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Study of the methanogenic potential of used grease and bleaching earth by conversion into biogas: The case of a wastewater treatment plant and a refinery

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ABSTRACT

The structure from which this study was carried out is a food processing company specializing in the transformation of crude palm oil into table oil. In order to comply with environmental standards when discharging the refinery's wastewater, it has its own wastewater treatment unit. The aim of this study is to contribute to environmental protection by recovering grease and bleaching earth through biogas production by anaerobic digestion. The characterization provided us with moisture, dry matter and volatile solids contents the values obtained are 32.31%, 67.69% and 97.57% respectively. In the same order, those for bleaching earth are 1.80%, 98.20% and 59.97%. The values obtained during the characterization of the bleaching earth are not conducive to good anaerobic digestion. To optimise biogas production from fat, we used inoculums such as cattle dung and broiler droppings. Biodegradability tests carried out with different proportions of substrates and inoculums led to the conclusion that the presence of a large quantity of microorganisms is necessary for optimum biogas production. In addition, the grease produced a good quantity of biogas when co-digested with the inoculums. The cumulative volume of biogas obtained over 30 days with the grease was 445 ml. The highest quantity of biogas obtained by optimising gas production is 780 ml.

Keywords: grease; treatment; anaerobic; biogas; recovery; bleaching earth

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1. Introduction

Worldwide, the demand for energy continues to grow to meet development needs. Fossil fuels, notably oil, natural gas and coal, currently account for over 80% of supply. However, this high demand is depleting non-renewable energy sources and increasing greenhouse gas (GHG) emissions, which are at the root of climate change^[1]. In addition, human activity has always generated waste, which is often a potential source of illness due to air, water and soil pollution^[2]. This situation raises a number of questions: how can we reduce the consumption of fossil fuels, which generate greenhouse gases, while meeting society's energy needs? How can we effectively treat the waste that is constantly being produced? Since then, climate change and the environment have become major preoccupations worldwide. In many

countries, fossil fuels are being phased out in favor of energy from local, renewable sources. This is why, in developing countries, the use of renewable energies and the preservation of the environment have become key issues^[3]. With this in mind, several studies on waste management methods have been carried out. They show that one of the most appropriate methods is methanization. It appears to be an efficient, environmentallyfriendly process, providing two valuable products: biogas and digestate. The company in question is part of this dynamic. It is interested in an alternative way of recovering the fats formed by the oil at its wastewater treatment plant, as well as the used bleaching earth rejected during the decolorization of the oil into a high value-added product. To date, the bleaching earth has been collected by an external company. The grease, on the other hand, is not evacuated and prevents easy treatment of the wastewater, which is sent to the sewage treatment plant. We thought it would be a good idea to recycle this waste using the methanization process. The abundance of grease and the non-use of the company's own bleaching earth were finally given the go-ahead to be valorized. In addition, the abundance and availability of cow dung and chicken droppings substrates from the central slaughterhouse in the vicinity of this company was one of the essential criteria for their use in our process. The overall aim of this study is to contribute to environmental protection by converting waste grease and bleaching earth into biogas. More specifically, our aim is to:

- Determine the physico-chemical parameters of grease and bleaching earth;
- Evaluate the methanogenic potential of used grease and bleaching earth using anaerobic monodigestion;
- Optimize biogas production by anaerobic co-digestion;
- Evaluate the composition of the resulting gas.

2. Materials and methods

This section includes study, sampling, laboratory and computer equipment.

2.1. Plant and animal material

The plant and animal material used in this study consists of:

- grease from the wastewater collector at wastewater treatment plant and bleaching earth taken from the refinery;
- cattle dung taken and broiler droppings from the INP-HB farm.

Fat and bleaching earth are the substrates, and cow dung and broiler droppings are the inoculums.

Figure 1. Raw materials: Grease (1), Used bleaching earth (2), Cow dung (3), Chicken droppings (4).

2.2. Sampling

For safety reasons, field equipment required a smock, helmet, safety shoes and nose plugs. Sampling was carried out using the following equipment (**Table 1**):

- Sterile plastic bottles and shovels for taking grease samples;
- Sterile paper used for the bleaching earth;
- Adhesive tape and permanent marker for labelling;
- A cool box for transporting the grease samples.

