SYSTEM CHARACTERIZATION AND CATALYTIC IGNITION OF PROPANE-AIR MIXTURES IN A PLUG FLOW REACTOR

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16. Abstract
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The catalytic plug flow reactor underdevelopment will allow the study of combustion properties of aqueous ethanol. Time response characterization of the mixing section was performed to understand how long it will take transient upstream conditions effect the reaction after the mixing section. Experiments were performed to determine what distance downstream from the mixing section actually had plug flow. A mathematical heat transfer model was also created to understand the energy flow around the catalytic ignition wire in the reaction section. This was followed by finite element modeling of the catalytic wire. The first tests of ignition temperature were conducted using propane/air mixtures over a heated platinum wire.

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**Table of Contents**

EXECUTIVE SUMMARY .......................................................... 1

INTRODUCTION ................................................................. 1

  Background and Motivation ................................................ 2
  Catalytic Plug Flow Reactor ............................................... 4

DESCRIPTION OF PROBLEM .................................................. 5

  Construction and Working ................................................ 7
  Mixing Section .............................................................. 7

APPROACH AND METHODOLOGY ........................................... 9

  Response Time Experiments .............................................. 9
  Verifying Plug Flow ...................................................... 11

Surface Temperature Model and Experimental Set up ..................... 12

  Microcalorimetry ......................................................... 12
  Data Acquisition .......................................................... 14
  Test Apparatus ............................................................ 15
  Finite Element Ignition Temperature Model ........................... 17

Finite Element Model (Algor) .............................................. 18

  Input parameters .......................................................... 18
  Sensitivity Studies ....................................................... 19

Finite Element Energy Balance Model ................................... 20

  Boundary Conditions ..................................................... 21
  Energy Balance ............................................................. 22
  Parameters Investigated ................................................ 23
  Theoretical Analysis ..................................................... 23

Finding ............................................................................... 26

  Plug Flow ................................................................. 26
  Surface Temperature ...................................................... 27

Summary of Equivalence Ratio Experiments ............................... 31

Finite Element Model Results ............................................. 33
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>CONCLUSIONS</td>
<td>38</td>
</tr>
<tr>
<td>Plug flow reactor</td>
<td>38</td>
</tr>
<tr>
<td>System Modeling</td>
<td>38</td>
</tr>
<tr>
<td>Catalytic ignition temperature</td>
<td>38</td>
</tr>
<tr>
<td>Recommendations</td>
<td>39</td>
</tr>
<tr>
<td>System modeling</td>
<td>39</td>
</tr>
<tr>
<td>Catalytic ignition temperature</td>
<td>39</td>
</tr>
<tr>
<td>REFERENCES</td>
<td>41</td>
</tr>
</tbody>
</table>
EXECUTIVE SUMMARY

This project advanced NIATT’s goal of using aqueous ethanol in vehicle transportation. This report describes further progress on the catalytic plug flow reactor;

The catalytic plug flow reactor underdevelopment will allow the study of combustion properties of aqueous ethanol. Time response characterization of the mixing section was performed to understand how long it will take transient upstream conditions effect the reaction after the mixing section. Experiments were performed to determine what distance downstream from the mixing section actually had plug flow. A mathematical heat transfer model was also created to understand the energy flow around the catalytic ignition wire in the reaction section. This was followed by finite element modeling of the catalytic wire. The first tests of ignition temperature were conducted using propane/air mixtures over a heated platinum wire.
INTRODUCTION

The purpose of this project was to develop and test an apparatus and methodology for studying catalytically ignited plug-flow combustion of aqueous fuels. We are especially interested in being able to address cold starting issues in Aquanol based vehicles involving catalytic ignition. Aquanol is a mix of 65 percent ethanol and 35 percent water by volume. Research has been conducted and patents issued on the subject of aqueous fuel combustion with catalytic assistance. Cold starting is one the least understood aspects of igniter operation and has motivated the construction of a catalytic plug flow reactor (Fig. 1) at the University of Idaho to explore the relationship between average catalyst surface temperature, fluid flow velocity, power supplied and equivalence ratio.

![Figure 1 Catalytic plug flow reactor](image)

This investigation started by characterizing the performance of a specially designed mixing nozzle and characterizing the plug flow region for insertion of platinum catalyst downstream of the nozzle. This was followed by more fundamental experimental and finite element studies to better understand the changes in average surface temperatures by varying parameters including the flow velocity, power supplied, and equivalence ratio. Ignition temperatures were also obtained as functions of flow velocity and equivalence ratio. These results shall form a platform
for future research with Aquanol air mixtures over platinum wires. In this introductory chapter, the background and motivation for this work are first presented followed by brief discussions on the benefits of Aquanol fuel combustion, catalytic igniter technology and on the catalytic plug flow reactor. Finally the specific objectives of this study are presented along with a brief outline of this thesis.

**Background and Motivation**

Previous studies, discussed below, indicate that lean mixtures can tolerate higher compression ratios leading to an increase in engine efficiency. Lean mixtures have the potential of reducing NOx and CO emissions in internal combustion engines. However, lean mixtures are not readily ignited and are characterized by a low flame propagation velocity. One effective method to improve flame initiation is catalytic assistance of ignition and combustion. The transition state of the molecule catalyst complex is then in a low energy configuration which provides an easier route for the reactants to proceed in subsequent reactions to form the final products. The fact that the catalyst can have strong influence on the reaction mechanism, in addition to lowering the overall activation energy, was suggested by the work of Patterson and Kembal [1], who obtained a negative reaction order in fuel for the oxidation of olefins on platinum films. Similar findings have been reported by Moro-Oka et.al. [2], by Schwartz et al. [3], and by Cardoso and Luss [4].

Bruno and coworkers [5] demonstrated experimentally that surface chemistry plays a vital role in the conversion of fuel to CO$_2$ and H$_2$O when lean preheated propane-air mixtures are passed over platinum catalyst. Cho and Law [6] performed propane-air mixture experiments at low Reynolds number in steady state flow conditions to achieve axial symmetry of the flow along the length of wire and to minimize convective heat transfer losses. The results of these experiments were reported for a flow velocity of 5-10 cm/s that corresponded to a Reynolds number of 0.1. They used 0.5 - 2.0 percent of propane by volume in the fuel-air mixtures and the experimental data was independent of the fluid velocity. Their paper mentions that since catalytic combustion is basically a three component system involving fuel/oxidizer/catalyst, with strong coupling between the gas and surface processes, simplified approaches in the above manner can lead to
ambiguities in attempts to understand the phenomenon under investigation. The experimental and finite element studies reported here explore the changes in catalyst surface temperature with the variations in flow velocity, fuel concentration, oxygen concentration, platinum wire geometry, power supply and preheated temperatures of fuel air mixtures at higher Reynolds number.

