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Alternative Fuels Research Laboratory

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Abstract

NASA Glenn has invested over $1.5 million in engineering, and infrastructure upgrades to renovate an existing test facility at the NASA Glenn Research Center (GRC), which is now being used as an Alternative Fuels Laboratory. Facility systems have demonstrated reliability and consistency for continuous and safe operations in Fischer-Tropsch (F-T) synthesis and thermal stability testing. This effort is supported by the NASA Fundamental Aeronautics Subsonic Fixed Wing project.

The purpose of this test facility is to conduct bench scale F-T catalyst screening experiments. These experiments require the use of a synthesis gas feedstock, which will enable the investigation of F-T reaction kinetics, product yields and hydrocarbon distributions. Currently the facility has the capability of performing three simultaneous reactor screening tests, along with a fourth fixed-bed reactor for catalyst activation studies.

Product gas composition and performance data can be continuously obtained with an automated gas sampling system, which directly connects the reactors to a micro-gas chromatograph (micro GC). Liquid and molten product samples are collected intermittently and are analyzed by injecting as a diluted sample into designated gas chromatograph units.

The test facility also has the capability of performing thermal stability experiments of alternative aviation fuels with the use of a Hot Liquid Process Simulator (HLPS) (Ref. 1) in accordance to ASTM D 3241 “Thermal Oxidation Stability of Aviation Fuels” (JFTOT method) (Ref. 2). An Ellipsometer will be used to study fuel fouling thicknesses on heated tubes from the HLPS experiments.

A detailed overview of the test facility systems and capabilities are described in this paper.

Introduction

Rising crude oil prices, energy independence, fluctuating global climate changes, and improving combustion performance while meeting advanced emission requirements are all major concerns in the area of commercial aviation. Reducing capital costs associated with synthetic jet-fuel production from non-conventional sources is also of concern. NASA Glenn’s Alternative Fuel research effort focuses primarily on improving combustion performance and reducing emissions in advanced jet engine designs that would use alternative fuels including but not limited to F-T fuels. In the 1920s the F-T fuel process was first developed by Franz Fisher and Hans Tropsch at the Kaiser Wilhelm Institute for Coal Research in Germany prior to World War II. This process was initially developed to help Germany’s need for fuel while in the middle
of a petroleum shortage. The initial process for producing F-T fuels was focused on converting coal into synthetic fuel, since Germany had an abundant coal supply.

Fischer-Tropsch jet fuel is produced from reacting synthesis gas (carbon monoxide and hydrogen) over the surface of a catalyst. F-T jet fuel is comparable to Jet A-1 fuel and has been shown to have superior properties in engine testing. Research has proven significant reductions in particulate emissions without affecting engine performance, excellent thermal stability at elevated temperatures, and superior low temperature properties. Since F-T fuels do not contain aromatics or sulfur contaminants, they are typically considered higher quality, and have shown to burn cleaner than petroleum-based fuels. This in turn results in lower emission concentrations of CO, unburned hydrocarbons, and harmful particulates being released into the atmosphere.

Although there are many positive aspects of F-T fuels, there are also technical, economic, and strategic challenges that come along with this type of fuel. Challenges that still need to be overcome include the interchangeability of F-T fuels with crude-oil derived kerosene-type fuels; sealing compatibility of fuel systems already conditioned in crude-oil derived kerosene-type fuels with succeeding exposure to F-T fuels containing no aromatics; demand and supply of F-T fuels at prices comparable to crude-oil derived kerosene-type fuels; and the modification of existing fuel specifications to allow for general approval of F-T kerosene-type fuels (Ref. 3). The standards set by Sasol in South Africa for supplying alternative fuel to commercial aircraft is a major factor driving today’s technology and investments in the direction of producing synthetic F-T fuels.

Research hardware at the NASA Glenn Research Center consists of three-1 L, high pressure, Fischer-Tropsch continuous stirred tank reactors (CSTR). Inside of these reactors, gaseous hydrogen and carbon monoxide are mixed in the presence of a catalyst and heated under pressure to cause the F-T synthesis reaction. The F-T reaction products are collected as molten wax, liquid oil, water, and light gas. Catalysts used in the F-T process are either iron or cobalt based materials with promoters and supports such as silica and alumina. The gas, liquid, and molten wax samples taken from the F-T reactors are thoroughly analyzed by Gas Chromatographs (GCs) to investigate F-T reaction kinetics and product distributions.

