

Tests to Reduce TorreCatTM Technology to Practice

Tyler Westover
Rachel Emerson

February 2016



The INL is a U.S. Department of Energy National Laboratory
operated by Battelle Energy Alliance

Tests to Reduce TorreCat™ Technology to Practice

**Tyler Westover
Rachel Emerson**

February 2016

**Idaho National Laboratory
Idaho Falls, Idaho 83415**

<http://www.inl.gov>

**Prepared for the
U.S. Department of Energy
Office of Energy Efficiency and Renewable Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517**

**Project Title: Tests to Reduce TorreCat™ Technology to Practice
Strategic Partnership Project 15820 Control No. 15-237**

Recipient: INL

Project Location: INL

Project end date: 02/26/2016

Written by: Tyler Westover, Rachel Emerson

Participating Researchers: Michael Clark, Matthew Anderson, Rodney Shurtliff, Corey Landon, Cody Scheer, Luke Williams, Austin Matthews

This report contains business sensitive and/or potential intellectual property information. Do not distribute beyond immediate partners until this report has been officially released by INL with an external report release number. Information in this report may appear in a subsequent journal article submission.

Background and Purpose

Torrefaction is the thermal treatment of materials in the absence of oxygen in the temperature range of 200 to 300 °C and has been shown to improve handling and grinding properties, hydrophobicity, volatiles content, energy density, and combustion performance of renewable energy biomass feedstock materials. The disadvantages of torrefaction are its relative high cost compared to the low value input feedstock material and the energy that can be lost to volatilized gases.

This work will demonstrate a new technology developed by Advanced Torrefaction Systems (ATS), known as TorreCat™ Technology, that uses an oxidation catalyst in a closed system to combust and destroy volatile organic compounds (VOCs) and other byproducts produced in the torrefaction process. An oxidation catalyst is a substance, or a combination of substances, that accelerate the rate of a chemical reaction without being consumed by the reaction. Catalytic combustion is a reaction that occurs at temperatures 50% lower than traditional combustion, such that essentially no NO_x is created. The output of the oxidation catalyst (flue gas) consists mainly of superheated steam and inert gases (carbon dioxide and nitrogen), which can be used for heat in the thermal treatment process.

INL has previously developed a pilot-scale Continuous-Feed Thermal Treatment System (CFTTS) that has 10 kg/hr capacity but does not reform the flue gas to reduce environmental concerns or capture all available heat from the biomass material. Using the TorreCat™ technology in INL's thermal treatment system will demonstrate increased thermal efficiencies during the treatment process as well as reduced environmental impact and clean-up costs. The objective of this project is to determine the effectiveness of the Torrecat™ technology to reform the flue gas and capture as much of its heat content as possible.

Materials and methods

Sample Preparation

Approximately 1,000 lbs of clean (debarked) southern loblolly pine microchips were obtained from Green Circle BioEnergy in Cottondale, Florida. The as-received wood chips had a moisture content of approximately 50% wet basis, and were dried to less than 10% moisture content in an oven at 105 °C for 24 hours. Prior to torrefaction, fine particles were removed by sieving the wood chips using a 3 mm sieve using a Sweco vibrating separator.

Continuous-feed thermal pretreatment experiments

The CFTTS is a flexible test system capable of providing drying and thermal processing of a variety of biomass types, including herbaceous, woody and algal materials as well as clean waste materials, such as plastics, paper and cardboard at temperatures as hot as 270 °C in either air or inert atmospheres. The graphical user interface of the CFTTS provides real-time access to process data and varying levels of automated operation allowing the operational parameters to be conveniently changed during testing to meet research objectives. The CFTTS process vessel is configured as a horizontal pipe with heating and cooling sections with controlled entry and exit points located at each end to allow transfer of solid material and process sweep gas into and out of the system.

A photograph of the system is shown in Fig. 1, while the general schematic of the system and controls is shown in Figure 2. Material enters the heated reactor at (A) and exits at (C). The material infeed and outfeed systems are equipped with knife gate air locks that allow the reactor atmosphere to be purged with inert gas. The section (A) to (B) is actively heated, while the section (B) to (C) is not heated to allow material to cool to less than 150 °C before exiting the reactor and being exposed to an air atmosphere. Independent augers, driven from each end move the material through the two sections and determine the total residence time of material inside the reactor. Inside the heated section, material is stirred by T-shaped fingers as shown in Fig. 3 or by auger screws. Heated inert gas enters the reactor at (D) and exits at (E) where a cyclone removes dust and fine organic particles from the process gas stream. Other major system components include a blower (F), oxidation catalyst (G), condenser (H), process gas heater (I), gas exhaust fans (J), and process gas lines to bypass the oxidation catalyst, the condenser, and the reactor. All components can be actively heated except the material infeed and outfeed mechanisms. During operation, the condenser is cooled to room temperature but is heated afterwards to assist in draining condensed oil and tar. In addition to process gas recirculation, inert gas is continuously injected into the unheated section of the reactor to maintain a slight positive pressure in the reactor. As shown in Fig. 2, excess gas is allowed to exit the system after the condenser by passing through a bubbler. The depth of the outlet line in the bubbler determines the pressure in the system and was set to approximately 8" of water.

