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Assessment of anaerobic co-digestion of agro wastes for biogas recovery: A bench scale application to date palm wastes

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Abstract
Anaerobic digestion is a technology widely used for treatment of organic waste to enhance biogas recovery. In this study, recycling of date palm wastes (DPWs) was examined as a source for biogas production. The effects of inoculum addition, pretreatment of substrate, and temperature on the biogas production were investigated in batch mode digesters. Results revealed that the effect of inoculum addition was more significant than alkaline pretreatment of raw waste materials. The biogas recovery from inoculated DPWs exceeds its production from DPWs without inoculation by approximately 140% at mesophilic conditions. Whereby, the increase of biogas recovery from pretreated DPWs was 52% higher than its production from untreated DPWs at mesophilic conditions. The thermophilic conditions improved the biogas yield by approximately 23%. The kinetic of bio-digestion process was well described by modified Gompertz model and the experimental and predicted values of biogas production were fitted well with correlation coefficient values > 0.96 suggesting favorable conditions of the process.

Keywords: Biogas; Anaerobic Co-digestion; Agriculture waste; Date palm wastes; Methane recovery.

1. Introduction

Renewable energy is a socially and politically defined category of energy sources. Among the different forms of renewable sources, biomass is undoubtedly one of the most promising [1]. About 16% of global final energy consumption comes from renewable resources, with 10% of all energy from traditional biomass, mainly used for heating, and 3.4% from hydroelectricity. When biomass is burnt or digested, the emitted CO_2 is recycled into the atmosphere, so not adding to atmospheric CO_2 concentration over the lifetime of the biomass growth [2]. Anaerobic digestion has been, and continues to be, one of the most widely used processed for the stabilization of biosolid waste, such as from the agro and municipal waste to industrial waste. The widespread use of this technology stems from its potential advantages including, the production of energy of methane, a reduction of 30–50% of waste volume requiring ultimate disposal, and a rate of pathogen destruction-particularly in the thermophilic process. The stabilized biomass can also be utilized as an excellent soil conditioner after appropriate treatment [3]. The composition of biogas varies depending upon the types and relative contents of different raw materials, as well as upon the different conditions and fermenting phases. The quality of biogas generated by organic waste materials does not remain constant but varies with the period of digestion [4]. Several studies have been reported about the co-digestion of lignocellulosic waste materials and agro wastes for biogas production. Rincón et al. [5] studied the methanogenic stage of a two-stage anaerobic digestion process treating two-phase olive oil mill solid residue (OMSR) at mesophilic temperature (35°C). A methane yield of 0.268 ± 0.003 L CH_4 at standard temperature and pressure conditions (STP) g^{-1} COD eliminated was achieved.

Jaafar [6] verified the possibility of using a special type of Iraqi date fruit named Zahdi (normally used for syrup production) as a resource for biogas production at thermophilic digestion with activated sludge as inoculum. Methane was produced with a yield of 570 mL/ VS of substrate. Addition of 1% yeast extract solution as nutrient increased methane yield by 5.9%. Marňóna et al. [7] studied the production of biogas co-digestion of cattle manure with food waste and sewage sludge mesophilic and thermophilic conditions using continuously stirred-tank reactors. Maximum obtained value was 603 LCH_4/kg VS feed for the co-digestion of a mixture of 70% manure, 20% food waste and 10% sewage sludge at 36°C. Lower methane yields were obtained when operating at 55°C. Kafle & Kim [8] evaluated the performance of anaerobic digesters using a mixture of apple waste (AW) and swine manure (SM). This mixture improved the biogas yield by approximately 16% and 48% at mesophilic and thermophilic temperatures, respectively, compared to the use of SM only, but no significant difference was found in the methane yield. Tampio et al. [9] compared the anaerobic digestion of autoclaved and untreated source segregated food waste (FW) over 473 days in semi-continuously fed mesophilic reactors with trace elements supplementation. Methane yields were 5–10% higher for untreated FW than autoclaved FW. However, none of the previously reported studies have dealt with the date palm wastes. The date palm *Phoenix dactylifera* has played an important role in the day-to-day life of the people for the last 7000 years. Today worldwide production, utilization and industrialization of dates are continuously increasing since date fruits have earned great importance in human nutrition owing to their rich content of essential nutrients. Tons of date palm wastes are discarded daily either as an agricultural by product of no economic wastes or by the date processing industries without proper waste management leading to environmental problems. Thus, there is an urgent need to find suitable applications for this waste [10]. Current study, aimed to assess for the first time the biogas production and recovery from the anaerobic co-digestion of date palm wastes.

