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Biogas Recovery from Refinery Oily Sludge by Co-Digestion Followed by Sustainable Approach for Recycling the Residual Digestate in Concrete Mixes

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ABSTRACT

This study investigated the potential of biogas recovery from refinery oily sludge (ROS) inoculated with animals' manure by co-digestion of in lab-scale biodigesters at mesophilic conditions. Cow dung (CD), cattle manure (CM), and poultry manure (PM) were utilized as co-substrates. The biogas production from the co-digestion process exceeds its production from uninoculated ROS by approximately 67.5, 22.13 and 21.6% for PM, CM, and CD, respectively. Kinetics of the co-digestion process was well described by the modified Gompertz model. The predicted and experimental values of biogas production were well fitted with $R^2 > 0.96$, suggesting favorable conditions of the digestion process. New approach for recycling the residual digestate to replace freshwater in concrete mixes was carried out. Results of examining the mechanical properties of the residual digestate-modified concrete mixes demonstrated a potential sustainable approach for the disposal of residual digestate in concrete mixes.

Keywords: co-digestion; biogas recovery; methane; gompertz model; refinery oily sludge; concrete.

INTRODUCTION

Energy security, environmental pollution and the increase in the emission of greenhouse gases is due to the consumption of fossil fuels as an energy source, which currently constitutes 88% of the total global energy consumption [1, 2]. Energy is the main factor in the implementation of domestic, commercial and industrial businesses, therefore, thinking about producing an environmentally friendly renewable energy source has become an alternative to fossil fuels. [3]. Biogas is one of the most important sources of renewable energies due to its ability to generate heat, steam, electricity and fuel for vehicles. Anaerobic digestion process is the major technology for biogas production [4]. It is considered as one of the best environmentally friendly processes for the disposal of complex organic wastes compared to the traditional methods such as incineration, burial and composting, in addition to its benefit of gas production and the control of CH_4 and CO_2 emissions in the anaerobic decomposition process in landfills [5]. Anaerobic digestion can be defined as the biological decomposition of complex organic matter including, fats and proteins into simple organic substances by the activity of anaerobic consortium under strict anaerobic conditions. The anaerobic bio-decomposition occurs through four subsequent steps which are hydrolysis, acidogenesis, acetogenesis and methanogenesis. Since the process is carried out by microorganisms, it is sensitive to the changes in temperature, acidity, and percentage C/N ratio [6, 7]. Methane is the main component (50–75%) of the produced biogas, CO_2 (25–50%) and trace amounts of other gases [1,8].

There are many sources of hydrocarbons that can be used in the anaerobic digestion and biogas production, one of these complex materials is the petroleum oily sludge. The primary source for oily sludge is exploration, transportation, storage and refining processes [9–11]. In general, the main components of petroleum oily sludge are water (30–85%), complex hydrocarbon compounds (15–50%), and solids (5–46%) [10, 12]. Refining 500 tons of crude oil produces a ton of oily sludge [13]. When dealing with these large quantities, especially with the increased global demand for crude oil consumption in the refining and petrochemical industry, the unsafe disposal or unsustainable improper treatment will pose a threat to the environment and human health [10]. Accordingly, reducing the risk and adverse effects of oil sludge to the environment has become a mandatory as it is one of the major problems in the oil industries. Petroleum oil sludge is a complex mixture of hydrocarbons and other recalcitrant compounds which may cause severe environmental pollution due to their persistence, wide distribution, and toxicity [14]. Numerous studies have considered some technical solutions for safe disposal of oily sludge including incineration, solidification, and biodegradation. However, there are many problems associated with incineration such as the emission of harmful gases from incineration as well as the high cost of fuel needed for burning and the disposal of the remaining ash [12, 15–16]. Anaerobic co-digestion is a well-established treatment technology in which a mixture of different feedstocks is processed into the fundamental anaerobic digestion technique to increase the biogenic methane content of produced biogas [17]. Numerous studies are available on biogas production from various waste materials such as, but not limited to olive pomace, faecal sludge, poultry manure, slaughterhouse waste, and cotton gin trash [18–21]. However, very limited studies dealt with biogas production from the digestion of petroleum oily sludge. The potential of biogas from the oily sludge by anaerobic digestion using methanogenic bacteria isolated from the intestine of a cow was investigated at mesophilic conditions (37°) and retention time 16 days [22]. Biogas production by the co-digestion of oily sludge with corn stover was assessed at thermophilic conditions (55°C) [23]. Biogas generation by co-digestion of oily sludge with sugarcane bagasse was investigated at mesophilic conditions (35–37°C) and retention time 33 days. The raw materials were pre-treated mechanically and thermo-chemically to further enhance the digestibility. The results demonstrated that the ideal ratio of C/N was in the range of 20–30 [24]. The effect of biochar dosage on methane production from the co-digestion of oily sludge (OS) with starch was studied at mesophilic conditions [25].

The current experimental and kinetic study aimed to; (1) assess the potential of biogas production from anaerobic co-digestion of real-field refinery oily sludge (ROS) individually inoculated with three types of animal manure including cow dung, cattle manure and poultry manure at mesophilic conditions, and (2) examine a novel application of the residual dilute digestate to replace fresh water in concrete mixes for complete sustainable product life cycle management.

MATERIALS AND METHODS

Substrate

Real samples of refinery oily sludge (ROS) were freshly grabbed from a local petroleum refinery in Baghdad, Iraq. This oily sludge is normally originated from three major sources which are; (1) API separators, (2) coagulation/flocculation unit of wastewater treatment plant in the refinery, and (3) the activated sludge resulted from the biotreatment of the refinery wastewater [26]. The pH value of ROS was 7.3 ± 0.1. Table 1 presents the major characteristics of the ROS.

