Thermochemical Processing

Learning Objectives:
- Enhance understanding of the four stages of combustion.
- Provide an introduction to thermochemical processing of biomass.
- Provide an introduction to thermochemical processing products and their uses.
- Enhance understanding of engineering tradeoffs between energy and co-products.

Learning Outcomes:
Upon completion of this laboratory, participants will be able to:
- Describe the four stages of biomass combustion.
- Match weight loss at given temperatures to biomass components.
- Describe different thermochemical processes and their solid, liquid, and gas product distributions.
- Describe advantages and disadvantages of different thermochemical processes.
- Perform a mass balance on a thermochemical reactor.

Pre-Lab

Background

Thermochemical Processing
Thermochemical processing is the use of heat to promote chemical transformations of biomass into energy and chemical products. The six processes discussed in this lab are combustion, slow pyrolysis, torrefaction, fast pyrolysis, flash pyrolysis, and gasification. These represent the processes receiving the most attention across the thermochemical platform for producing heat, power, fuels, biochars, and chemicals. All of these processes create some amount of three products: solid (char and/or ash), liquid (bio-oil or tar), and gas (syngas or producer gas). Each process uses different reaction conditions (temperature, pressure, heating rate, residence time, reactive or inert atmosphere, purge gas flow rate, etc) to optimize the production and/or quality of one or more specific products.

Stages of Combustion
Key to understanding thermochemical processing is an understanding what occurs during the four steps of combustion (i.e. burning in the presence of sufficient oxygen). Some or all of these steps occur in the other processes, but often to a lesser extent.
1. Most biomass contains at least some moisture so the first step in combustion is drying. As water boils at a relatively low temperature, steam is the first thing to be removed. Fires are more difficult to get started than to maintain because water evaporation is an endothermic (energy-requiring) process. Energy must be added to start a fire before any energy can be extracted from that fire.
2. The second combustion step is volatilization or pyrolysis (no oxygen needed yet). As heat breaks the chemical bonds within the biomass, smaller molecules vaporize and escape from the biomass particle.

3. It is not until the third step: gas phase oxidation that one sees a flame. As hot volatile molecules leave the biomass particle, they come in contact with oxygen and burn (oxidize), releasing heat and light. If there is enough oxygen present, the only products are carbon dioxide and water. If there is not enough oxygen, however, these volatiles do not burn completely and can result in heavy smoke and tar or gas-phase polymerization to soot.

4. When all of the volatile parts of the biomass have been oxidized and removed, only a very hot, slow-burning solid shell is left to undergo the final step of combustion: solid-phase oxidation. These glowing “coals” are still reacting with oxygen, but because the oxygen has to diffuse to the surface of the solid rather than react with gas-phase volatiles, the process is much slower and does not give off a visible flame. Eventually, all of the carbon is oxidized to carbon dioxide and only the non-combustible mineral material, the ash, is left.

The extent to which each combustion process occurs depends on the amount of energy available (i.e. the temperature), the amount of oxygen, and the residence time of the biomass particle and product fractions in the oxidizing atmosphere. In combustion chambers and boilers, for example, high temperatures and excess oxygen are used to drive all reactions to completion.

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**Slow Pyrolysis**
Charcoal for heating and other purposes is traditionally made by slow pyrolysis: heating in the absence of oxygen to moderate or high temperatures. The process is characterized by slow heating rates and long residence times. Necessary heat to start
and drive the reaction is usually provided internally by combusting a portion of the feedstock. The goal of slow pyrolysis is a high-carbon, energy-dense solid char product. The co-products are a watery, low-molecular-weight acidic liquid called pyroligneous acid or “wood tar”, and a low-energy, combustible gas.

Charcoal production has existed in the repertoire of human technologies for thousands of years, most likely since humans learned how to control fire. In early fire pits, bits of charcoal would have been left over after a fire, especially if the center of larger pieces did not burn completely. Humans gradually learned that they could produce more of this black, light and friable material if they covered burning wood or debris. Some of the first techniques to produce charcoal, pit kilns and mound kilns, were used through the early 20th century and are still used in developing countries around the world.