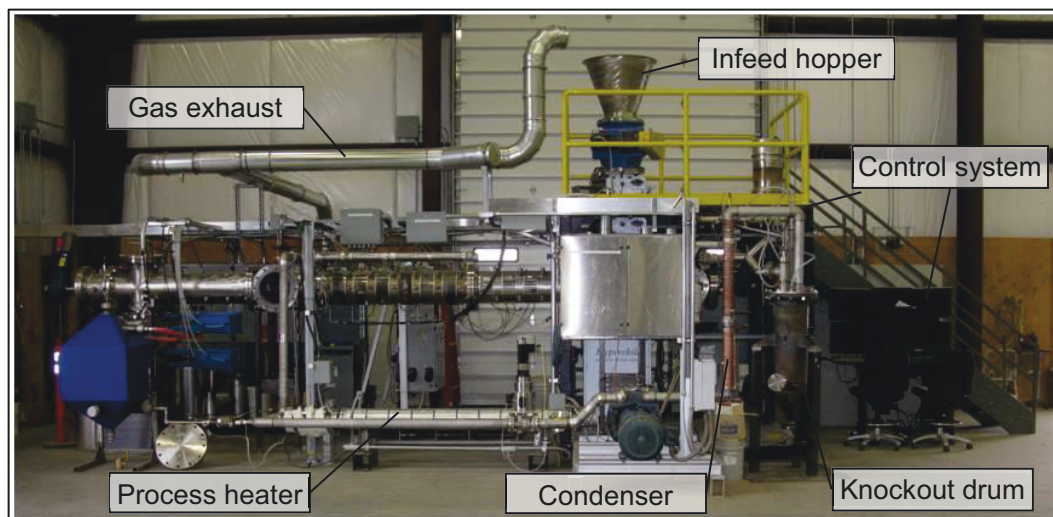


Figure 1. Side photograph of Continuous-Feed Thermal Treatment System (CFTTS) at INL.