The ultimate goal is to explore a possible solution for cold starting problem while using Aquanol fuel in vehicles. Ethanol, a renewable fuel, is produced primarily from grains and other agricultural crops. Previously research has been conducted to reap the benefits of alcohol-based fuels. The addition of water to ethanol in a combustion chamber reduces flame temperatures and thus reduces NOx emissions. Studies of aqueous transportation fuels, discussed below, indicate that in majority of the cases, engines had cold starting problems so they were always started on gasoline and once the engine was warm enough they were changed to aqueous fuels. The primary drawback to Aquanol is the difficulty of initiating combustion. Conventional sources of ignition such as spark plugs cannot ignite these aqueous ethanol air mixtures because of the high water concentrations involved extinguish the flame. Likewise, compression ignition of aqueous fuels has been largely ineffective due to problems in controlling the ignition timing. In conventional design combustion chambers with commonly used fuels, it is very difficult to increase the compression ratio due to problems of knocking, throttle and mechanical losses. Earlier research by Jarosinski and Podfilipski [7] has been done using modified design combustion chambers with typical fuels such as gasoline and diesel. Also, research using alternative alcohol based fuels but with conventional design combustion chambers has been done.

The research reported here is unique since it combines features of both of the above approaches and uses alcohol based fuels in a modified design combustion chamber. Work with water-naphthalene and water-alcohol mixtures pioneered the use of water as a combustion diluent and temperature reducer for NOx reduction in diesel and SI engines [8]. If this technology were to be commercialized, gasoline or diesel would have to be delivered to the engine during start-up,
necessitating two separate fuel-handling systems. This would not be cost effective and could not be retrofitted to existing engine.

Hence to eliminate double fuel handling systems and to address engine cold starting issues, catalytic ignition research was initiated at University of Idaho. This ignition source is elementary in the conversion of an engine for operation on Aquanol. Initial experiments were performed with propane-air mixtures over platinum wires and gradually we shall shift to Aquanol-air mixtures. These studies provided the ground and motivation for pursuing this research on catalytic assisted ignition and combustion.

**Catalytic Plug Flow Reactor**

As discussed the catalytic plug reactor has many features that make it a promising combustor design for overcoming many difficulties associated with conventional combustor chamber designs. This specially designed catalytic reactor can handle both liquid and gaseous fuels. It operating pressures will range from 1 atm to 15 atm and it will be able to withstand temperatures up to 1700 K.
DESCRIPTION OF PROBLEM

Introducing catalytic surfaces in combustion systems supplies an additional control parameter, namely the surface reaction rate. Diffusion of energy and intermediate species released from exothermic surface reaction can dramatically accelerate the gas phase reaction and initiate ignition at low temperatures.

This work is one of a series of experimental and modeling investigations designed to help understand the ethanol-water gas phase and surface mechanisms. The experimental approach includes the design, construction and testing of a plug flow reactor that can be pressurized to typical pre-ignition engine conditions. The modeling approach includes thermodynamic calculations at different ethanol-water volume factions, equivalence ratios, and pressures. Detailed chemical kinetic modeling of gas-phase and surface reactions, coupled with experimental results, will permit us to develop a simplified model that accurately predicted ignition.

A literature search for flow reactor systems provided information about standard designs features of reactors for combustion studies. Table 1 below summarizes the nominal operating ranges for the reactor based on the work of Koert, Hoffman, Dryer, Karim, Vermeersch, Mao, Mitchell [15 - 21] and operating ranges for the catalytic plug flow reactor at University of Idaho.

The combustion section of the reactor will contain catalysts in different arrangements as needed for the experiments. The work reported here used a platinum catalyst wire for igniting the fuel-air mixture. Instrumentation and calculations related to the wire will be used to determine the transition temperature from kinetic to diffusion control of the ignition process, based on the experimental method of Schwartz, et al. [3]. The flow section ensures plug flow – a uniform cross section of temperature, velocity, and concentration.
Table 1 Summary of design parameters

<table>
<thead>
<tr>
<th>Operating Parameter</th>
<th>Units</th>
<th>Range</th>
<th>University of Idaho Catalytic Plug Flow Reactor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>cm</td>
<td>2.2 – 10.0</td>
<td>10.0</td>
</tr>
<tr>
<td>Reactor Length</td>
<td>cm</td>
<td>30.0 - 80.0</td>
<td>85.0</td>
</tr>
<tr>
<td>Mixing Length</td>
<td>cm</td>
<td>20.0</td>
<td>11.0</td>
</tr>
<tr>
<td>Maximum Pressure</td>
<td>atm</td>
<td>1 – 28</td>
<td>1 – 15</td>
</tr>
<tr>
<td>Maximum Temperature</td>
<td>K</td>
<td>1000 – 1700</td>
<td>1700</td>
</tr>
<tr>
<td>Reynolds Number</td>
<td></td>
<td>Turbulent: Koert, Dryer, Hoffman Laminar: Karim</td>
<td>Laminar and turbulent</td>
</tr>
<tr>
<td>Residence Time</td>
<td>ms</td>
<td>2 – 250</td>
<td>70</td>
</tr>
<tr>
<td>Mixing Time</td>
<td>s</td>
<td>30 – 50</td>
<td>10 - 20</td>
</tr>
<tr>
<td>Velocity</td>
<td>cm/s</td>
<td>50 – 700</td>
<td>900</td>
</tr>
<tr>
<td>Cooled Sampling Probe</td>
<td>Type</td>
<td>Used by Koert, Dryer, Hoffman, Dagaut, Mao, Marr [15 – 21]</td>
<td>Under construction for future work</td>
</tr>
<tr>
<td>Quartz Tube</td>
<td></td>
<td>Used by Koert, Dryer, Dagaut, Vermeersch</td>
<td>Used for flow section</td>
</tr>
<tr>
<td>Analysis Systems</td>
<td>Type</td>
<td>Gas Chromatography, Mass Spectroscopy, FTIR, NDIR, NID</td>
<td>Gas Chromatography Mass Spectroscopy (GCMS)</td>
</tr>
</tbody>
</table>
**Construction and Working**

The schematic in Fig. 2 illustrates the fuel inlet, mixing, flow, and combustion sections of the pressurizable reactor.

