Syngas Evolutionary Behavior during Chicken Manure

Pyrolysis and Air Gasification

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Abstract

The evolutionary behavior of syngas composition during the pyrolysis and gasification of chicken manure was examined at different temperatures and \( \text{O}_2 \) concentrations. A gas chromatography was used to quantify the syngas evolved. Pure nitrogen was used for pyrolysis while two different oxygen concentrations (21\% and 10\%) in nitrogen were used for gasification. Five specific temperatures examined during pyrolysis and gasification were from 600-1000 °C in steps of 100 °C. Higher \( \text{O}_2 \) concentration (21\%) produced higher energy yields compared to lower \( \text{O}_2 \) concentrations. Initial 8-10 minutes’ yield produced \( \text{CO}_2 \) dominant syngas from decarboxylation after which the compositions changed to

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Keywords: Chicken manure, Air gasification, Pyrolysis, Syngas production.

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equilibrium. High temperature and low O\textsubscript{2} concentrations yielded higher CO flow rates and amounts. Equilibrium H\textsubscript{2} content was reduced with an increase in O\textsubscript{2} concentration due to the rapid oxidation of H\textsubscript{2} in the presence of oxidative environment. CH\textsubscript{4} was obtained from thermal cracking with its evolution being similar to that of other higher hydrocarbons evolved, albeit in smaller concentration.

1. Introduction

Waste management is amongst the most important global challenges facing the society for seeking improved healthy environment. The amount of wastes generated continues to increase so that seeking sustainability requires increased reuse of all waste materials for further use as feedstock or reformed to usable energy. Global energy security and energy sustainability requires harvesting clean energy from wastes and low-grade fuels that will help reduce fossil fuel consumption and extend their availability. This also helps in the mitigation of greenhouse gases as energy produced from wastes and biomass is nearly carbon neutral. Use of solid organic wastes including municipal solid wastes, animal wastes along with biomass as an energy source is key to sustainable energy production and waste disposal. Such energy source has a carbon-neutral effect on greenhouse gas emissions due to their renewable production and disposal [1]. Livestock manure which includes poultry and other animal litter (excretes, bedding material) was traditionally land spread for its use as fertilizer and soil amendment. But with increased amounts of wastes composting due to large volume involved from increased population
causes eutrophication of water bodies including ground water by spreading leachates of nitrates, potassium and phosphorous present in the wastes to pollute both land and water and spreading into associated pathogens [2]. Environmentally safe disposal techniques can be equipped with energy production capability. Energy harnessing techniques such as anaerobic digestion, direct combustion, and gasification are environmentally safe for the disposal of animal wastes as well as municipal solid wastes. Pyrolysis and Gasification of wastes is an efficient solution over direct combustion, as it can produce liquid/syngas fuels of medium to high heating value, which can be utilized with the currently available infrastructure for power generation and transportation. Even though anaerobic digestion can produce methane its relatively slow production rate makes it less viable strategy compared to pyrolysis and gasification.

Gasification is the thermal conversion of solid carbonaceous material at high temperatures in the presence of gasifying agents, such as air, O₂, steam, CO₂ and their mixtures to form product gas of high heating value containing H₂, CO, CO₂, CH₄, C₂H₆ and other higher series of hydrocarbons. Tar, containing heavy aromatic hydrocarbon residues, is also formed as an undesired byproduct which can be further thermally or catalytically cracked or reformed to enhance syngas yield. The composition of syngas evolved from gasification depends to a large extent on the gasifying agent, temperature, feedstock chemical properties and other reactor operational parameters. Production of uniform syngas constituents from versatile sources including chicken manure, poultry litter, biomass, solid wastes, and coal makes it an attractive solution over direct combustion of these low-value heterogeneous feedstocks. Literature is available on the
investigations of biomass with and without catalysts, using different gasifying agents, temperatures, and gasifiers [1,3–17]. Equations (1–5) show the major reactions that occur during gasification.

