Original Research Article

Thermogravimetric Characterization of Cattle Manure as Pyrolysis and Combustion Feedstocks

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A B S T R A C T

K e y w o r d s Cattle manure, Biomass, Thermogravimetric analysis	Cattle manure is a biowaste with bioenergy recovery potential for heat and power generation. However, there is less kinetics data available in literature to date. In this work, a kinetic study of the pyrolysis process of cattle manure is investigated through the use of thermogravimetric analyses. The samples were heated over a range of temperature from 15 to 800 ^{0}C with heating rates of 10 ^{0}C /min. The weight loss was measured by a thermogravimetric analyser in an inert atmosphere. Experimental results showed that an animal manure pyrolysis process can be divided into three stages: dehydration, pyrolysis, and carbonization. These stages may produce differed features on end residuum, weight loss rate, and peak features, as indicated by thermogravimetric (TG) and derivative thermogravimetric (DTG) curves. The thermal gravimetric (DTG) thermogram shows that the highest reaction rate occurred at between 200 and 450 °C where the devolatilisation process was initiated to overcome the activation energy barrier of the manure. Results showed four steps for both the pyrolysis and the combustion reactions, with the second step being the most critical one and during which most thermal decomposition of cellulose, hemicelluloses, starch and protein occurred.

Introduction

The emergent need for energy sources alternative to fossil fuels led to a huge investigation to find new and renewable energy resources. The share of biomass in meeting world energy demand is currently one of the largest, if compared with the other renewable energy resources, e.g. solar, wind, hydropower, and geothermal energy (Huang and Yuan, 2015). Biomass can be any type of organic material derived from plant, microorganisms, and animals. In recent years, the use of animal manures as energy resources has attracted promising attention at the global and national scales, since it could not only provide a renewable source of energy but also alleviate pressures on the environment pollution (Shen *et al.,* 2015; Cao *et al.,* 2016). Conversion of animal manures

into the energy can be made mainly by two methods. One is biochemical conversion and the other is thermochemical conversion. In the biochemical conversion biological catalysts and organisms are used to obtain the energy rich products from the manures while in the thermochemical conversion heat and chemical catalysts are used to produce energy. One of the major challenges in the biochemical conversion is the rapid and complete conversion of manures into the energy efficiently. Compared with biochemical conversion, thermochemical conversion has higher efficiency by the shorter reaction time required and the superior ability to degrade the animal manures (Cao *et al.,* 2014; Mezzullo *et al.,* 2013; Thanapal *et al.,* 2012). Thermochemical conversion can be sub divided further into combustion, gasification, and pyrolysis. Combustion is not an environment friendly method attributed to generating too much CO_2 and some other hazardous gas, and gasification is high-cost for consuming a large number of energy to maintain high operating temperature. However, pyrolysis causes less pollution emission and has a reasonable cost and simple operation. Consequently, among these technologies, pyrolysis has been accepted as a potential method for the disposal of animal manures (Lawrence *et al.,* 2009; Baniasadi *et al.,* 2016; Heidenreich *et al.,* 2015). During pyrolysis, the manures are decomposed in an oxygen deficient environment to produce gas, liquid and char. The pyrolysis gas can be directly used as a gaseous biofuel for power generation or converted to other products by a syngas based route, and the liquid fraction has a potential use as biofuel after appropriate upgrading (Ertaş *et al.,* 2010; Font-Palma 2012). The char is the solid residue remaining after the pyrolysis process, which is mostly composed of carbon and inorganic materials (ash), and which can have further application as soil amendment and for activated carbon

production (Qian *et al.,* 2007; Uzoma *et al.,*2011; Ro *et al.,* 2014). In addition, pyrolysis is not only as a single process, but also as the first step to combustion and gasification. Therefore, the knowledge on the pyrolysis conversion of animal manures and then the kinetic and thermodynamic parameters in the conversion process are greatly important for the proper design of a pyrolysis reactor in large scale conversion process. Thermogravimetric analysis (TGA) is an important approach to address the pyrolysis process, and has been proved to be one of the best methods for conducting the kinetics of pyrolysis (Damartzis *et al.,* 2011). By analyzing the TGA curve, one can understand the change process of samples with the increase of temperature and assess its thermal properties.

