Optimization of Anaerobic Co-Digestion of Cattle Manure and Anaerobic Biological Wastewater Treatment Sludge

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Abstract
In this study; effect of pH and amount of anaerobic biological treatment sludge (ABTS) on biogas production rate was investigated by co-digestion of cattle manure with ABTS. Laboratory experiments were performed in batch tank reactors at mesophilic conditions using Box-Wilson experimental design method. Temperature and total solid concentration was kept constant. The amount of ABTS and pH were varied between 50–150 g and 6.8–7.8 respectively. Statistical analyses were carried out by the use of Design Expert Trial 7.0.0 program. Maximum biogas production rate obtained at a pH value of 7.28 and the amount of anaerobic biological treatment sludge of 108.17 gram was 1.805 mL/day.g.

Keywords
Biogas
Co-digestion
Cattle manure
Anaerobic biological treatment sludge
Optimization
Statistical method

1. INTRODUCTION

Anaerobic digestion (AD) is a technology widely used for treatment of organic waste for biogas production [Budiyano, 2010]. Natural degradation of manure, leading to emissions of CH4 during storage, is very undesirable because of the global warming effects resulting from the release of greenhouse gases [Moller, 2004]. However, by controlled anaerobic digestion of animal manure capturing the biogas, the CH4 emissions during storage of manure can be reduced and the energy from manure can be used to substitute fossil fuels, as a CO2-free energy source [Moller, 2004]. The anaerobic fermentation of manure for biogas production does not reduce its value as a fertilizer supplement, since nitrogen and other useful substances remain in the treated sludge [Budiyano, 2010].

The anaerobic digestion of materials to biogas is basically a two stage process. In the first stage, acidogens bacteria break down the complex substances in to simpler soluble organic compounds such as volatile organic acids. At the second stage, methanogens convert these acids in to methane and carbon dioxide [Ghaly and Damkumar, 1999]. Because biogas production is bacterial process; temperature, pH, particle size, agitation, carbon/nitrogen ratio, total solid percent etc. are very important parameters for the efficiency of the process [Kashyap et. al,2003; Demirci and Türkavcı, 2001; Gökçay, 2002; Tanyolaç et.al, 1992; Dönmez, 1981; Erarslan, 1981; Santosh et. al, 2004].

Anaerobic treatment performance is adversely affected by slight pH changes in their optimum values. Methanogens are much more susceptible to pH variations than other microorganisms. Anaerobes can be grouped into two separate pH groups: acidogens and methanogens. The optimum pH is 5.5–6.5 for acidogens and 7.8–8.2 for the methanogens. Optimum pH for the combined cultures ranges from 6.8 to 7.4 with neutral pH being the ideal value. Since methanogenesis is considered to be the rate-limiting step, the maintenance of the pH value of the reactor close to the neutral value is important. Acidogens are
significantly less sensitive to low or high pH values and acid fermentation will predominate over methanogenesis, which may result in the decay of the reactor contents [Khanal, 2008].

Although particle size is not as important as temperature or pH of the digester contents, it still exerts a certain influence on gas production. The size of the feedstock should not be too large otherwise it clogs the digester and makes the bacterial digestion highly difficult. Smaller particles on the other hand would provide large surface area for the adsorption of the substrate which results in increased bacterial activity and hence increased biogas production [Santosh et.al, 2004].

Solid concentration is another important parameter for biogas production [Ekinci and Mutlu, 2009]. According to the literature the best concentration of the cattle manure total solids is 7-9% for conventional biogas plants [Santosh et.al, 2004]. Baserja reports that the anaerobic digestion of cattle dung is unstable below total solids concentration of 7% while solids concentration above 10% causes overloading in the reactor [Baserja, 1984].

Sanchez et.al. investigated the effect of temperature and pH on the kinetics of methane production and organic nitrogen and phosphorus degradation in the anaerobic digestion process of cattle manure using two laboratory scale batch complete-mix reactors operating at 35°C (mesophilic temperature), and two laboratory scale batch complete- mix reactors operating at 60°C (thermophilic temperature). The initial pH of the waste was 7.6. The pH value of employed at the selected temperatures was 7.0 and the apparent kinetic constant of the biomethanization process was observed to increase by 2.3 times when the initial pH of the influent was increased from 7.0 to 7.6 at mesophilic temperature. The values found at thermophilic temperature were similar [Sanchez et.al, 2000].

Bodiyan et. al. studied the use of rumen fluid as inoculums for biogas production from cattle manure. They used seven laboratory 400 mL biogas digesters in batch operation mode. 100 g fresh cattle manure was mixed with 50 g rumen fluid and different volumes of tap water obtaining six different total solid contents between 2.64% – 12.30%. The control reactor total solid was 18.40%. The best performance was obtained with 7.4 and 9.2% of total solid with a biogas production of 184.09 and 186.28 mL/gVS, respectively after 90 days. These results suggest that, based on total solid content effects to biogas yield; rumen fluid inoculums exhibit the similar effect with other inoculums [Bodiyo et. al, 2010].