\[
\begin{align*}
\text{Char Oxidation:} & \quad C + O_2 &\Rightarrow& \text{CO}_2 \\
\text{Boudouard Reaction:} & \quad \text{CO}_2 + C &\Rightarrow& \text{2CO} \\
\text{Water-gas shift Reaction:} & \quad \text{CO} + \text{H}_2\text{O} &\Rightarrow& \text{CO}_2 + \text{H}_2 \\
\text{Water-gas Reaction:} & \quad \text{C} + \text{H}_2\text{O} &\Rightarrow& \text{CO} + \text{H}_2 \\
\text{Water-gas Reaction:} & \quad \text{C}_n\text{H}_m &\Rightarrow& \text{C}^* + \text{C}_x\text{H}_y + \text{H}_2
\end{align*}
\]

Poultry litter is one of the major waste sources from the poultry industry that has only increased with time due to our quest for increased production. Manure, waste beddings, waste food, and feathers constitute poultry litter that has high ash content. Manure contains a considerable amount of organic nitrogen from proteins and amino acids. Abundant literature is available on pyrolysis and gasification of biomass, but differences exist in the amounts of various elements present in chicken litter than biomass. These include low heating value, high ash content, and high nitrogen content. These differences cause many challenges that have motivated many researchers to study the pyrolytic behavior of chicken litter [18]. Thermogravimetric analysis of poultry litter and comparison with coal revealed that decomposition of poultry litter starts at lower temperatures than that of coal and has a maximum weight loss rate at around 370 °C [19]. TGA results on the pyrolysis of chicken litter showed three stages of weight loss, unlike woodchips pyrolysis which occurs in two stages because of lignocellulose content [18].
Chicken litter pyrolysis involves three stages of decomposition of cellulose and hemicellulose with a peak at a temperature less than that of wood chips, followed by manure and lignin decomposition and the last stage of residual char devolatilization. Higher manure content assists in faster decomposition of waste litter. Increase in temperature increases syngas yields, and decreases liquid and char yields [20]. At temperatures above 700 °C, CO$_2$ yields increased and it was attributed to decarboxylation of minerals carbonates in the ash and reached a stable value above 1000 °C [19]. Highly viscous bio-oils were produced from chicken litter by fast pyrolysis. The results showed that bio-oil from chicken litter was relatively less acidic than that obtained from hardwood [21]. These pyrolysis results also showed that higher heating value (HHV) of bio-crude oil from chicken litter to be higher than that produced by hardwood due to the conversion of its protein content into higher hydrocarbons from the absence of CO$_2$ and N$_2$.

The high inorganic content in poultry litter (>20%) aids in fertilizing capabilities. For syngas production, the inorganic components can provide catalytic activity. However, a major drawback of dealing with chicken litter lies in the handling difficulty due to the low fusion temperature of inorganics present since the chicken litter ash contains high amounts of Ca, Na, and K. Such high amounts of ash content in chicken litter with phosphorous and potassium oxides makes it an efficient fertilizer even after gasification [22]. While the effect of other alkali and alkaline earth metal compounds on product gas is not known, decarboxylation of CaCO$_3$ was observed at temperatures above 700 °C [19]. The presence of gas evolution from inorganic matter in manure and the low
melting points of its constituents calls for the inclusion of ash chemistry into the modeling of high-temperature gasification and pyrolysis models [23]. Presently ash is usually considered inert in biomass gasification models.

Chicken litter is characterized by high nitrogen content due to the presence of protein and amino-acids compared to woodchips or other biomass feedstock [19]. The amine and carboxylic groups in the amino acids increase the complexity of gasification reaction with releasing CO$_2$ by decarboxylation and nitrogen compounds (such as NH$_3$, HCN, and HCNO as gases) which are NO$_x$ precursors and N-bound char and tar compounds. Studies on the evolution of nitrogenous species revealed NH$_3$ as the dominant nitrogenous gas species and rest of N contained in char at low temperatures (<600 °C) while at higher temperatures, HCN was the main nitrogenous gas product and high yields of tar containing nitrogen [19]. Nitrogenous gas species yield was found to reduce with increase in heating rate which may be due to an increase in N-bound tar yields. It is suggested that the presence of volatile nitrogen makes such feedstock a viable option for NO$_x$ control since pyrolysis of chicken litter releases nitrogenous gaseous species which separates most of the nitrogen bound in the feedstock, unlike coal.