2. Materials and methods

2.1 Materials

The date palm wastes (DPWs) used in this study involved mixed petiole, rachis, fronds, and leaflet waste materials resulted from the tapping and trimming processes of the date palm trees. This type of solid waste materials is abundantly available in Iraq without proper management and application.

The average measured values of total solids (TS), volatile solids (VS), and pH for the mixed date palm wastes samples were found to be 45.91 ± 2.57, 41.42 ± 1.04, and 7 ± 0.2, respectively. Cattle manure which is known to be rich in methanogenic anaerobic bacteria was used to inoculate the digesters. Cattle manure was freshly collected from a local slaughter house, prepared as slurry, and then added to the digesters as a supplementary material to enrich the bacterial activity and enhance the anaerobic co-digestion process.

2.2 Pretreatment of wastes materials

The pretreatment of the collected date palm wastes, was carried out to facilitate the hydrolysis of cellulose component existing in the substrate. Cellulose and lignin has a highly crystalline structure due to the presence of an extensive hydrogen bond and inter-chain in the cellulose structure. After cleaning manually the collected DPWs samples to remove dirt and dust, the cleaned materials were crushed, and sieved to different particle sizes. Chemical pretreatment included the addition of $Ca(OH)_2$ to the sieved DPWs at concentrations ranged from 0.1 to 0.2g $Ca(OH)_2$/g TS of waste was carried out then the mixtures were autoclaved at 121°C for 20 min. The calcium will precipitate and removed as $CaCO_3$ by flushing the autoclaved mix with CO_2 [11]. Inoculum slurry was prepared by mixing 50g of cattle manure with 400 mL distilled water and was manually homogenized with glass rod.

2.3 Digesters set up

As the main objective of this study was the anaerobic co-digestion of date palm wastes (DPWs) for biogas production, four bench-scale digesters operated in batch mode were set up in duplicate as given in Table 1. The digesters were of 500-mL Pyrex borosilicate heatproof code glass bottles. The components of each digester were maintained at 1:10 which is equivalent to 40 g solid waste material: 400 mL (inoculum slurry or distilled water). Each digester was tightly plugged with rubber stopper contains 2 holes each of 4mm diameter through which a piece of glass tube was submersed into the digester and the other end of the glass tube was connected with rubber tube for the produced biogas transfer to the gas measuring apparatus. The rubber stoppers were tightly wrapped with parafilm to prevent any release of the produced gas. Digesters were immersed in a thermostatic water bath to maintain the required temperature conditions. Manual shaking of digesters were performed daily to insure that substrate molecules and bacterial come into close. Sodium bicarbonate ($NaHCO_3$) was used for pH adjustment and phenolphthalein was used for coloring water in the displacement bottle. The digesters were flushed with nitrogen for 10 min to provide anaerobic environment conditions.

Table 1. Digesters with waste setup material and temperature condition

Digester No.	Waste materials mix in digester	Temperature condition
1	Pretreated waste inoculated with cattle manure	Mesophilic (38ºC)
2	Pretreated waste with distilled water	
3	Untreated waste inoculated with cattle manure	
4	Pretreated waste inoculated with cattle manure	Thermophilic (55ºC)

2.4 Methods of analysis

The measurement of total solids (TS) and volatile solids (VS) were carried out in triplicate according to the procedure outlined in the *standard methods* [12]. pH was measured using pH meter (Model: WTW, Inolab 720). The recovered biogas was measured by three approaches; the manometer which is a simple apparatus consisted of glass U-tube shape with 10mm internal diameter filled with potassium hydroxide solution. The U-tube hitched with tap to adjust the level of solution with atmospheric pressure after CO_2 removal. The tube was provided with two ports, one for a biogas injection, and the other for gas outlet after removal of CO_2. The released gas was fractioned in a percentages (i.e. methane and CO_2 percentages) using the 4% potassium hydroxide. All measurements were carried out at room temperature and atmospheric pressure. The volume of gases was recalculated for standard temperature and pressure (STP: 0^oC and 1 bar) according to Hansen et al. [13]. The other gas measuring approach is the water displacement method in which the gases were first passed through an airtight washing bottle containing 1 molar sodium hydroxide solution in order to eliminate the carbon dioxide. Then the remaining methane passed to a 500-ml glass container; displacing the water which overflowed into a measuring cylinder. The volume of displaced colored water represents the volume of produced methane. Gas chromatography was used to determine the major components of the produced biogas.