Table 1 summarises the equipment used to carry out the measurements.

Table 1. Equipment used for physico-chemical analyses.

In addition, the research methodology consisted of sampling the grease from the collector at the treatment plant and the used bleaching earth, determining their physico-chemical parameters, evaluating and optimising the methanogenic potential in the laboratory and determining the composition of the biogas obtained.

2.3. Sample collection

The grease wastewater treatment plant and the used bleaching earth were collected from the collecting area and the used earth area respectively, using a shovel. The grease was transported to the laboratory in a cool box, and the used bleaching earth was transported using sterile bleaching earth paper. The inoculums were placed in sterile plastic jars to facilitate transport.

Once in the laboratory, a series of analyses were carried out to characterise the different samples.

2.4. Determination of physico-chemical parameters

2.4.1. pH

The hydrogen potential (pH) of each waste sample was determined by dissolving the waste in a waste/distilled water ratio of 1:100. 2.5 g of waste was suspended with 250 mL of distilled water in a plastic beaker under constant stirring for 5 minutes using a magnetic stirrer. The suspension was left to stand for 30 minutes before pH measurements were taken. Readings were taken using a HANNA HI 9525 pH meter.

2.4.2. Humidity

The moisture content (%H) is determined by oven drying at 105°C for 24 hours. The percentage humidity of the various samples is determined by **equation 1**:

$$
\%H = \frac{100 \times (M_0 - M_1)}{M_0} \tag{1}
$$

% H: percentage of humidity;

M0: initial mass of the sample before drying;

M1: final mass of the sample after drying.

2.4.3. Total Solids (TS) or Dry Matter (DM)

The moisture content is used to determine the Total Solids (TS) content.

$$
\mathcal{V}_0 \mathbf{ST} = \mathbf{100} - \mathcal{V}_0 \mathbf{H} \tag{2}
$$

 $% ST$: total solids content

2.4.4. Volatile Solids (VSS)

The volatile solids (VSS) or organic matter (OM) content is obtained by weighing the difference between the mass of dry waste (M1) and the mass of waste calcined at 550° C (M2) for 5 hours.

$$
\%SV = \frac{100 \times (M_1 - M_2)}{M_1} \tag{3}
$$

M2: final mass of the sample after calcination.

2.4.5. Chemical Oxygen Demand (COD)

This involves oxidation of the oxidisable organic matter contained in the sample to be analysed by an excess of potassium dichromate $(K_2Cr_7O_7)$ in an acid medium (H_2SO_4) and boiling in the COD meter (2 hours at a temperature of 150°C). It was carried out in the presence of silver sulphate (Ag2SO4) as a catalyst. The solution obtained is assayed using a solution of iron (II) ammonium sulphate (Mohr's salt) (AFNOR NF T 90- 101, 1988). The COD (mg O_2/L) is calculated using the following formula:

$$
DCO = \frac{8000 C (V_1 - V_2)}{V_0}
$$
 (4)

With:

 $V1$: volume of blank used for the test (mL);

V2: volume of burette drop for each sample (mL);

0: volume of test sample before dilution (mL);

: concentration of Mohr's salt used (mol/L).

2.4.6. Total Alkalimetric Titration (TAC)

The aim here is to measure the alkalinity in the digesters. A 10mL volume of this mixture is taken and diluted in 250mL of distilled water. Next, a volume of 50mL was taken for titration with a sulphuric acid solution (H2SO4 at 0.04N) in the presence of a few drops of methyl orange. Finally, the volume of the burette drop (V1) is noted and this is used to calculate the TAC. The TAC is expressed in mg/L and obtained by the following expression:

$$
TAC = \frac{V_1 \times N \times 1000}{V}
$$
 (5)

With:

 $V:$ volume of test sample (mL);

 $V1$: volume of burette drop containing acid (mL);

N: normality of the acid solution.

2.4.7. E Practical evaluation of biogas production (BMP test)

Figure 2. Experimental set-up for the BMP test.