A Hot Liquid Process Simulator (HLPS) enables thermal stability experimental studies to be conducted with conventional and alternative aviation fuels, as well as blended fuel derivatives (Ref. 1). This system provides a rapid thermal stability screening test for candidate fuels before conducting expensive experiments in combustion test facilities.

**Facility Overview**

The Alternative Fuels Laboratory located at the NASA Glenn Research Center in Cleveland, Ohio is the only test facility at NASA Glenn solely used for Alternative Fuels Research. Figure 1 shows an external view of the Alternative Fuels Laboratory.

The Alternative Fuels Laboratory consists of a control room, power supply room, and a high bay test cell. The control room contains a portion of facility control systems, an operator’s control station, and the primary data acquisition system. A separate room within the control room contains a Hot Liquid Process Simulator (HLPS), an Ellipsometer and associated data acquisition hardware for Fuel Stability Testing. The power supply room contains various mechanical systems, ventilation controls and facility power panels. The 840 ft² test cell contains the Fischer-Tropsch Reactor hardware, system components, an activation reactor, a mini-lab housing three GCs, and ventilation system. The test cell also includes a mezzanine area in which individual reactor gas control panels and a catalytic combustor are installed. The test cell also contains individual reactor control systems which are operated by Programmable Logic Controllers (PLCs). Figure 2 shows all three CSTRs at floor level.
Figure 1.—External view of Alternative Fuels Laboratory.

Figure 2.—Three CSTR reactors units at floor level, control panels and combustor on mezzanine level.
A small, partially enclosed miniature laboratory (mini-lab) used for sample analysis is also located directly inside of the test cell. The mini-lab consists of a sink, cabinetry, countertops, three gas chromatographs, a computer and printer. Figure 3 shows the mini-lab enclosure.

Figure 3.—Mini-lab enclosure.
Building Utilities

Gaseous nitrogen, compressed shop air, gaseous argon, and domestic water are supplied directly into the building from surrounding test facilities. The 2400 psig gaseous nitrogen supply is used to pressurize and purge the F-T reactor systems and auxiliary equipment with flow capabilities from 0 to 120 SLPH for each reactor. Compressed shop air is also available at 125 psig in multiple locations throughout the laboratory. Ultra high purity hydrogen, ultra high purity carbon monoxide, and nitrogen/argon gas feed systems are also supplied to the test facility with a flow range from 0 to 120 SLPH for each reactor.

Facility Research Systems

The alternative fuels research hardware consists of three CSTRs, a fixed-bed reactor and a catalytic combustor system. All three CSTR reactors have identical specifications. The basic reactor units were procured from Pressure Products Industries (PPI), model FC-1 series, 1-liter reactors. The CSTR reactor vessels are rated for up to 6,000 psig (408 atm) at 343 °C (650 °F) (Ref. 4). Current operating pressures for the CSTR reactors are limited to 450 psig at 280 °C, based on the system designs employed by Glenn Research Center and by the operational requirements for running F-T synthesis studies. Figure 4 shows a CSTR unit (Ref. 4).

![Figure 4.—One (1) liter capacity CSTR (Ref. 4).](image-url)
A block flow diagram in Figure 5 depicts the F-T rig process configuration. Each integrated reactor system includes individually controlled gas feed systems, a continuous stirred reactor unit, and individual sampling ports for removing refinery gas, liquid, and molten wax samples. Each CSTR is equipped with a dedicated programmable logic controller (PLC) for control and operation monitoring.

The internal components of each F-T reactor consist of an agitator, a gas feed sparger and sample filter. The F-T reactor itself is constructed of 316 SS material and has internal body dimensions of 3 in. diameter by 10.25 in. long and has a capacity of 1 liter. The reactor is custom fitted with an internal agitator driven by a 1/2 HP AC inverter duty variable speed explosion proof motor, typically controlled to run at 500 to 1250 rpm. The reactor agitator can be operated as low as 100 rpm for catalyst activation purposes.