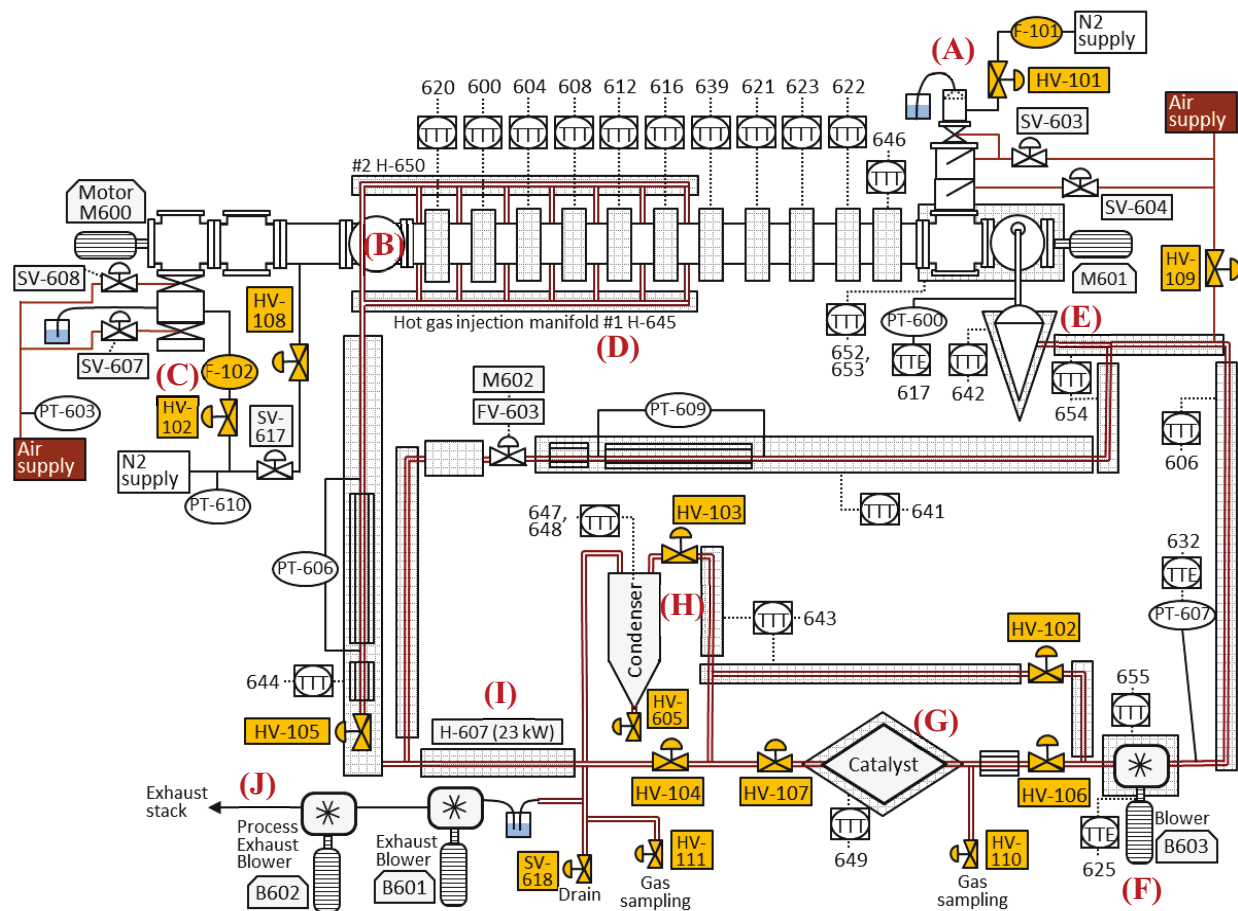


Figure 2. Schematic of Continuous-Feed Thermal Treatment System (CFTTS) including controls.

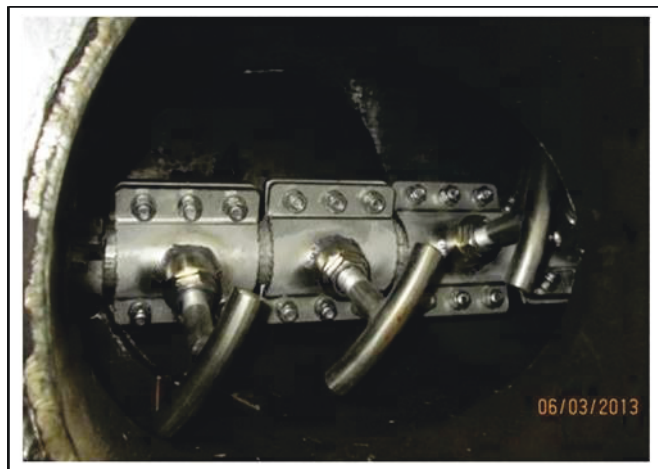


Figure 3. Photograph of T-shaped fingers that stir material inside the reactor.

Gas analysis

Process gas is sampled before and after the catalyst for analysis. Fixed gases CO, CO₂, CH₄, H₂, and O₂ are measured using a multi-gas industrial process gas analyzer (Model 7905AN45, NOVA Analytical Systems, Niagara Falls, NY). Gas samples are also collected using thermal desorption

tubes, which are analyzed offline using an Agilent 7890B gas chromatograph equipped with dual FID channels and connected to an automated Markes International thermal desorption system (TD-100).

Tests are conducted by feeding a small amount of material into the infeed at (A) and injecting air into the process gas stream at point (E) after the cyclone (upstream of the blower and catalyst). Initially, excess air is injected to ensure that sufficient oxygen is available for complete oxidation of hydrocarbon vapors and carbon monoxide in the catalyst. Oxygen content in the process gas is monitored downstream from the catalyst and used to inform decisions to either decrease the rate at which oxygen is injected or increase the rate at which feedstock is fed so that the oxygen content downstream from the catalyst can be decreased to the target range. Ideally, the oxygen content downstream from the catalyst should be as low as possible to minimize combustion of sample in the thermal treatment reactor; however, oxygen injection must always be greater than the stoichiometric requirement to avoid coking the catalyst. Reducing the oxygen content below 1% in the process gas stream after the oxidation catalyst requires good control of steady-state operation of the system in order to avoid coking the catalyst.