To build a pit kiln, workers would dig a hole, pack it with dry material (mostly wood), leaving room at each end for an air inlet and outlet, and ignite the material on one end. Once a strong fire was going, less dense material (branches, leaves, etc.) was piled on top, followed by a layer of soil thick enough to keep out the air (~20 cm). Air would be allowed to enter on one side of the pit and exit on the other, causing the combustion region to gradually move across the pit. Workers would tend the kiln constantly over the next two or three days, opening and closing holes in the soil layer to control the amount of air. Once the carbonization process was complete, the pit would be uncovered and the charcoal allowed to cool. Yields of charcoal from pit kilns are generally very low (~10-30%), have wide variations in quality due to differing conditions within the pit, and often contain significant amounts of contaminants such as the soil used to cover the pile. Pit kilns tend to be energy inefficient and create large amounts of air pollution from the venting of the volatiles (smoke), non-condensable gases (carbon monoxide, methane, low molecular weight hydrocarbons, etc.) and particulate matter. For this reason, pit kilns are essentially aboveground pit kilns, using similar burn-and-cover methods and being susceptible to many of the same problems.

Building kilns from brick, concrete or metal was the next step in improving charcoal making technology. Not only are these kilns more permanent, they also allowed for greater heat insulation and control of conditions, thus increasing char yield, consistency and quality. Brick kilns are made of bricks sealed together with mortar or mud set on top of a brick base and are shaped like mounds or beehives. One opening is used to load in wood, while another on the opposite side is used to unload the finished charcoal. Vents along the bottom of the kiln can be opened or closed depending on the color of the smoke leaving the “eye” hole in the top center of the kiln (white = drying, yellow/brown= volatilization, bluish/clear = carbonization complete). Carbonization generally takes a week of adding air through the vents, followed by a couple days leaving just the “eye” hole open to vent volatiles, and finally, a cooling period with the kiln completely sealed. This method allows for a slower, more even burn that means less carbon is lost during the combustion phase. Also, by using bricks instead of loose soil, the charcoal is less likely to be contaminated with mineral matter. Rectangular, reinforced concrete kilns with
steel doors and clay pipe stacks, also called Missouri kilns, were very common in regions where a lot of charcoal was produced for the steel industry and several are still commercially operational today, especially for the production of grill charcoal briquettes. Future designs of kilns for clean and efficient large scale char production will likely focus on continuous (rather than batch) process kilns and will use afterburners to lower emissions.

**Biochar**
Biochar is charcoal that is used as a soil amendment and/or a carbon sequestration agent. As soil amendments, chars have been shown to increase soil fertility by improving nutrient and water retention, lowering soil acidity and density, and increasing microbial activity. In addition, energy production from biomass that stores carbon as biochar can be considered *carbon negative* due to biochar’s high recalcitrance.

**Torrefaction**
Torrefaction can be thought of as low temperature (200-300°C) slow pyrolysis. One example of torrefaction is the roasting of coffee beans. Torrefaction removes water and some volatiles from biomass, making the biomass easier to grind, transport and store. The products are much the same as those from regular slow pyrolysis except that the solid product is browner in color than black. This brown “char” is easy to grind, has a higher energy density than biomass, and is slightly hydrophobic, making it less likely to absorb water and less likely to decay in storage. The ability to transform raw biomass into a more easily-managed feedstock that is available year-round is potentially critical to the economical implementation of other thermochemical processes.