The agitator shaft is hollow in order for the vapor phase product to be re-circulated from top to bottom in the reactor. There are two sets of agitator blades on the shaft. The first set of pitched impellers is equally level with the gas/liquid product interface to force downward gas action and to reduce foaming. The second set of impellers consists of six vertical turbine blades located at the bottom of the shaft. A gas feed mixture consisting of CO, H₂, and Ar is injected into the reactor below the turbine blades to maximize contact and to break up gas feeds into small diameter bubbles. An internal baffle is installed in the reactor to improve contact efficiency and to avoid swirl.

Liquid product draw-off is taken from the top of the reactor liquid volume across a sintered metal filter. The filter selection is based on the catalyst to be tested. Internal reactor temperatures can be controlled through reactor wall heaters by a dedicated PLC for each reactor. Reactor pressure is individually controlled by a manual back-pressure regulator on each reactor. Figure 6 shows a cut-away view of the internal reactor components (Ref. 4).
Figure 6.—Cut-away view of internal components of CSTR from pressure products industries (PPI)—(Ref. 4).

Each F-T reactor assembly has three stainless steel cylindrical traps installed downstream of the reactors, which are used to collect F-T liquid products as three different fractions including molten waxes, light oil and water condensate. Each trap is 500 cc in capacity. Two traps are heated with electric heat tracers which are controlled by the reactor PLCs at the following conditions: hot trap at 200 °C (wax); warm trap at 100 °C (light oil). Water by-product is collected in the cold trap which is wrapped with a copper cooling coil where chilled ethylene glycol is circulated to maintain an internal temperature of 0 °C. Liquid sample level is maintained inside the reactor by manually drawing off liquid samples daily. To prevent wax from plugging the sample lines, all liquid lines are heated with heat tracing tape to approximately 175 °C.

Fixed-Bed Reactor R-4

The R-4 reactor is a fixed-bed reactor designed by NASA GRC based on requirements of a similar unit used by the Center for Applied Energy Research at the University of Kentucky (Ref. 5). This separate reactor is used for the activation of cobalt catalysts. The reactor is designed to process catalyst materials up to 225 psig and 430 °C with controlled flows of gaseous hydrogen, carbon monoxide, and either argon or nitrogen as an inert gas. Independent gas flow rates to the reactor can range from 0 to 120 SPLH. The average amount of catalyst loaded into the reactor is 10 to 20 g, although the amount varies according to the test parameters. The reactor is capable of holding approximately 50 g of catalyst, depending on catalyst density.

The reactor is fabricated from a 1 in. o.d., seamless 316 stainless steel tube with a wall thickness of 0.083 in. and is 12 in. long. A catalyst bed is supported by packed glass wool that is inserted inside of the reactor and is replaced for every test. The reactor is heated by an external electric mighty-band coil heater rated up to 940 W.

The reactor instrumentation monitors reactor internal and wall temperatures along with pressure. Two type K thermocouples are inserted internally from the bottom of the reactor and are at fixed lengths. One thermocouple is near the bottom of the catalyst bed (~1/4 in. above the glass wool), while the other is near the center of catalyst bed. Multiple thermocouple locations verify that the catalyst bed is uniformly heated to enable full catalyst activation. Figure 7 shows a diagram of the fixed-bed reactor with glass wool and catalyst loading.
The fixed-bed reactor is controlled by both a PLC and Labview (Ref. 7) software, which regulates gas flows, temperatures, and automatic alarms and shutdowns for high temperatures and pressures. The reactor temperature control includes a ramp and soak PID controller and an optional manual control with the use of the Labview control software. Figure 8 shows pictures of the fixed-bed reactor (R-4) with and without an insulation jacket, which is typically used during operation.