Two separate tests were performed for this work. For the first test, the reactor and all heated system components were maintained at 260 °C, while in the second test, all temperatures were reduced to 240 °C. Approximately 5 liters of sample material was fed through the infeed knife gates at intervals of 18 minutes and 12 minutes for the tests conducted at 260 °C and 240 °C, respectively (corresponding approximately to 4 and 6 kg/hr, respectively). In these tests, the response time of the oxygen analyzer was approximately 1 minute, and the target oxygen content downstream from the catalyst was 1%. Oxygen injection upstream of the blower was approximately 3.7 scfm and 2.7 scfm for operation at 260 °C and 240 °C, respectively. During both tests, nitrogen was injected in the material outfeed section of the reactor at a constant rate of 2.3 scfm. During both tests all valves were open, except for hand valve HV-104, so that process recycle gas passed sequentially through the blower, catalyst, condenser, process gas heater (H-607) and was then split approximately in equal parts with one split flowing through the reactor and another split by-passing the reactor to flow directly into the blower. The total process gas flowing through the blower was approximately 60 scfm.

Results and Discussion

Table 1 contains data that was collected real time upstream (up) and downstream (down) from the catalyst at different times during which the system appeared to be operating in steady state conditions. The process gas temperature increased approximately 177 and 158 °C across the catalyst while operating at 260 °C and 240 °C, respectively, while the oxygen concentrations across the catalyst decreased by 2.7% and 0.4%, respectively. For both tests, the carbon monoxide concentrations decreased from approximately 1% to below the detection limit of 0.1%, while carbon dioxide concentrations more than doubled to more than 10%. For both tests, hydrocarbons, measured as methane (CH₄), decreased from approximately 7% to below the detection limit of 0.1%.

Table 1. Data collected real time upstream (up) and downstream (down) from the catalyst at different times during which the system appeared to be operating in steady state conditions.

Op. Temp. (°C) → Sample location ↓	260			240		
	Up	Down	Diff.	Up	Down	Diff.
Temperature (°C)	249	426	177	239	397	158
O ₂ (%)	5.0	2.3	-2.7	2.0	1.6	-0.4
CO (%)	1.0	<0.1	-1.0	1.0	<0.1	-1.0
CO ₂ (%)	5.0	9.8	4.8	3.6	10.3	6.7
CH ₄ (%)	7.40	<0.1	-7.40	7.28	<0.1	-7.28
H ₂ (%)	0.00	0.07	0.07	0.00	0.04	0.04

Multiple process gas samples were collected using thermal desorption tubes upstream and downstream from the catalyst. Figure 4 shows example gas chromatograms (one collected upstream from the catalyst and two collected downstream from the catalyst). For the gas sample collected upstream from the catalyst, many peaks appear in the chromatogram at times less than 55 minutes. None of these peaks are evident in gas samples collected downstream from the catalyst, indicating that all of these compounds were destroyed by the catalyst. A small number of peaks were observed at times greater than 55 minutes in chromatograms of gas samples collected downstream from the catalyst. These peaks could be due to contamination because they appear in the large molecular weight area (time > 55 minutes) and are not seen in any of the gas samples collected upstream from the catalyst. Some of the primary peaks have been identified qualitatively from previous similar studies, and the calculated areas of those peaks are shown in Table. 2, which also shows that none of the identified compounds were observed in the samples collected downstream from the catalyst.