Inoculums (co-substrates)

Three types of inoculums including cattle manure (CM), poultry manure (PM) and cow dung (CD) were alternatively used as co-substrates with ROS to inseminate the activity of bacteria, improve the efficiency of composting, and boost the anaerobic co-digestion process in the bio-digesters. The analysis of CM, PM,

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

Constituents	Units	Average concentration (mg/L)
Chemical Oxygen demand (COD)	mg/L	30,520
Total petroleum hydrocarbons (TPH)	mg/L	980
Oil & grease (O&G)	mg/L	2,300
Total dissolved solids (TDS)	mg/L	2,970
Total suspended solids (TSS)	mg/L	3,270
Pb^{+2}	mg/L	6.35
Zn^{+2}	mg/L	30.1
Cu^{+2}	mg/L	2.40
V^{+2}	mg/L	94

and CD samples indicated that the dominant bacterial cells were *Escherichia Coli, Serratia fonticola,* and *Escherichia Coli*, respectively. It is known that the animals' manure contains different microbial communities especially methanogenesis, so a more stable mixture of higher nutrient content for the co-digestion process can be obtained by inoculation of the substrate with animals' manure [27].

Experimental setup and digesters operation

In this experimental investigation, the anaerobic co-digestion of ROS with animals' dung and manure was performed in bench scale-digesters operated in a batch mode. The experimental system comprised of 500 mL Pyrex borosilicate heatproof code glass bottles which were used as the anaerobic digesters. Four digesters were setup in duplicate (a total of 8 digesters). Each biodigester contained 320 ml refinery oily sludge (ROS) and 80 ml of inoculum to maintain 1:4 volume ratio resulted in a total volume of 400 ml. Every digester was tightly plugged with a rubber stopper. Each stopper contained 2 holes of 4mm diameter for each hole. These holes were utilized to introduce into the digester a piece of the glass tube and the other end of the glass tube was connected with a rubber tube for the transfer of biogas to the gas measuring system. Parafilm was used to wrap the rubber stoppers tightly to prohibit the release of produced gas. To keep and maintain anaerobic conditions in the digesters, they were flushed with nitrogen for 15 min. The biodigesters were placed in a thermostatic water bath to achieve and preserve mesophilic conditions at 37°C. The digesters were regularly manually shaken to provide better contact between the biomass and the substrate. pH adjustment was performed using sodium bicarbonate ($NaHCO_3$) [28, 29]. Food grade dye was used to color the water in the displacement bottle. Table 2 illustrates the contents of each digester.

Analysis

Total volatile solids (TVS), and total suspended solids (TSS) measurements were performed based on the procedures of the Standard Methods [30]. Chemical oxygen demand (COD) was measured using a COD analyzer (Type: Lovibond COD/RD/125). Heavy metals concentrations were detected using atomic absorption spectroscopy (Model: GBC A.C.N. 005 472 686, Australia). The analysis for C, N, P and K concentrations was carried out according to a reported procedure [31].

The produced biogas was measured by using the liquid displacement method. At first, the biogas passed through a glass vial containing 1M NaOH solution to remove the CO_2 from biogas. The CO_2 –saturated alkaline solution was periodically replaced by a new solution. Then the remaining CH_4 passed to another glass vial, displacing the colored water which overflowed into a volumetric cylinder. Volume of the displaced colored water was equal to the volume of produced CH_4 (Figure 1). All measurements were conducted at atmospheric pressure and room temperature [32–34]. Volumes of the produced biogas were recalculated based on the standard pressure and temperature (STP: 273 K and 1 atm) [35]. Cross checking measurements of the biogas components were accomplished using Gasmet DX4040 analyzer. Major characteristics of the uninoculated and inoculated ROS are given in Table 3.

Preparation of residual digestate-modified concrete mixes

To affirm the sustainability and validity of the suggested path for ROS treatment and to achieve the product life cycle as well, a decision was made to experimentally investigate the reuse of residual digestate of liquid texture to replace the fresh water in concrete mixes. The decision was made based on; (1) establish a safe approach to get rid of the residual digestate

Table 2. Details of digesters set-up (prepared in duplicate)

Digester No.	Symbols	Experimental conditions
1	ROS-CD	ROS inoculated with cow dung
2	ROS-CM	ROS inoculated with cattle manure
3	ROS-PM	ROS with poultry manure
4	ROS-C	Uninoculated ROS (without animals' inoculum) considered as the control