**Catalytic Combustor**

A catalytic combustor system is installed on the exit side of the reactors to oxidize refinery gases generated during the Fischer-Tropsch reaction prior to venting into the atmosphere. The combustor system is an in-house GRC design which has been successfully utilized in other facilities at NASA Glenn. This combustor takes electrically preheated air from the 125 psig facility shop air system and mixes it with the refinery gases prior to the introduction into the catalytic combustor. The combustor operating temperature is controlled nominally between 750 to 850 °C by varying the air feed flow that is sent to the combustor. The percent excess air used in the oxidation process ranges from 480 to 540 percent (above stoichiometric). The combustor exhaust gases are air quenched down to 150 to 260 °C prior to release into the atmosphere through the facility vent stack. The combustor operates below 15 psig and is less than 6 in. in diameter. This gas clean-up system effectively eliminates CO, CH₄, and residual light hydrocarbon emissions for a more environmentally friendly discharge into the atmosphere. Figure 9 shows the catalytic combustor.
Figure 8.—Fixed-bed reactor R-4 with and without insulation jacket.

Figure 9.—Catalytic combustor.
Hot Liquid Process Simulator (HLPS)

The Alternative Fuels Laboratory also houses a Hot Liquid Process Simulator (HLPS), model HLPS-400 manufactured by Alcor (Ref. 1). The HLPS analyzer is used to determine fuel breakpoint according to the ASTM D3241 specification test called Jet Fuel Thermal Oxidation Test (JFTOT). Breakpoint can be defined as the highest temperature at which a fuel can pass the JFTOT (Ref. 2).

The HLPS uses the idea of running fluid through a resistance heated tube-in-shell heat exchanger while monitoring flow, temperature, and pressure. An air saturated fuel sample is loaded into a high-pressure reservoir. The fuel reservoir is pressurized to ~500 psig with gaseous nitrogen and is set to a constant flow rate. At the outlet of the reservoir the fuel flows through a pre-filter and then over the heated tube. The heated tube is controlled by a ramp and soak controller which increases tube temperature to the specified set-point. At the outlet of the heated tube, a small filter is in place to capture any particulates in the heated fuel. The HLPS analyzer measures differential pressure (DP) over this filter. At the end of 150 min test, the tube is assessed by tube discoloration in a visual tube rater which is an internally lit black box consisting of a standard ASTM color chart. The tube is optically compared to the color chart and is assigned a color number ranging from 1 to 4 (1 is metallic silver, 2 is slightly tan, 3 and 4 are shades of brown). A color rating of three or above or a DP greater than 25 mmHg constitutes a failed test. This test is repeated in 5 °C increments until a failed test is observed. The HLPS analyzer is equipped with a visual tube rater to rank tube discoloration; a differential pressure transducer to quantify heat induced particulate formation; and a heater system to handle a wide range of fluids including highly viscous materials. Figure 10 shows a diagram of the hot liquid process simulator unit. Figure 11 shows the test tube apparatus cutout views.

Figure 10.—Hot liquid process simulator.
The HLPS components are mainly constructed of stainless steel. The HLPS reservoir can hold up to 1 liter of fuel and is capable of testing fuel fouling tendencies at conditions up to 650 °C. Various types of heater tubes can be used in order to reach temperature requirements for each individual test. JFTOT tests require the use of an Aluminum tube, which has an upper limit of 380 °C. Steel or stainless steel tubes are also available for tests requiring higher temperatures of up to 650 °C. The flow rate can be varied from 0.25 to 5 mL/min for each test, although JFTOT requires a set flow rate of 3 mL/min. Typical tests use approximately 600 mL of fuel per test, and take approximately 4 hr to assess one temperature point. The number of tests needed per fuel sample depends on what fuel is tested, and where the actual breakpoint temperature is found.

**Gas Analysis Systems**

The Gas Analysis equipment is located in the mini-lab enclosure located inside of the test cell. Gas Analysis equipment consists of an Agilent 3000A Micro Gas Chromatograph (Micro GC) and two Agilent 6890 Gas Chromatographs (GC) (Ref. 6). The Micro GC is used to analyze refinery gas samples taken directly from the F-T reactors on a continuous basis. One of the Agilent 6890 GC is designated for oil analysis, and another is designated for wax analysis.
All three gas chromatographs separate and identify compounds in the given sample. Agilent’s EZChrom (Ref. 6) software is used to control the GCs and generate a detailed report for all samples sent to or injected into the gas chromatographs. The chemical compositions and quantities are used to study the overall mass balance, product distribution, and reaction kinetics. The gas chromatographs are capable of measuring compositions of light refinery gas mixtures \((H_2, CO, CH_4, C_1 – C_4, CO_2, \text{ and } N_2)\), light oils \(C_4 – C_{44}\), and heavy hydrocarbons \(C_{11} – C_{80}\) (alkanes and alkenes).

