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Impact of Iron Oxide (Fe2O3) Nanoparticles on Biogas Yield from Co-digestion of Food Waste and Cow Dung

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A B S T R A C T

In the pursuit of efficient biomass management and enhanced renewable energy security, there is a global exploration of diverse methods and processes to optimize biogas yield. The escalating issue of food waste necessitates sustainable solutions for its management, and anaerobic digestion (AD) emerges as a promising approach in addressing this challenge. The addition of Nanoparticles (NPs) has been carried out to study its effect as a catalyst on the production of biogas via anaerobic digestion. This study investigates the effect of using iron oxides (Fe_2O_3) NPs on the co-digestion of food waste (FW) and cow dung (CD) in a lab-scale batch reactors under ambient conditions. Synthesis of iron oxide NPs was achieved through the sol-gel method. Three lab-scale reactors with varying NP concentrations ranging from 5 to 50 mg/L, were compared with a control reactor without NPs. The results show that the addition of iron oxides (Fe_2O_3) NPs at concentrations of 5, 20, and 50 mg/L obtained cumulative biogas yield 160, 242, and 188 mL/gVS respectively compared to 135 mL/gVS without NPs. Notably, at 20 mg/L, the highest daily biogas output from the feedstock was observed on the 20th day, with total biogas production increasing significantly by about 78%.

1. Introduction

Anaerobic digestion, a critical process in the quest for sustainable energy, involves the microbial breakdown of organic waste to produce biogas, primarily composed of methane (CH_4). This technology presents a promising approach to mitigating climate change by converting waste into a valuable energy resource. The AD process consists of four stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. To maximize the benefits of this technology, careful consideration must be given to factors influencing its performance, such as feedstock type, operating conditions, and microbial ecology. The addition of additives is one of the most promising ways to improve the anaerobic digestion process [1].

Zinc (Zn), cobalt (Co), iron (Fe), nickel (Ni), and molybdenum (Mo) are among the trace metals that are vital sources of nutrients. These metals increase the anaerobic bacteria's activities during the AD technique by facilitating the creation of essential enzymes and co-enzymes[2-4]. One of the noteworthy additions are carbon-based conductive materials, for example, Granular Activated Carbon (GAC), Powdered Activated Carbon (PAC), and Carbon Nanotubes (CNTs) [5]. These materials have large specific surface area, which helps with microbe adherence, strong electron conductivity, and the ability to adsorb potentially hazardous substances [6]. The yield and biogas production have been increased by the application of metallic and non-metal oxide nanoparticles, such as copper, nickel oxide, hematite, palladium, silver, etc. [7]. Ajao et al.[8] investigated the effect of silicon oxide nano additive on the biogas production and methane yield of anaerobic digestion using cow and sheep dung as substrates. The addition of silicon oxide nano additive (30 mg/l) increased methane content in cow dung biogas from 64.9% to 65.7% and in sheep dung biogas from 59.3% to 60.2%.

Boscaro et al. 2022 [9] explored the potential of iron oxide nanoparticles (Fe_3O_4 NPs) to enhance the anaerobic digestion of crude glycerol. The researchers synthesized Fe_3O_4 NPs and added them to anaerobic reactors containing granular sludge and crude glycerol. Results showed that the addition of Fe_3O_4 NPs significantly increased methane production and the rate of methane formation.

Zhang et al. 2020 [10] investigated the impact of iron oxide nanoparticles on biogas production and waste sludge reduction in a two-stage anaerobic digestion process. The optimal concentration of Fe_3O_4 NPs was found to be 100 mg/L, which produced substantial growth in hydrogen and biogas production compared to the control. Additionally, Fe_3O_4 NPs reduced the accumulation of volatile suspended solids (VSS) and inhibited the production of ammonium nitrogen (NH4+-N).

Furthermore, Numerous investigations have demonstrated that nanoparticles of iron oxide, such as maghemite (Fe_2O_3) and magnetite (Fe_3O_4), can significantly improve the yield and quality of biogas[11-17].

