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Biogas Production Potential of Rose Oil Processing Wastes and Quail Manure in Turkiye: Assessment by Hohenheim Batch Test

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Abstract: Biological conversion of biomass to methane has received increasing attention in recent years. In the present study anaerobic digestion of rose oil processing wastes (ROPW) and quail manure, which are unique for only a few regions in the world, was investigated. Since the rose oil production is a water distillation process, thus rose oil processing wastes have a high moisture content (90% w.b.) which is naturally suitable for biogas generation. For this purpose, methane generation potential of the ROPW was first determined by "Hohenheim Batch Test" procedure at fermentation temperature of 37 °C. In addition, total theoretical energy generation potential of a single stage continuous digestion, treating ROPW and quail manure were calculated based on experimental results. According to the results of this study, energy potential that could be produced from ROPW and quail manure was calculated as 3,15 GWh_{el}/year. Results indicate that the quality of the substrates with high methane yield is significantly important.
Key words: Biogas, anaerobic digestion, rose oil processing wastes, quail manure

INTRODUCTION

Biogas process, which is a method of recycling through anaerobic digestion of organic materials and an important source of renewable energy, is evaluated as the most convenient process for the utilization of wastes for the sustainable development. Biogas process results in a gas having methane content of 55-75% and 25-45% carbon dioxide as well as trace gases such as hydrogen sulphide and hydrogen (Angelidaki et al., 2003) and fermented manure. Table 1 presents the typical composition of biogas.

Table 1. Typical composition of biogas

Methane, CH_4	55–75%
Carbon dioxide, CO_2	25–45%
Carbon monoxide, CO	0–0.3%
Nitrogen, N_2	1–5%
Hydrogen, H_2	0–3%
Hydrogen sulphide, H_2S	0.1–0.5%
Oxygen, O_2	Traces %

Every agro-industrial system produces a wide variety of organic feedstock, such as lignocellulosic materials, crop residues, vegetable oils, animal fats, protein-rich waste, pre-digested wastewater sludges, animal slurries and manures, waste paper, household waste, etc., as by-products. Biogas production from these wastes both reduces the adverse effects of waste on environment and generate electric and heat. Biogas production by anaerobic digestion (AD) of biomass can help in partially replacing fossil fuel-derived energy and thereby in reducing environmental impact by providing a clean and diffused fuel from renewable feedstock. Anaerobic co-digestion of wastes may be a good integrated solution by the means of the disposal problem of such waste materials and the problem of improving the quality of soils can simultaneously be solved in an environment-friendly way (INTUSER, 2007).

Commercially cultivated rose (R. damascena Mill.), one of the most important commercial essential oil sources, is a perennial bush rose, much branched, thorny, with large, highly scented, roseate flowers. The main rose growing areas are Bulgaria, Southern Russia, Turkey and Morocco where the environment is similar

(Weiss, 1997). Although there are 25 rose cultivars in Turkey, R. damascena is cultivated for the production of essential oil (Konur, 1990; Ercisli, 2004). The climatic properties of the Isparta and Burdur region in Turkey such as temperature, humidity, cloudiness, and precipitation in the flowering season (May and June) have important contribution in obtaining rose oils with high quality in accordance with the world standards (Ağaoğlu et al., 2000). Three main products obtained from rose plants after processing are: oil, concrete, and absolute, which vary in their characteristics according to origin (Kaur et al., 2007; Konur, 1990).

Rose oil is steam distilled from the flowers of the R. damascena. Rose oils, extracts and derivates are among the most important of natural perfume ingredients used in perfumery (Kurkcuoglu, 1988). The rose petals generally contain very little essential oil in comparison with the other essential oil crops. One kg of rose oil can be obtained from about 3000 kg of rose petals (Baser, 1992), and so oil content is only about 0.03%. Due to processing rose for oil, 27000 tons of rose oil processing waste per year is produced. Although official complaints from neighborhood of processing facilities are recorded, no waste management practices are applied. Considering the fact that higher heating energy demand of the rose oil distillation process, taking into account of generating biogas from waste could be a "double" solution of both heating applications and waste management.

A number of studies concerning anaerobic digestion of wastes such as grape marcs, maize drying up residues and tomato skins and seeds (Dinuccio et al., 2010); leather fleshing wastes (Shanmuga and Horan, 2009); cotton wastes (Isci and Demirer, 2007), banana waste (Caballero-Arzapalo et al., 2010); sugar cane residues (Salomon and Silva Lora, 2009); sisal pulp and coffee residues (Kivaisi and Rubindamayugi, 1996); olive mill waste water (Gelegenis et al. 2007); olive mill effluents (Angelidaki et al., 2000) were conducted. However, studies on anaerobic digestion of rose oil processing wastes are limited and much less are known of the methane potential of this waste.