**Fast Pyrolysis**
Fast pyrolysis, like slow pyrolysis, is the heating of biomass in the absence of oxygen. Unlike slow pyrolysis, however, fast pyrolysis uses very high heating rates (~1000°C/s), short residence times, and the rapid quenching of vapors to maximize the production of the liquid product, bio-oil. The theory behind fast pyrolysis design highlights the difference between a thermodynamics-controlled process and a kinetics-controlled process. In a thermodynamics-controlled process, reactants and products are allowed sufficient contact time to reach thermodynamic equilibrium. The final distribution of products depends on process conditions such as temperature and pressure, but not on reaction rate. Slow pyrolysis represents a thermodynamics-controlled process; the amount of char or gas products varies with temperature, pressure and feedstock, but would be the same regardless of whether the reaction lasted for a few hours or a few days. In fast pyrolysis, a kinetics-controlled reaction, the goal is to create and separate vapors as quickly as possible before they can condense and carbonize as secondary chars or crack into low-molecular-weight, non-condensable gases. Fast pyrolysis wants to avoid thermodynamic equilibrium. This is accomplished through a high rate of heat transfer to the biomass, causing the drying and volatilization steps to occur almost instantaneously. Methods to achieve such high heat transfer rates include: reducing the biomass particle size, selecting an effective heat carrier (such as sand or steel shot), and using a fluidized bed, heated blade (ablative pyrolysis), or screw mixer (auger pyrolysis) reactor design. Once heated, the large amount of created volatile molecules and
aerosols quickly expand out of the biomass particles (sometimes causing the particles to fracture apart) and are removed from the reaction zone by inert sweep gas. Outside of the reaction zone, the hot vapors are quickly separated from the solid char by cyclones or filters. Finally, the vapors and aerosols are condensed out of the gas phase by cooling, scrubbing, electrostatic precipitation, etc. while the non-condensable gases are sent on to an afterburner for energy or heat recovery. To achieve a maximum yield of oil (~70% by weight), fast pyrolysis reactors are designed to achieve moderate temperatures (400-600°C) and vapor residence times of no more than a few seconds.

**Bio-oil**

Bio-oil from fast pyrolysis is a complicated mixture of water and oxygenated organic compounds including organic acids, aldehydes, alcohols, furans, pyrans, anhydrosugars and aromatics. Approximately 300 different compounds have been identified in bio-oil from the decomposition of hemicellulose, cellulose and lignin. Currently, bio-oil can used as a heavy oil replacement in commercial boilers and some steam turbines for heat and electricity, as an energy-dense and pump-able biorenewable feedstock for gasification, and as a petroleum replacement in the production of asphalt (i.e. “bio-asphalt”).

As a feedstock for the production of organic chemicals and transportation fuels, bio-oil has been compared to crude petroleum in that can provide a wide variety of products but requires fractionation and upgrading. There are three key differences between crude oil and bio-oil that pose a significant problem for its direct use in existing refineries, namely water content, oxygen content and high acidity. Bio-oil is also unstable, especially when stored at high temperatures. It tends to separate into aqueous and hydrophobic phases, and the high acidity and oxygen content catalyze polymerization reactions that dramatically increase oil viscosity. Research aimed at improving bio-oil properties has included bio-oil collection system designs that separate the oil into fractions, catalytic reforming of aqueous bio-oil to produce hydrogen, and bio-oil upgrading through hydrogenation to remove carboxylic acids and oxygen.

**Flash Pyrolysis**

Flash Pyrolysis™ is a batch pyrolysis process that uses moderate pressures (2-25 atm) to minimize reaction time and maximize biocarbon yield. In this process, biomass is packed into canisters and loaded into a high pressure chamber. Compressed air is pumped into the chamber and the reaction is initiated by electric heaters on the bottom of the reactor. The biomass at the bottom begins to burn, heating the biomass above it. After about 30-45 minutes, the oxygen in the chamber has been depleted and all of the biomass has been transformed into biocarbon. Vented gases are sent to an afterburner to produce heat and/or electricity. The increased pressure shifts the thermodynamic equilibrium of the reaction to favor char formation and increases the rate of reaction, making the overall throughput rate only slightly slower than continuous fast pyrolysis process. Current marketing of the process is focused on more traditional uses of charcoal (coal replacements and activated carbons) but has strong potential in the areas of waste management (waste-to-carbon), and biochar horticulture and agriculture applications.
Gasification
As the name implies, the primary product of gasification is the non-condensable gas fraction. The process is characterized by higher temperatures (750-1800°C) and the presence of some oxygen, measured in equivalence ratio or the fraction of the amount of oxygen needed for stoichiometric combustion (typically around 0.25 or 25%). The product gas, called syngas (short for “synthesis gas”), or producer gas if it contains nitrogen, consists mostly of carbon monoxide (CO) and hydrogen (H₂) with smaller amounts of carbon dioxide, methane and other low molecular weight hydrocarbons. Overall, gasification is very similar to combustion, but due to the limited oxygen, it is not able to complete the gas-phase and solid-phase oxidation steps which would yield carbon dioxide (CO₂) and water (H₂O).