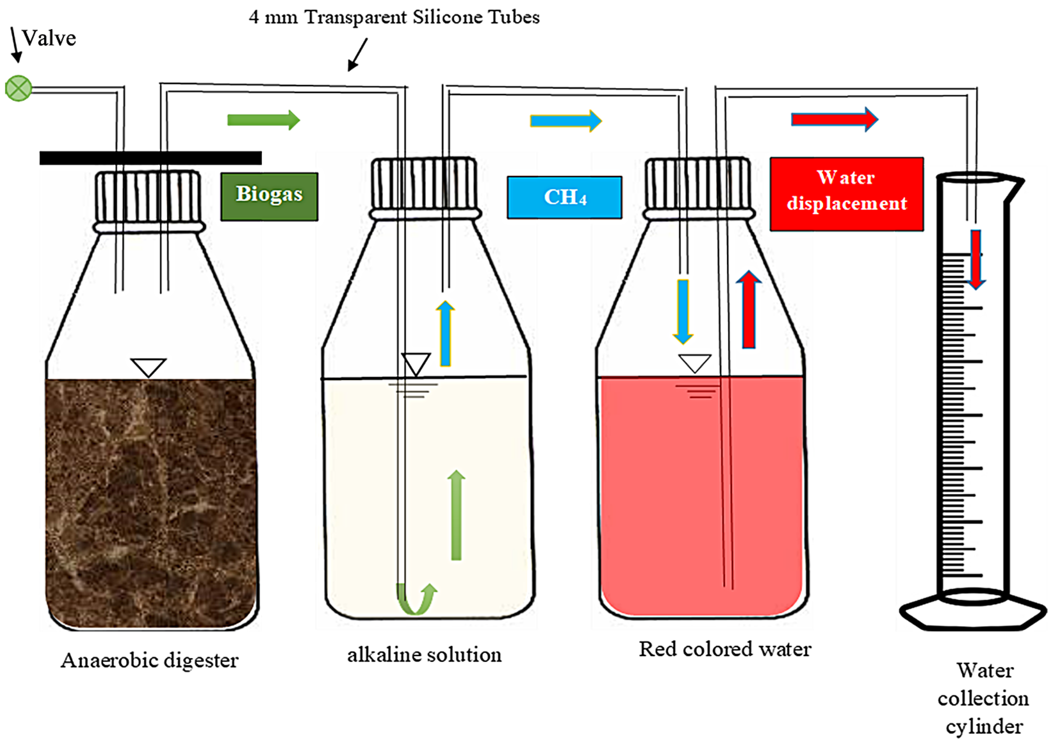

Figure 1. Schematic diagram of the experimental system

rather than being released to the water resources or randomly dumped, and (2) achieve a sustainable approach for preparing green concrete mixes modified with waste material, meanwhile preserving the fresh water from excessive usage. Fifty-four concrete moulds of the digestate-modified concrete mixes were prepared to examine their mechanical properties including workability (slump) test, compressive and flexural strengths, dry density, as well as the leaching test. The concrete mixes were designed according to the British Standards with cement: sand: gravel ratio of 1:1.5:3 [36]. Table 4 presents the details of concrete mixes. Ordinary Portland cement was utilized for preparing of concrete mixes. Coarse aggregate was natural crushed stone of 20 mm maximum, whereby, the fine aggregate was natural sand of desert origin and maximum size of 4.75 mm.

RESULTS AND DISCUSSION

Influence of inoculum type

The influence of inoculum type on biogas production and methane yield is shown in Figures 2–5 and Table 5. It is well observed that the highest biogas production was obtained with ROS-PM compared to ROS-CM and ROS-CD. Also, it is worth to mention that the biogas production and methane yield from the uninoculated oily sludge (ROS-C) were the lowest compared to the inoculated ROS. These observations were attributed to the availability of methanogenic species at different concentrations in the digested mixes which in turn affected the anaerobic co-digestion process and subsequently the amount of biogas production. Also, C/N ratio could be another major factor which affected the digestion process. The C/N

Table 3. Major characteristics of the uninoculated and inoculated ROS

Constituents	Unit	Concentration			
		ROS-C (control)	ROS-CD	ROS-CM	ROS-PM
COD	mg/L	30,520	39,150	38,526	38,410
TSS	mg/L	3,275	3,540	3,620	3,540
TVS	mg/L	11,980	12,650	12,150	12,000
pH	-	7.30 ± 0.1	7.18 ± 0.3	7.02 ± 0.2	7.76 ± 0.3

Table 4. Details of the digestate-modified concrete mixes

Mixes symbols*	Material				W/C
	Cement (kg/m³)	Sand (kg/m³)	Gravel (kg/m³)	Water (kg/m³)	
MC	394	591	1182	197 (fresh water)	0.50
MS1	394	591	1182	177.3 (residual digestate)	0.45
MS2	394	591	1182	197 (residual digestate)	0.50
MS3	394	591	1182	216.7 (residual digestate)	0.55

Note: *MC – control mixes control prepared with fresh water, MS – digestate-modified concrete mixes.

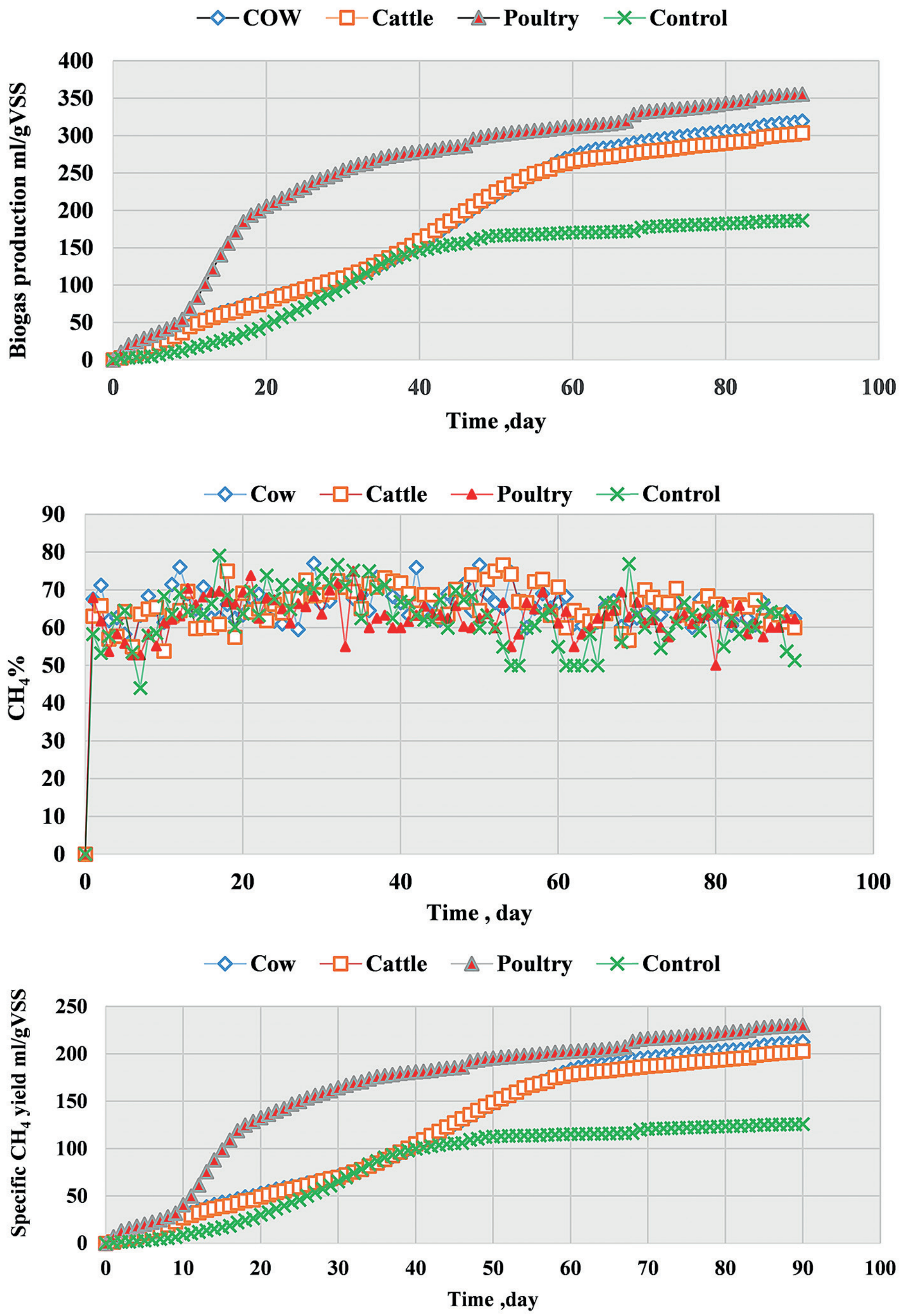

Figure 2. The profiles of biogas and methane production from ROS with and without inoculums