![Diagram of catalytic plug flow reactor](image)

**Figure 2 Sections of the catalytic plug flow reactor**

Fuel nitrogen mixture is introduced from the inlet port as shown in Figure 12. Oxygen is introduced from the side port where it mixes with the fuel nitrogen mixture to have the desired equivalence ratio.

**Mixing Section**

The mixing section is designed in such a manner that it minimizes the mixing times and ensures complete mixing. In the mixing nozzle, the nitrogen-fuel mixture tapers into a tube and is vented outward through six small holes, similar to a design developed at Drexel University [6]. Oxygen enters the middle ring where it fills a large cavity and is mixed with N₂ via 18 small holes. A sleeve creates the outside wall of the cavity, forming a plenum, where oxygen enters.
A prototype-mixing nozzle was machined from polymethyl methacrylate and tested for fit with the evaporator section. The gas-mixing nozzle design aimed to preserve a step-change when the concentration of one gas stream — the fuel-nitrogen stream — was suddenly changed. Experiments described in the next section used two CO$_2$-N$_2$ gas streams of different compositions verified complete mixing.
**APPROACH AND METHODOLOGY**

The aim of these experiments was threefold. First, we wanted to verify the operation of the mixing nozzle in terms of response time. Second, we needed to find the axial limits of the plug flow region for insertion of the platinum catalyst. For this purpose, a hot wire anemometer was used for obtaining the velocity profile at various radial positions and at different axial positions. Third, we needed to characterize the average surface temperature of the Pt wire in plug flow for comparison with an FEA model.

**Response Time Experiments**

Tests were performed to determine how the sampling probe length and flow rate of the gases affect the response time. This procedure was performed for sampling probe lengths of 0.889 m and 2.565 m. The flow rate was kept constant at 13.2 percent total for the calibration gas and 16.4 percent total for the oxygen. This corresponds to a volumetric flow rate of about 43 cm$^3$/sec.

Figure 3 shows the experimental set up for studying the effect of changing the sampling probe length on the response time. Gases from two fifteen-gallon tanks were flowed through the flow meters.
Calibration and nitrogen gas entered the reactor as shown in Fig. 3. Initially the calibration gas is started and is allowed to reach steady flow conditions. After 70 seconds the nitrogen gas regulator is opened. The nitrogen gas flows through the 18 small holes of the middle ring of the mixing nozzle and mixes with the calibration gas. From this point the gas proceeded through the mixing nozzle and to the probe located in the exhaust pipe. This probe was connected to the five-gas analyzer used to collect data.

![Figure 4](image)

**Figure 4 Change in N\(_2\) when the sampling probe length is 2.565 m**

The volume concentration of nitrogen changed from steady state 92.4 percent to 95.77 percent within time interval of around 15 seconds. The time constant \(\tau\) is calculated by using the following relationship

\[
X = e^{t/\tau}
\]  

(1)

where \(X\) is the change in concentration, \(t\) is the time interval of change and \(\tau\) is the time constant. The time constant \(\tau\) determined using this relationship is 9.87 sec.
Table 2 Effect of changing the flow rates on response time

<table>
<thead>
<tr>
<th>Length (m)</th>
<th>Pressure (atm)</th>
<th>Flow Rate, m³/min</th>
<th>Response Time, s</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Calibration gas</td>
<td>N₂ gas</td>
</tr>
<tr>
<td>2.565</td>
<td>3.40</td>
<td>13.6</td>
<td>16.0</td>
</tr>
<tr>
<td>2.565</td>
<td>3.40</td>
<td>13.6</td>
<td>16.0</td>
</tr>
<tr>
<td>2.565</td>
<td>6.8</td>
<td>23.5</td>
<td>26.5</td>
</tr>
<tr>
<td>2.565</td>
<td>6.8</td>
<td>23.5</td>
<td>26.6</td>
</tr>
</tbody>
</table>

**Verifying Plug Flow**

A systematic procedure for acquiring data was created that could accurately describe the fluid flow conditions downstream of the mixing nozzle. The pressure in the nitrogen supply tank was kept at 3.7 atmospheres and pressure of the oxygen gas tank was kept at 3.4 atmospheres for all sets of experimental data recorded. This insured uniformity of flow. The nitrogen gas was turned on first. After 20 seconds the oxygen was added. The hot wire anemometer was initially placed at 3.6 diameters downstream and was traversed across the flow section at several locations. The same procedure was adopted for recording fluid flow velocity at 5.625, 7.625 and 11.625 diameters of the mixing nozzle and the results are shown are shown in Fig. 5.
Initially when the fluid flows out of the mixing nozzle, the flow is non-uniform across the cross section of the tube. The mixing nozzle is designed in such a manner to create uniform flow across the cross section. In the previous chapter we studied the response time taken by the gases for complete mixing. Here we were trying to find an axial location downstream of the mixing nozzle that would have uniform flow across the cross section.

**Surface Temperature Model and Experimental Set up**

**Microcalorimetry**

Studies, discussed below, indicate that microcalorimetry experiments have been conducted in two different ways: the heated gas technique and the heated wire technique. In the heated-gas technique [23], the pre-heated gas mixture determines the temperature of the catalyst. In the absence of catalytic reactions the catalyst surface temperature is same as that of the gas mixture. But in the presence of catalytic reactions, the surface temperature of the catalyst changes and becomes higher than the ambient temperature. The lowest point at which the temperature of the catalyst changes is defined as the ignition point and the corresponding temperature as the ignition...
temperature. In the heated-wire technique, the gas mixture is heated locally at the catalyst surface by the wire that is the active heating element. Of these two methods, the heated-wire technique has been favored by most of the researchers in extracting kinetic and temperature data out of catalytic reactions.