Fe_2O_3 NPs allow methanogens and bacteria to transport electrons directly between species, which has a favorable effect on methanogenic archaea activity and biogas production [18]. However, Fe_2O_3 can suppress the methanogenic consortia, nevertheless, this is very dependent on its concentration[19].

The aim of this study is to produce low-cost, non-toxic Fe_2O_3 nanoparticles and evaluate their characteristics. Then investigate their impact on the anaerobic co-digestion of cow dung and food waste, as well as their different concentration effect in producing biogas

2. Experimental Test Rig

An experimental test rig is created to study the effects of Fe_2O_3 NPs on biogas production. There are four symmetrical small plastic digesters. Each digester is provided with a valve to control the produced gas. The biogas production was measured using the water displacement method through a graduated laboratory immersed in a water vessel as shown in Fig. 1.

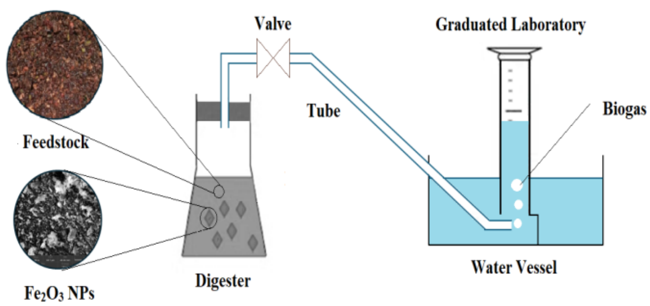

Figure 1: A schematic diagram of the biogas plant

3. Material and methods

3.1. Preparation of Fe2O3 NPs in the laboratory

The preparation process shown in Fig.2 was conducted by preparing one liter of ferric nitrate solution with 0.15 mol/m^3 concentration, the molar mass of the ferric nitrate nonahydrate is 403.9 g/mol and the solution was prepared by performing the following steps:

- 20 grams of Fe_2O_3 are added gradually and dissolved to 100 ml of nitric acid with 0.15 mol/m^3 of concentration.
- A glass rod is used for stirring until a yellow-green ferric nitrate solution with 3 mol/m^3 concentration is produced.
- The solution is diluted to the desired concentration by adding 1900 ml of water to reach the desired concentration.
- The solution is dissolved in 50 mL of ethylene glycol ($C_2H_6O_2$) at 50 °C.

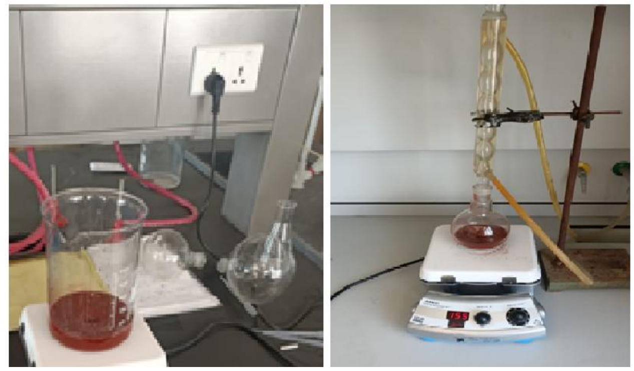

Figure 2: Preparation of NPs in the laboratory

- The solution is stirred for 90 min using a hot plate magnetic stirrer.
- Then the solution is transferred to a heater at 80 °C until a brown semi-solid jell is produced.
- After this process is completed the jell is dried inside a hot oven at 800 °C for 150 mins.
- The final nanoparticle powder is deposited at the bottom of the test vessel and is crushed to form a reddish powder.

3.2. Characterization of Fe2O3 NPs

The characterization of NPs is measured at the Center of Excellence for Membrane Testing and Characterization (CEMTC) at Port Said University. Figure 3 shows the Scanning Electron Microscopy (SEM) images of Fe2O3 NPs at four different magnifications (50 µm, 20 µm, 1 µm, and 500 nm).