Investigations on fixed-bed air gasification of chicken litter and coal blends found no significant effect on the produced syngas heating value and demonstrated the feasibility of chicken litter usage as gasification feedstock [24]. Kinetics of ‘auto-gasification’ based on using the ultimate analysis of the sample to conjecture that the amount of carbon can be gasified by the amount of feed bound O$_2$ in the sample in O$_2$
gasification [25]. They also indicated that a share of carbon reacts to hydrocarbons while another share participates in water gas reaction which leads to scarcity of carbon to react with fuel bound O₂ causing further oxidation to form CO₂. Catalytic steam gasification of chicken manure in a fluidized bed at temperatures around 600 °C revealed an increase in H₂ content and total gas yields with the addition of Ni-Al₂O₃ catalyst [26]. Super-critical gasification of chicken manure was also investigated [27,28]. Literature regarding the parametric effects in gasification of poultry manure is limited although, abundant literature is available on such studies using biomass.

The present paper investigates the pyrolysis and O₂ gasification of chicken manure at temperatures ranging from 600 °C to 1000 °C in steps of 100 °C with a specific focus on the evolution of product gas yield and composition. Product gas parameters such as gas composition, yield of H₂, gaseous CₙHₘ, and heating value are evaluated to reveal the effects of temperature and O₂ content in the gasifying agent. The data obtained augments to the fundamental knowledge of syngas evolution from chicken manure gasification which assists in the design of gasifiers as well as validation of computational models and model development for high-ash, low-energy content feedstocks containing significant amounts of fuel bound nitrogen.

2. Experimental

2.1. Reactor Facility

A schematic diagram of the laboratory scale semi-batch reactor facility used for investigating the pyrolysis and gasification behavior of chicken manure is given in Fig. 1.
N₂ of known flow rate was used as a tracer gas for all the experiments that helped to quantifying the amounts of various gas species evolved. Pyrolysis was investigated by flowing only N₂, while two different flow rates of O₂ in N₂ were used to examine O₂ gasification. The gasifying medium along with N₂ was passed through a preheating zone consisting of a furnace that was set to a temperature equal to that of the main reactor. Preheated mixture of different gasification agent gases was then allowed to enter the main reactor furnace located immediately downstream of the preheating furnace where it reacted with the chicken manure sample in quasi-isothermal conditions. The sample was filled in a quartz sample holder and inserted through the quick disconnect coupling after the reactor reached the set-point temperature. Time was counted starting from the time of sample insertion into the reactor.

Evolved syngas mixture from the reactor was first condensed in an ice followed by a water bath to remove tar and moisture. A portion of the tar-free syngas was sent through sampling line while the remaining (excess) gases were vented out through the exhaust duct after dilution. Moisture was removed using anhydrous calcium chloride in a desiccant. A majority of the syngas yield was evolved during first 5 minutes so that the product gas mixture samples were collected in sampling bottles for each minute to determine the evolutionary behavior of syngas yield. After the first 5 minutes, sampling time was at longer time duration so that gas sample was directly introduced into the gas chromatograph for the remaining duration of the reaction. Micro gas chromatograph was used to determine mole fractions of evolved gas species which can analyze samples at a sample rate of approximately three minutes during continuous acquisition. Micro GC was
calibrated to quantify \( \text{H}_2 \), \( \text{CO}, \text{CO}_2 \), \( \text{CH}_4 \), \( \text{C}_2\text{H}_6 \), \( \text{C}_2\text{H}_4 \), \( \text{C}_2\text{H}_2 \), \( \text{N}_2 \) and \( \text{O}_2 \) to an accuracy to \( \pm 0.1\% \).

2.2. Operating conditions

Chicken manure used for the present investigation was a dried chicken manure fertilizer sample in granular form. The ultimate and proximate analysis along with the higher heating value (HHV) [29] of the sample is given in Table 1. Tracer gas flow of 2.1 slpm of \( \text{N}_2 \) was used for all the experiments reported here at temperatures of 600 °C to 1000 °C. Three different \( \text{O}_2 \) gasification tests were analyzed having 0, 10, and 21% of \( \text{O}_2 \) concentration at fixed \( \text{N}_2 \) flow rate (2.1 slpm).