All the tests were carried out in batch digesters for 30 days. The reactors (digesters) were plastic laboratory models, to ensure anaerobic conditions. They are 1200 ml with a useful volume of 1000 ml and a headspace of 200 ml. They each have two openings, the first for taking liquid samples using syringes. The second is for collecting and measuring the volume of biogas produced. After incubating the substrates, the reactors are placed in a water bath at a thermophilic temperature of between 50°C and 55°C. This temperature range improves the fluidity of the lipids (fats). The lipids are then connected to 1000 ml graduated test tubes which are inverted into a container containing water. Once the mixture is in the digester, the final volume is adjusted to 1000 ml with distilled water. The digester was then hermetically sealed. To monitor the reactions, each digester is shaken 4 times for two minutes to ensure that the anaerobic medium is homogenous. When the biogas is produced, it exerts pressure on the water present in the gasometer (test tube), which is inverted to occupy its upper part. The water expelled from the top of the test tube leaves a void that is filled by the biogas. This displacement makes it possible to read the volume of gas produced. In terms of the sample, the mass used for each substrate is determined according to the following formula:

$$
\mathbf{m_0} = \frac{\mathbf{M}}{\frac{\%M\mathbf{S}V}{100} \times \frac{\%S\mathbf{T}}{100}}\tag{6}
$$

With:

m0: mass of sample to be taken (g);

M: mass of volatile solid fixed as a function of the quantity of digester volatile matter;

%MSV: volatile solid content (%);

%ST: total solids content (%).

2.5. Anaerobic digestion of substrates

We set a total mass of 16g (SV) per trial due to the volume of the digester. The different proportions of substrates for the mixtures are determined by the Design Expert software relative to Henry Scheffé's classical centred augmented mixing design [4]. This mixing plan requires ten trials for the three substrates, including three monodigestions and six codigestions. The different mixing ratios and the masses of the substrates used are given in **Tables 2** and **3**.

Proportions						
Tests	Grease	Cattle dung	Chicken droppings			
1	1	$\mathbf{0}$	$\boldsymbol{0}$			
2	θ	1	$\mathbf{0}$			
3	$\overline{0}$	Ω	1			
$\overline{4}$	1/2	1/2	Ω			
5	1/2	$\overline{0}$	1/2			
6	θ	1/2	1/2			
7	1/3	1/3	1/3			
8	2/3	$1/6$	1/6			
9	1/6	2/3	1/6			
10	1/6	1/6	$\frac{2}{3}$			

Table 2. Proportions of inputs.

```
Table 3. Input weights.
```


2.6. Analysis of biogas composition

When anaerobic digestion is used, it is a good idea to analyse the composition of the biogas in order to get an idea of it, and to know what type of treatment to apply to the biogas in order to purify it and direct it into a given value chain. To do this, we set up an experimental system consisting of digesters and air chambers. The digesters are treated under the same conditions as the BMP test, except that in this case, instead of being connected to test tubes, they are connected to the air chambers used to retain the gas. Once the 30 days were up, we connected the BOSEAN biogas analyser to each chamber to determine the composition of the gas inside.

Figure 3. A: Device for analysing the composition of biogas; B: Biogas analyser.

3. Results

This section presents the results of our various experiments and analyses, as well as a discussion of these results.

3.1. Input characteristics

3.1.1. Physico-chemical parameters

The biodegradability of organic matter depends directly on the biochemical composition of the substrate to be treated [4]. The physico-chemical parameters of the inputs are summarised in the following table:

Parameters	рH	%H	$\%MS$	$\%$ MSV
Grease	5.668	32.3125	67.6875	97.5739
Bleaching earth	5.063	1.7969	98.2032	59.9719
Cow dung	8.408	80.8698	19.1302	85.9618
Chicken droppings	8.465	73.5543	26.4457	89.8935

Table 4. Physico-chemical parameters of inputs.