Materials and Methods

Cattle Dung in this study was collected from dairy farm of GB Pant University of Agriculture and Technology, Pantnagar as the raw material. The moisture content of initial collected dairy manure was more than 50%. The cattle dung samples were dried in a furnace at 120 °C for 24 h to remove moisture before being grinded into fine particles. During pretreatment, samples were dried, ground and sieved. The particle size of the manure sample was less than 0.5 mm.

Proximate analysis

Proximate analysis was carried out to determine the moisture, volatile matter, fixed carbon and ash content of cattle dung. Moisture content, volatile matter and ash content were determined according to ASTM D 3173, ASTM D 3175 and ASTM D 3174 protocols, respectively on dry basis. Volatile matter was determined by establishing the loss in weight resulting from heating a coal or coke under rigidly controlled conditions. For

analysis, 1 g of sample was placed in ceramic crucible with a cover which fitted closely enough so that the carbon did not burn away and kept in a muffle furnace (make: Sonar, Associated Scientific Technology, New Delhi) at 950°C for 7 min. The weight loss was calculated which represented the volatile matter. Ash was determined by weighing the residue remaining after burning the biomass samples under rigidly controlled conditions. In a similar way, 1 g of sample was kept in muffle furnace at 750°C for 4 h and remaining weight was the ash content. The fixed carbon content was calculated by difference of weight.

Moisture Content

Moisture is defined as the amount of liquid per unit mass of the wet solid. According to the standardized test method for moisture analysis (ASTM D 3173), 1 g of the sample was retained in a hot-air-oven at 105°C for 1 h. Next, the oven-dried sample was weighed. Moisture content was estimated as

$$\text{Moisture content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \ (1)$$

Where W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample (g) and W_3 is the weight of the empty crucible + sample after drying (g).

Volatile matter

Volatile matter determines the percentage of the gaseous products which is released under the specific conditions of the test in the analysis of the sample free from moisture content. As per the standard test method for volatile matter analysis (ASTM D 3175), the oven-dried sample enclosed in a crucible with a lid was placed in a muffle furnace, and maintained at 950° ± 20°C for 7 min. Therefore, the crucible was first cooled in the

surrounding air and then in a desiccator, and weight loss was calculated

$$\text{Volatile matter (\%)} = \frac{W_3 - W_4}{W_2 - W_1} \times 100 \ (2)$$

where W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample (g) and W_3 is the weight of the empty crucible + sample after oven-drying (g). W_4 is the weight of the empty crucible + weight after heating in muffle furnace (g).

Ash content

Ash content was determined by weighing the residue remaining after burning the coal under rigidly controlled conditions of sample weight, temperature, time, atmosphere and equipment specifications.

According to the standardized test method for ash content analysis (ASTM D 3174), the remaining sample residue after volatile matter test, was heated in the muffle furnace at 700° ± 50°C for 4 h. The ash content in each sample was estimated using the formula

$$\text{Ash content (\%)} = \frac{W_5 - W_1}{W_2 - W_1} \times 100 \ (3)$$

where W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample, after volatile matter (g) and W_5 is the weight of the empty crucible + ash left in the crucible (g).

Heating value

The Heating value of cattle dung is determined using the bomb calorimeter, according to the standard test method for calorific value (ASTM E711-87). The bomb calorimeter consists of a solid cylindrical, stainless-steel bomb inside which the

combustion of fuel takes place. Less than 1 g of fuel sample is placed in the crucible, a fuse wire is used to ignite the fuel and the bomb filled with oxygen gas at 25–30 atmospheric pressure. The electrode was connected to electrical supply and initial water temperature was noted. After combustion of the fuel sample, the increase in water temperature was noted for determination of calorific value. The calorific value determined by the formula

$$\text{Calorific value} \left(\frac{\text{kcal}}{\text{kg}}\right) = \frac{W \times \Delta T}{M} \quad (4)$$

where M is the Mass of fuel placed in the crucible (g), W is the water equivalent of the bomb calorimeter or heat capacity (cal/°C), $\Delta T = t_2 - t_1$, where t_1 is the initial temperature of water in the calorimeter (°C) and t_2 is the final temperature of water in the calorimeter (°C).