2.5 Soil conditioning with digestate

To examine the overall validity of the selected treatment approach, the digestate resulted from the anaerobic digestion process was used for soil conditioning. Cress seeds were selected for this test. The seeds were planted in a digestate-conditioned soil contained in suitable pots. The pots were irrigated and observed on a daily basis for a period of one week.

3. Results and discussion

In order to determine the best conditions for maximum biogas production from DPWs material, the effect of key parameters including inoculum addition, chemical pretreatment of the digestive waste materials, and temperature were carefully considered in this study.

3.1 Effect of inoculum addition

This part of work was carried out to study the effect of inoculum on biogas production. The biogas production in digesters No. 1 and 2 for pretreated DPWs with inoculum and pretreated DPWs without inoculum respectively was monitored for 117 day. The profiles of biogas production are given in Figures 1-3. Results of the specific biogas production revealed that the use of inoculum improved the co-digestion process and anaerobic biodegradation of waste materials (Table 2). The increase of biogas production associated with the inoculum addition is significantly related to the increase of active microorganism since the cattle manure is a rich source for bacteria. However, the existence of cellulose digestive bacteria could be another potential assumption for the increase of biogas generation rates. This type of bacteria is capable to attack the tight association between lignin and cellulose bond. These results are in a good agreement with the previously outlined findings for biogas production from anaerobic digestion of cattle manure as a substrate [14]. They found out that rumen fluid inoculum increased the biogas production rate two to three times compared to the substrate without rumen fluid.