This research makes use of heated wire technique for obtaining temperature data out of catalytic reactions. Figure 6 shows the microcalorimetry heat transfer model for the platinum wire. Here

- $Q_{\text{In}}$ -- Heat flowing into the system due to the propane-air mixture
- $Q_{\text{Out}}$ -- Heat leaving the system in form of losses
- $Q_{\text{Conduction}}$ -- Heat lost due to conduction
- $Q_{\text{Convection}}$ -- Heat lost due to convection
- $Q_{\text{Radiation}}$ -- Heat lost due to radiation
- $Q_{\text{Generated}}$ -- Heat generated by the electrical circuit
- $Q_{\text{Steady}}$ -- Steady state condition
- $T_{\text{wall}}$ -- Temperature at the walls of the reactor

![Figure 6 Microcalorimetry heat transfer model](image)

\[ Q_{\text{In}} + Q_{\text{Generated}} - Q_{\text{Out}} = Q_{\text{Steady}} \]
The amount of energy entering into the system is the sum of $Q_{In}$ and $Q_{Generated}$. The total amount of energy lost due to conduction, convection and radiation is included in $Q_{Out}$. In the absence of gas flow over the platinum wire, the heat input to the system is solely the term representing the heat generated by the electrical system. The total heat lost by the system is $Q_{Out}$. The temperature of the platinum catalyst can be determined from the equilibrium of heat generation and the heat loss due to conduction, convection and radiation. The power required to maintain the platinum catalyst at temperature $T$ will be

$$P = I^2R$$  \[2\]

where $I$ is the current through the platinum wire and $R$ is the resistance across the platinum wire at temperature $T$. Here the quantity $I$ is measured at the power supply unit and voltage $V$ by using the data acquisition control unit. Previous experimental studies [1] by researchers indicate that wire geometries give more accurate measurement of surface temperature. Wire geometries are easier to construct and are less susceptible to physical change with time. This research has adopted wire geometry for its experimental investigation.

**Data Acquisition**

The experimental facility consists of catalyst configuration, digital flow system, variable power supply unit, and computer data acquisition control unit for monitoring changes in voltage across the circuit.

The reactor is designed for pressures ranging from 1-15 atmospheres and temperatures up to 1700 Kelvin. Its core diameter is 10 cm and it can handle both liquid and gaseous fuels. For the work here, the reactor was operated at atmospheric pressure and a quartz tube replaced the metal combustion section. Fuel nitrogen mixture was made to enter the reactor from the inlet port and oxygen flows through the side port. Porter Mass Flow meters, Model 112 (flow meter)/ Model 202 and 202A (Flow controller) were used for measurement and control of gas flows.

A turbulence grid injection system was used as a mixing nozzle. The mixing nozzle ensured timely and complete mixing. The design of the mixing nozzle was crucial; it needed to provide a
homogeneous mixture in a short period of time. A platinum catalyst was inserted downstream of the mixing nozzle across the fluid flow in a quartz tube.

The most interesting feature of the experimental setup is the electronic circuitry that enables an accurate measurement of ignition temperature. In these experiments the platinum wire performs many functions simultaneously. It acts as a catalyst enabling surface reactions of propane-air mixtures. It is connected to a variable power supply unit and acts as a constant source of heat supply to the system. The 508-micron diameter platinum catalyst is connected to a data acquisition control unit and computer for monitoring the changes in temperature and acts as a temperature transducer.

Due to an increase in the functionality of the catalyst, the data acquisition control unit becomes an integral part of the heating power system. The data acquisition control unit used for our experiments has enough capacity to heat the catalyst to a high temperature favorable for chemical reaction and at the same time and has enough sensitivity to respond to and detect small changes in the power consumption.

For non-reactive conditions where no gas flows over the platinum wire, the voltage and current readings represent the power required to heat and then maintain the platinum catalyst at a constant temperature. Under the influence of fluid flow over the platinum wire the thermal equilibrium is disturbed due to the chemical reactions at the surface of the catalyst.

**Test Apparatus**

The platinum catalyst used for these experiments was purchased from Surepure Chemetals. It is highly resistant to corrosion and electrochemical attack and its purity is 99.99 percent. A simple straight platinum wire of 508-micron diameter was used for these experiments. The axis of the platinum wire was perpendicular to the direction of fluid flow. The average surface temperature of the platinum catalyst was calculated from the measurement of the change in its resistance.
Figure 7 is a flow diagram of propane-air mixture ignition by a platinum catalyst. With the Pt wire connected to a variable power supply and monitoring of voltage, power to the wire was slowly increased as a propane-air mixture was passed over the wire.

As the temperature of the wire increased, the corresponding change in resistance was calculated and the average wire temperature found from Equation 3. The average surface temperature obtained was used for comparison with the FEA model results.

The electrical resistance of platinum varies as

\[ R = R_0 (1 + \alpha (T - T_0)) \]  \[3\]

where \( R \) is the electrical resistance at temperature \( T \), \( R_0 \) is the electrical resistance at temperature \( T_0 \), and \( \alpha \) is the resistivity of Pt, a property of the metal.

Figure 8 shows the plug flow reactor with the platinum catalyst inserted 5.625 diameters downstream of the mixing nozzle. The 508-micron diameter platinum catalyst is connected to a variable power supply unit and the voltage changes with the flow of propane-air mixture over the wire are recorded using the data acquisition control unit.
Figure 8 Plug flow reactor with the platinum insertion

**Finite Element Ignition Temperature Model**

While the propane-air mixture experiments provided average surface temperature, we wanted to model the temperature distribution and use the model to predict average surface temperature for both simple and complicated catalyst geometries. To verify our model, we compared it with the experimental surface temperatures measured with the platinum catalyst wire.

The Algor software package was used for conducting FEA heat transfer analysis. The reasons for using Algor are listed below:

- Built-in precision FEA model-building capabilities with structured meshing tools for building designs from scratch and refining CAD designs.
- It provides support for CAD universal files such as ACIS, IGES and STEP.
- Full association with each design change for AutoDesk Inventor, Pro/ENGINEER, Solid Edge and Solid Works.
- Automatic, unstructured FEA meshing for single parts and assemblies with a built-in aspect ratio check, using bricks, tetrahedral or a hybrid of bricks on the model surface.
- Complete and easy-to-use finite-element modeling, results evaluation and presentation interface, FEMPRO.
This section contains information on three-dimensional FEA studies conducted to understand the relationship between the surface temperature and power supply. Relative significance of fuel/air surface heating, axial conduction to the catalyst supports, surface heat generation, supplemental electrical heating, radiative losses, and convective losses have been documented from the finite element studies and preliminary experiments. Of particular interest is the temperature distribution along the length of the platinum catalyst and comparison of the calculated and measured average wire temperature.