To determine the heat capacity of the bomb calorimeter, wherever nichrome fuse wire and cotton thread are used simultaneously, pure benzoic acid is used in the bomb.

In this case, cotton thread was also used along with nichrome fuse wire. The heat capacity for nichrome fuse wire is 333.68 cal/g and for cotton thread it is 4180 cal/g(Parray,2012)

$$W = \frac{M \times H + (E_w) + (E_t)}{\Delta T} \quad (5)$$

where W is the water equivalent of the bomb calorimeter (2283.32 cal/°C), M the mass of the test sample, ΔT the rise in temperature, E_w the correction of heat of combustion for nichrome fuse wire and E_t is the correction of heat of combustion for cotton thread

$$E_w = M_w \times H_w, \quad (6)$$

where M_w is the mass of nichrome fuse wire and H_w is the heat capacity per gram of nichrome fuse wire

$E_t = M_t \times H_t$, where M_t is the mass of cotton thread and H_t is the heat capacity per gram of cotton thread.

Elemental analysis

Elemental analysis (EA) refers to the process of determination of mass fraction of carbon, hydrogen, nitrogen and sulphur to find out the structure of an unknown compound. EA of cattle dung was carried out in a CHN analyzer. Oxygen content was determined by difference of weight considering that the whole biomass was made of C, H, N, O and ash.

Composition analysis

Hemicelluloses, lignin and cellulose content of cattle dung (Pinusroxburgii) were determined by direct methods described by Lin *et al.,* (2010). Before determination of cellulose, hemicelluloses and lignin, extractives in cattle dung were determined using a Soxhlet extractor for 5.0 g of biomass sample by adding 300 ml acetone. Sample was kept in a cellulose thimble placed inside the receiver of Soxhlet apparatus and acetone was poured into the heating flask. Acetone was heated at a temperature of 90°C for 2h during which many cycles of extraction were repeated. The extracted sample was then oven dried at 105°C to constant weight. The extractives content was taken as the difference in weight between the raw biomass and extractive-free biomass.

In order to determine hemicelluloses content, 30 ml 0.5 M NaOH solution was added to 1 g of extractive free biomass in a 100 ml erlenmeyer flask and heated at 80°C for 3.5 h as hemicellulose is soluble in 0.5 M NaOH

solution. It was then filtered and washed C in a hot air°with de-ionized water until pH reached 7.0. The residue was dried at 105°C in hot air convective oven for two hours. The difference between the sample weight before and after this treatment was the hemicellulose content. The lignin content was determined by adding 30 ml, 98 wt.% H_2SO_4 to 1 g of dried extractive free biomass in a 100 ml erlenmeyer flask. The sample was kept at ambient temperature for 24 h with periodic shaking for complete hydrolysis of cellulose and C for 1 h in a water bath. Residue washemicellulose. After that it was boiled at 100 filtered through a filtering crucible.

The residue was then washed with de-ionized water C. Obtained content was incinerated in°until neutral pH and dried to constant weight at 105°C for ash content. The difference weight after ash deduction from a muffle furnace at 575 residue was taken as lignin content. The cellulose content was calculated by difference, assuming that extractives, hemicellulose, lignin, ash, and cellulose are the only components of the entire biomass.

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hemicellulose, lignin, ash, and cellulose are the only components of the entire biomass

Thermogravimetric analysis

Thermogravimetric analysis (TGA) was applied to investigate the thermal degradation behaviour of cattle manure. Thermogravimetric analysis (TGA/DTG) of cattle dung was carried out using a Netzsch STA (Model: 409 PC Luxx, Selb, Germany) under N_2 atmosphere with a flow rate of 50 ml min^{-1} and heating rate of 10°C. Diameter of samples of less than 0.4 mm was used with an initial mass of approximately 10 mg. Cattle manure sample with size of 2 ± 1 mg was weighted and placed on the holder for analysis. Prior to the test, the chamber was purged by nitrogen (Megamount: 99.8% purity) to ensure inert environment for pyrolysis. The sample, placed on an aluminium holder, was heated from room temperature to 800 °C with nitrogen carrier gas flow of 20 ml/min. The weight loss and temperature change during the process was recorded in thermogravimetric (TG) and derivative thermogravimetric (DTG) formats. The substance that remained after the completion of the pyrolysis process was assumed to be ash or char originated from the cattle manure sample.

