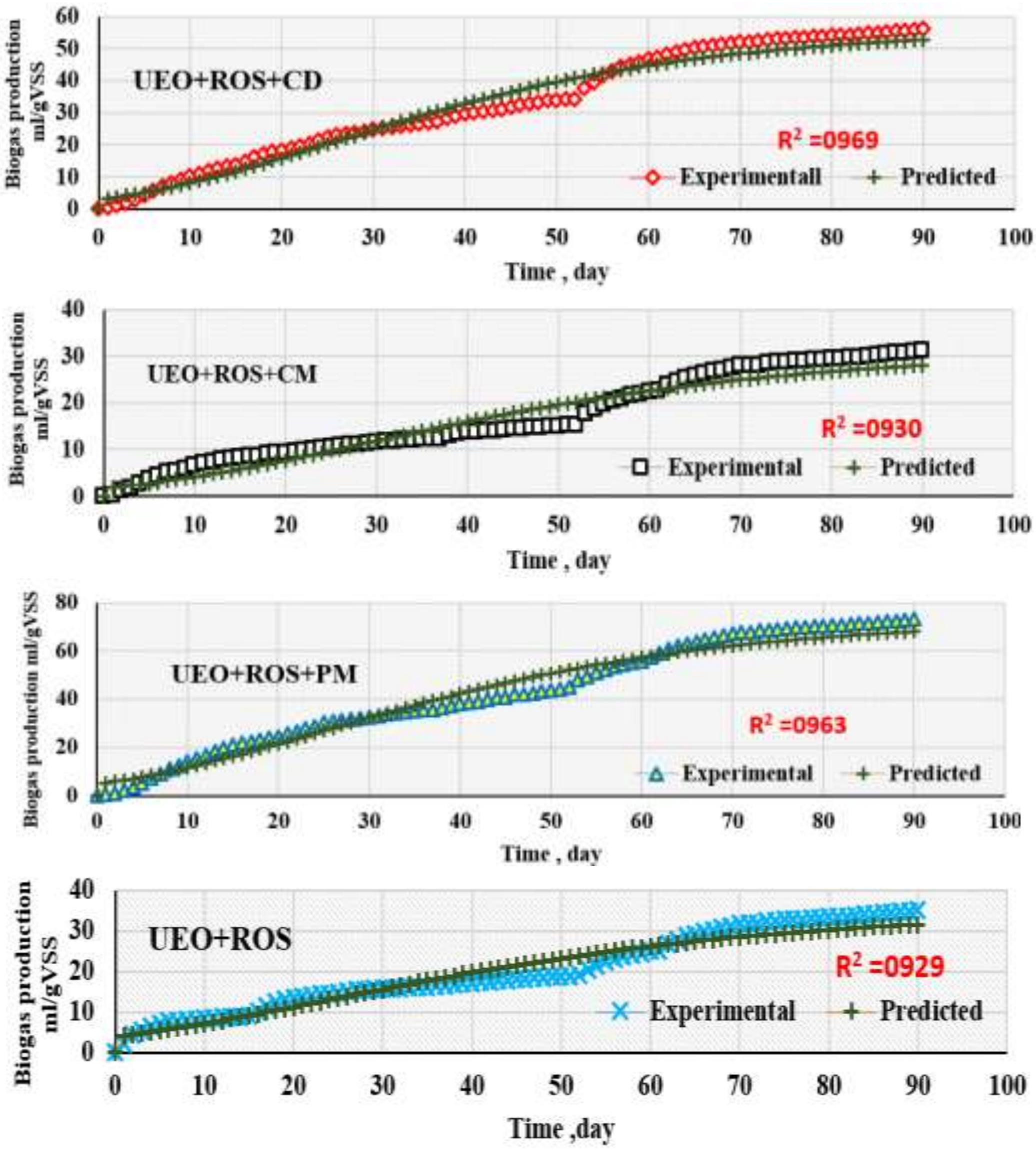

Figure 5. Predicted and experimental data for specific biogas production for UEO +ROS

4. CONCLUSIONS

This work was carried out to assess the validity and significance of the anaerobic co-digestion process for biogas generation and methane yield using UEO as the substrate and animals' dung, including poultry manure, cow dung, and cattle manure as co-substrates. The experimental work revealed that adding inoculum at mesophilic conditions notably affected the volume of generated biogas (37°C). During the 30 days observation period for the first group of biodigesters, maximum biogas production was 0.98, 1.23, 1.93, and 0 ml/g VS from UEO±CD, UEO±CM, UEO±PM, and UEO, respectively, whereby during the 90 days' observation period for the second group of biodigesters, maximum biogas production was 3.49, 2.47, 3.64 and 2.44 ml/g VS from UEO+ROS±CD, UEO+ROS±CM, UEO+ROS±PM, and UEO+ROS, respectively. The Modified Gompertz Model fairly described the kinetics of the digestion process. The measured and predicted values of biogas generation were well-fitted, with the coefficient of determination values higher than 0.92.

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