3. Results and discussion

This work focuses on examining syngas yield at different concentrations of \( \text{O}_2 \) in the gasifying medium, including the pyrolysis case. Product syngas is characterized by composition and yield of defined major gas species. The evolutionary behavior of syngas yield and its composition for different pyrolysis and gasifying conditions was studied. CO and \( \text{CO}_2 \) compositions were analyzed to determine the dominating reaction for different \( \text{O}_2 \) concentrations in the gasifying medium. Hydrocarbon evolution (\( \text{CH}_4 \) and \( \text{C}_2 \)) was compared to investigate the effect of \( \text{O}_2 \) and reaction temperature on the thermal cracking reactions.

3.1. Evolution of Product gas flow-rate
Figures 2–4 show the evolution of various components in the total syngas yield which includes H₂, CO, CH₄, CO₂, C₂H₆, C₂H₄, and C₂H₂. Temperature has a positive and dominating effect on the net yield of syngas. An increase in temperature enhanced the conversion to gas over char/liquid products, which supports the findings given in the literature on different feedstocks. The peaks are caused due to de-volatilization of the chicken manure sample. With the increase in temperature, the peaks of the flow rates shift towards lower residence times due to increased heating rates that enhance reaction kinetics of decomposition. The dependence of total gaseous flow rates on O₂ concentration in gasifying medium is more complex as it also depends on the reaction temperature. Pyrolytic yield corresponds to first 8-10 minutes during which the sample is converted into char and the duration of pyrolysis has low a dependence upon reaction temperature. The yield after 10 minutes corresponds to the char-gasification reactions which includes oxidation with composition dependent on Boudouard reaction equilibrium. While for the case of 10% O₂, the char gasification yield is less relative to 21% O₂; the char gasification is negligible in 0% O₂ case. This shows that the yield is from enhanced char gasification as also observed from the literature [30]. The char gasification reaction is relatively slow as observed from Fig. 3. The results show that while the de-volatilization took around 10 minutes, the gasification took around three times of that for pyrolysis time. This reaction was not observed for temperatures below 800 °C meaning that gasification reactions are important at higher temperatures (above 700 °C). Although the manure gasification can be broadly separated into regimes of de-volatilization and char-gasification, there exists an intermediate zone between these two phases, during which both the phases co-exist which can be observed by the difference
between 0% and 21% cases as seen from a partial overlap of de-volatilization and char-gasification, compare Fig. 2 with Fig. 4.

3.2. *Product gas composition*

The evolution of dry gas composition in the syngas without the tracer gas is presented in Figs. 5–7. During the first 8-10 minutes, which is a thermally transient period until the sample reaches thermal equilibrium with the reactor, the evolution of CO$_2$ is the dominant constituent, see Fig. 5. From Figs. 5 and 6, the dominance of CO$_2$ during early period is consistent and it can be attributed to both the decarboxylation of amino acids, acetic acid and other carboxylic groups generated during lignocellulose decomposition. After the decarboxylation and when the temperature of the sample exceeds 500 °C, the sample starts breaking other CH, CO, and C=O groups to generate CH$_4$, CO$_2$, CO, and other gases [31]. Pyrolytic water [19], which is released during this process, also accounts for H$_2$ production through reforming of hydrocarbons apart from thermal cracking. The evolution of C$_2$ hydrocarbon gases is similar to CH$_4$, through pyrolysis of higher hydrocarbons and other alkyl groups. Results shown in Fig. 7 for 0% O$_2$ at 1000 °C case shows that the initial period is dominated by syngas (H$_2$ and CO) as opposed to CO$_2$ unlike the other cases examined here. This can be attributed to a very high heating rate of the sample and cracking hydrocarbons groups to be faster than carboxylation. After the initial pyrolysis, the gases evolve according to the slow char gasification. Examination of Fig. 5 reveals that amount of H$_2$ produced for the 0% O$_2$ case is considerably higher than the evolved CO$_2$, although the total yield is almost negligible after the pyrolytic stage. Comparing Figs. 5–7, such behavior is observed at all
the investigated temperatures. Low CO₂ content in the syngas can be attributed to the absence of sufficient amounts of O₂ to result in high H₂ yield. The presence of O₂ at 1000 °C, the high concentration of CO₂ can be due to the oxidation of some of the hydrocarbons evolving during this early period.

