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Product compositions from sewage sludge pyrolysis in a fluidized bed and correlations with temperature

Alejandro Jaramillo-Arango[a,*], Isabel Fonts[b,c], Farid Chejne[a], Jesús Arauzo[c]

[a] Grupo de Termodinámica Aplicada y Energías Alternativas, Facultad de Minas, Universidad Nacional de Colombia – Sede Medellín, Cr 80 N° 65-223 Medellín, Colombia
[b] Centro Universitario de la Defensa de Zaragoza, Ctra. Huesca s/n, 50090 Zaragoza, Spain
[c] Thermochemical Process Group (GPT), Aragon Institute for Engineering Research (I3A), Universidad de Zaragoza, Mariano Esquilor s/n, 50018 Zaragoza, Spain

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ABSTRACT

The increasing trend for the production of sewage sludge has made it necessary to evaluate thermal disposal alternatives, such as pyrolysis, gasification and combustion. In these processes, the devolatilization step or primary pyrolysis determines the chemical compounds that may ultimately react in the homogeneous and/or heterogeneous phase to form the final products. The effect of temperature on the pyrolysis of an anaerobically digested sludge was studied by means of chemical characterization of the gases, liquids and solids released in a fluidized bed reactor between 300 and 800 °C. It was observed that in mass fractions, the gases were mainly composed of CO and CO_2, while liquids were mostly pyrolytic water. Atomic species balances were performed to analyze the distribution of the different elements in each phase and as a result, the possibility of oxide reduction reactions in ash was found. Finally, the yields of char, H_2O, CO_2, CO, CH_4, H_2 and organic liquids were fitted to a cubic function of temperature.

1. Introduction

Demographic growth and urban densification have increased the need to implement wastewater treatment systems to mitigate the impact on natural resources. Nowadays, more than 75% of the total population in developed countries, Latin America and the Caribbean is living in urban areas and urban population growth was around 1.2% per year for the last decade [1]. Hence, the predictable future of sewage sludge production growth must be met with the necessary research of the alternatives for its disposal.

Thermal utilization of sewage sludge, through combustion, gasification and pyrolysis, has been studied as an alternative to agricultural use or landfill disposal, generating propitious options with regards to energetic, environmental and economic factors [2–7]. Pyrolysis is by one side a valorization treatment itself, and by other side, the first step of combustion and gasification. The advancement of these thermal processes by means of using modeling as a tool for analysis, design and scale-up for sewage sludge, allows us to confront the challenge of dealing with a complex and heterogeneous material [8–11] whose kinetic devolatilization and organic compounds released at high heating rates have not yet been studied. Pyrolysis or devolatilization is demonstrated to be a key step, affecting greatly the model results [12]. The modeling of the conversion of solid fuel in combustion or gasification systems needs a description of the composition of the volatile gases that leave a fuel particle of typical size in a fixed or fluidized bed during devolatilization [13] and these measurements are also required at high heating rates [12]. To develop a kinetic model that predict not only the solid and the volatiles generated but also the specific production of other products is an extremely complicated work. For this reason, pyrolysis is usually described simply either based on experimental measures or based on correlations developed also under a certain set of operational conditions [14].

In the case of coal, empirical relationships have been developed to describe the stoichiometry of the pyrolysis products according to the amount of volatiles in the proximate analysis [15] or to the temperature [16–20], and have been used in reactor modeling with either a fixed [21] or fluidized bed [22–25]. Also, in the area of lignocellulosic biomass [13,26–29] and residues [27], some works with the same conceptual approach to coal devolatilization have been published.

Abbreviations: Daf, dry ash free; GC, gas chromatograph; FID, flame ionization detector; MS, mass spectrometer; ICP-AES, inductively coupled plasma atomic emission spectroscopy.

* Corresponding author.
E-mail address: jaajaramilloar@unal.edu.co (A. Jaramillo-Arango).

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For the pyrolysis kinetics of sewage sludge, the most rigorous analyses have been focused on the consumption of the solid fraction without volatiles being released during characterization, and they were carried out in thermobalance (TGA) at low heating rates [30–36]. Studies of fluidized bed reactors under conditions close to the operation of real equipment have shown that the performance and properties of char, liquid and gases are mainly affected by temperature in the intervals of study (450–650 °C) [37,38] and empirical expressions to predict the yield and composition of gases [27] have been proposed, although the species present in liquids have been rarely correlated [39]. Moreover, in the case of sewage sludge, there are few works that correlate the pyrolysis results as a function of the operational conditions [27,37–39], the intervals of study of the operational conditions are short [37,40–42] and many times these correlations do not include all the most significant pyrolysis products [27].