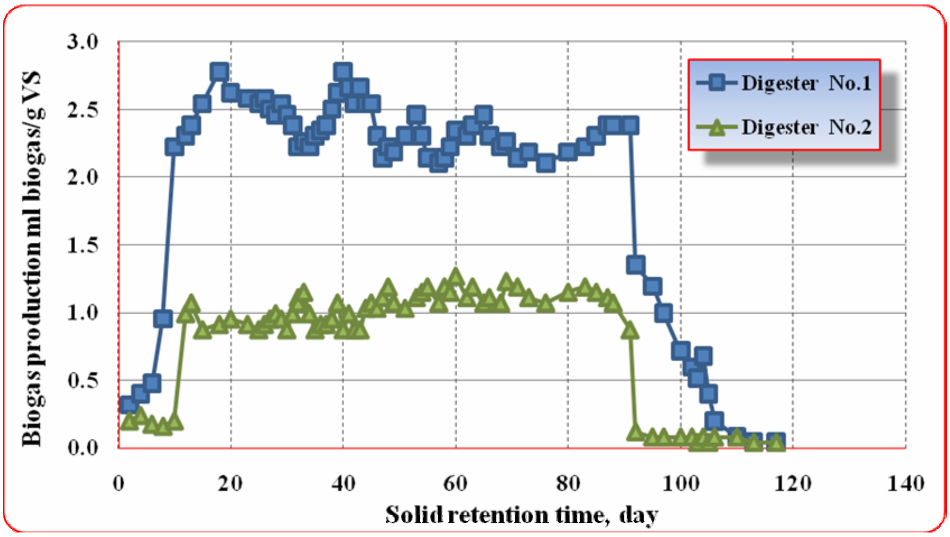

Figure 1. Biogas production profile for digesters No.1 and 2

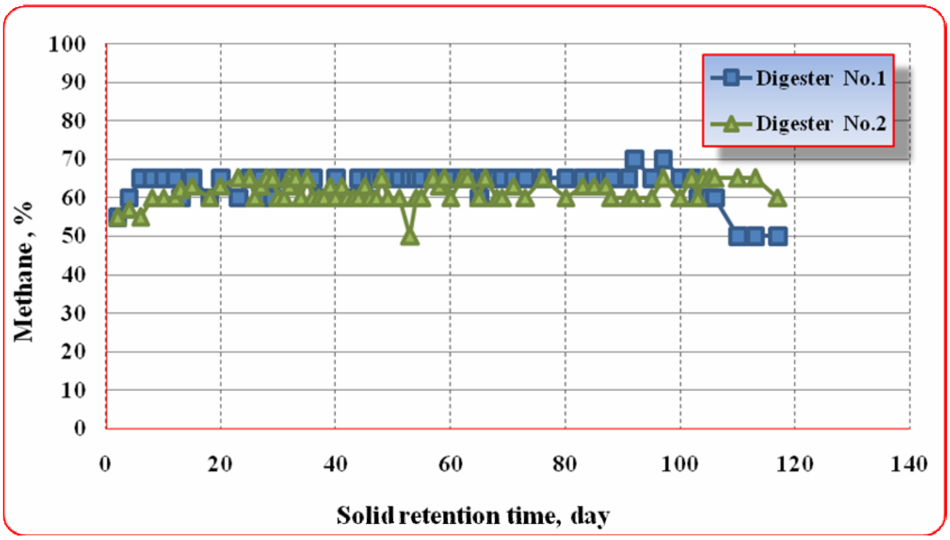

Figure 2. Percentages of CH_4 production for digesters No.1 and 2

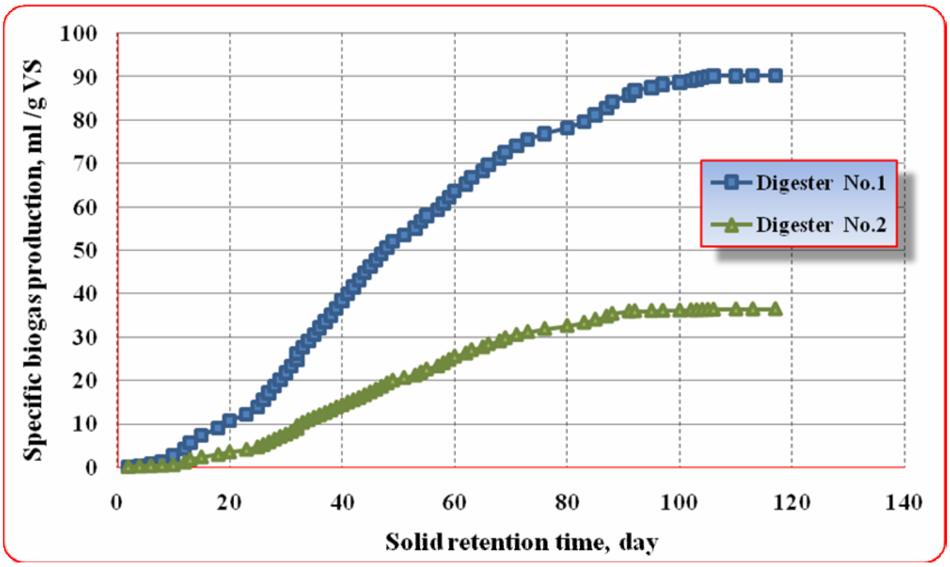

Figure 3. Specific cumulative biogas production profiles for digesters No. 1 and 2

Table 2. Effect of inoculum addition on biogas production

Digester No.	Inoculum	Maximum specific biogas production (mL/g VS)	Maximum specific CH_4 production (mL/g VS)	Biogas Increase (%)
1	Applicable	141.667 ± 8.1	90.381	
2	NA*	59.103 ± 2.4	36.493	139.7

** Not applicable*

3.2 Effect of chemical treatment

This section of work was devoted to investigate the effect of chemical pretreatment of DPWs on biogas production. The profiles of biogas production in digesters No. 1 and 3 for pretreated inoculated DPWs and untreated inoculated DPWs, respectively are given in Figures 4-6. These profiles indicate that the effect of alkaline pretreatment of DPWs was significant with respect to the enhancement of co-digestion process and the subsequent biogas production (Table 3). However, anaerobic digestion of lignocellulosic materials is a challenge because of the complex, rigid, and fibrous structure of these matters which under anaerobic conditions poorly degrades. Abdulkarim [15] reported that the addition of alkaline buffer based on total solid contents increased the biodegradability of the organic fraction of solid waste.