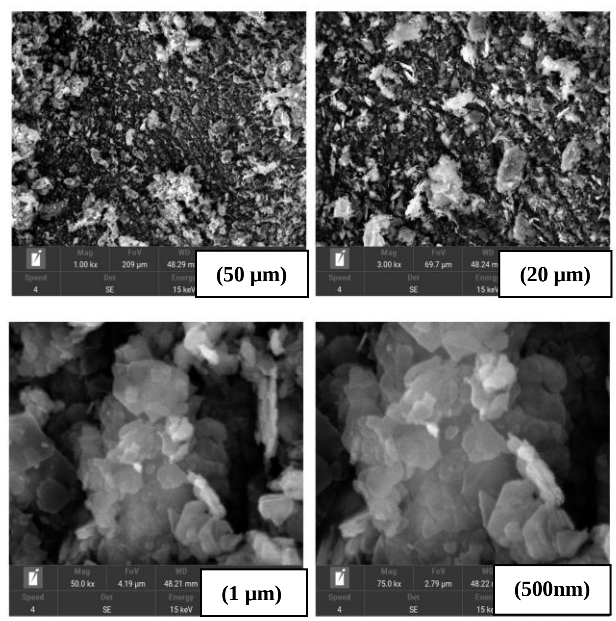

Figure 3: The SEM images of Fe_2O_3 nanoparticles at four different magnifications (50 µm, 20 µm, 1 µm, and 500 nm).

Figure 3 depicts the surface morphology of Fe_2O_3 nanoparticles. The SEM photos show agglomerated Fe_2O_3 nanoparticles. Because of their high surface energy, nanoparticles tend to agglomerate and develop into bigger assemblies. The particle size of Fe_2O_3 nanoparticles increases in the region of 200 nm. Van der Waals force and magnetic interactions between particles can produce agglomeration. SEM pictures, however, revealed no distinct form or size distribution of Fe_2O_3 particles.

The zeta potential shown in Fig. 4 offers information on the stability of nano formulations, and a negative value indicates decreased particle aggregation in the continuous phase. The particle is stable if its potential is between +30 and -30 millivolts. The zeta potential value was discovered to be -8.09 mV, indicating that the nanoparticles are stable. Iron oxide nanoparticles are in the specified range, implying that they are not aggregating and are stable under the conditions in which they will be employed.

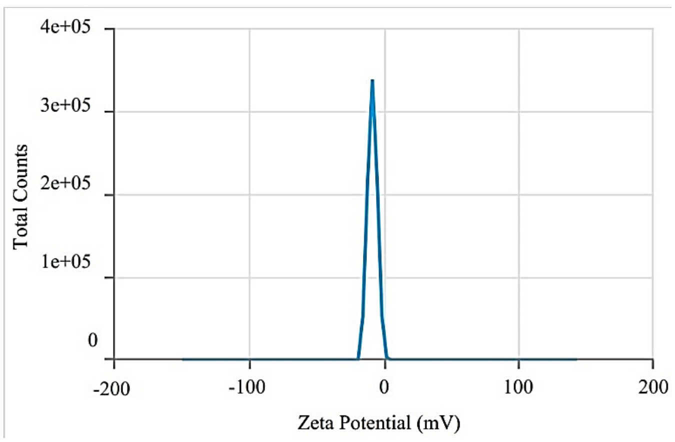

Figure 4: Zeta potential distribution of Fe_2O_3 nanoparticles

X-ray powder diffraction (XRD) was performed using (BRUKER D8 DISCOVER XRD) equipped with Cu target (wavelength 1.5406 Å) step size 0.2º used to determine the Fe_2O_3 NPs crystal structure as plotted in Fig. 5.