In an ideal gasification situation, the reaction is thermodynamically controlled. The gas composition and carbon conversion can be predicted based on temperature and pressure, and the only co-product is char. In reality, there is not sufficient time for the reaction to reach equilibrium, resulting in sticky, viscous tars that can clog reactor plumbing and cause significant problems in downstream gas applications. Much research has been devoted to the development of methods to address this tar problem such as the use of steam and/or catalysts to promote tar cracking, tar filtering or scrubbing systems for downstream gas cleaning, and raising the reaction temperature and/or residence time.

The oxygen needed for the gasification can come from air (air-blown gasification) or from a mixture of steam and oxygen (steam/oxygen-blown gasification). Steam/oxygen-blown gasification has three advantages over air-blown. First, the product gas stream is not diluted with nitrogen. Second, steam can easily be separated from the gas stream by condensation. Finally, steam in the reaction can be used to accomplish a water-gas-shift (WGS) reaction to increase the hydrogen content of the product gas. The WGS reaction is based on the equilibrium between water, carbon monoxide, carbon dioxide, and hydrogen:

\[
\text{CO} + \text{H}_2\text{O} \leftrightarrow \text{CO}_2 + \text{H}_2
\]

One downside of steam/oxygen-blown gasification is that it requires the use of expensive gas separation equipment to produce pure oxygen from air.

Syngas Uses
There are several uses for syngas and producer gas. The most direct use is as an alternative to natural gas (i.e. methane). Prior to the widespread use of natural gas, “town gas” from the gasification of coal was commonly used in heaters, stoves and light fixtures. Syngas, which contains carbon that has already been mostly oxidized, is much less energy dense than natural gas especially when the product gases were diluted with nitrogen. Transportation fuels and chemicals can be synthesized from syngas. One important reaction is the production of methanol from carbon monoxide and hydrogen:

\[
\text{CO} + 2\text{H}_2 \leftrightarrow \text{CH}_3\text{OH}
\]
**Fisher-Tropsch Synthesis**

Hydrocarbons can be produced from syngas through the catalytic Fisher-Tropsch process which uses low-moderate temperatures, high pressures and transition metal catalysts to produce a distribution of alkanes and paraffin waxes. Depending on the reaction conditions, the alkanes can range from the shorter-chain gasoline fraction to the medium-length jet fuels to the longer diesel fuels and waxes:

\[(2n+1)H_2 + nCO \leftrightarrow C_nH_{(2n+2)} + nH_2O\]

One challenge of Fisher-Tropsch synthesis is that its optimal H₂:CO ratio is around 2, while the ratio in the syngas from the gasifier is lower (generally closer to 1), meaning that significant amounts of CO must be converted to CO₂ by the WGS reaction to provide the necessary hydrogen. Another challenge to this and other catalytic processes is the coking or fouling of the catalyst. Even tiny amounts (on the parts per billion scale) of some fouling species can be enough to ruin a process. Therefore, compounds containing sulfur, nitrogen, halides (fluoride, bromide, chloride and iodide), and tars or particulate matter that can coke on catalysts must be meticulously removed from the product gas stream prior to the catalysis reactor. Gas cleaning/conditioning is therefore one of the most complicated and expensive gasification system components.

**Pre-Lab Tasks**

1. Brainstorm a few ways that thermochemical processing of biomass is different than biochemical processing of biomass.
2. List some ways thermochemical processing was used to provide energy and materials before the 20th century when petroleum and coal became dominant.
3. Considering the stages of combustion that charcoal briquettes have already been through, why do they require lighter fluid?
4. Compare and contrast bio-oil and crude oil. What are some concerns with bio-oil that have to be addressed if it is to be a substitute for crude oil?
5. If biomass is composed of carbon, hydrogen, oxygen, nitrogen and ash (metal oxides), which of the solid, liquid or gas products do you think each element ends up in for fast pyrolysis? Slow pyrolysis? Gasification?
6. Why might you want to use torrefaction as a pretreatment for another thermochemical process? How does this relate to what you know about biomass properties?
Combustion and Thermogravimetric Analysis (TGA)

Safety Checklist
- Proper attire is worn (long pants and closed-toe shoes).
- Food and drinks are stored and consumed outside the laboratory.
- Lab coat and safety glass are worn.
- Latex or nitrile gloves are used when handling samples and chemicals.
- Insulated gloves are used when handling hot materials.