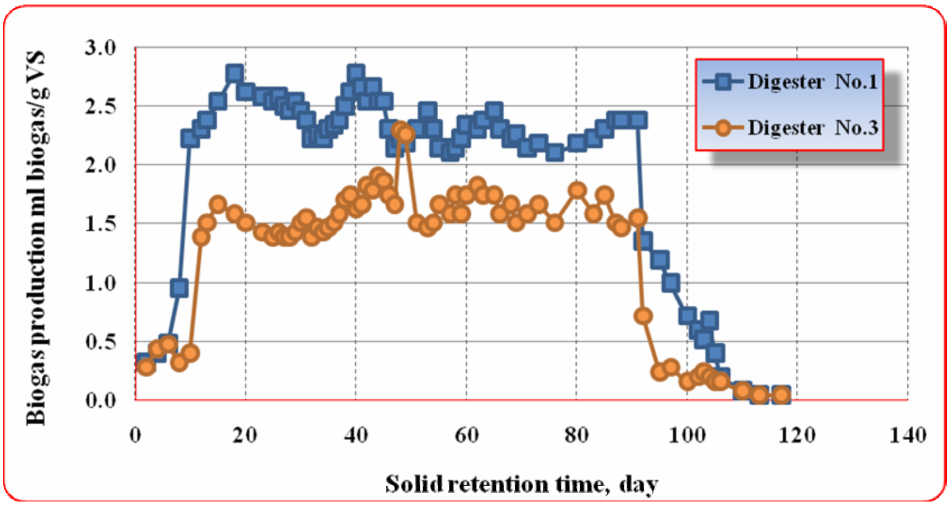

Figure 4. Biogas production profile for digesters No.1 and 3

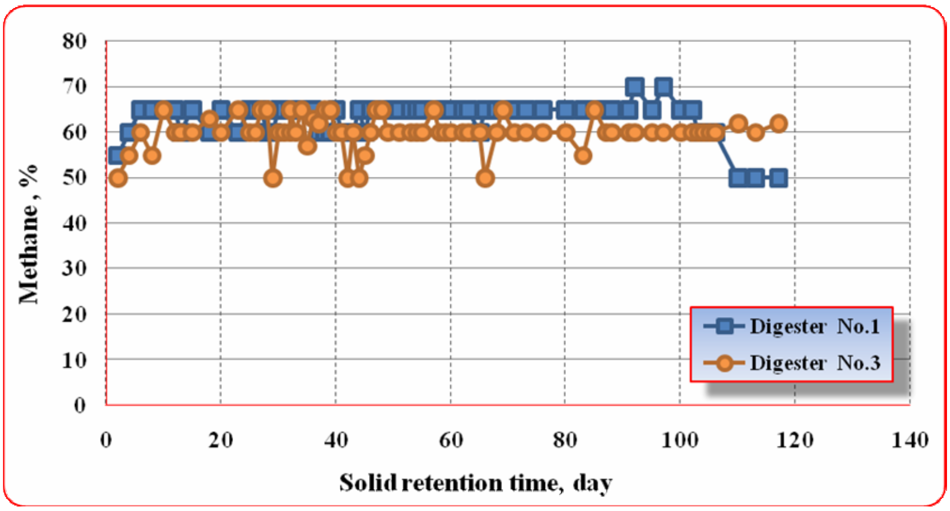

Figure 5. Percentages of CH_4 production digesters No.1 and 3

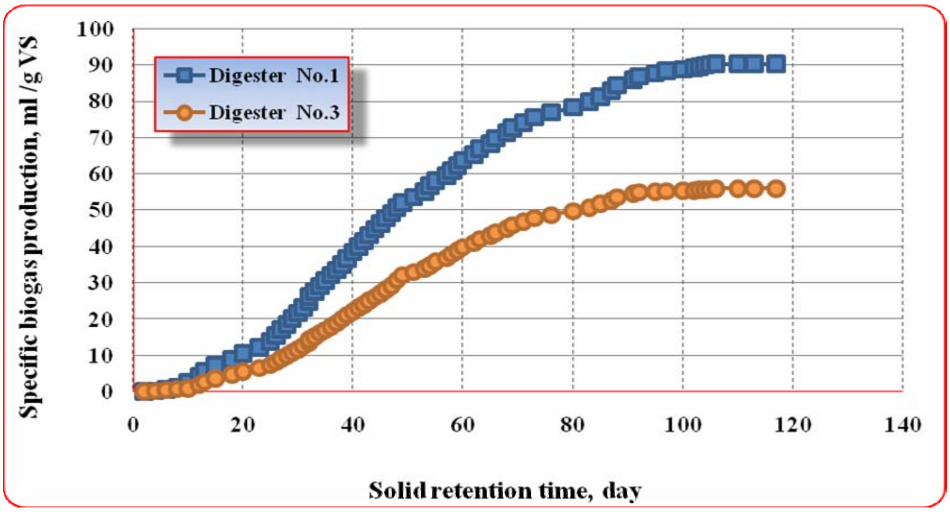

Figure 6. Specific and cumulative CH_4 production profiles for digesters No. 1 and 3

Table 3. Effect of pretreatment process of DPWs on biogas production

Digester No.	Pretreatment	Maximum specific biogas production (mL/g VS)	Maximum specific CH_4 Production (mL/g VS)	Biogas increase (%)
1	Applicable	141.667 ± 8.1	90.381	51.92
3	NA*	93.254 ± 4.2	56.107	

** Not applicable*

3.3 Influence of temperature

Results revealed a significant effect of temperature on biogas production. This is due to the fact that temperature is a very important operational parameter in anaerobic digestion processes. As given in Figure 7, the biogas recovery at thermophilic conditions was relatively higher than at mesophilic conditions. Table 4 summarizes the effect of temperature condition on the specific biogas production during 90 days-period observation indicating that biogas production at thermophilic conditions exceeds its production at mesophilic conditions by 92%. In conclusion, biogas yield with respect to methane content produced at thermophilic conditions is more favorable than its quality produced at mesophilic temperature range