Refinery gas samples are delivered to the Agilent 3000A Micro GC through stainless steel tubing, which directly connects the product gas line to the inlet of the GC. A gas liquid separator is attached to the inlet of the Micro GC. This serves as a flow controller and filter for samples before they enter four internal capillary columns and pass through a Thermal Conductivity Detector (TCD) for component identification. The Agilent 3000A Micro GC contains four internal columns which all have unique separation capabilities and therefore require specific carrier gases to move the samples through the columns. Columns currently being used consist of a “MS 5A" column which uses Argon as a carrier gas, and a “Plot U”, “Alumina Plot”, and “OV-1” columns which helium as a carrier gas. Calibration is performed daily using one of two catalyst defined calibration gas mixtures. Figure 12 shows the Agilent 3000A Micro GC.

The Agilent 6890 GC’s are used to analyze liquid hydrocarbon products that are manually collected from the reactors. Light oil samples and wax samples are analyzed by two separate GC’s each equipped with an Automatic Liquid Sampler for direct injection into a column. Once a sample is injected into the GC, helium carrier gas moves the sample through either an Agilent “DB-5" or "HP5" column before it is sent to a hydrogen Flame Ionization Detector (FID) to be identified. Calibration is performed periodically using applicable chemical standards. Figure 13 shows the Agilent 6890 GC.
Facility Control Systems

Facility process systems are operated by two main control systems: Programmable Logic Controllers (PLCs) and Labview software which utilizes National Instruments hardware (Ref. 7). Each F-T reactor is independently controlled and monitored by a designated PLC. The fixed-bed reactor, catalytic combustor and all other components are controlled by the Labview software. PLCs are primarily used to control the three main reactor units and do not save data.

EZChrom software is used to control Gas Chromatograph operations, generate sample data reports, and provide automated sampling and analysis sequences (Ref. 6). The EZChrom control system operates all gas chromatographs according to user defined parameters.

The HLPS can be operated either by using a push button control panel located directly on the HLPS unit or by the Labview program installed on the HLPS computer in the laboratory. The software for the HLPS operation was designed by Alcor-PAC and can only be modified by Alcor (Ref. 1).

Instrumentation/Emergency Shutdowns

The F-T reactor systems include numerous types of instrumentation used to monitor and control facility and system operations. Temperatures, pressures, and flows are reported by the PLCs and Labview and are stored as data files. Both the PLCs and Labview contain integrated alarm features to ensure orderly and safe shutdowns. Facility and/or reactor shutdown sequences are instantaneously initiated when any programmed alarm or shutdown point is reached. The current Labview configuration for instrumentation consists of 278 Labview channels which are available for data acquisition. The expansion of additional modules and channels is available.

Temperature measurements are obtained with type-K thermocouples, while pressure measurements are taken with high accuracy diaphragm-type pressure transducers. Gas supply flow rates are controlled by mass flow controllers for each individual gas supply system. Refinery gas flow rates are measured by the use of wet test meters. All components are calibrated on a regular basis to ensure accuracy.

Data Acquisition

One computer has been designated as the primary data acquisition system and is directly connected to the National Instruments hardware and internal facility network. This computer executes the Labview software which collects data from the facility instrumentation. Data is transferred from the PLCs to the Labview computer for data logging purposes. The Labview software does not have control authority over the reactor PLCs other than to initiate emergency shutdowns. The second computer in the control room is used for monitoring the gas chromatographs processes, data retrieval, and monitoring individual reactor operations.

The EZChrom software is solely used for GC data acquisition (Ref. 6). All liquid and gas sample results are stored in the GC computer.