In this study; effect of pH and amount of anaerobic biological treatment sludge (ABTS) on biogas production rate was investigated by co-digestion cattle manure with ABTS by using laboratory scale batch reactors. Temperature and total solid percentage was kept constant and their values were selected according to the literature. Experiments were designed by Box-Wilson method and evaluated by Design Expert Trial 7.0.0 program to find the optimum results. Effect of pH value is selected because it is one of the important factors for producing biogas. To the best of our knowledge there is not any study in the literature about co-digestion cattle manure with ABTS. In addition to this, in the literature biogas production conditions are not modeled and optimized by using statistical method. Therefore, the main objective of this study is developing a rate model for biogas production for cattle manure and ABTS co-digestion system.

2. MATERIAL AND METHODS

2.1. Materials

Cattle manure used in these experiments was obtained from Atatürk Orman Çiftliği (AOC) Dairy Plant. In AOC 1 ton of cattle food used for feeding animals was consisted about 400 kg of wheat, 250 kg barley, 100 kg of maize, 200 kg of sunflower meal and the remaining 50 kg additives (mineral powder, vitamins, salt, etc.). It was also reported that the amount of biogas produced was dependent also upon the particle size [Santosh et.al, 2004]. The material was grounded for 3 minutes with a blender in order to reduce the particles to appropriate dimensions. ABTS used in the experiments was obtained from ASKİ Ankara Central Wastewater Treatment Plant anaerobic treatment tanks. The temperature of the tanks was kept constant at 35°C which was the most suitable temperature for mesophilic conditions. The most important factors mentioned in the literature were Nitrogen/Phosphorus ratio, total solid percentage, COD value
Therefore, cattle manure and ABTS were analyzed before the experiment by using standard methods [APHA, 1998] and the results were tabulated in Table 1.

**Table 1. Characteristics of ABTS and cattle manure used in the experiments**

<table>
<thead>
<tr>
<th></th>
<th>Cattle Manure</th>
<th>ABTS</th>
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<tbody>
<tr>
<td>% Total Solid</td>
<td>19.68%</td>
<td>8.68%</td>
</tr>
<tr>
<td>% Volatile Solid*</td>
<td>89.79%</td>
<td>38.83%</td>
</tr>
<tr>
<td>% Fixed Solid*</td>
<td>10.21%</td>
<td>61.17%</td>
</tr>
<tr>
<td>% Carbon*</td>
<td>30.22%</td>
<td>19.94%</td>
</tr>
<tr>
<td>% Nitrogen*</td>
<td>1.63%</td>
<td>-</td>
</tr>
<tr>
<td>% Phosphorus*</td>
<td>0.65%</td>
<td>-</td>
</tr>
<tr>
<td>Amount of Acetic Acid</td>
<td>150.24</td>
<td>193.34</td>
</tr>
<tr>
<td>(mg/L g solid)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>COD (mg O₂/L g solid)</td>
<td>412.48</td>
<td>270.72</td>
</tr>
<tr>
<td>COD:N:P</td>
<td>635:2.51:1</td>
<td></td>
</tr>
</tbody>
</table>

*Analyze results were based on total solid.

### 2.2. Experimental Design

Box-Wilson experimental design method was used to develop statistical model for biogas production rate. The independent variables selected were pH ($X_1$) and the amount of ABTS ($X_2$) while the Biogas production rate ($Y$, mL/day. g manure) was chosen as dependent variable. ABTS was kept at pH 7.30 at ASKİ Ankara Central Wastewater Treatment Plant anaerobic treatment tanks. This is an appropriate value according to the literature, so the center point for Box-Wilson method was determined to be pH value of 7.30 and its step size was chosen as 0.50. Another factor which effects the biogas production is the amount of ABTS. When the ABTS amount was increased up to a limit, biogas production was also observed to reach to its limits. When this limit was exceeded beyond the total organic load, the biogas production was observed to decrease. Based on these findings the center point and step size were selected as 100 grams and 50 grams respectively. Box - Wilson method of experimental design was carried out under the experimental conditions listed in Table 2. In Box-Wilson experimental plan, 14 experiments with six replicates at the center point and four α points ($α = 1.414$) were employed. There were six replicates made at the center point (0,0) to measure the error involved in the experiment.

**Table 2. Coded and real values of independent variables in the experimental plan**

<table>
<thead>
<tr>
<th></th>
<th>Coded Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Real Values</td>
<td>-1.414</td>
</tr>
<tr>
<td>$X_1$ (pH)</td>
<td>6.59</td>
</tr>
<tr>
<td>$X_2$ (ABTS amount, g)</td>
<td>29.30</td>
</tr>
</tbody>
</table>

### 2.3. Experimental Procedure
The cattle manure and water were placed in fourteen 250 mL ABTS glass batch reactors according to the specifications of Box-Wilson method (Table 3). The total content and the solid percentage in each reactor were 250 grams and 8.68%, respectively. The pH values were adjusted by the use of 0.1 M–5 M H₂SO₄ and 0.1 M–5 M NaOH solutions in the reactors according to the conditions specified by Box-Wilson method. Reactors were mixed thoroughly mixed before the addition of water at 35°C. Biogas produced by the microorganisms during the anaerobic digestion was collected in graduated burettes which contain concentrated sulfuric acid and saturated sodium sulphate solution [APHA, 1998] (Figure 1). In the experiment, reactors were agitated 10 seconds per hour to obtain uniform mixture and to release produced biogas. The effects of pH (6.8–7.8) and amount of ABTS (50–150 g) upon biogas gas production rate were elucidated. The experimental period was chosen as 10 days.