This table shows the pH, moisture content, dry matter content and volatile matter content of the various substrates.

pH is an important parameter for anaerobic digestion. The table shows that the pH values for fat, bleaching earth, cattle dung and broiler droppings are 5.882, 5.063, 8.408 and 8.465 respectively. It should be noted that the optimum pH range for waste for anaerobic digestion is between 6.5 and 8.5 and the values obtained are not within this optimum pH range except for cow dung and chicken droppings. In addition, cattle dung with a pH above 7 favours the growth of methanogenic bacteria **[**6**]** . Since our basic substrates (fat and bleaching earth) are acidic, this would be due to the fact that they contain organic acids **[**7,8**] .** This was also confirmed by Wassila ARRAS (2017) during an experimental and modelling study of the anaerobic digestion of residual organic matter ^[9,10]. The acidic pH of basic waste negatively influences biogas production, as it affects the activity of microorganisms to degrade organic matter into methane. The use of inoculum with basic pH levels will raise the pH in the digesters and ensure that anaerobic digestion proceeds smoothly.

(1) Humidity

The moisture content (%H) of fat, bleaching earth, cattle dung and chicken droppings are 32.3125, 1.7969, 80.8698 and 73.5543 respectively. For fat, cattle dung and broiler droppings, these high values reflect their high water content. This large amount of water shows that these wastes are fermentable so anaerobic digestion is therefore appropriate for this type of waste **[**11,12**] .** On the other hand, the low moisture content of bleaching earth offers it resistance to fermentation. Its dry matter content of over 20% means that it has to undergo the dry methanisation process. Above a dry matter content of 20%, microbial equilibria and kinetics are disturbed. It is therefore necessary to combine dry digestion with a thermophilic regime, as this increases hydrolysis kinetics **[**13-15**] .**

(2) Dry matter

Determination of the dry matter (DM) content (%) is a criterion that makes it possible to classify the substrate according to its ability to be more or less degradable by biochemical means**[**16**]** . The dry matter content of our study substrates was 67.6875, 98.2032, 19.1302 and 26.4457 for grease, bleaching earth, cattle dung and chicken droppings respectively. The values obtained compare with anaerobic digestion by the dry route with a percentage between 15% and 40% ^[17].

(3) Volatile solids

The volatile solid matter (VSM) contents (%) are 97.574; 59.9719; 85.962 and 89.894 are for fat, bleaching earth, cattle dung and broiler droppings respectively. These high volatile solids contents suggest that the organic load is high, which also implies a high energy potential^[18-21]. Consequently, this waste is favourable for methanisation. As for bleaching earth, its moderately high volatile solids content (%VS) of 59.972 comes from the fact that its organic load is not too high. However, it is suitable for methanisation, as this value is higher than that obtained when recovering energetic food waste by anaerobic digestion, which was 21.85 %^[22].

3.2. Monitoring anaerobic digestion

3.2.1. Changes in certain parameters in digesters during anaerobic digestion

Table 5 shows the initial and final values of pH, COD and TAC for all the tests over the experimental period.

Table 5. Characteristics of some parameters at the start and end of the tests.

As far as pH is concerned, the table shows that it fell in all the digesters. This can be explained by the fact that, basically, anaerobic digestion tends to lower the pH of the medium. In addition, acidification of the medium, sometimes linked to the additional quantities of VFA released into the anaerobic medium**[**23-26**]** , may be the cause. In addition, the methanogenesis step can be inhibited at low pH**[**27-28**]** .

As far as COD is concerned, we can say that this methanisation process has helped to reduce it. Other authors such as**[**29-30**]** have confirmed the reduction of COD by methanisation. This reduction in COD is thought to be due to the oxidation of a large proportion of the organic matter contained in the substrates.

Measuring the TAC provides information about the alkalinity in the digesters. It was noted that at the end of digestion, there was an increase in TAC at all levels**[**31**]** . In fact, this increase in alkalinity is explained by the activity of methanogenic bacteria in making the medium alkaline by synthesising carbon dioxide, ammonia and bicarbonate^[18, 32-35]. This alkalinisation of the medium makes it possible to correct the VFA supplement, which has a direct influence on the biogas volume yield**[**36**]** .

3.2.2. Daily biogas production kinetics

Biogas production in the digesters was monitored. The gross biogas production kinetics curves for the various tests were illustrated. The gross daily biogas production recorded from each digester is assessed below.

3.2.3. Daily biogas production kinetics for each substrate

Figure 4. Daily biogas production from fat and another substrates.