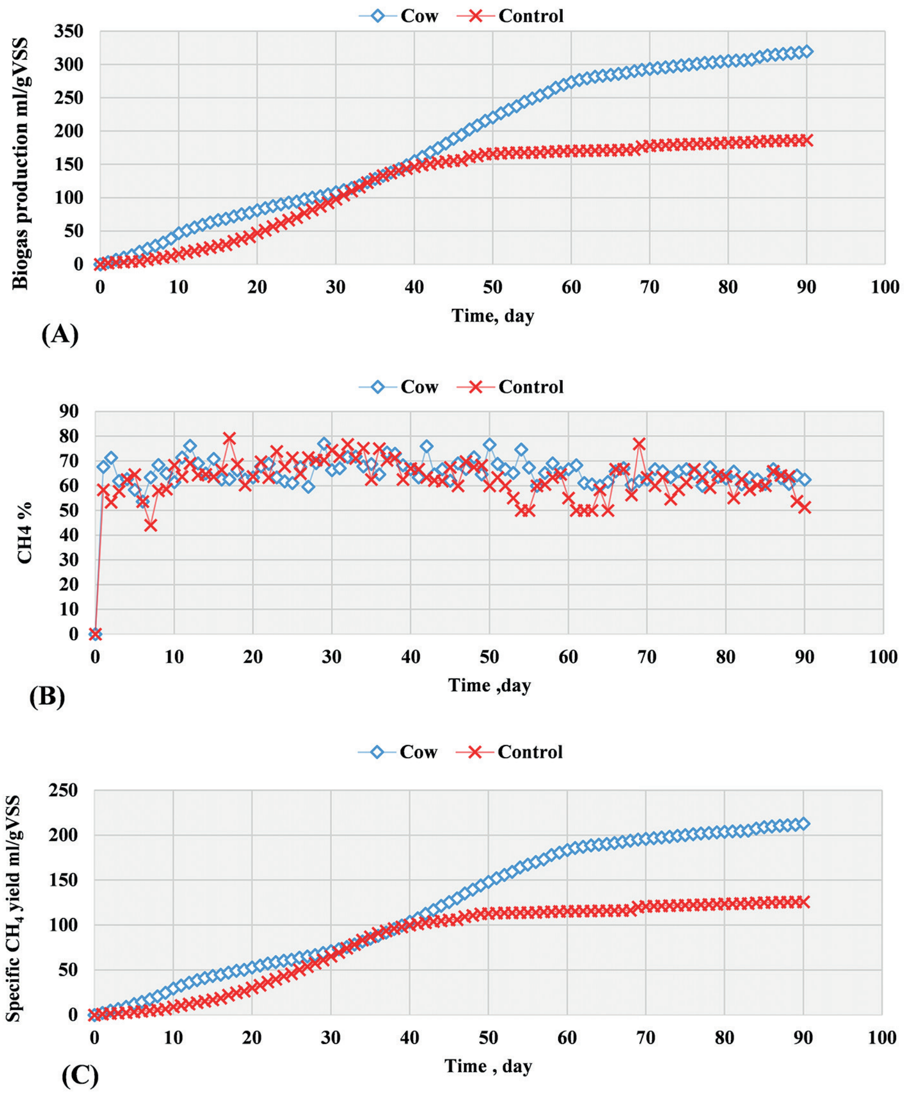

Figure 3. ROS-CD versus ROS-C; (a) biogas production (b) percentages CH_4 production (c) specific CH_4 production

ratios for the ROS-C, ROS-PM, ROS-CM, and ROS-CD were 20.47, 21.90, 28.64, and 27.96, respectively. The variations in C/N values were due to the differences in the nitrogen and carbon contents in the manures (Table 6).

It is well observed that, the nitrogen contents in the 3 types of manures were comparable with slight differences. ROS-C had the lower C/N indicating that the inoculation process with different types of animals' manures increased the C/N ratios, and subsequently enhanced and improved the co-digestion process. The C/N ratio in the manures is considered as a principal factor that affect the digestion process [37]. The organic fraction of poultry manure had C/N ratio varied from 1:1 to 27:1 [38]. reported that the optimum range for C/N ratio for anaerobic digestion is from 20:1 to 30:1 [3]. Higher C/N ratio will cause low biogas production. This could be attributed to the fact that nitrogen will be rapidly utilized by methanogenesis to achieve their protein needs and will no longer react on the remaining carbon in the material.