**Finite Element Model (Algor)**

A 3-D element representing a platinum catalyst was first created in SUPERDRAW and then exported to FEMPRO’S FEA Editor environment. Initially, I planned to achieve the temperature distribution using a steady state heat transfer analysis. Modeling in FEMPRO’S FEA Editor environment involves the following steps.

We imported the already developed SUPERDRAW file into FEA, created a meshed model, specified data needed for analysis including the analysis type, element type, and element definition. Next we selected the material, added the applied temperatures, specified surface properties, which included calculating the convection coefficient, ambient temperature, heat flux, fluid properties and wall temperature. Finally we checked the model geometry and finite element data to verify that it is ready for analysis.

**Input parameters**

We considered a platinum catalyst (cylinder) of 254-micron diameter and 0.015 m long in SOLID WORKS. The surface in contact with the wire supports was kept at room temperature, 25°C. The entire outer surface was exposed to convection cooling by the fluid flow. For this particular case study, the Reynolds number was taken as 2,400 to have a similarity with the experimental conditions. The temperature profile is symmetrical; the bottom surface of the FEA model corresponds to the intersection of the catalytic wire and the axial centerline of the reactor.
tube. The temperature profile is symmetrical; the bottom surface of the FEA model corresponds to the intersection of the catalytic wire and the axial centerline of the reactor tube.

The average surface temperature obtained using 3-dimensional FEA analysis for a power supply of 3.87 W was 725 K. The experimental results indicate that for the same amount of power supply the average surface temperature was 662 K. The 3-dimensional propane-air mixture experiments are offset from the experimental results by 9.5 percent.

Figure 9 shows the Algor surface temperature across the length of the platinum catalyst. Here length 0 indicates the center of the platinum catalyst and it is at a temperature of 1082 K and length 15 indicates the top edge of the platinum catalyst wire, which is at a prescribed ambient temperature. The average Algor surface temperature for a end prescribed at 273 K as calculated in Algor comes out to be 725 K.

![Figure 9 FEA surface temperature profile](attachment:figure9.png)

**Sensitivity Studies**

Sensitivity studies were conducted using the Algor FEA package to understand the effects of changing fluid flow velocity and power input to the wire on average surface temperature of the platinum catalyst.
The fluid velocity for this particular case study was 1.52 m/s. We were interested in studying the changes in temperature with respect to the changes in fluid velocity. The other parameters—mesh size, heat flux, and wire geometry—are kept constant. The results indicate that as fluid flow velocity is increased, convective heat losses also increase and so there is a decrease in the average Algor surface temperature. This agrees with the theory and experimental results and this is verification that the FEA model is correct.

Calculations were carried out to study the average temperature variation with the changes in the power supply. The results show an increase in average catalyst temperature with the changes in the power supply. As the heat input to the system increases, the average catalyst temperature increases. In the laboratory experiments we observed a similar trend. This concurs with the theoretical analysis. As the heat input to the system is increased the catalyst temperature increases. In laboratory experiments a similar trend was observed. This makes us to believe that our model is correct.

**Finite Element Energy Balance Model**

An analytical model was derived to obtain a temperature distribution along the length of the platinum catalyst in terms of temperature of inflowing fuel air mixture, thermal conductivity of platinum catalyst, length and cross-sectional area of the platinum wire, coefficient of heat transfer from the gas to the surface, perimeter of the platinum wire and temperature of the walls.

The following assumptions were made for this model

Assumptions

- The temperature of the propane-air mixture surrounding the platinum catalyst is uniform.
- The temperature variation in the radial direction is the platinum wire is negligible compared to the temperature gradient along its length.
- The ends of the platinum catalyst are at known temperatures.
- We assume a steady state heat transfer process involving heat generation.
- Negligible radiation exchange with the surroundings.
o Uniform convection coefficient over outer surface of the Pt wire.

o We then developed a differential equation for the temperature distribution at various points along the length of the platinum wire by means of a balance of the following quantities

○ Conductive heat flow into and out of a differential section \( dx \), of the platinum catalyst.

○ Heat loss from the wire due to convection cooling by the inflowing fuel air mixture.

○ Heat generated per unit volume in the differential section, \( dx \), by the constant temperature anemometer circuit.

**Boundary Conditions**

The boundary conditions were obtained by expressing mathematically the following

○ The temperature at the ends of the platinum catalyst is equal to the wall temperature.

○ There is heat generation due to electrical heating. The BC is that the maximum temperature of the wire occurs at its geometrical center.

○ Hence, at that location,  \( \frac{dT}{dx} = 0 \)

Based on these assumptions, applying laws of heat transfer and specifying the above boundary conditions, we obtained a temperature distribution along the length of the platinum wire.
Energy Balance

Let

\( dx \) - Differential element
\( x \) - Distance along platinum catalyst, m.
\( L \) - Length of platinum wire, m.
\( A \) - Cross sectional area of platinum catalyst, \( m^2 \).
\( P \) - Perimeter of wire, m.
\( k \) - Thermal conductivity of wire, W/mK
\( h \) - Heat transfer coefficient, W/m\(^2\)K.
\( I \) - Current electrical supply system, amp
\( R_0 \) - Resistivity at reference temperature, ohms
\( \alpha \) - Temperature coefficient of resistivity, 1/K

\( t_w \) - Temperature of walls, K
\( t \) - Temperature of platinum wire, K.
\( t_{amb} \) - Temperature of inflowing fuel air mixture, K

Energy entering the system = Energy leaving the system

\[ q_{(\text{Condn})\text{In}} + q_{\text{Gen}} - q_{(\text{Condn})\text{Out}} - q_{\text{Convection}} = q_{\text{steady}} \]
Parameters Investigated

Ignition of propane-air mixtures over platinum wires at higher Reynolds number was experimentally studied by using microcalorimetry. Parameters investigated included average surface temperature, flow velocity, and equivalence ratio. The meaning of the word ‘ignition’ used in the combustion field is much different than that used in day-to-day life. In the combustion field, the word ignition can signify different moments in the process of producing an explosion. The theory of ignition has been defined by two different approaches: kinetic theory and thermal theory. According to kinetic theory, ignition is defined as the point at which the production of radicals due to chain branching exceeds the destruction due to chain termination and product formation [25]. From the thermal point of view, ignition temperature is defined as the lowest temperature at which a measurable amount of heat is released from chemical reactions [6]. The propane-air mixture experiments described here take the thermal approach into account for analysis.