The regression analysis of the experimental results and the estimation of the coefficients of the regression equation were carried out by the use of The Design-Expert Trial 7.0.0 program. The statistical significance of the model was determined by Fisher’s F-test. The shape of the curve generated by the model was predicted by the canonical analysis. A two-dimensional graphical representation of the system behavior known as the response surface was used to describe individual and cumulative effects of the variables as well as the mutual interactions between the independent and the dependent variables [Li et.al, 2007].

3. RESULTS AND DISCUSSION

The major aim of this study was to develop a model for biogas production and determine its optimum conditions for cattle manure and ABTS co-digestion. The volume of the biogas produced was measured as shown in Figure 1. The daily amount of gas produced was assumed to be the same throughout the study. Therefore the gas production rate was calculated by dividing total gas volume obtained from each reactor during the experimental period (10 days) and mass of manure remained in it. The experimental data are tabulated in Table 3.

Table 3. Experimental design and experimental gas production rates
Using Design Expert Trial 7.0.0 program, biogas production rate (Y) were expressed as a function of independent variables; pH ($X_1$) and amount of ABTS ($X_2$) as given below:

$$Y \text{ (mL/g manure. day) } = 1.76 - 0.18X_1 + 0.35X_2 + 0.12X_1X_2 - 0.34X_1^2 - 0.54X_2^2$$

The parameter value (p-value) for all parameters was below 0.05, so all parameters in this equation was significant. Model equation determined by analysis of variance (ANOVA) and its regression coefficient ($R^2$) was calculated as 0.9987. It can be seen from this high regression coefficient that the model fits the system well. By using this model equation, all the biogas production rates were calculated. Figure 2. shows the observed biogas production rates versus those calculated from model equation. The figure proves that calculated value of the biogas production rate was in good agreement with the value observed in the experiment. This model gives similar biogas production rate similar to the experimental procedure.

The 3D response surface plots described by the regression model were drawn to illustrate the effects of the independent variables and the interactive effects of each independent variable on the response variables. The shape of the corresponding contour plots indicates whether the mutual interactions between the independent variables are significant or not. The elliptical nature of the contour plots reveals that the interactions between the independent variables are significant. From the 3D response surface plots and the corresponding contour plots, the optimal values of the independent variables could be determined and the interaction between each independent variable’s pair can be easily distinguished [Li, 2007]. Figure 3 illustrates the 3D plot and its corresponding contour plot which shows the effects of ABTS amount and pH on the biogas production rate.

Figure 2. Observed biogas production rate versus predicted biogas production rate

Figure 3. Response surface plot and contour plot of the combined effects of ABTS amount and pH on the biogas production rate

By the analysis of Figure 3 using the Design-Expert software, the maximum biogas daily production rate was found to be 1.805 mL/day.g. Under these conditions the optimum pH and amount of ABTS were 7.28 and 108.17 gram respectively. According to Figure 3, minimum biogas production rate was observed at high pH and low ABTS values. The value of the biogas production rate increased up to optimum values of independent variables and then decreased.

Optimum value of pH (7.28) is in agreement with the literature [Khanal, 2008]. In addition to this, most of the microorganisms were come from ABTS and they are at pH value 7.30 similar to the value in the tank. Sanchez also reported that, biomethanation is increased when pH increased from 7.0 to wastes’ initial pH value of 7.6 [Sanchez et.al, 2000]. So, as expected optimum pH value is close to the value in the tank (from which ABTS was taken).
The production of biogas from the cattle manure only is a very slow process due to the fact that it does not contain enough microorganisms to carry out this process in an efficient manner. Therefore it is necessary to use ABTS with high microorganism content to increase the production up to limit. However, the solid content of ABTS is low and it decreases the total solid content of the digester. That is why the determination of the optimum ABTS amount in the reactor is of paramount importance. If optimum value is exceeded total solid content is decreased, so biogas production rate is low.

4. CONCLUSION

The effect of pH and amount of ABTS on biogas production rate was investigated by co-digestion of cattle manure with ABTS by performing a series laboratory experiment designed by Box-Wilson experimental design method. The most important finding from this research is that the best performance for biogas generation was the digester with pH value of 7.28 and the amount of anaerobic biological treatment sludge of 108.17 gram was 1.805 mL/day.g after 10 days observation. This result shows that in order to shorten the start up period and to increase the gas production efficiency, the ABTS addition to the cattle manure with certain amounts will be suitable for the operation of a biogas-producing biogas plant from cattle manure.

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REFERENCES