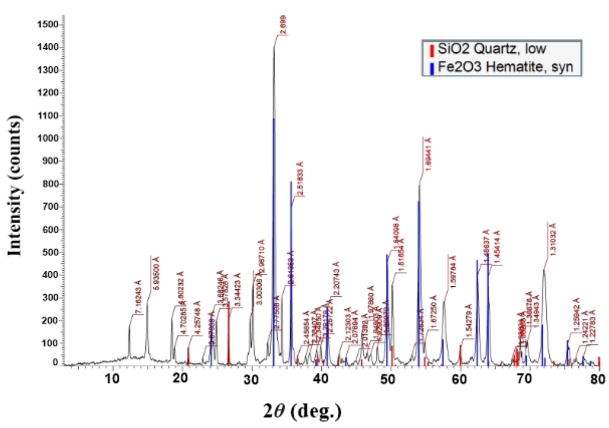

Figure 5: XRD pattern of Fe_2O_3

From Figure 5, the maximum peak of Fe_2O_3 occur at 2θ = 33.15º and 35.6º with net intensity of 1490 counts and 694 counts respectively. During the first 10 degrees there is no peaks appeared due to noise . In order to obtain the crystallite size (D), Scherrer formula was employed as follows [20]:

$$D = \frac{K\lambda}{B\cos\theta} \tag{1}$$

where *D* is the crystalline size, *K* is a constant whose value is approximately 0.9, λ is the wavelength of the X-rays (0.15406 nm), *B* is the full width at half-maximum (FWHM) (in rad).

The crystallite size was determined by Scherrer formula for the maximum 10 peaks of Fe2O3 range between (31.78 nm and 71.93nm) and the average value equal to 47.52 nm. Hematite nanoparticles are evidently well-crystallized and primarily exist as granules with small and large spherical shaped particles [21].

Feedstock material and its characteristics

The substrate used in this study is composed of a mixture of 0.5 kg fresh cow dung and 1 kg food waste. The cow dung was supplied from an animal farm located in the south of Port Said city. Food wastes are composed of leftover food including rice, pasta, cooked meat, watermelon peels, cooked peas, and spinach. The food waste was shredded into small piece using an electrical shredder for homogeneity. This would make the digestion easier before the material is loaded into the digester. Specifically, shredding improves the waste's surface area, which facilitates handling, transportation, and processing. After shredding, the feedstock was mixed with 1 Liter of hot water at 52 ℃ to have a good mixing of the feedstock. The total volume of the feedstock is about 2.5 Liters. Then the feedstock loaded into the digester with the required NPs mass to obtain the desired concentration. Table 1 represents the initial feedstock characteristics.

Table 1: Initial feedstock characteristics.

Cow dung (kg)	**0.5**
Food waste mass (kg)	**1**
Water (L)	**1**
C/N	**17.3**
TS (%)	**19.4**
VS (%)	**49.9**
pH	**5.7**

4. Results and Discussions

The experiments were conducted to evaluate biogas yield from the anaerobic co-digestion of FW and CD at ambient temperature. As demonstrated in Fig. 6, the daily biogas production from different concentrations of NPs is compared. The biogas production reaches the maximum value from the concentration of 20 mg/l after 20 days from feeding. The cumulative biogas yield shown in Fig.7 has a significant increase during this period compared to the other concentrations and the control reactor without NPs. As shown in Figs 5-6 the daily biogas production and cumulative biogas yield from the reactor with 50 mg/l concentration and the control reactor without NPs are almost identical during the first 34 days, then the cumulative biogas started to increase from the 50 mg/l reactor during the remaining days. At a concentration of 5 mg/l the biogas production has a bad effect during the first 45 days which has a value lower than the control reactor without NPs. Then starts to increase and finally has a cumulative value higher than the control reactor.

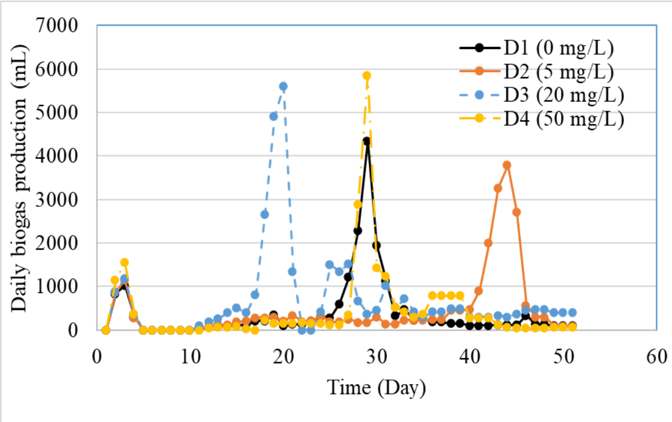

Figure 6: Daily biogas production for different NPs concentrations.