in this study. These observations are in a good agreement with the previously reported data regarding the biogas production at mesophilic and thermophilic conditions. Vindis et al. [16] reported a decrease in solid retention time and increase in biogas production from anaerobic digestion of maize silage under thermophilic conditions. Achu & Liu [17] realized higher biogas productivity under thermophilic conditions.

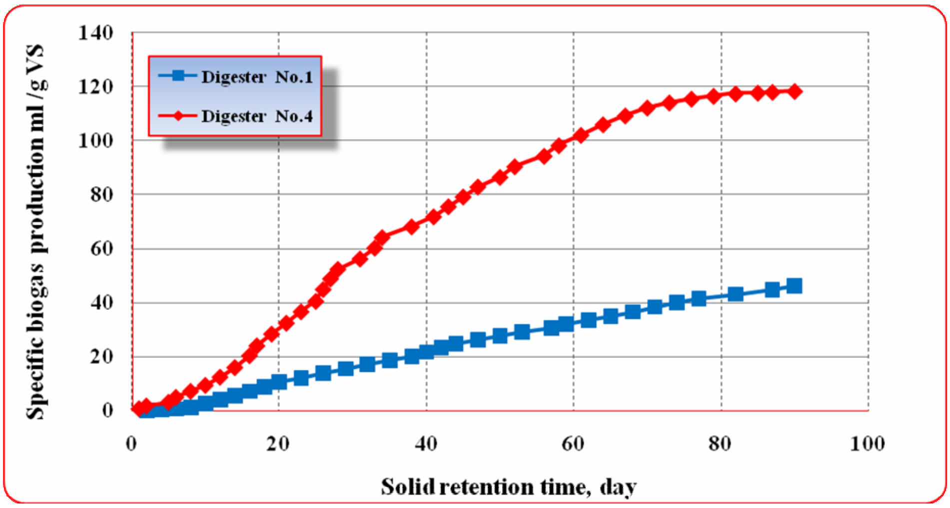

Figure 7. Specific and cumulative biogas production profiles in digesters No. 1 and 4

Table 4. Effect of temperature on the specific biogas production from pretreated inoculated DPWs

Digester No.	Temperature condition	Specific biogas production (mL/g VS)	Specific CH_4 production (mL/g VS)
1	Mesophilic	134.880	85.966
4	Thermophilic	166.468	118.389

3.4 Kinetic model

Biogas production rate in batch condition is corresponding to specific growth rate of methanogenic bacteria in the bio-digester. Accordingly, the predicted biogas production rate will obey Modified Gompertz Model [18] as follows:

$$G_{(t)}=G_0.\exp\{-\exp[((R_{max}.e)/G_0)(\lambda-t)+1]\} \quad (1)$$

where: $G_{(t)}$ = the cumulative biogas yield at a digestion time (mL/g VS), G_0 = the biogas potential of the substrate (mL/g VS), R_{max} = maximum methane production rate (mL/g VS-d), λ = lag phase (day) t = time (day), e = exp (1) = 2.7183.