Problem Statement
As the Middle School Academy participants, and biorenewables "experts" the President has requested that you plan a Biorenewables-Themed picnic. You will be focusing on thermochemical processing of biomass available as you've looked into biochemical processing but found it would be too expensive and time-consuming to adjust processes for all of the different feedstocks. Your team has been challenged to come up with this picnic, and you've been provided some materials and tools, but will need to obtain those items NOT included to make your summer-time Biorenewables-Themed picnic a success. Additionally, the President requests that you explain the 4 stages of combustion based on a fire-pit demo, and the inputs and steps taken to produce biodiesel that's being used in the generator you'll be using for your picnic. You will also need to have two poster size displays outlining: 1.) the 4 stages of combustion, and 2.) the biodiesel making process. This will become clear from the next paragraphs! Some ISU and other resources will be available for limited consultations!

Biomass Fire-Pit Demonstration

Background
The best way to understand the four stages of combustion, the underlying process of all thermochemical processes, is to watch a fire. Most people have had experience with a campfire or burn pile, but rarely do they pay attention to the stages of combustion. For this activity, a backyard portable fire pit will be used to demonstrate a wood fire process starting with the ignition of kindling, through the addition of new fuel, and ending with the extinguishment of glowing coals.

Pre-experiment Checklist
The following checklist should be performed before beginning the demonstration:

- Environmental Health & Safety Department has been notified of the date and time of the demonstration, and has provided an appropriate fire extinguisher.
- PROVIDED:
  - Fire pit is set up on appropriate non-flammable surface at a safe distance from the building. A weber grill/pit will be provided for your team to use.
  - Lighter provided
  - Smoothie maker
  - Microwave
- Diesel Generator and Fuel Grade Biodiesel from Biobus as Fuel!
- Tables (located in 1115 BRL)
- Fire extinguisher
- Limited ISU and Other resource personnel for consultation
- Limited utensils in 1st floor kitchen (ice cream scoop, etc.)
- Two-tripods and poster sized post-it paper for displaying your information on
  1.) The 4 stages of combustion (far away from the fire, please!), and 2.)
  the making of biodiesel.

**YOU NEED TO OBTAIN:**
- Wood fuel, kindling, twigs, ignition materials such as paper.
- Serving items such as ice, paper plates, utensils, cups, napkins, paper
towels and table coverings (tables used are long tables located in 1115
BRL) that are made from biorenewable materials (bioplastic, recycled
material, etc.)
- Sticks/skewers for roasting hot dogs and marshmallows
- Food and refreshment items:
  - Your friends request the following smoothie recipe:
    - Vanilla ice cream + diet lemon/lime soda + berries
  - Your friends request the following food items:
    - All Beef Hot dogs, buns, Big Marshmallows, ketchup,
      mustard, & pickle relish
  - Your friends request the following drink items:
    - Water, iced tea (unsweetened or diet), diet/regular soda
- Setup time worked out so that food is served at NOON, and concluded by
  1pm.
- List of invitees, and ensure you have enough serving materials and food for
  # of invitees! ISU resource can help with identification and invitation email!
- Have a bucket of water on hand (just in case!)
- Have a cleanup plan! Use Gloves, paper towels and water/cleaner!

**Procedure (Ensure you have safety glasses on and don’t stand right over the fire)**

**The FIRE PIT**

1. Stack kindling and ignition materials in fire pit. Be prepared to observe and explain
   the 4 stages of combustion to your guests…you never know who will ask!
2. Light ignition materials and monitor burn.
3. Add larger pieces of wood gradually to build up fire.
4. Record careful observations of how fire progresses, different colors of smoke are
   emitted, flame appears and disappears, etc., especially as each new piece of
   wood is added.
5. Continue augmenting fire until bottom layer of glowing coals forms.
6. Roast hot dogs and marshmallows to desired doneness.
7. About 1 hour before fire is to be completely extinguished, discontinue adding
   wood. Don’t add any more wood @1pm! Fire needs to diminish
8. Make careful observations as fire gradually diminishes.
9. Once fire is extinguished and coals have cooled, clean out fire pit and collect ashes for disposal.
10. If the water bucket is still available use it to “wipe out/clean the fire pit and dump into the grass. Use paper towels to clean it out so that it is fairly clean and dry!