From the above-mentioned facts and due to the higher nitrogen and phosphorous content in the ROS-PM, it exhibited higher biogas production compared to ROS-C, ROS-CM, and ROS-CD. An increase in biogas production due to the increased percent of nitrogen and phosphorous in poultry manure compared to cattle manure was reported [39]. Also, the moisture content could be another important influencer since increasing the moisture content facilitate the mixing process and enhanced the transfer of bacteria. The presence of trace metals in animals' manure results in an increment in the methanogenic activity and accelerates the formation of

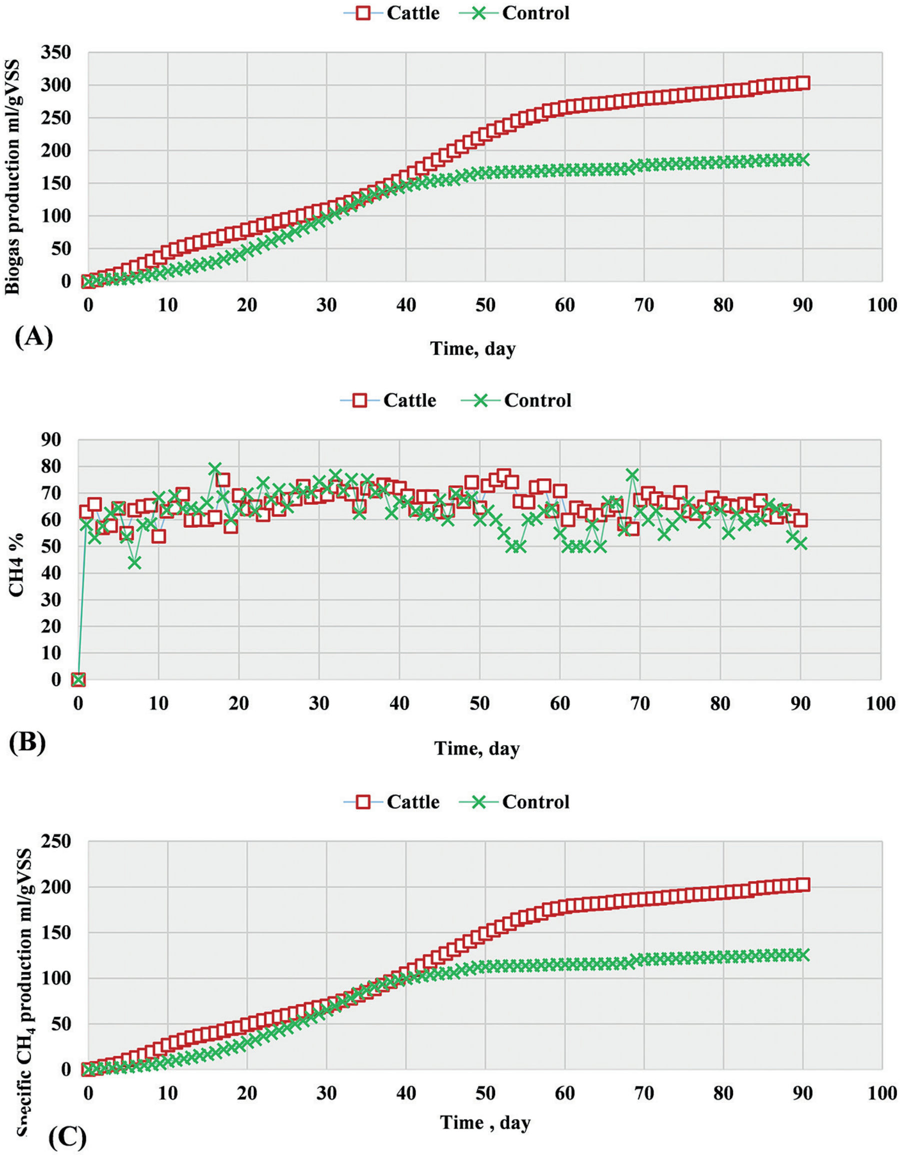

Figure 4. ROS-CM versus ROS-C; (a) biogas production (b) percentages CH_4 production (c) specific CH_4 production

methane [4]. In this study, the analysis of the animals' manures indicated the presence of potassium, calcium, magnesium, manganese, zinc, and copper in their texture. The higher contents of some metals were observed in the poultry manure which may explain the higher production of biogas from ROS-PM compared to other tested mixes. In addition, to methanogenic bacteria, the existence of *Serratia fonticola* as a dominant type of microorganisms in the PM as mentioned in section 2.2 could be another reason behind the higher degradation of hydrocarbons and organic content in the ROS since *Serratia fonticola* is considered as hydrocarbon degrading bacteria.

However, biogas production observed in the uninoculated digester (ROS-C) could be attributed to the fact that one of the main components of ROS is the residue of crude oil from the API separators as well as the sludge from the storage tank. The presence of the crude oil in the ROS may explain the source of methanogenesis in the control digester. Oil and hydrocarbons degradation in deep reservoir environments and geosphere has been attributed to methanogenic microbial consortia [40, 41].

Removal of organic content and total suspended solids

The effect of anaerobic co-digestion on the removal of organic content as COD and TSS is shown in Table 7. The high removal efficiencies up to 92% indicated the potential of this process, in particular the ability of methanogenesis to

