Metadata for: Data for: Effect of feedstock moisture content on biochar yield and carbon recalcitrance during autothermal pyrolysis.

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Abstract

This study investigates the influence of feedstock moisture content on biochar yield and carbon recalcitrance during autothermal pyrolysis of anaerobic digestate. Biochar is a product of pyrolysis that can serve as a carbon sequestration agent – the fraction of recalcitrant carbon in the biochar contributes to its stability and allows it to store carbon in the soil long term. Digestate samples with moisture contents of <5%, 10%, 20%, 30%, and 40% were tested in duplicate. The resulting product distribution and composition of biochars produced were analyzed for yield and carbon stability. Increasing feedstock moisture content had no significant effect on biochar yields produced but significantly affected the yield and composition of the bio-oil and noncondensable gases (NCGs). The Van Krevelen Diagram and the volatile matter-to-fixed carbon (VM/FC) diagram were used to evaluate biochar's composition to determine its degree of carbonization and stability. Analysis of biochar composition indicates that an increase in moisture content had no significant effect on biochar composition, and thus, its carbon recalcitrance.

Keywords

autothermal pyrolysis, bio-oil, biochar, digestate, fluidized bed reactor, moisture content

Award(s)

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Maintenance

Update frequency: not planned

This dataset is complete and no maintenance is planned.

Geographic coverage

No specific geographies are presented in this dataset.

Temporal coverage

begin date	end date
2024-12-01	2025-12-31

Taxonomic coverage

Kingdom	Phylum	Class	Order	Family	Genus	Species
Animalia	Chordata	Mammalia	Artiodactyla	Bovidae	Bos	Bos taurus

Methods

Related publication

These methods are based on those published in: McMahon, Madeleine E. 2025. “Effects of Feedstock Moisture Content on Biochar Yield and Carbon Recalcitrance During Autothermal Pyrolysis of Anaerobic Digestate.” In ProQuest Dissertations and Theses. M.S., Iowa State University. 3298629944. Dissertations & Theses @ Iowa State University; ProQuest Dissertations & Theses Global. https://www.proquest.com/dissertations-theses/effects-feedstock-moisture-content-on-biochar/docview/3298629944/se-2?accountid=10906.

Preparation of Pyrolysis Feedstock

Solid anaerobic digestate was obtained from a commercial dairy farm in Eastern Iowa, USA. The facility operates two continuously stirred tank reactors (CSTR) each with a working volume of 3700 m3. The inputs for the CSTRs include cow manure, off-farm municipal food waste, and corn stover. After removal from the digesters, the digestate is mechanically dewatered to produce a liquid and a solid fraction. For this study, the solid fraction was collected, and its moisture content was measured in triplicate using a Sartorius Moisture Analyzer, yielding an average of 70 ± 0.7 wt. % on a wet basis (w.b.).

The collected digestate was oven-dried at 105˚C until the moisture content was reduced to below 10 wt. % w.b. The dried material was then milled using a knife mill to guarantee a uniform particle size of 1/16”. The milled digestate was stored in sealed 5-gallon containers indoors. Ultimate and proximate analyses of the dried digestate are provided in Table 1.

On the day of the pyrolysis trials, the digestate was rehydrated to the target moisture content using deionized (DI) water. A syringe pump connected to an Erweka paddle mixer delivered water at a rate of 100 mL/min onto the digestate while the paddle mixer ensured uniform distribution of moisture. Target moisture contents of <5%, 10%, 20%, 30%, and 40% were prepared and tested.

Fluidized Bed Pyrolysis System

The experiments were conducted using a pilot-scale bubbling fluidized bed reactor integrated with a biomass feed assembly, two hot gas cyclones, a multistage condenser train (including electrostatic precipitators and shell-and-tube heat exchangers), and a micro gas chromatograph (MicroGC).

The feed system includes a metering auger for consistent biomass delivery and an injection auger which introduces material directly into the reactor bed. Prior to each experiment, the feed system was calibrated to ensure a biomass feed rate of 500 g/hr.

The 3.8 cm diameter fluidized bed reactor consists of a plenum, fluidized bed, and freeboard section, all enclosed by ceramic clamshell heaters to minimize parasitic heat loss. Within the reactor bed, there is 250 g of a -16+30 mesh quartz sand which is fluidized with 20 standard liters per minute (SLPM) of a preheated nitrogen/air mixture at 500˚C. The gas flow rate is approximately twice the minimum fluidization rate for the bed material, ensuring that the bed enters a bubbling fluidization regime. The air-to-nitrogen ratio was adjusted for each moisture content tested to target a constant reactor temperature of 500˚C.