Comparing the syngas composition during char gasification for different O₂ concentrations show significant differences in the mole fractions of H₂, CO, and CO₂. From Figs. 8–10, at increased temperatures, the observed increase in CO concentration is attributed to the Boudouard reaction that is driven towards dry gasification portion of char. This dependence is significant only for 10% and 21% O₂ cases, compare Figs. 5, 8, and 11. The H₂ content in syngas decays to zero during char gasification for these two cases, unlike for 0% cases in which H₂ concentration only stabilized (i.e., no increase). This absence of H₂ corresponds to the presence of excess O₂ to oxidize H₂ and thus a decrease in H₂ content. Figures 9 and 12 reveals that the trend of H₂ evolution is similar to the evolution of CH₄ and C₂H₆, which means that the H₂ released for the O₂ content of 10% and 21% is mostly from cracking of hydrocarbons. Hydrocarbons formed thermally crack to give lighter hydrocarbons which decompose to generate H₂ as seen from Eq. (5). Comparing the results from Figs. 10 and 13, increase in O₂ content in the gasifying medium reduced the CO content in syngas during char gasification implying more char oxidation than char gasification that leads to more CO₂. Temperature and O₂ concentrations play a major role in the CO and CO₂ evolution during gasification. For low temperatures and high O₂ content, CO₂ is favored over CO.
3.3. Cumulative energy yield

The energy yield (total heating value of evolved gas) was calculated to investigate the effect of gasifying conditions. As seen from the Fig. 13, the effect of an increase in temperature was to increase the heating rate for an effective breakdown of hydrocarbons into gases which increased the gaseous hydrocarbon content and gasification kinetics to increase the energy yield. At lower temperatures, more tar was formed which cannot be quantified by microGC. A notable feature from the comparison between different O\textsubscript{2} content in gasifying medium is the differences in energy yield with an increase in temperature between 0\% case (pyrolysis case) and the other gasification cases. For 0\% case, the total energy yield for temperatures lower than 1000 °C is lower compared to the 21\% O\textsubscript{2} case. This is due to the dependence of effective pyrolysis of the sample (majorly thermal decomposition for 0\% case), while for the 10\%, and 21\% O\textsubscript{2} gasification cases, the presence of excess O\textsubscript{2} provided improved char gasification even at lower temperatures. The release of more CO compared to CO\textsubscript{2} at higher temperatures increased the energy yield, but the increase in energy yield with temperature is highest for 0\% case. At 1000 °C, it can be seen that the energy yield of 10\% case is the lowest as compared to other temperatures. This observation reveals that the dependence of O\textsubscript{2} content on total energy is not simple because for pyrolysis (0\%) case, the high energy yields due to efficient gaseous hydrocarbon yield and thermal cracking of hydrocarbons to H\textsubscript{2} which has high heating value, while for 21\% case the increase is due to increase in CO content. For the 10\% case, the energy yield is lower because the O\textsubscript{2} content is relatively high to oxidize the tar content and the increase in CO yield is lower for this case compared to 21\% O\textsubscript{2} because of low O\textsubscript{2} content. This infers that partial oxidation of sample to produce
CO is efficient at lower temperatures, and if higher temperatures are used, O\(_2\) content in the gasifying medium must be controlled to make it comparable to pyrolysis condition.

### 3.4. Residue from gasification

Figure 15 shows a comparison between the chicken manure sample before and after gasification with non-zero O\(_2\) (gasification cases). An important parameter that needs to be considered here is very high ash content in chicken manure compared to any major biomass sources. This high ash content with low fusion temperatures calls for better gasifier coatings with corrosion resistance. This ash produced can be utilized as a fertilizer, which means the gasifiers built for chicken manure gasification should account for the collection of high volumes of ash in their design with due consideration to corrosion and deposition resistance. This also reveals better material destruction capabilities from gasification to reduce the waste products into 80% gaseous fuels and 20% fertilizers by wt. neglecting the CO\(_2\) production compared to composting which is used for only as fertilizer and its over-usage can cause eutrophication of groundwater.

### 3.5. Error Analysis

The accuracy of the calibrated gas compositions from the microGC was estimated be 0.1%. The data uncertainty of flow rates and composition of product gas species lies within the bounds of the symbol sizes used in the figures.