Properties of feedstock

The physicochemical properties of the cattle obtained through the proximate and ultimate analyses are shown in Table 1 in weight percentage dry basis. The carbon content in the manure is approximately 40.89 wt%. The combined nitrogen content in the manure is less than 2%, which is desirable from the point of view of bio-oil and synthesis gas production (Friedl.,2005)The determined calorific value of the cattle manure is 17.70 MJ/kg, of which the value is comparable to the data as reported by Nanda *et al.,* [28].

Table.1 Properties of cattle manure

Proximate Analysis (wt%)	
Moisture Content	5.6
Volatile matter	70.5
Ash content	14.2
Fixed carbon	15.3
Composiotion Analysis (wt%)	
Cellulose	28.1
Hemi cellulose	11.9
Lignin	12.9
Ultimate Analysis (wt%)	
C	40.89
H	6.72
N	1.51
O	50.12
HHV(MJkg^{-1})	17.70

Fig.1 Thermogravimetric (TG) and Derivative Thermogravimetric (DTG) Curves of cattle dung at 10^0C min^{-1} heating rate
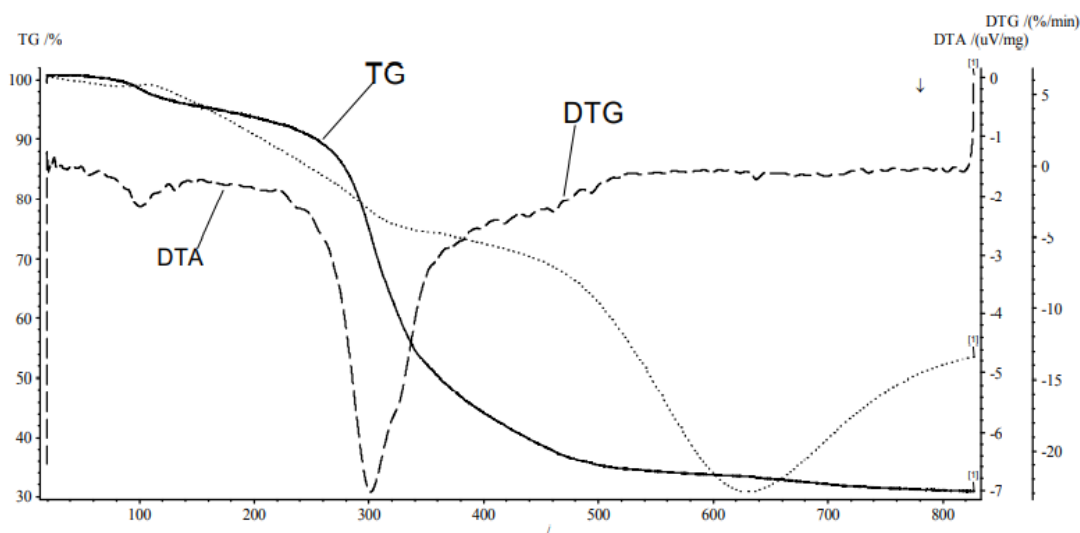
The low heating value of the manure is largely attributed to the high level of oxygen and ash content of 50.12 %wt and 14.2 wt% respectively. The proximate analysis shows that cattle manure still contains 6.5% of moisture despite being pre-dried in oven, most probably due to the remaining moisture that is present in the cellulose. The volatile matters in the sample is about 70.5 wt%, while the fixed carbon is 15.3wt%. The high level of volatile matter indicates the tendency of reactive agents being produced during thermal degradation process. Manures typically contain high organic content and low cellulosic composition owing to the bacterial and chemical reactivity along animals digestive track (Yuan et., 2017;). Cellulose, hemicelluloses and lignin content

are were found as 28.1, 11.9, 12.9% respectively.