Pyrolysis vapors and biochar exiting the reactor passed through two hot gas cyclones for biochar removal, followed by a four-stage condenser train which separated the pyrolysis vapors according to molecular weight. Stage fractions (SFs) 1 and 2 (“heavy ends”) contain higher molecular weight anhydrosugars and phenolic compounds. SFs 3 and 4 (“light ends”) primarily consist of aqueous-phase components such as ketones, furans, and carboxylic acids.

SF1 was condensed with a shell-and-tube heat exchanger operating at 130˚C. Following SF1, SF2 was collected using an electrostatic precipitator (ESP) operating at 120˚C which consists of aerosols and vapors. SF3 was condensed with a second shell-and-tube heat exchanger operating at 0˚C and SF4 is the final condensing stage where the second ESP operates at -10˚C. The NCGs are then passed through a desiccant and measured using a Ritter® TG5/4-ER-1 bar drum-type gas meter to determine gas flowrate. A slipstream was continuously analyzed via a Varian CP-49000 MicroGC to quantify nitrogen (N2), oxygen (O2), carbon dioxide (CO2), methane (CH4), ethane (C2H6), and ethylene (C2H4).

Each moisture content was tested in duplicate. The bio-oil was collected and massed immediately following the run and the biochar was recovered after the system cooled to ambient temperature to prevent combustion.

Product Analyses

Mass balances for the biochar, bio-oil, and NCGs yields were completed following each experiment. Bio-oil and biochar masses were determined by weighing the condensing train and cyclone components before and after the run. The NCGs were quantified using the Varian® CP-4900 MicroGC-measured gas compositions and the total volumetric flowrate measured by a wet test meter. All yields are reported on a dry biomass basis. Bio-oil moisture content was measured using a Mettler Toledo Karl Fischer Titrator V30S equipped with a Mettler Toledo InMotion KF Flex Oven Autosampler. The solvent was Hydranal Working Medium K® and the titrant was Hydranal Composite 5 K®. Total water yield was calculated by multiplying the total

oil yield by the measured moisture content. Dry bio-oil yield, reaction water, and organics yield were calculated using the equations provided by.

Elemental analysis was used to determine the carbon, nitrogen, hydrogen, sulfur, and oxygen (by difference) content of the anaerobic digestate and the produced biochar. The analysis was performed using an Elementar vario Micro Cube Analyzer. Approximately 4-6 mg of sample was weighed into tin boats along with an equal mass of combustion aid. After combustion, the resultant gases passed through a cell that measures the change in thermal conductivity relative to the reference gas, enabling elemental quantification.

Proximate analysis was performed using a Mettler Toledo TGA/DSC 1® system with STARe® software. Samples of 20-40 mg were placed in ceramic crucibles and heated from 25˚C to 105˚C at a rate of 10˚C/min under 100 mL/min of N2 and held at temperature for 40 minutes to remove moisture. The temperature was then increased to 900˚C at 10˚C/min and held for 20 minutes to measure volatile content. Air flowing at 100 mL/min was then introduced at 900˚C for 30 minutes to combust remaining material. Fixed carbon was calculated from the final weight loss and the remaining weight is considered ash.

Determination of Total Organic Carbon in Biochar

Total organic carbon (TOC) was determined by subtracting inorganic carbon from total carbon. Inorganic carbon was quantified using a modified NaOH trap method. 1 g of biochar was placed in a 100 mL open vial with a stir bar inside a sealed 1 L mason jar fitted with septa. A second 20 mL vial containing 10 mL of 1 M NaOH was also placed inside the jar. After the jar was sealed, 50 mL of 0.1 M HCl was injected through the septa into the vial containing the biochar. After 72 hours, the NaOH vials were removed, treated with 10 mL of $BaCl_2$ to precipitate carbonates, filtered, and titrated with 0.1 M HCl using phenolphthalein as the endpoint indicator. The amount of CO_2 evolved from the biochar-acid mixture was calculated as twice the difference in titrant volume required to titrate the blanks and samples. All measurements were performed in triplicate and corrected using a blank NaOH trap.

Data tables

This section contains summary information for each data table (bolded) in this repository, starting with the file name and a textual description of the table. Following that is a table where each row represents one column in the data table with metadata for that column. If the data table contained any categorical columns, there will then be a table containing descriptions of each category.

dataset_bio_oil_water_content. This table contains the partitioning of liquid pyrolysis products into heavy organic compounds, light organic compounds, and reaction water for anaerobic digestate subjected to autothermal pyrolysis at varying feedstock moisture contents. Heavy organics correspond primarily to high-molecular-weight compounds recovered in the first two condensation stages, while light organics represent lower-molecular-weight compounds recovered in later condensation stages after correction for water content. Reaction water represents water generated during pyrolysis and oxidation reactions rather than water

originally present in the feedstock. Each row represents a single pyrolysis trial conducted at a specified feedstock moisture content and summarizes the resulting liquid product composition on a dry biomass basis. Number of records: 10.