Coturnix japonica is a terrestrial bird, inhabits grassy areas, is indigenous to Japan, China, Korea and Indochina and was introduced into Hawaii in 1921 (Mills et al., 1997). Similarly quail rising for both meat and egg production was started in late 70s in Turkiye. After that period Alarslan (2006) implies that the interest on quail production decreased by lower production costs of layers. But author informs that the breeding of Japanese quail (Coturnix cot. Japonica) is given in high interest which resulted in high capacity (over 20000 animals) farms in Turkiye.

The data on the methane potential of substrates can be used for design parameters for anaerobic digestion plants. This paper concentrates on the anaerobic digestion of rose oil processing wastes in the above mentioned region. Additionally, ın our comphrensive research on literature, we haven't found any results of biogas potential of quail manure. Therefore, we want to introduce the methane generation potential of quail manure in this paper.

This study aimed to determine the biogas generation potential of rose oil processing wastes and quail and cow manure separately which can be co-fermented with together. Methane potential of these substrates will be determined using the patented test method of Hohenheim Batch Test, simply known as HBT.

MATERIALS and METHOD

Materials

This study involved anaerobic digestion of rose oil processing wastes (ROPW), quail and cow manure separately. ROPW was collected from wild disposal pond of Gülbirlik Rose Oil Factory, Isparta, Turkiye. Substrate was air dried for 6 months. Fresh quail and cow manure were collected from Unterer Linderhof Research Farm at the University of Hohenheim.

Experimental Unit

This research study was conducted at the Biogas Research Laboratory of State Institute of Agricultural Engineering and Bioenergy, Hohenheim University Stuttgart. Figure 1 shows the patented Hohenheim Batch Test Unit. The system consists of 100 ml glass syringes with a 1/1 graduation as well as a capillary extension, gas chamber, aperture for gas analysis, and plunger. A gas-tight hose is put onto the capillary extension, which can be closed with the aid of a hose clamp. Lubricating grease, which is inert against anaerobic degradation, was used to seal the gap between plunger and the glass syringe. This method does not need an additional gas sampling tube. Between measurement periods, the biogas is collected

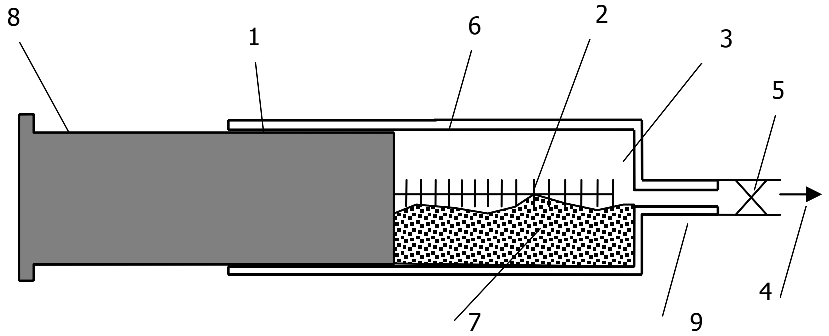

Figure 1. Test syringe of Hohenheim Batch Test. (1) Sliding and sealing agent, (2) Graduation 1/1 ml for volume determination, (3) Gas chamber, (4) Aperture for gas analysis, (5) Hose clamp/Glass tap, (6) Glass syringe, (7) Fermentation substrate, (8) Plunger, and (9) capillary extension

in the syringe sampler which also serves as the fermentation chamber. Gas loses via the connection hoses to a gas sampling tube are thus avoided (VDI 4630, 2006).

Glass syringes are placed for the mixing of the substrate in a motor-driven carrier circle, which have approximately up to 130 syringes can be put into. The entire unit is placed into an incubator, where the desired fermentation temperature can be chosen (Figure 2).

Experimental Procedure

Depending on the dry matter content, a representative sample was taken from approximately one kilogram of fresh mass. The sample was examined for the moisture and ash content. Then it was gently dried in a drying oven at 50 to 60 °C for 48 hours. Afterwards, the sample was grounded such that it was able to pass through a one-millimeter sieve. The processing of the sample allows the fermenter to be filled with a representative weighed-in quantity of 500 mg of test substrate.

For the experiment, approximately 30 ml of inoculated substrate was first put into the prepared flask, and the weighed-in quantity was determined with a precision of 1/100 gram. Subsequently, 500 mg of the test substrate was weighed in with a precision of 1/1000 gram using an analytical balance. With the aid of the greased stopper, syringe held in vertically and residual air was evacuated from the flask, and the latter was closed gas-tight by hose clamp. Then, the syringes were put into the rotating circle horizontally. Gas measurements were made by holding the syringe vertically and releasing the biogas produced.