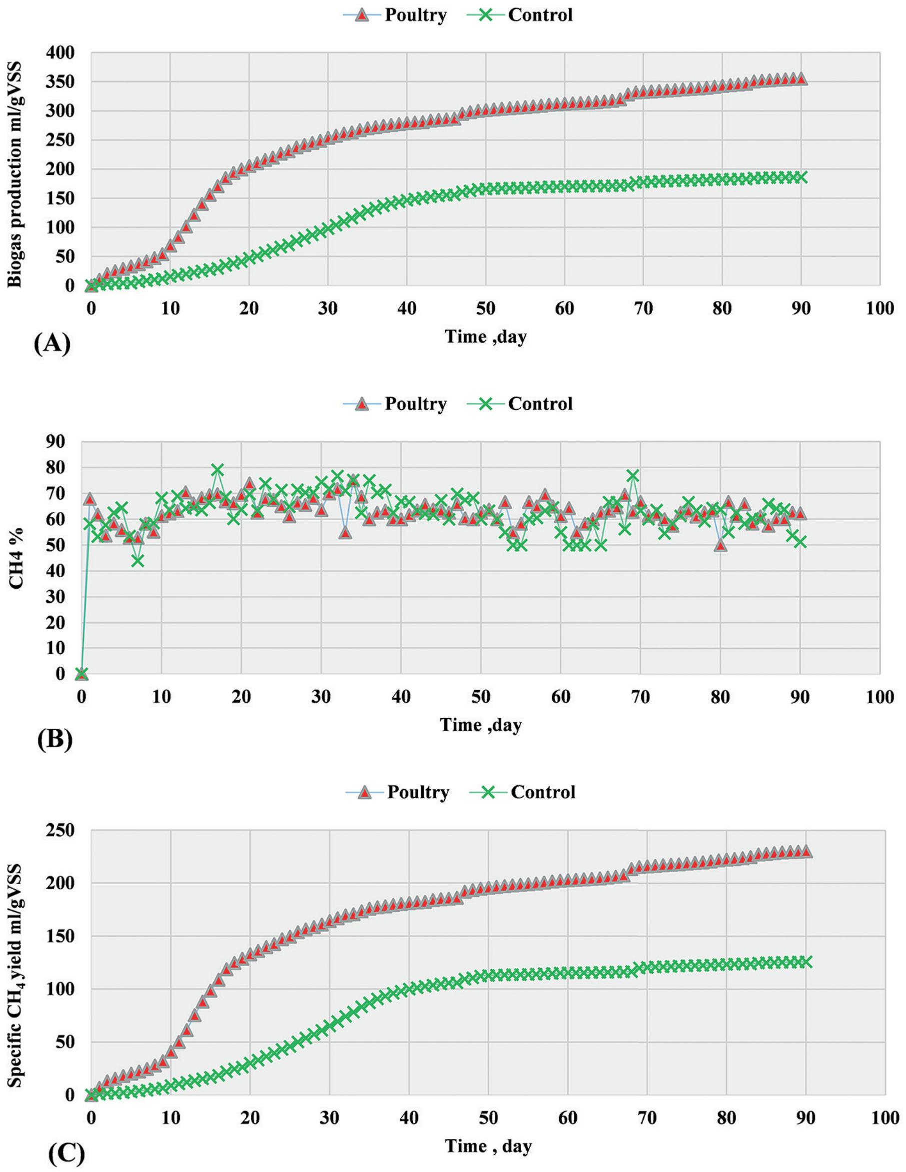

Figure 5. ROS-PM versus ROS-C; (a) biogas production (b) percentages CH_4 production (c) specific CH_4 production

degrade the organic constitutes including the hydrocarbon complex compound in the ROS. These results well agreed with the previous observations [14, 42].

Kinetic study

The rate of biogas production at batch conditions is related to the specific growth rate of methanogenic bacteria in the digester. Modified Gompertz Model can be applied for the prediction of biogas production rate as follows [43]:

$$P_{(t)} = P_0 \cdot \exp\{- \exp [(R_{max} \times 2.7183/P_0) (\lambda - t) + 1]\} \quad (1)$$

where: $P_{(t)}$ – cumulative biogas yield at the time of digestion (mL/g VS),
P_0 – the substrate potential for biogas (ml/g VSS);
R_{max} – maximum production rate of CH_4 (ml/g VSS.d),
λ – Lag phase (day),
t – time (day).

A nonlinear least-square regression analysis was applied using SPSS [IBM SPSS statistics V26, (2019)] to assess the values of λ, R_{max}, and the predicted biogas and CH_4 yield. The predicted kinetic parameters of Gompertz model are presented in Table 8. The plots of the experimental and predicted values of biogas production are

Table 5. Influence of inoculum addition on biogas production

Digester No.	Inoculum	Maximum specific biogas production (mL/g VSS)	Maximum specific CH_4 production (mL/g VSS)	Biogas increase (%)
1	ROS-CD	8.76	6.086	21.6
2	ROS-CM	8.82	5.493	22.1
3	ROS-PM	21.16	15.695	67.5
4	ROS-C (control)	6.87	5.036	-

Table 6. Carbon and nitrogen contents in the ROS mixes

Constituents	ROS mixes			
	ROS-C	ROS-CD	ROS-CM	ROS-PM
Organic carbon (C) %	47.50	79.74	80.65	83.58
Nitrogen (N) %	2.38	1.19	1.05	3.22
Phosphorous (P) %	0.01	0.50	0.44	0.53
C/N	20.47	27.96	28.64	21.90

Table 7. Removal efficiencies of COD and TSS in the biodigesters

Digester No.	Mixture symbol	Removal efficiency %	
		COD	TSS
1	ROS-CD	92.8	80.8
2	ROS-CM	91.5	82.9
3	ROS-PM	91.9	84.5
4	ROS-C (control)	87.9	85.3

Table 8. Kinetic study results at mesophilic conditions after 90 days

Type of mixes	$G_{(t)exp.}$ (mL CH_4/g VS)	Gompertz model parameters				R^2
		λ (day)	$R_{max.}$ (mL CH_4/g VS)	G_0 (mL CH_4/g VS)	$G_{(t)\ predicted}$ (mL CH_4/g VS)	
ROS-CD	304.0	8.563	5.760	319.70	174.47	0.979
ROS-CM	202.95	8.032	5.705	303.72	291.41	0.985
ROS-PM	230.43	0.233	7.849	355.47	351.68	0.965
POS-C	125.94	10.550	5.252	186.38	185.23	0.996

presented in Figure 6. It is clearly noted that both measured and predicted values of biogas production were well fitted.

Sustainable approach for the residual digestate management

As mentioned earlier in this study, a novel implementation of the residual digestate was carried out to replace the fresh water in concrete mixes to achieve a complete sustainable management of the ROS. The most significant criterion for the validity of the suggested approach is the influence of the residual digestate on the fundamental mechanical properties of the digestate-modified concrete mixes. The properties of these digestate-modified concrete were as follows:

Slump test and workability – the slump values were found to be 20, 28, 40 and 65 mm for specimens MS1, MS2, MS3, and MC, respectively on a dry basis (Figure 7). The results revealed that for concrete mixes prepared with the residual digestate, the slump increased with increasing the W/C to 0.55. However, the slumps values of all modified concrete mixes were less than its value for the control mix which was prepared with fresh water. In spite of the decline in the slump, the digestate- modified concrete mixes were considered workable since the acceptable slump values range is 20–80 mm [44].