When an ignitable gas stream is made to flow over the wire, some chemical heat is generated at the surface of the wire due to the surface reactions occurring between the fuel air mixture and the platinum wire. Hence, less electrical heating is needed to maintain the surface at the same temperature \( T \). In order to have greater control on the accuracy and precision of the analysis, instead of a bare platinum wire a catalyst/sensor prepared in a thin film by depositing catalytic material on a ceramic substrate can be used. Robben et al. [24] have used the heated-film microcalorimetric technique to study catalyzed combustion of hydrogen/air mixtures in a flat plate boundary layer. Activation energy and the pre-exponential constants are determined from the heat release rate measurements in conjunction with numerical computation of the boundary layer combustion model.

Theoretical Analysis

Figure 21 shows a theoretical model of the energy fluxes resulting from heat generation by (electrical heating and exothermic reaction) and heat loss due to conduction, convection and radiation. As shown in Figure 21, for a given concentration of reactants the heat generation curve as a function of catalyst temperature exhibits the typical sigmoidal shape, while the heat loss...
curve is given by a straight line [1]. Since the heat transfer coefficient remains constant, an increase in catalyst temperature due to external heating causes a shift in the heat loss curve from A1 to A2 without any change in the slope. This process continues as long as there is no heat generation by the exothermic chemical reaction occurring on the catalyst surface.

But when the exothermic heat is generated by surface catalytic reactions the heat loss curve and heat generation curve no longer intersect but they are tangent to each other as seen on heat loss curve A3. The steady state conditions that used to be there due to the kinetic controlled regime no longer exist and the system shows the process of ignition and the corresponding ignition temperature is shown in Fig. 11. During ignition there is a rapid transition to the diffusion controlled steady state regime as evident.

![Figure 11 Theoretical analysis of energy flux](image-url)
Figure 12 Methodology for obtaining ignition temperature

In this research, the propane-air mixture set of experiments is focused on the average ignition temperature rather than on measurement of chemical heat release rates. Figure 12 shows the methodology for determining ignition temperature. Initially power is supplied to the platinum catalyst connected to a variable power supply unit. Initially the power was set up to a definite level (power setting 1) and the propane air mixture is made to flow over the heated platinum wire. With no change being detected in voltage on the data acquisition control unit, the power level is increased to another level, which is power setting 2. This gradual electrical heating through incremental power settings slowly increased the surface temperature of the platinum catalyst. Again with no change being detected in the voltage, the power level is changed to another incremental level. This process is continued until a stage is reached when at a definite power setting there is a sudden rise in voltage recorded on the data acquisition control unit. This transition phase is defined as ignition for these particular sets of experiments and the corresponding temperature as the ignition temperature.
In addition to electrical heating of the platinum wire, additional heat is generated due to the chemical reactions occurring on the surface of the catalyst. Hence, the temperature of the platinum catalyst increases. This small increase in temperature of the catalyst is reflected as an increase in its resistance. For the same amount of current flowing through it, the final voltage reading becomes greater than the initial voltage reading. This change in voltage reading is successfully detected by using a data acquisition control unit connected to a computer.

**Finding**

Successful completion of experimental and finite element studies on plug flow combustion of propane-air mixtures completes a major milestone in the exploration of catalytic ignition of Aquanol air mixtures over platinum wires. This section summarizes results for plug flow experiments, surface temperature experiments, and the finite element model.

**Plug Flow**

Figure 13 shows the velocity profile for hot wire axial positions of 3.625, 5.625, 11.625 and 13.625 diameters downstream of the mixing nozzle. It is evident from the chart that the platinum catalyst can be inserted in the region between 5.625 and 11.625 diameters downstream of the mixing nozzle.

![Figure 13 Velocity across cross section at various distances from mixing nozzle](image-url)
The volumetric flow rate was estimated at each of the axial positions by summing the product of the velocity at each radial position and the area of the annular region that the probe was centered on. These calculations were within 10 percent of expected and the difference is suspected to be uncertainty in the probe velocymeasurement. Based on these results, the platinum catalyst was inserted at 5.625 diameters downstream of the mixing nozzle.

**Surface Temperature**

*Equivalence Ratio – 0.1*

For these set of propane-air mixture experiments, a variable power supply unit was connected to the ends of the platinum catalyst wire. The distinguishing feature of the power supply unit was that it was possible to regulate small changes in the current flowing through the circuit.

Figure 14 shows the average surface temperature as a function of power input to the catalyst. It is clear from this figure that higher amount of power is needed to be supplied to achieve higher temperatures. The propane-air mixture experiments were conducted at higher Reynolds number of 2,400, fuel volume percentage of 5 percent and an equivalence ratio of 0.1.
The plot shows that the surface temperature of the catalyst wire rises from 362 K to 715 K when the power input is increased from 1.48 W to 4.36 W at an equivalence ratio of 0.1. The plot shows the 95 percent confidence intervals for all the data points. Figure 15 shows significant variation in the average surface temperature recorded at 2.2 W and at 2.6 W. The reasons for this particular erratic behavior are discussed in detail below.

![Figure 15 Average surface temperature for 1st and 2nd sets of experiments](image)

Here, the 1st and 2nd sets of experiments were performed continuously with no intermittent shutdown of the apparatus. During the 2nd set of experiments, the first three readings are nearly concurrent. But later there is a significant discrepancy in the fourth and fifth data recording. It is due to the fact that while the 1st set of experiment data was obtained, carbon particles and non-volatile impurities had started accumulating on the surface of the platinum. This residue deposits on the platinum surface restricted the direct contact of propane-air molecules with the platinum surface, thereby inhibiting the catalytic activity. Hence, we saw a significant increase in the surface temperature for the fourth and fifth readings.
In order to avoid further discrepancies, the platinum catalyst was carefully taken out of the quartz tube and was replaced by a new platinum wire. The new wire helped to eliminate the surface temperature differences and it can be seen in the subsequent data recordings.