The materials of ROPW, quail manure, cow manure, three forms of maize silages, which are reference materials (KF-Standard, Standard 600, and Maize silage 600), and inoculum with three replications were run for 35 days at mesophilic temperature of 37±2ºC for anaerobic digestion process. Three flasks were filled with pure inoculated substrate to determine the biogas generation of inoculum. In order to secure the results further, the reference substrates (3 forms of maize silage) were digested at the same time. The added inoculum was collected from the laboratory type digester operating at the same temperature.

During the course of the test, to ensure sufficient mixing of the fermentation material, test flasks were placed in a circle carrier which was also placed in the incubator. The carrier circle was turned with the speed of 10 rpm, thus mixing of the substrates was ensured. Figure 2 shows the schematic view of the carrier circle and the incubator.

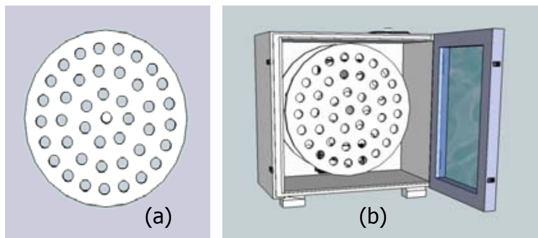

Figure 2. Schematic view of the (a) carrier circle and (b) circle in the incubator

The biogas quantity was measured by observation via 1 mm graduation of the flask. Biogas quantity readings were read off in 4 times per day (every 6th hours) for the first 6 days, and then reduced to 3, 2 and once per day for the rest of experiment.

The methane content of the biogas was analyzed by CH_4 analyzer only if there is 20 ml of gas produced in the flask.

The following equation was used to calculate the normal volume of the gas produced (VDI 4630, 2006).

$$V_0^{tr} = V \cdot \frac{(p - p_w) \cdot T_0}{p_0 \cdot T} \qquad (1)$$

Where;

V_0^{tr} : Volume of the dry gas in the normal state (ml_N),

V : Volume of the gas as read off (ml),

p : Pressure of the gas phase at the time of reading (hPa),

p_w : Vapour pressure of the water as a function of the temperature of the ambient air (hPa),

T_0 : Normal temperature (273 K),

p_0 : Normal pressure (1013 hPa),

T : Temperature of the fermentation gas of the ambient air (K)

Methane concentration of recorded biogas was calculated as follows (VDI 4630, 2006).

$$C_{CH_4}^{tr} = C_{CH_4}^{f} \cdot \frac{p}{(p - p_w)} \qquad (2)$$

Where,

$C_{CH_4}^{tr}$: methane concentration in the dry biogas (% by volume),

$C_{CH_4}^{f}$: methane concentration in the moist biogas (% by volume),

p : Pressure of the gas phase at the time of reading (hPa),

p_w : Vapour pressure of the water as a function of the temperature of the ambient (hPa).

Cumulative methane generation was calculated as follows by integration of methane generation over time (Ekinci, 2001).

$$M_{CH_4}(t) = M_{CH_4}(0) + \int_{t_1}^{t_2} M_{CH_4}(t)dt \qquad (3)$$

Where,

$M_{CH_4}(0)$: Methane generation (Nm^3 CH_4/kg ODM_{in}) at t=0,

$M_{CH_4}(t)$: Cumulative methane generation Nm^3 CH_4/kg ODM_{in},

t_2-t_1 : Sampling duration time between two sampling.

RESULTS and DISCUSSION

The volume of experimental units designed for biogas batch fermentation tests are, for example, 250 ml (Isci and Demirer, 2007), 0,4 l (Tosun et al., 2004) 2 l (Dinuccio et al., 2010;) 2,5 l (Mahanta et al., 2004), 3,73 l (Karim et al., 2005), and 10 l (Rajeshawari et al., 1998). Basic advantage of HBT is probably, the small dimension of the experimental test unit, consisting of about 130 syringes with a volume of 100 ml, which allows an accomplishment out of a large number of repetitions or parallel tests with a reduced spare requirement (Helffrich and Oecshner, 2003).

Since the produced biogas was relatively in small amounts, gas analyzer device should be able to measure composition of very little amounts of biogas (in this case CH_4 analyzer was calibrated to minimum 20 ml of biogas).

The quantity of the required substrate for each syringe was 0.5 g, thus sampling and sample preparation needed particular importance. It is important to note that the results of three forms of maize silages, which are reference materials (KF-Standard, Standard 600, and Maize silage 600) in this study were compared with the results of previous studies for the same materials at the same conditions in terms of mean biogas generation data for the accuracy and reliability of the experiment. This approach increases the consistency of the obtained data.