Figure 4 shows the evolution of daily biogas production for each input. It can be seen from the graph that digester E3 (Manure) obtained its highest peak in biogas production on the 3rd day of anaerobic digestion when we collected 120 mL of biogas. The highest biogas peak for digester E2 (Dung) was on day 9, when we collected 40 mL of biogas. The second highest peak was for the anaerobic digestion of fat (E1), also recorded on day 9, with a volume of 70 mL.

3.2.4. codigestion of fats and other substrates

Figure 5 shows the evolution of the volume of biogas produced as a function of time in digesters E4 (1/2 Dung+1/2 Grease), E5 (1/2 Grease+1/2 Manure) and E6 (1/2 Manure+1/2 Dung).

Figure 5. Daily biogas production from two-by-two inoculum-substrate and another substrates-another substrates mixture.

It can be seen that for all the digesters, biogas production was not constant. In addition, there were at least two peaks on the graph representing the maximum specific biogas production for each digester. The dung $+$ manure mixture from day 1 to day 2 gave a quantity of biogas that marked the highest peak on the graph. From day 3, this daily volume falls and is cancelled out until day 6. From day 7 to the end of the experiment, the quantity of biogas produced is irregular. For the grease + dung and grease + droppings mixtures, biogas production was irregular from the beginning to the end of digestion.

Figure 6. Daily biogas production from binary mixtures.

The graph shows a similar trend for all the curves. There were also several biogas peaks in all the mixtures. The most significant peak for digester E7 occurred on day 20, when 60 mL of biogas was produced. The largest peak for digester E8 occurred on day 22, when 100 mL of biogas was recorded. Digesters E9 and E10 reached their peak in terms of biogas output on day 22, when they produced 150mL and 190 mL respectively.

3.3. Cumulative biogas production kinetics

Based on the daily biogas production, the cumulative biogas production kinetics are represented for each digestion case.

3.3.1. Monodigestion of substrates

Figure 7 shows the cumulative biogas volumes per gram of organic matter during 30 days of anaerobic digestion. The tests were carried out on fat, cattle dung and chicken droppings in digesters E1, E2 and E3. The figure shows a biogas volume of 445 ml, 340 ml and 410 ml for fat, dung and droppings respectively. Fat therefore has the highest cumulative quantity of biogas, followed by droppings and then dung. As far as the chicken droppings are concerned, over the first four days, biogas production in digester E3 started very quickly. However, from the 5th day onwards, biogas production was almost non-existent, before gradually increasing exponentially from the 20th to the 30th day. This long pause in production may be due to inhibition caused by excessive VFA generation in the digester. In the case of cattle fat and dung, biogas production was very slow from day 1 to day 8. This phase corresponds to the latency phase. This period corresponds to the start of anaerobic digestion. The microorganisms are adapting to the anaerobic environment to break down the organic matter. From the 9th day onwards, there is an exponential increase in the quantity of biogas produced until the end (Day 30). This is the exponential growth phase.

Figure 7. Cumulative biogas production kinetics for substrates and other substrates.

Figure 8. Cumulative biogas production kinetics for substrate and another substrates.

3.3.2. Anaerobic digestion of binary mixtures

In this graph, the codigestion between cattle dung and manure in experiment E6 records the largest quantity of biogas with a cumulative volume of 430 ml. The mixture of fat and cattle dung in E4 gave a cumulative biogas volume of 230 ml. The mixture of fat and dung in E5 produced 215 ml. It should be noted that the quantity of gas observed in digester E6 is double that obtained in digester E5.

For the E6 mixture, biogas production was high for the first two days. From day 3 to day 8, biogas production slowed down. Production resumed exponentially from day 9 until the end of digestion. As for the other two mixes, E4 and E5, their biogas production evolves in almost the same way. This is reflected in their representative curves, which are almost superimposed. From day 1 to day 8 in both cases, biogas generation was slow. Exponential growth was observed from day 9 to day 23. A slowdown in biogas production was observed from day 24 until the end of digestion. This corresponds to the plateau phase, which results in low biogas production due to substrate exhaustion^[37].

3.3.3. Anaerobic digestion of ternary mixtures

Figure 9. Cumulative biogas production kinetics for substrate and another substrates mixtures (ternary digestions).