This set of experiments helped to understand the effect of aging of the catalyst on ignition. Figure 25 shows that the average surface temperature is affected by the history of the catalytic surface.

**Equivalence Ratio – 0.3**

Figure 16 shows the average surface temperature as a function of power input to the catalyst. For these sets of data, the propane-air mixture experiments were conducted at higher Reynolds number of 2,400, fuel volume percentage of 5 percent and an equivalence ratio of 0.3.

![Figure 16 Average surface temperature at an equivalence ratio of 0.3](image)

Here, the 1st, 2nd and 3rd set of experiments were performed intermittently with shutdown of the apparatus. The results indicate that there is very good agreement between all the three set of experiments performed after definite intervals of time. All 3 different sets of data were recorded at an interval of 1 hour to understand the effect of cold starting on catalytic activity. Hence, these experimental results are compared with the results obtained by conducting FEA studies. The
overall aim here is to determine the amount of electrical heating needed to reach a desired surface temperature.

*Equivalence Ratio – 0.7*

Figure 17 shows the average surface temperature as a function of power input to the catalyst. For this sets of data, the propane-air mixture experiments were conducted at higher Reynolds number of 2,400, fuel volume percentage of 5 percent and an equivalence ratio of 0.7.

![Figure 17 Average surface temperature at an equivalence ratio of 0.7](image)

The plot shows that the surface temperature of the catalyst wire rises from 347 K to 581 K when the power input is increased from 1.46 W to 3.76 W at an equivalence ratio of 0.7. The plot has 95 percent confidence intervals for at least 3 data points. Figure 27 shows significant variation in the average surface temperature recorded at 2.74 W and at 3.02 W. The reasons for this particular erratic behavior are discussed in detail below.
Here, the 1st and 2nd set of experiments was performed continuously with no intermittent shutdown of the apparatus. Figure 18 shows close agreement in the two sets of experimental data. However for the fifth reading, the surface temperature and corresponding power supply needed increased drastically. The surface of the platinum wire was checked for residue deposits. Then the platinum wire was carefully taken out of the quartz tube. After the contaminated catalyst wire was mechanically cleaned it was then placed in the quartz tube for further data recording. The results in Figure 18 indicate that when the same wire is used there is less difference in the data recordings in the 1st and 2nd set of experiments as compared to replacing the used wire with a new platinum wire. Based on these experiments it was found that new and aged catalysts are less active than a moderately used catalyst.

**Summary of Equivalence Ratio Experiments**

Figure 19 shows a comparison of the effect on average surface temperature with the variation in power supply at equivalence ratios of 0.1, 0.3 and 0.7.
It is clear that in the initial stage of the heating of the platinum catalyst, there is no significant change in the average surface temperature of the platinum wire. It is evident from the figure that higher amount of external heating is needed at lower equivalence ratios.

**Comparing experimental and analytical results**

Figure 20 compares the average catalyst wire temperature obtained experimentally with that calculated by the 3-D FEA models.
The average surface temperature obtained at the experimental power level where surface reactions began using a 3-D propane-air mixture FEA analysis is 725 K, only 9.5 percent higher than the experimentally obtained temperature of 662 K.

Differences between experiment and model may be due to modeling assumptions (the end temperature of the wire is prescribed and thermal radiation is neglected). Because 3-D FEA results differ from the experimental results, it is an indication that physical assumptions such as a prescribed top surface temperature and negligible thermal radiation, and steady state conditions are critical for matching experimental conditions.

**Finite Element Model Results**
Oxygen molecules are readily adsorbed on the platinum surface [1]. Hence, the ignition of propane/air mixture significantly depends on the amount of fuel adsorbed by the platinum surface. An increase in the fuel concentration in the fuel air mixture is likely to facilitate ignition. This theory is tested by conducting experimental studies of catalytic ignition of propane-air mixtures.

**Effects of Equivalence Ratio**
Equivalence ratio is typically defined as the actual mass fuel/air ratio divided by the stoichiometric mass fuel/air ratio. However, for these set of experiments, equivalence ratio is defined as the actual mass fuel/oxygen ratio divided by the stoichiometric mass fuel/oxygen ratio. The Reynolds number of the fuel air mixture was 2,400 and fuel volume percentage was 5 percent. The need to extend the range of study from lean to richer conditions, and at the same time to suppress the gas-phase ignition, is accomplished by diluting the fuel air/mixture with nitrogen. The same equivalence ratio can be obtained with different volume fractions of fuel and oxygen with the use of nitrogen dilution.

Figure 21 shows the changes in average ignition temperature as a function of equivalence ratio. The results indicate that at lower equivalence ratios, since the amount of fuel content is comparatively less, the average ignition temperature required is higher. That is, higher heating of
the platinum by the electrical system is needed to achieve ignition of propane-air mixtures at lower equivalence ratios. The plot indicates that the average ignition temperature decreases with the increase in equivalence ratio.

The average ignition temperature was recorded while going from fuel lean to fuel rich conditions (forward) direction and to verify the accuracy of the results the data was recorded in (reverse) direction. Figure 31 shows that there is an error of about 3 percent in the average ignition temperature data in forward and reverse directions at higher equivalence ratios.

![Plot showing average ignition temperature as a function of equivalence ratio.](image)

**Figure 21 Average ignition temperature as a function of equivalence ratio**

The probable reason for this discrepancy is during the reverse data recording, a few propane molecules had been already adsorbed by the platinum catalyst from the previous set of data recordings. But an error of 1.9 percent is seen in the average ignition temperature data in forward and reverse directions at lower equivalence ratios. The reason for this is that at lower equivalence ratios the amount of propane molecules available for catalytic activity on the platinum surface are less as the experiments were conducted from richer to leaner mixtures.
**Effect of Reynolds Number**

Figure 22 shows the changes in average ignition temperature at different equivalence ratios at Re < 1 and at Re = 2,400. It is clear from this figure that at Re =2,400, the fluid velocities are higher and so correspondingly higher amount of external heating is needed to achieve ignition.

![Figure 22 Average ignition temperature at different Reynolds numbers](image)

The heat loss due to convection at higher Reynolds number is a major reason for supplying additional power when compared with conditions when Re is less than 1. One probable reason apart from the fluid velocity is wire diameter, for our experimental data we used a platinum catalyst wire of 508 micron in diameters whereas the results reported in [6] make use of 127-micron diameter platinum wires. The figure indicates that there is a clear similarity in the pattern in which the average ignition temperature decreases with the increase in equivalence ratios.