Pyrolysis characteristics

Thermogravimetric analysis (TG) and differential thermogravimetry (DTG) curves for the 10°C min^{-1} heating rate under a N$_2$ atmosphere are shown in Figure 1. In the context of TGA and DTG curves, the pyrolysis process with anascending temperature was divided into three stages: drying (0-200°C), main pyrolysis (200-450°C), and carbonization (450-800°C). Cattle manure had TGA plots similar to those of corn Residue (Fei Yu, *et al.,* 2006), mustard straw and stalk (S. Maiti, *et al.,* 2007), and microalgae (Peng W, *et al.,* 2001). However, manimal manure in three stages had a temperature range that was different from other samples.. As indicated by the TG and DTG curves, they differed on end residuum, weight loss rate, and peak features. Pyrolytic properties are summarized in Table 1.

The profile of dairy manure weight loss exhibited four stages during the degradation process (Figure 1). During the first stage (from room temperature to around 200°C), the weight loss was 10–12% of the original weight. The moisture content of the dried manure sample was approximately 6.5%, as shown in Table 1. Therefore, although volatile compounds may have contributed to the weight loss, the major weight loss was mainly due to the evaporation of moisture during the stage I (Liu *et al.,* 2009). The weight loss between the temperatures of 200°C and 450°C was the major reaction area where most of the organic matter was lost. As two dips in the DTG curve were observed, this reaction zone was divided into additional two stages (stage II and stage III). In the second stage (200–310°C), around 35% of the original weight was lost. A sharp weight loss was observed in this stage— the highest

weight loss rate was reached at 10°C. Consequently, the second stage was considered to be the critical stage in the pyrolysis process (Domínguez, *et al.,* 2008). The weight loss during this stage results mainly from the thermal degradation of cellulose, hemicelluloses, protein, starch and microbial cell walls (Wu *et al.,* 2011). For the third stage (450–800°C), the mass loss rate was slower than for the second zone—about 27% of the original weight was lost. Lignin in dairy manure may contribute to this weight loss (Wu *et al.,* 2011) because the pyrolytic decomposition of lignin occurs between 300°C and 500°C. Hemicellulose decomposes at 250–300°C and cellulose at 300–350°C (Carrier *et al.,* 2011). Most of the starch and protein were lost during the second stage. The thermal degradation of lignin was reported to be slow and over a wide range (up to 800°C) (Huang *et al.,* 2011), which may have been due to the extremely wide temperature range of the activity of chemical bonds and functional groups in lignin (Wang *et al.,* 2009). The 6% weight loss during stage VI (500–800 °C) may have contributed to further charcoal devolatilization (Font-Palma, 2012). In the last stage, the weight loss rate became stable and near zero. The remaining solid residue at the end of pyrolysis was char (including ash and fixed carbon), which was 21% of the original mass. The ash content given in Table 1 was 23.89% higher than the pyrolysis residue content. Two reasons may explain above phenomenon. The first is the variety of the manure samples and the second is the traces of oxygen remaining in the thermal analyzer before experiment operation.

Combustion characteristics

TG and DTG curves for dairy manure oxidized in air with a heating rate of 10°C min^{-1} are shown in Figure 1. In combustion reactions, the air was sufficient for complete

combustion. Similar to pyrolysis, four stages were observed. The first stage (from room temperature to 200°C) resulted mainly from the evaporation of water (11% weight lost). The second oxidation zone, which was the most significant zone, ranged from 200°C to 450°C, and around 34% of the original mass was lost. The highest weight loss rate was observed around 300°C (the sharpest peak), which was a little higher than the temperature with the maximum weight loss rate during pyrolysis. Analogous to pyrolysis, weight loss in the second zone resulted from combustion of cellulose, hemicelluloses, protein and starch. The third oxidation zone, which started at 450°C and ended at 800°C, may have been due to the lignin oxidation (Wang *et al.,* 2011). The fourth oxidization zone may be explained by the further oxidization of char. The weight loss rate was slow with only 3% of the original weight being lost. At the end of the combustion, the remaining solid was ash, with a weight of about 19% of the initial weight.

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