The Biodiesel Generator
1. You need to use the Diesel Generator and Biobus Biodiesel to power your smoothie maker. There is an SOP by the generator and the remote starter is secured to the outside structure of the generator. Please seek an ISU undergrad. Lab student to assist you.
2. Be prepared for your guests to ask you about how to make biodiesel!
3. You may need to power the microwave if you feel you need it…located in 1115 BRL.

Data Analysis
1. Compare observations of what happens to a new piece of wood between it being added to the fire and completely reducing to ashes. Describe what you observed during each stage of combustion.
2. What would you have expected to be different if a wet piece of wood were added to the fire?
3. What do you think causes the crackling/popping noises during a fire?
4. Approximately how much ash remained relative to the amount of wood that was used? How much would you expect to be left if a similar amount of switchgrass or corn stover were burned instead?
5. Were there any pieces of wood that didn’t burn or only partially burned? What might you do to correct this problem in the fire pit? What about in an enclosed furnace? How would you control the burn?

OPTIONAL - Thermogravimetric Analysis (TGA)

Background
In the properties of biomass lab, a thermogravimetric analyzer (TGA) was used to measure the moisture, volatiles, fixed carbon and ash content of biomass, i.e. to perform proximate analysis. Such an analysis is concerned with weight differences at the start and finish of various heating cycles. A TGA can also be used is to measure continuous weight loss curves, where the mass of a sample is tracked over a range of temperatures for a set period of time. Exactly when biomass decomposes and gases evolve provide clues about that biomass' composition. For example, two samples of biomass may have the same proximate composition, but one may start to pyrolyze at a lower temperature. This would generally mean that the more reactive sample contains more hemicellulose than cellulose relative to the other sample since the chemical bonds in hemicellulose require less energy to break than those in the more crystalline cellulose.

In this lab, weight loss curves for several feedstocks over the 25-800°C range will be provided. One curve for each feedstock will have been produced under an inert gas
(nitrogen), while another will have been produced under air. The instrument used to produce the curves is a Mettler Toledo Thermogravimetric Analyzer with Differential Scanning Calorimetry capabilities (TGA/DSC) Model 1 Star® system located in the 3rd floor labs. Unlike the NAVAS TGA 1000 used in the biomass properties lab, this TGA uses much smaller sample sizes (20-30 mg) and has a much smaller, more precise furnace. Each weight loss curve displays weight percent remaining versus temperature. At a constant heating rate of 5 °C/min, temperature can also be used determine the time elapsed since heating began.

Two research articles provided may be helpful in identifying which components of biomass decompose at which temperatures:


**Pre-experiment Checklist**

- Printouts of TGA weight loss curves for at least two feedstocks made under inert and oxidizing atmospheres are available.
- Printouts of the Raveendran et al. and the Yang et al. articles are available.

**Data Analysis**

1. On each weight loss curve, find and label important features such as temperatures where weight loss started, where weight loss was the fastest, and where the weight leveled off.
2. Calculate approximately what percentage of weight was lost over each 100° interval. Which temperature intervals were the most interesting?
3. Compare the weight loss curves for a single feedstock made under the inert oxidizing intervals. Which temperature regions are similar? Which are different? How do the final weights compare? If you could see the samples after they had completed the heating programs, would you expect to see ash or char?
4. Compare the weight loss curves of two different feedstocks. Based on the temperatures at which different biomass components decompose, which biomass do you think has more hemicellulose? Cellulose? Lignin? Ash?
5. As described in the two articles, some biomass decomposition reactions are exothermic while others are endothermic. How do you think this would affect the design of a biomass thermochemical reactor? At which points in the process would you need to add extra heat? Do you think it would be possible to run a reactor under adiabatic conditions (no heat added or removed)?