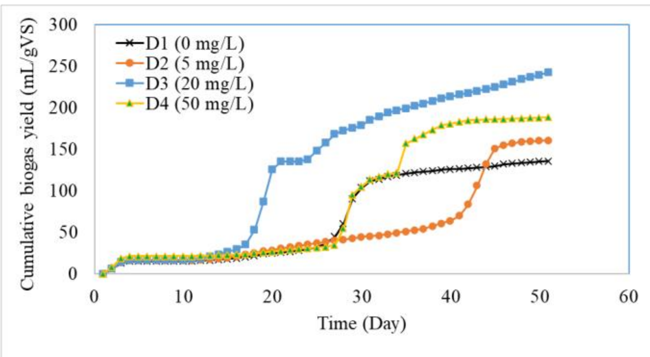

Figure 7: Cumulative biogas production for different NPs concentrations.

Table 2: A comparison between the present study and the previous works..

Feedstock	NPs type	Optimum concentration (mg./L)	Enhanced Biogas yield (%)	Ref
Manure	**Fe_3O_4**	**20**	**95**	**[22]**
Cow manure	**Fe_3O_4**	**16.67**	**45**	**[23]**
Food waste +cow dung	**Fe_2O_3**	**20**	**78**	**Present work**

A comparison between the current study and previous researches is shown in Table 2. The addition of various forms of iron NPs can increase biogas production in the range of 45% and 95%. Abdelsalam et al. [22] found that using iron NPs increased the amount of biogas produced by anaerobic digestion of manure by 95% at 20 mg/L concentration. Moreover, Juntupally et al.[23] found this percentage to be 45% by adding 16.67 mg/L of iron NPs with cow manure as a feedstock of AD. In the current study, the addition of Fe2O3 at 20 mg/L NPs concentration the production of biogas yield increased by about 78% for AD of food waste with cow dung digestion. The optimum concentration achieved in this present work shows a good agreement with both [22, 23].

The partial conversion of generated CO2 to methane in the presence of iron NPs by electron transmission may be the cause of these increases in the biogas production [24, 25] as shown in Equation (2) as follows:

$$8H^{+} + 4Fe^{0} + CO_2 \rightarrow CH_4 + 4Fe^{2+} + 2H_2O \qquad (2)$$

5. Conclusions

This study used four symmetrical small plastic digesters to evaluate the effect of the addition of Nanoparticles (NPs) as a catalyst on the production of biogas via anaerobic digestion. Below are the key findings:

- The Iron oxide (Fe_2O_3) NPs are stable under the conditions at which will be employed.
- The iron oxides (Fe_2O_3) NPs are an effective catalyst in the production of biogas via anaerobic digestion in a lab-scale reactor under ambient conditions.
- The cumulative biogas yield are135,160, 242, and 188 mL/gVS at Fe_2O_3 NPs concentrations of 0, 5, 20, and 50 mg/L respectively.
- The high cumulative biogas yield is obtained at Fe_2O_3 NPs concentration of 20 mg/L.
- The highest daily biogas output from the feedstock was observed on 20th day at NPs concentration of 20 mg/l.

Abbreviations

AD	**Anaerobic digestion**
CD	**Cow Dung**
CEMTC	**Center of Excellence for Membrane Testing and Characterization**
CNTs	**Carbon Nanotubes**
FW	**Food Waste**
GAC	**Granular Activated Carbon**
NPs	**Nanoparticles**
PAC	**Powdered Activated Carbon**
SEM	**Scanning Electron Microscopy**
VSS	**volatile Suspended Solids**
XRD	**X-ray powder diffraction**

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