Compressive strength – the results of compressive strenth test for MC, MS1, MS2, and MS3 at 7 and 28 days are shown in Figure 8. It is well obseved that MS1 and MS2 exhibited the

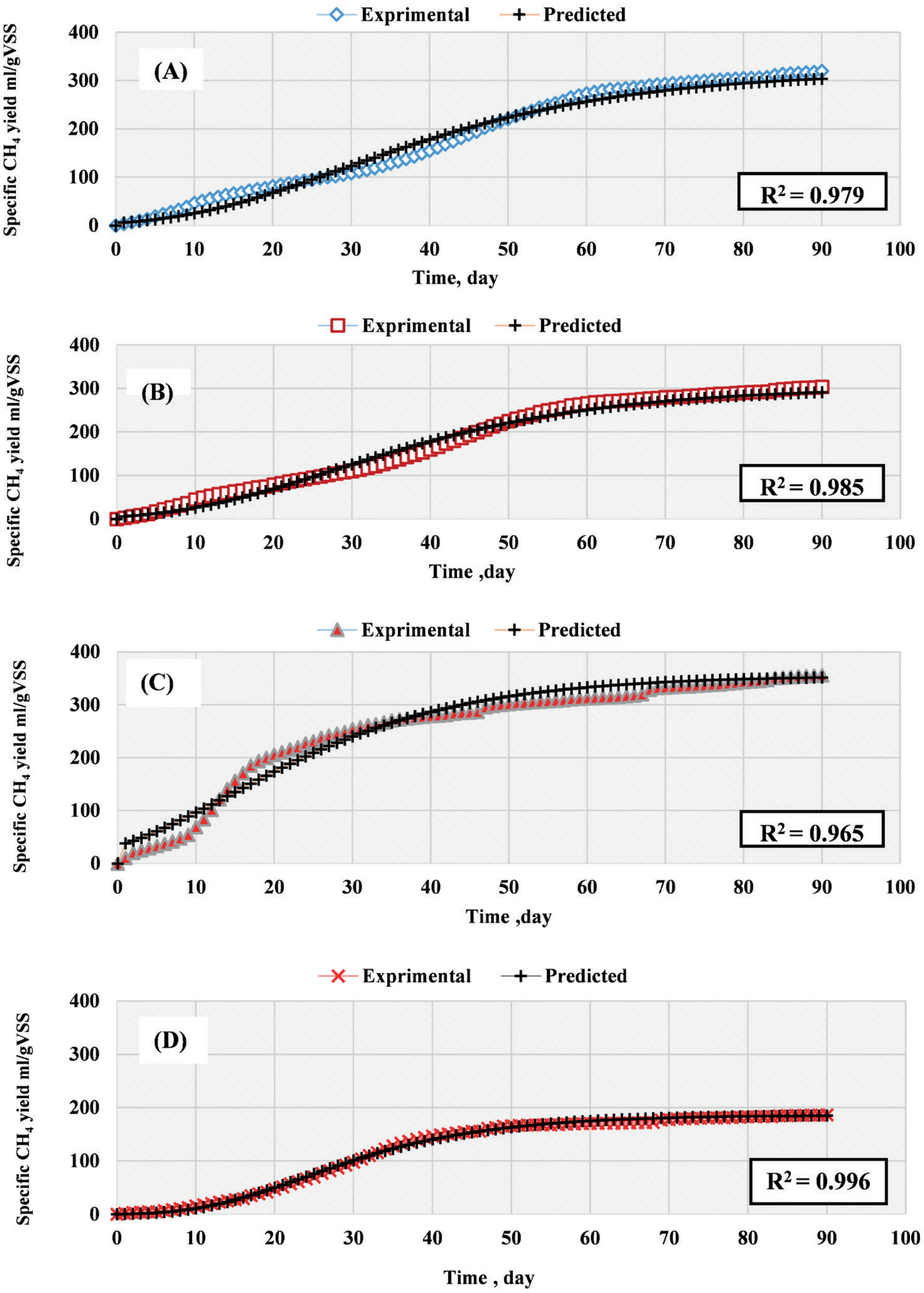

Figure 6. Measured and predicted data for; (a) ROS-CD, (b) ROS-CM, (c) ROS-PM, (d) ROS-C

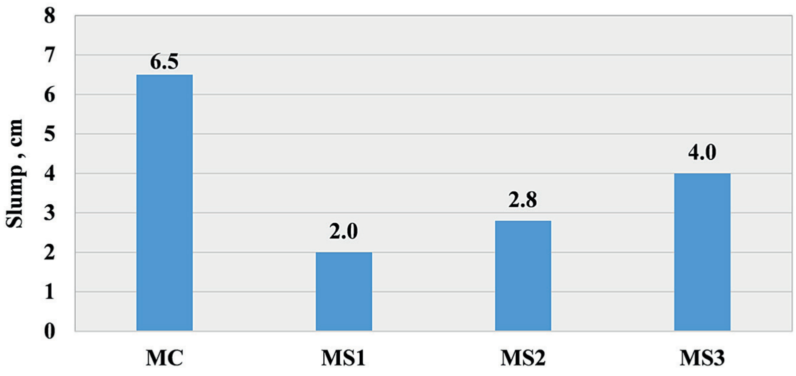

Figure 7. Slump values for concrete mixes

higher compressive strength values which were considered acceptable because they are within the allowable range of compressive strength according to the Brithis Standards for lightweight concrete which can be used in domestic floors, workshop bases, garages, driveways and internal floor slabs where achived the requirement of lightweight concrete [36].