The thermal stability data obtained from the Hot Liquid Process Simulator is saved directly on the HLPS computer through the Labview program.

Test Duration

Fischer-Tropsch research consists of long duration reactor tests which run continuously 24 hours a day, 7 days a week. Once F-T testing is initiated, the facility runs in an automated, unattended mode with the exception of daily sample collection. Approximately two months of the year is used for facility maintenance, upgrades, and instrument calibration.
Thermal Stability tests conducted with the HLPS unit take approximately 4 hr per sample. Multiple sample tests are needed to determine the actual thermal stability of the fuel being analyzed. A maximum of two tests can be performed per day, and it typically takes about 1 to 3 days of testing to determine the actual “breakpoint” of a fuel. Thermal Stability tests can be performed at any time and do not have a designated maintenance shutdown period.

**Future Installation**

A steam generator system which can be used to vaporize de-ionized water for research purposes has already been designed for future installation. The design has incorporated a Chromolox (Ref. 8) vertical steam boiler, model CHPES which is ASME Section I ‘S’ stamped pressure vessel. The system will be able to generate 60 lb per hour of steam at 235 psig. It is a commercial off the shelf process system that consists of a condensate flash vessel, piping, valving, pump, electric vaporizing heater, associated instrumentation and controls with factory preset shutdowns for safe operation.

A de-ionized water (DI) system which will provide conditioned water to the steam generator system and the mini-lab has also been planned and designed for future installation. The de-ionized system is an electrically powered, commercial off the shelf unit that will be provided by Siemens Water Technologies (Ref. 9). This system will provide between 0.15 to 3 gpm of de-ionized water flow. It will provide De-ionized water at 50 to 200 KΩ-cm to the steam generator and de-ionized Industrial-Grade water at >1 MΩ-cm to the mini-lab.

| TABLE 1.—FACILITY CAPABILITY SUMMARY |  |
| F-T reactors |  |
| Reactor capacity | 1 liter |
| Total number of reactors | 3 |
| Materials of construction | 316 SS |
| Operating pressure | 0 to 450 psig |
| Operating temperature | 0 to 280 °C |
| Gas feeds | UHP H2, UHP CO, N2 and Argon |
| Gas feed flow rates | 0-120 SLPH per feed |
| Shop air | 125 psig |
| Reactor motor speed | 100 to 1250 rpm |
| Catalyst type | Cobalt or Iron |

| Fixed bed reactor |  |
| Material of construction | 316 SS |
| Operating pressure | 0 to 225 psig |
| Operating temperature | 0 to 430 °C |
| Gas feeds | UHP H2, UHP CO, N2 and Argon |
| Gas feed flow rates | 0 to 120 SLPH per feed |

| Hot liquid process simulator |  |
| Materials of construction | 316 SS |
| Reservoir capacity | 1 liter |
| Amount of fuel used per test | 600 ml |
| Pressure | 500 psig |
| Maximum temperature | 650 °C |
| Flow rate range | 0.25 to 5 ml/min |
| Single test duration | 4 hr |

| Gas chromatography |  |
| Agilent 3000A Micro GC (gas) | TCD, C1 to C4 compounds |
| Agilent 6890 GCs (liquid) | FID, C6 to C80 compounds |
Concluding Remarks

The F-T reactors are capable of determining the conversion of syngas into synthetic crude from non-conventional resources. By using the F-T reactors to develop novel catalysts coupled with the development of the innovative F-T process, new pathways can be identified to reduce future fuel production costs. F-T process promotes fuel production from non-petroleum sources such as coal, natural gas, and renewable biomass. Alternative jet fuels thermal stability can be assessed using HLPS in this facility.

Future research plans include the testing of novel catalysts with unique types of supports and promoters. Catalyst developments will be performed at NASA Glenn along with Universities and external businesses specializing in catalyst development.

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**SUBJECT TERMS**
Alternative fuels; Fischer-Tropsch; Thermal stability; F-T reaction kinetics; Product yields; Hydrocarbon distribution; Catalyst; Thermophysical properties; Aviation fuels; JFTOT

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