**Effects of Fluid Flow Velocity**

Apart from the fuel and oxygen concentrations, the fluid velocity has a significant influence on the ignition temperature at higher Re flows. An increase in fluid flow velocity helps the reaction to stay in the kinetically controlled regime for a longer period. But the drawback of increased fluid flow velocity is that the time a given propane-air molecule spends near the catalyst surface
decreases [6]. The residence time of the reactants within the reacting environment decreases. The propane-air molecules have less time to react and consequently it is difficult to achieve ignition. However, a higher catalyst surface temperature can facilitate a more intense reacting environment, wherein the molecules of the propane-air mixture are equally excited in a shorter period to time [6]. Also, when the fluid velocity is increased, the heat losses due to convection increase. Hence, a mixture flowing at a higher flow velocity would require a correspondingly higher temperature of ignition. Figure 33 shows the effect of fluid flow velocity on the ignition temperature of propane-air mixtures. For these sets of data, the propane-air mixture experiments were conducted at fuel volume percentage of 5 percent at an equivalence ratio of 0.3.

![Figure 23](image)  
**Figure 23** Average ignition temperature as a function of fluid flow velocity

It is evident from Figure 23 that as the propane-air mixture flow velocity is increased from 1.14 m/s to 1.96 m/s the corresponding ignition temperature required increases significantly. These fluid flow velocity results shall form the platform for further research on Aquanol-air mixtures at varied fluid flow velocities. Figure 23 indicates that as fluid velocity increases the uncertainty associated with the average ignition temperature also increases.
The plot shows that the average ignition temperature of the catalyst wire rises from 586 K to 715 K when the fluid velocity is increased from 1.14 m/s to 1.96 m/s at an equivalence ratio of 0.3. The plot has 95 percent confidence intervals for all the data.
CONCLUSIONS

Plug flow reactor

This work produced a robust, reliable platform for evaluating average catalytic ignition temperatures of fuel-air mixtures. A specially designed catalytic plug flow reactor was modified to conduct catalytic ignition of propane-air mixtures over platinum wires. Considerable effort was made to characterize the reactor and catalyst system. Experiments on gases mixing response time were conducted to determine the effectiveness of the mixing nozzle. Hot wire anemometer experiments were conducted to determine the axial position downstream of the mixing nozzle for insertion of the platinum catalyst. Surface temperatures were obtained as a function of equivalence ratio and power supplied to the catalyst.

System Modeling

Finite element steady state heat transfer analysis of propane-air mixtures over platinum wires has helped to develop a correlation between the amount of power needed to be supplied to reach a desired average catalytic surface temperature. Further, the results of the FEA sensitivity analysis shall provide an insight into accommodating system modifications. The FEA sensitivity studies on the platinum wire geometry has been very much helpful in investigating the changes in the wire temperature without purchasing platinum wires of different diameters and lengths. Since 10 inches of platinum wire costs around $140, FEA studies has proved to be beneficial in eliminating costs. Although the FEA results don’t match up the experimental results hundred percent, we used the 3-D FEA results to get a closer range of changes in average catalytic surface temperature with the corresponding alterations in the system geometry, fluid flow and heat supplied.

Catalytic ignition temperature

Propane-air mixture experiments over platinum catalyst were performed using microcalorimetry to investigate various parameters associated with catalytic ignition of propane-air mixtures over platinum wires at higher Reynolds number. Parameters investigated included flow velocity and equivalence ratio.
Recommendations

The work described here represents a first phase at developing and understanding catalytic ignition of fuel-air mixtures.

Plug flow reactor

An interesting exercise would be flow visualization of two gas streams in the mixing nozzle. Aqueous ethanol experiments will first require verification that the liquid fuel evaporator works correctly. Development of the reactor should continue with the design of a quartz-lined pressure-vessel reaction section, a catalyst bed, water-cooled sampling probe, and operation of a gas chromatograph and mass spectrometer for product species quantification. These additions will permit measurements of conversion efficiency of fuel to complete combustion products for comparison with detailed chemical kinetic modeling.

System modeling

The immediate next step is to include radiation losses in the 3-D model that we have already generated, perform parametric studies for model sensitivity to a prescribed end temperature and convective heat transfer coefficient, and compare with the experimental results. Efforts shall be made to conduct transient heat transfer analysis for this model using Algor. Further, Algor may be used to obtain temperature information in more complex geometries such as the catalytic igniter core/pre-chamber. Algor may also be used to model the fluid mechanics and heat transfer of the actual igniter.

The capabilities of Algor should be explored to determine if the program accepts chemical reactions as heat generation terms. If so, a simplified catalytic ignition mechanism should be developed so that the FEA model can predict ignition temperature.

Catalytic ignition temperature

The experimental methodology for average surface and ignition temperatures described herein should be expanded for propane-air mixtures and conducted with other fuels including aqueous ethanol-air mixtures. For example, ignition temperatures at the similar conditions used by Cho and Law [6] (wire diameter, Re, equivalence ratio, fuel and oxygen mole fractions) should be
conducted to gain additional confidence in the apparatus and methodology. More than one set of fuel and oxygen percentages can have the same fuel-oxygen equivalence ratio; this would permit study of the influence that fuel and oxygen partial pressure have on the catalytic reactions. These tests should be extended to high Re flow as well.

Propane-air mixture ignition experiments have helped to understand the effect of catalyst aging on ignition temperature. A very helpful insight is provided by these particular sets of experiments. It brings into account an issue that needs to be addressed in future studies and analysis. If residue formation on the platinum surface occurs during use in vehicles, then efforts have to be made to minimize this trouble of changing the platinum catalyst frequently. One possible reason for the embrittlement of platinum catalyst may be the increased porosity due to the presence of hydrogen at high temperatures. The catalyst aging effect has opened up a new field for further investigation changes in surface morphology of the platinum exposed to high temperature reactions.

Future experimental work with aqueous ethanol-air mixtures should collect data to determine the temperature of the platinum catalyst required for ignition of a particular volumetric content of water in ethanol at a specified equivalence ratio. Further investigation should be carried out in order to understand the changes in ignition temperature for different volumetric concentrations of aqueous ethanol at a constant excess air coefficient.
REFERENCES