Flexural strength – the results of flexural strength test at 7 and 28 days are given in Figure 9. The results demonstrated comparable values of 4.1± 0.2, 4.3 ± 0.06, 4.2 ± 0.09, and 4.0 ± 0.06 for MC, MS1, MS2, and MS3 respectively. However, the highest value of flexural strength was obtained with MS1 (W/C= 0.45), whereby, the lowest value was observed

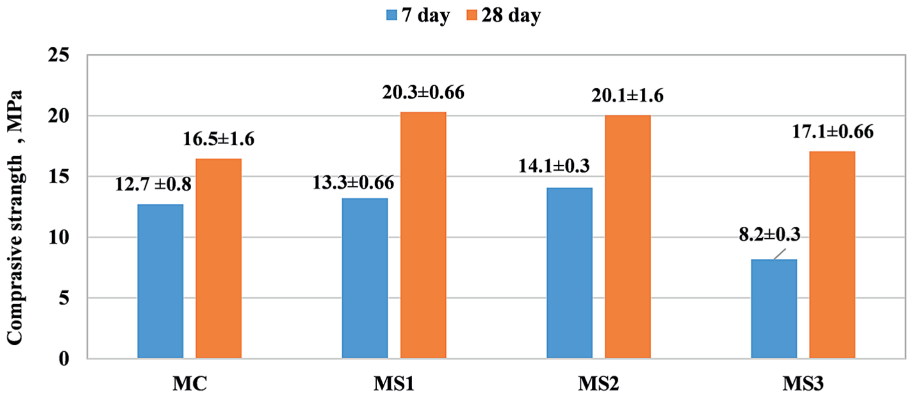

Figure 8. Compressive strength for the concrete mixes

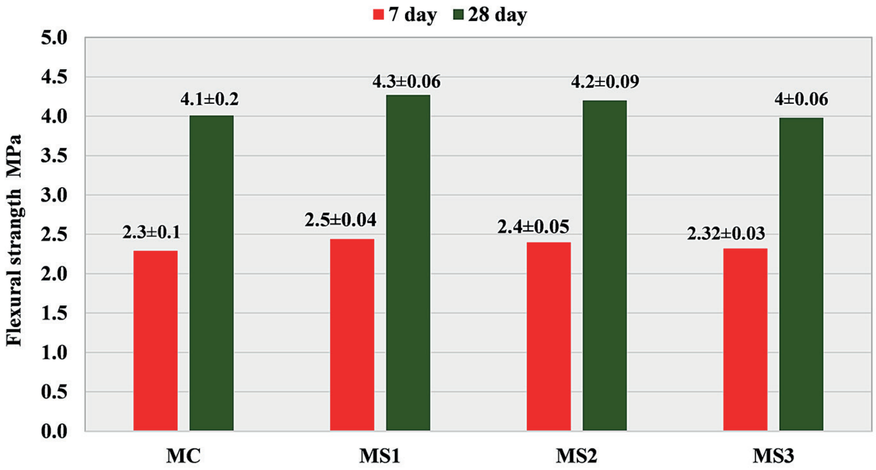

Figure 9. Flexural strength for the concrete mixes

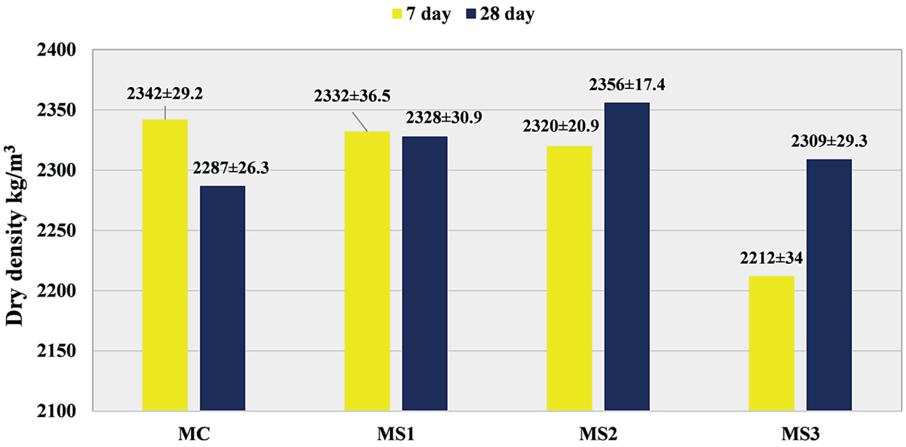

Figure 10. Dry density for the concrete mixes

Table 9. Quality of the leachate from the digestate- modified concrete after 90 days

Constituent	Unit	Quality of the residual digestate introduced into the digestate-modified concrete mixes	Quality of the leachate from the digestate-modified concrete mixes
Total suspended solid (TSS)	mg/L	850	< 3
Total dissolved solid (TDS)	mg/L	1,300	< 50
Chemical oxygen demand (COD)	mg/L	3,000	Nil
pH	-	7.01	7.03
Viscosity	Ns/m^2	$1.003x10^{-3}$	$1.003x10^{-3}$
Color	mg/L	Light brown	Clear, colorless
V^{+2}	mg/L	95	Nil
Zn^{+2}	mg/L	32.5	Nil
Pb^{+2}	mg/L	7.65	Nil
Cu^{+2}	mg/L	3.50	Nil

with MS3 (W/C= 0.55) which agreed with results of the compressive strength.

Dry density – the dry density values for MC, MS1, MS2, and MS3 mixes were 2287 ± 36.3, 2328 ± 30.9, 2356 ± 17.4, and 2309 ± 29.3 kg/m^3, respectively (Figure 10). Those results excedded the range of dry density for lightweight concrete which is from 320 to 1920 kg/m^3 according to [45].

Leaching test – the leaching test was carried out according to a previosly reported procedure [46]. The results of leaching test demonstrated none of the residual digestate contents were detected in the leachate. Also, the pH of leachate didn't notebly changed (Table 9). Therefore, the disappearance of the target contaminants in the leachate could be attributed to the presumption that a possible retention of these constuients occured within the concrete matrix. The results of the leaching test indicated that using the residual digestate is non-hazardous for concrete mixes.

CONCLUSIONS

Results of the experimental study for biogas production from refinery oily sludge revealed that biogas production was affected by the type of inoculum. Maximum biogas production from the ROS-CD, ROS-CM, ROS-PM and ROS-C were 8.76, 8.82, 21.16, and 6.86 mL/g VS, respectively. Modified Gompertz model was applied for studying the kinetic of co-digestion process. The predicted results by this model were well fitted with the experimental values of biogas production with correlation coefficient values > 0.96. The current study was extended for a novel application of the residual digestate to replace fresh water in green concrete mixes The mechanical properties of the digestate –modified concrete mixes implied that recycling and reusing the residual digestate was significant.

Future work will consider the co-digestion of the refinery oily sludge in a continuous mode.

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