attribute name	attribute definition	storage type	measurement scale standard unit
dataset bio oil water content pkey	A unique primary key identifier for each row in this table.	integer	dimensionless
feedstock moisture percent	Moisture content of the feedstock on a dry biomass basis.	float	percent
heavy organics percent	Fraction of high boiling point organic compounds in the bio oil, reported on a dry biomass basis.	float	percent
light organics percent	Fraction of low boiling point organic compounds in the bio oil, reported on a dry biomass basis.	float	percent
reaction water percent	Water generated during conversion (excluding feedstock moisture), reported on a dry biomass basis.	float	percent

dataset_biochar_composition. This table contains elemental and proximate analyses of biochar produced during autothermal pyrolysis of anaerobic digestate prepared at multiple feedstock moisture contents. Each row represents a single analytical replicate of biochar generated from one pyrolysis treatment moisture level. Three replicate measurements were performed for each moisture treatment, allowing evaluation of within-treatment variability in elemental composition and carbon stability characteristics. Number of records: 30.

attribute name	attribute definition	storage type	measurement scale standard unit
carbon percent	Elemental content of the biochar, reported on an ash-free dry basis.	float	percent
dataset biochar composition pkey	A unique primary key identifier for each row in this table.	integer	dimensionless
feedstock moisture percent	Moisture content of the digestate used to produce biochar sample.	float	percent
fixed carbon percent	Fixed carbon content of the biochar on an ash free dry basis.	float	percent
hydrogen percent	Elemental content of the biochar, reported on an ash-free dry basis.	float	percent
nitrogen percent	Elemental content of the biochar, reported on an ash-free dry basis.	float	percent
oxygen percent	Elemental content of the biochar, reported on an ash-free dry basis.	float	percent
replicate	Sample replicate of pyrolysis run at a specific feedstock moisture content.	float	number
sulphur percent	Elemental content of the biochar, reported on an ash-free dry basis.	float	percent
volatile matter percent	Volatile matter content of the biochar on an ash free dry basis.	float	percent

dataset_pyrolysis_yield_dry_biomass. This table contains pyrolysis product yields from autothermal fluidized-bed pyrolysis of anaerobic digestate prepared at different feedstock moisture contents. Each row represents one replicate from the results from a single pyrolysis

trial conducted using digestate prepared at a specified moisture content. Number of records: 10.

attribute name	attribute definition	storage type	measurement scale standard unit
char yield percent	Yield of solid char produced during conversion, reported on a dry biomass basis.	float	percent
dataset pyrolysis yield dry biomass pkey	A unique primary key identifier for each row in this table.	integer	dimensionless
feedstock moisture percent	Moisture content of the feedstock on a dry biomass basis.	float	percent
heavy ends percent	Fraction of high boiling point organic compounds in the bio oil, reported on a dry biomass basis.	float	percent
light ends no h2o percent	Light ends fraction corrected to exclude water content, reported on a dry biomass basis.	float	percent
light ends percent	Fraction of low boiling point organic compounds in the bio oil, reported on a dry biomass basis.	float	percent
liquid yield percent	Yield of total liquid phase products produced from conversion, reported on a dry biomass basis	float	percent
non condensable gases percent	Yield of non condensable gases produced during conversion, expressed as mass fraction of dry biomass.	float	percent
organics percent	Total organic fraction in the bio oil (sum of organic components excluding water), reported on a dry biomass basis.	float	percent
reaction water percent	Water generated during conversion (excluding feedstock moisture), reported on a dry biomass basis.	float	percent

dataset_pyrolysis_yield_gasses. This table contains the composition of non-condensable gases (NCGs) produced during autothermal pyrolysis of anaerobic digestate at different feedstock moisture contents. Gas yields were calculated from continuous micro gas chromatograph measurements combined with volumetric gas flow measurements and are reported as percentages of the dry biomass feedstock. Each row represents the yield of a single gas species measured during a pyrolysis trial at a particular feedstock moisture content. Each row represents one NCG at one feedstock moisture content. Number of records: 90.

attribute name	attribute definition	storage type	measurement scale standard unit
dataset pyrolysis yield gasses pkey	A unique primary key identifier for each row in this table.	integer	dimensionless
feedstock moisture percent	Moisture content of the feedstock on a dry biomass basis.	float	percent
gas	Name of the gas species measured.	string	
gas yield percent	Yield of the specified gas species at the specified feedstock moisture content on a dry biomass basis.	float	percent

Other entities

This section contains summary information for any other files in this repository, including the file name, description, and a download link.

This dataset has no accompanying entities.