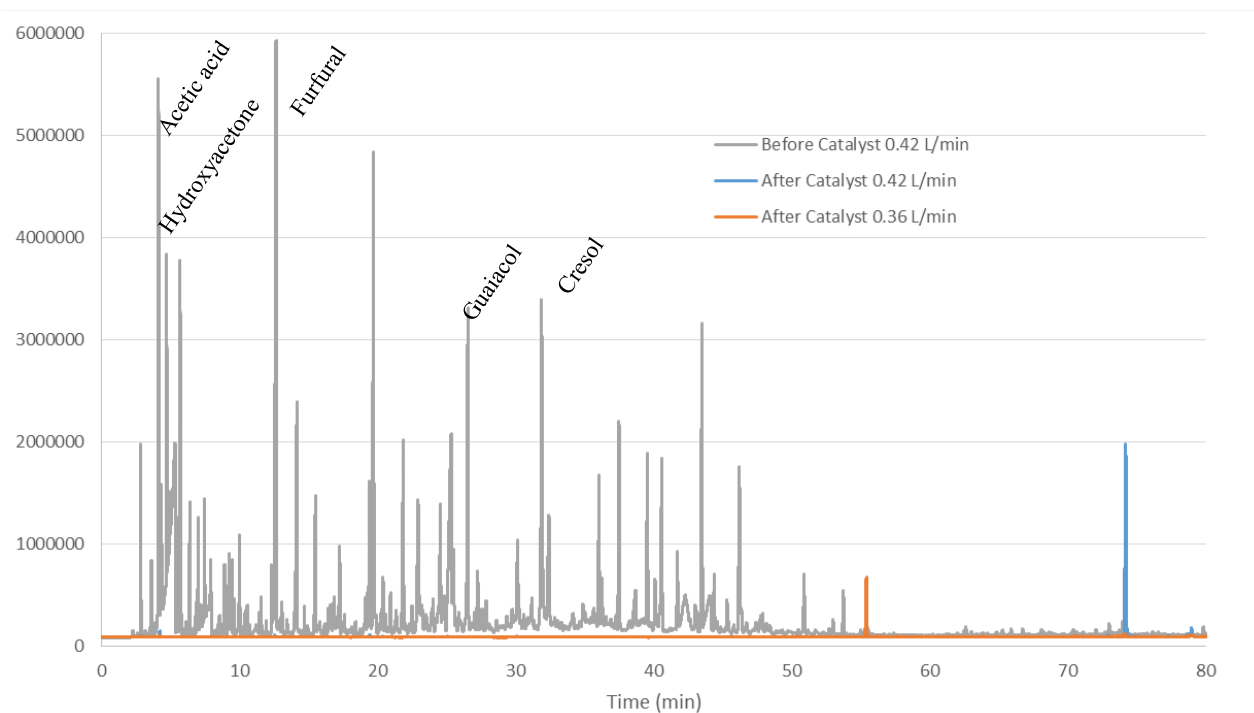


Figure 4. Chromatograms of gas samples collected upstream and downstream from the catalyst.

Table 2. Peak areas for primary identified compounds and total areas of identified peaks of gas samples collected upstream and downstream from the catalyst.

Primary Compounds	Upstream (area)		Downstream (area)	
	Sample 1	Sample 2	Sample 1	Sample 2
Acetic acid	12,108,540	164,481,582	No peak	No peak
Hydroxyacetone	19,170,287	59,123,002	No peak	No peak
Furfural	15,339,160	42,877,240	No peak	No peak
Guaiacol	9,104,895	31,593,750	No peak	No peak
Cresol	9,277,178	24,444,720	No peak	No peak

Future Work

The tests conducted for this work were intended to be preliminary tests to evaluate the suitability of the available experimental equipment to demonstrate the TorreCat™ technology. The tests were successful and additional tests are planned to acquire additional data needed to inform techno-economic analyses (TEA) of the process. Specifically, commercial application of the TorreCat™ technology will be much more favorable if the process can be better controlled to reduce the oxygen concentration of the process gas exiting the oxidation catalyst to less than 200 ppm. Commercial systems that use oxidation catalysts in similar applications have shown that low oxygen levels downstream from the catalyst are feasible with good control of the process parameters.

Future tests are planned that will incorporate modifications to the system to improve control of the process parameters. First, a General Electric Delta F 100 E Series oxygen analyzer will be obtained that has a response time of approximately 10 seconds and a measurement resolution of 100 ppm. Second, an additional knife gate will be installed at the material infeed to provide a two-stage air-lock with improved purge capability. The improved infeed air-lock system should make it possible to introduce material into the reactor more frequently to better approximate steady-state operation. The goal of the next tests will be to maintain the oxygen level downstream of the catalyst below 800 ppm.

Future tests will also vary the amount of process gas flow in the line that by-passes the reactor. This clean gas stream serves two important functions during operation. First, as noted above, it dilutes the process gas entering the blower to increase the dew point of the gas and reduce the potential to condense tars and oils in the blower. Second, and equally as important, it allows control of the temperature rise of the process gas stream across the catalyst. If the process gas entering the catalyst is too rich in hydrocarbons, the oxidation reactions within the catalyst section can excessively heat the process gas, potentially damaging the catalyst. By diluting the process gas before it enters the catalyst, the temperature of the gas exiting the catalyst can be controlled. Future experiments will adjust valves HV-105 and FV-603 (see Fig. 2) to control the flow of process gas through the reactor by-pass line to determine the relationship between gas flow rates in this line and the temperature of the process gases exiting the catalyst. Tests may also be conducted at different temperatures and using different materials in order to determine the amount of inert process gas that can be produced using the TorreCat™ technology under different experimental conditions.