### 4. Conclusions
The pyrolysis and gasification of chicken manure at two different O₂ concentration in air at different isothermal temperature conditions have been examined. The results provided detailed information on the evolutionary behavior of syngas composition and yield, energy yield and residual material. The evolution of syngas flow rates suggests increased gaseous products yield with the increase in temperature and O₂ content in the gasifying agent. The quality of gases produced was determined from the chemical composition of the gases evolved. Higher O₂ content in gasifying medium produced higher CO₂ content in the syngas at low gasification temperatures. However, at higher temperatures (more than 800 °C) CO₂ decomposed into CO by reaction with the char residuals. The gaseous evolution occurred in approximately two stages. They included rapid decarboxylation and cracking of hydrocarbons followed by gasification of residue char formed after de-volatilization. The cumulative energy yields showed that gasification by partial oxidation produces more energy compared to pyrolysis and very high temperatures are required for pyrolysis to match the energy yield from air gasification. The results also showed that at higher temperatures, due to better conversion kinetics, higher O₂ content in gasifying medium assists to enhance carbon oxidation. At higher temperatures, due to Boudouard reaction equilibrium, higher CO is favored – more O₂ content produces more CO₂ which then reacts with char to enhance CO yield. Residues after gasification were ash (mineral matter) that is high in chicken manure compared to other biomass feedstocks so that better and corrosive resistant hardware will be required to handle high ash amounts that also has low melting points to provide issues of fouling and deposition on heat exchange equipment in chicken-manure-fired feedstock systems.
Acknowledgments

The support from the Office of Naval Research (ONR) in this research is gratefully acknowledged.

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Figure captions:

Fig. 1. A schematic diagram of the semi-batch reactor

Fig. 2. Product gas evolution for pyrolysis case (0% oxygen gasifying medium)

Fig. 3. Product gas evolution for 10% oxygen in gasifying medium

Fig. 4. Product gas evolution for 21% oxygen in gasifying medium

Fig. 5. Evolutionary behavior of gas species mole fraction at 800 °C for 0% O2 concentration (pyrolysis case)

Fig. 6. Evolutionary behavior of gas species mole fraction at 900 °C for 0% O2 concentration (pyrolysis case)

Fig. 7. Evolutionary behavior of gas species mole fraction at 1000 °C for 0% O2 concentration (pyrolysis case)

Fig. 8. Evolutionary behavior of gas species mole fraction at 800 °C for 10% O2 concentration in air gasification case

Fig. 9. Evolutionary behavior of gas species mole fraction at 900 °C for 10% O2 concentration in air gasification case

Fig. 10. Evolutionary behavior of gas species mole fraction at 1000 °C for 10% O2 concentration in air gasification case

Fig. 11. Evolutionary behavior of gas species mole fraction at 800 °C for 21% O2 concentration in air gasification case

Fig. 12. Evolutionary behavior of gas species mole fraction at 900 °C for 21% O2 concentration in air gasification case

Fig. 13. Evolutionary behavior of gas species mole fraction at 1000 °C for 21% O2 concentration in air gasification case

Fig. 14. Cumulative energy yield from the syngas at different temperature and O2 content

Fig. 15. Original chicken manure sample (left) and gasified residue sample (right)
Figure 2
Figure 3

Product gas flow rate (g/min) vs. Time (minutes)

- 600 °C
- 700 °C
- 800 °C
- 900 °C
- 1000 °C
Figure 4

Product gas flow rate (g/min) vs. Time (minutes) for different temperatures:
- 600 °C
- 700 °C
- 800 °C
- 900 °C
- 1000 °C
Figure 6
Figure 7

The diagram shows the mole fraction of various gases over time in a chemical process. The x-axis represents time in minutes, and the y-axis represents the mole fraction (%). The lines indicate the behavior of different gases:

- Red line: H₂
- Green line: CH₄
- Blue line: CO
- Orange line: CO₂
- Black line: C₂H₅

The diagram illustrates the transient behavior of these gases as the process progresses.
Figure 8
Figure 9
Figure 10
Figure 12
Figure 13
Figure 14
<p>| | |</p>
<table>
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<tr>
<td><strong>Proximate Analysis (wt. % dry)</strong></td>
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<tr>
<td>Volatile content</td>
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<tr>
<td>Ash content at 550°C</td>
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<tr>
<td>Fixed carbon</td>
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<tr>
<td>HHV (in MJ/kg)</td>
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Table 1. Proximate and Ultimate analysis of chicken manure [33]