Cumulative methane productions of substrates of ROPW, quail manure, cow manure, three forms of maize silages (KF-Standard, Standard 600, and Maize silage 600) as function of time are given in Figure 2. All material showed peak at days of between 2 and 11. All fermenters used exhibited initially strong methane production, which slowly declined after approximately eleven days. The cumulative methane production of quail manure reached 0,26 Nm^3/kg ODM_{in} which is considerably close to the methane production of there maize silage reference materials (approximately 0,32 Nm^3/kg ODM_{in}). The cumulative methane production of ROPW and cow manure resulted lower values 0,136 and 0,115 $Nm^3/kg\ ODM_{in}$ respectively. The cumulative methane production obtained from quail manure was almost two times higher than that of ROPW. The relatively low biogas generation potential of ROPW (0,136 $m^3\ CH_4/kg$ ODM) was found to be lower than the results found by Tosun et al. (2004) which concerned on anaerobic digestion of rose residues, resulted with the value of 0,26 $m^3\ CH_4/kg$ ODM. The difference between two research findings could be due to the fact that the ROPW material was stored 6 months at uncontrolled environment for this study, which resulted in possible decomposition of ROPW material during the specified time.

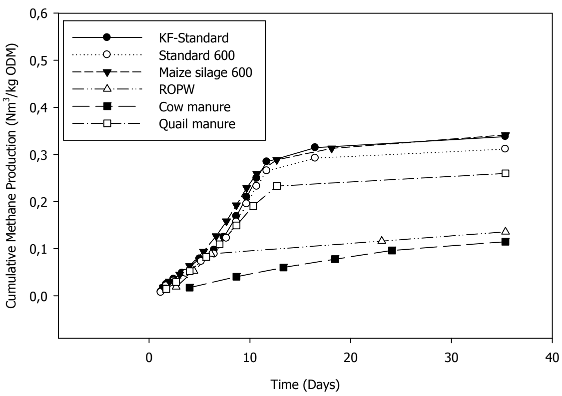

Figure 2. Cumulative methane production of substrates of ROPW, quail manure, cow manure, three forms of maize silages (KF-Standard, Standard 600, and Maize silage 600)

Figure 3 shows cumulative biogas and methane productions of substrates of ROPW, quail manure, cow manure, three forms of maize silages (KF-Standard, Standard 600, and Maize silage 600).

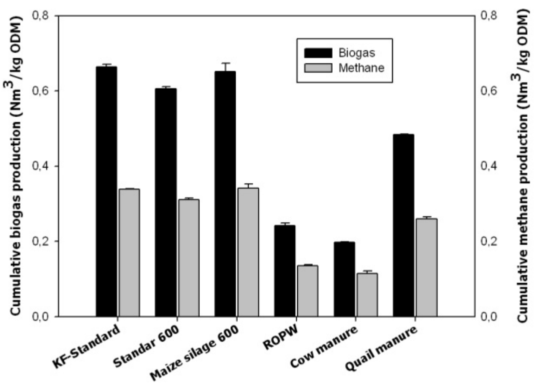

Figure 3. Cumulative biogas and methane production of substrates

The highest methane fraction in produced biogas was measured for ROPW material recorded as 56 %. For the other materials, the fraction of methane varied from 50 % to 53 %. As can be seen from Figure 3, the standard errors of biogas and methane productions of each substrate were averagely lower than 0,5 %. It can be concluded that the patented experimental unit yielded reliable results even though very small volume of the test syringe.

Energetic potential of the quail manure and ROPW was given in Table 2 considering biomass availability.

Total theoretical energetic potential of quail manure and ROPW was estimated as approximately 3 GWh_{el}/year as shown in Table 2. This is equivalent to approximately 10 000 tons of maize silage.

CONCLUSIONS

The followings were concluded from the study:

- Quail manure has a relatively high biogas potential with the value of 0,260 $Nm^3\ CH_4/kg\ ODM_i$.
- Rose oil processing wastes have methane potential of 0,136 $Nm^3\ CH_4/kg\ ODM_i$.
- HBT unit is useful and practical to determine the methane potential of organic substrates.

Table 2. Energetic potential of the quail manure and ROPW

Substrate	Biomass availability (t FM/Year)	Ton ODM/year	Energetic equivalent		
			(TJ /year)	GWh_{el}/year)[2]	(TES[3] /year)
ROPW	27000	3645	19.33	2.15	7,247
Quail manure	1506[1]	904	9.16	1.02	3,438

[1] Estimation based on data given by Alarslan, 1999

[2] Energy efficiency of combined heat and power unit is assumed to be %40 (Dinuccio et al., 2010)

[3] TES: Tons equivalent of maize silage. 1 GJ=3,371 TES.

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