Operating parameters of a fluidized bed such as the bed temperature, particle size, fluidization velocities, residence times of solids and gases influence pyrolysis products [37,38,43]. However, bed temperature is the most sensitive parameter for pyrolysis of a specific material [5,37,42]. From elsewhere, a detailed kinetic model that accounts for the species released during pyrolysis of various sludge at high heating rates and that also applies to the conditions of a fluidized bed requires a costly and complicated experimental effort. Consequently, the implementation of semi-empirical modeling of sewage sludge pyrolysis based on the bed temperature, in response to its decisive operational influence, it is considered an appropriate strategy to advance toward stricter modeling of thermochemical processes. This kind of models or even more simple approximations is commonly used as sub-models in the simulation of gasification and combustion [14,29].

The aim of this work is to develop an empirical model for the devolatilization stage of a high-ash sewage sludge, which predicts the amount of the main pyrolysis products released at temperatures among 300 and 800 °C, with a potential use as a sub-model to include in the modeling of pyrolysis, gasification and/or combustion. In order to do that, the products obtained from steady state pyrolysis experiments, carried out in a fluidized bed regime, have been quantified, characterized and checked by means of atomic species balances. Moreover, the values predicted by the models have been compared with literature experimental data.

2. Materials and methods

2.1. Materials

Anaerobically digested sewage sludge from a wastewater treatment plant located in Medellin (Colombia), which treats domestic and industrial discharges, was dried under ambient conditions, crushed, grinded and sieved to obtain uniform particles with sizes of $289 \pm 54\,\mu m$. Through experiments on a cold fluidizer, a bulk density of $0.66\,g/cm^3$ and a minimum fluidization velocity of 3.8 cm/s were measured.

Sewage sludge was characterized by proximate and ultimate analyses (Table 1). Ash composition was analyzed at facilities at the Instituto de Carboquímica (CSIC, Zaragoza, Spain) with the ICP-AES technique in a spectrometer, HORIBA Jobin Ybon 2000.

2.2. Experimental rig

Fig. 1 shows the experimental system used for pyrolysis runs. Experiments were carried out in a fluidized bed reactor made of stainless steel (AISI 310) with a reaction zone controlled by a lateral overflow with an inner diameter of 4 cm in the reaction zone, a height of 31 cm from the distributor plate and a freeboard of 7 cm in diameter and 22 cm in height.

Sewage sludge was introduced continuously to the fluidized bed reactor by means of a screw feeder. The reactor was operated at an atmospheric pressure with external heating provided by electrical furnaces and independent temperature control in the bed, the freeboard and the cyclone.

The system was continuously fed with a stream of nitrogen, guaranteeing three times the minimum fluidization velocity. For this, the gas density was corrected with temperature for each experiment and according to the data, the set point of the mass flow controller was modified. Entrained solids were separated from the gas stream using a cyclone and a hot filter.

Two ice condensers, an electrostatic precipitator and a cotton filter were used to collect the liquid. The flow of non-condensable gases was measured with a volumetric counter and their composition was analyzed by gas chromatography (GC) using a Micro-GC Agilent G2801A, provided with calibration conditions for identification and quantification of CO, CO_2, CH_4, H_2, N_2, C_2H_4, C_2H_6,C_2H_2 and H_2S.

2.3. Pyrolysis experiments

For each test, the reactor was initially fed with a charge of about 150 g of char prepared at 100 °C above the experimental temperature, in order to prevent the non-steady state occurring due to the change in the bed composition at the beginning of the experiment if it is initially fed with sand. The experiments were performed according to the following procedure:

1. Once the whole installation is assembled, the existence of leakage is verified by checking that the controlled volumetric flow introduced in the cold reactor by means of a flow controller is the same to that measured at the exit of the plant using a volumetric counter. During these measurements and also during the whole experiment, two-thirds of the nitrogen flow is introduced by the distributor plate and the rest through the screw feeder for assisting entry of the solids into the bed.
2. The reactor is heated to the experimental temperature in the reaction zone, freeboard and cyclone.
3. Upon reaching the experimental temperature, it is waited for 15 min to start feeding sewage sludge at a rate of 3 g/min.
4. Throughout the test, the composition of gas products is recorded by the Micro-GC. Moreover, char and liquid products are collected, corresponding to char in the char pot, the bed and the cyclone, and liquid in the two ice-cooled condensers and the electrostatic precipitator.
5. After 90 min, the power is suspended and the reactor is allowed to cool down.
6. Once the run has finished and the system is cold, the solids and liquids are quantified by weighing of the vessels where they have been collected.
7. The three liquid vessels are washed and liquids recovered using a known amount of methanol, reaching a liquid recovery of around 90% in mass.

2.4. Product characterization

The amount of char and liquid generated are determined by the mass difference of their recipients and vessels at the start and the end of the experiment. Proximate analyses were carried out on the samples of char collected from the char pot and from the bed. Negligible differences were found between them.

The liquid product fractions collected with methanol from the three vessels were stored jointly in a bottle and deposited in a fridge at 3–5 °C until they was analyzed. The water content of the