These graphs show the cumulative volume of biogas produced during the ternary digestion of the substrates. The ternary mixture E9 gave a cumulative volume of 780 ml, which is the highest volume of biogas. This was followed by E10 with a volume of 660 ml, then the proportional mixture E8 with a volume of 385 ml and finally mixture E7 with a volume of 370 ml. The curves showing the evolution of the cumulative volume of biogas in digesters E9 and E10 evolve very closely. We can see that from day 1 to the end, there is an increasing trend in the quantity of biogas in these digesters. At the same time, however, a different phenomenon is observed in digesters E7 and E8. The last two curves show low biogas production from the start, followed by an acceleration in production, before declining or slowing down at the end. This kinetics is consistent with the results of several other authors^[12, 38-39]. On these graphs, three distinct phases are noted as in some authors^[40].

1st phase: latency phase, marked by low biogas production. The duration of this phase depends on the nature of the substrate. From day 1 to day 8, biogas generation slows to an estimated 22 ml and 5 ml respectively for digesters E7 and E8.

2nd phase: exponential phase, marked by increasing biogas production. It corresponds to the central part of the production curves [40]. From day 9 to day 27, significant biogas production begins in variable proportions. The values obtained from digesters E7 and E8 are 330 mL and 365 mL respectively.

3rd phase: plateau phase, corresponding to a decrease in biogas production. It begins on day 28 and continues until the end of the experiment.

3.3.4. Assessment of the composition of the biogas obtained

In the final analysis, the quality of the gas obtained is the best key to assessing each registered production. Analysing the composition of the biogas would therefore give a more precise idea of the nature of the gas, i.e. whether the gas obtained is indeed biogas.

		Composition of biogas		
Tests	CH ₄	CO ₂	$\bf CO$	H_2S
	$\frac{0}{0}$	$\frac{0}{0}$	ppm	ppm
E1	20	78	831	6
E2	89	9	5	1
$\mathop{\mathrm{E3}}$	$80\,$	18	3	$\overline{2}$
E ₄	18	80	θ	1
E ₅	19	79	Ω	1
E ₆	98	θ	9	\overline{c}
E7	18	80	θ	1
E8	18	80	Ω	
E ₉	18	80	Ω	1
E10	19	79	0	

Table 6. Composition of the biogas obtained in each digester.

From **Table 6**, which shows the composition of the biogas in each digester, we can see that the proportion of methane contained in the raw biogas varies from one digester to another. The biogas obtained in most of the digesters in our study is mainly composed of CO2. However, digester E6, which contained a 50% mixture of chicken droppings and cattle dung, produced more methane than the other digesters. Then we have digesters E2 and E3 containing the inoculums. The lowest yields were observed in digesters E1, E4, E5, E6, E7, E8, E9 and E10. The proportion of flammable gas varies according to several factors (pre-treatment, dose, nature of the substrate, etc.) $[41]$.

5. Conclusion

The work carried out concerns the study of the methanogenic potential of grease and bleaching earth by conversion into biogas: the case of a company's wastewater treatment plant and refinery. The results of the characterisation of the raw materials showed that grease can still be recovered by methanisation, while the results for bleaching earth were not favourable for this recovery method in the proportions used. To optimise the biomethanisation of grease, a number of factors need to be controlled, including water content, agitation, the quantities and activity of the bacteria present, pH, temperature and composition. Suitably moistened material can be homogenised to promote material transfer. The medium can be buffered to avoid major variations in pH. Nutrient deficiencies or excesses can be limited by mixing the material to be degraded with other materials with suitable characteristics. The grease used in our study gives a good quantity of biogas when co-digested with the inoculums. The cumulative volume of biogas obtained over 30 days with the grease is 445 ml and the highest quantity of biogas obtained by optimising biogas production with the inoculums is 780 ml.

The biogas obtained during fat methanisation is composed of approximately 78% inert $CO₂$, approximately 20% CH₄ and a few trace elements such as H_2S and CO.

In our study, the production of biogas from fat constitutes an alternative way of recovering this waste, and contributes to the circular economy. The bleaching earth used in the proportions studied is not directly suitable for recovery by anaerobic digestion.

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Conflict of interest

The authors declare that they have no conflict of interest.

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