A nonlinear least-square regression analysis was performed using SPSS [IBM SPSS statistics 18 (2009)] to determine λ, Rmax, and the predicted biogas and methane yield (Table 5). Plots of the measured and predicted values of biogas production are given in Figures 8-10. It is well observed that the predicted values of biogas production using modified Gompertz model is well fitted with the measured values. Results of this section are in a good agreement with the previously outlined findings. Kafle et al. [8] reported that the measured values of biogas produced from the bio-digestion of fish waste are well fitted with the predicted values using modified Gompertz model. Budiyono et al. [14] proved that the measured values of biogas produced from the digestion of cattle manure in batch mode are well fitted with the predicted data obtained by modified Gompertz model.

Table 5. Results of a kinetic study using Gompertz model at mesophilic conditions after 90 days

Digester No.	$G_{(t)}$ 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)	$G_{(t)}$ model (mL CH_4/g VS)	
1	85.967	15.407	1.597	90.381	83.860	0.985
2	35.969	19.172	0.687	36.493	34.060	0.979
3	54.604	18.181	1.069	56.107	52.530	0.986

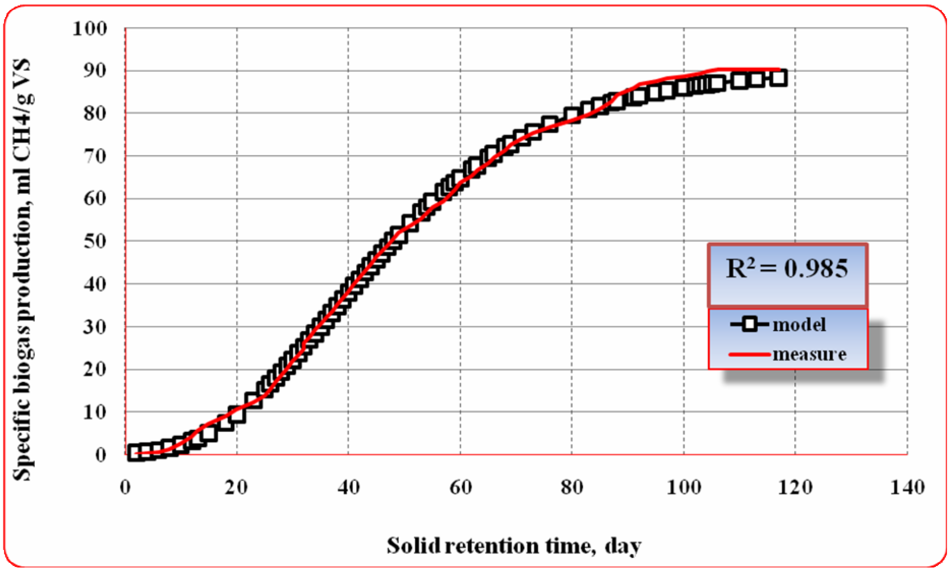

Figure 8. Measured and predicted results for biogas production from pretreated inoculated DPW

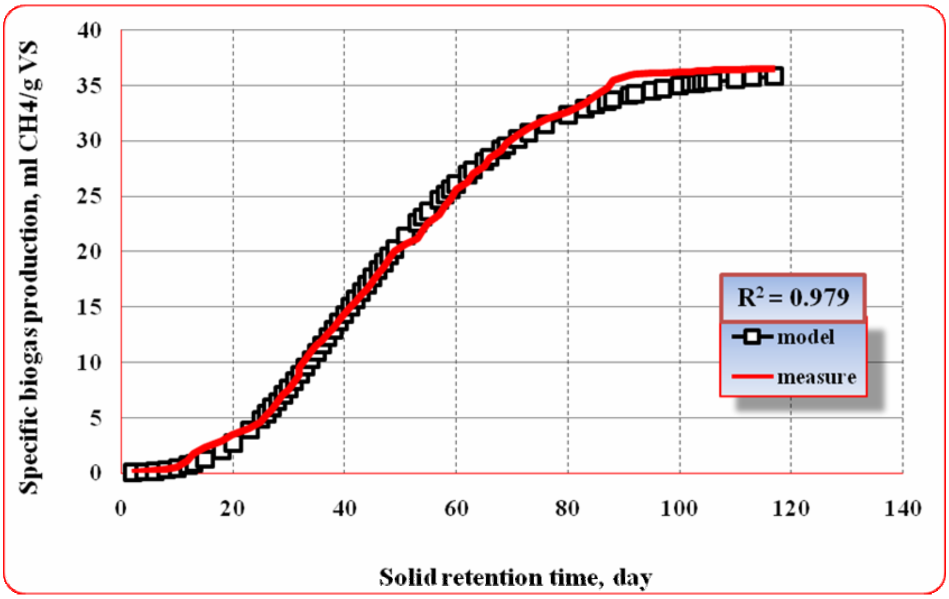

Figure 9. Measured and predicted results for biogas production from pretreated DPW

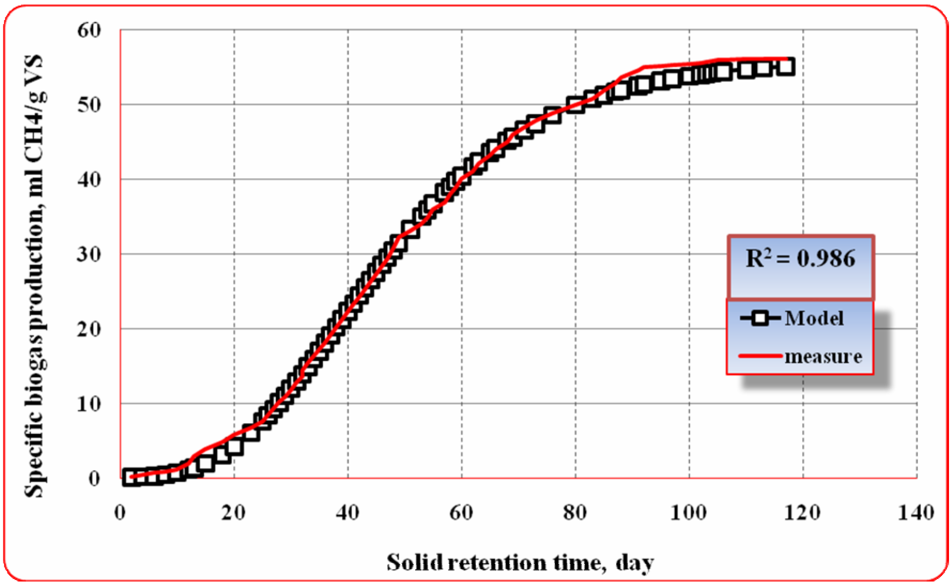

Figure 10. Measured and predicted results for biogas production from untreated inoculated DPW

3.5 Soil fertilization with residual digestates

The results of this part of work demonstrated that the selected process is a potential approach to treat the digestate resulted from the digestion process of DPWs. Figure 11 presents the growth progress of cress seeds after one week observation period. A healthy favorable growth of the planted Cress seeds was observed indicating that this approach is potential method to treat residues of digestive process.

4. Conclusion

This study was devoted to investigate the potential of anaerobic co-digestion for biogas production using abundantly available date palm waste materials of no economic value as the substrate. The experimental work demonstrated that the volume of produced biogas significantly affected by inoculum addition, pretreatment of waste materials, temperature conditions. The ultimate biogas yield from co-digesting of inoculated DPWs was estimated to be 141.667 ± 8.1mL/g VS, whereby without inoculation it was 59.103 ± 2.4 mL/g VS. Maximum biogas production from co-digestion of alkaline pretreated DPWs was estimated to be 141.667 ± 8.1 mL/g VS, whereby, it was 93.254 ± 4.2 mL/g VS for untreated DPWs. The kinetic of bio-digestion process was well described by Modified Gompertz Model and the experimental and predicted values of biogas production were fitted well with correlation coefficient values > 0.96 suggesting favorable conditions of the process.

Figure 11. Growth observations for the planted cress seeds after one week, pot (A) is for non-conditioned soil, and pot (B) for digestate-conditioned soil

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