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Mixed Heterogeneous-Homogeneous Combustion of Dodecane in a Parallel Plate Microreactor

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Conversion of chemical energy to electrical energy using commercial thermoelectric (TE) devices typically yields efficiencies of ~1%. An alternative and largely overlooked approach is to use thermophotovoltaics (TPV) to convert IR radiation to electrical output. TPV devices offer potential benefits in size, efficiency and steady state output compared to TEs. Both platforms benefit from low pressure drop, heat recirculation and mild temperature gradients in the power harvesting section, however, they operate under very different conditions. TEs have shown promise to convert heat at relatively low temperatures (~500 °C) and achieve operating efficiency approaching 10%. To achieve a comparable operating efficiency in a TPV device, the combination reactor/emitter walls must be very hot (~1000°C) so that there is a sufficient flux of in-band (>0.5eV) photons to be harvested. For a low-power TPV device with 100W chemical input, reaching these conditions limits emitter size to ~10 cm². Confining reactions to catalytic surfaces can provide a stable reaction zone with a mild temperature gradient under conditions of high flow velocity and considerable heat loss. In the limit of highly confined heterogeneous dominant operation, heat is efficiently transferred to the reactor walls, however, small residence times (several ms) results in fuel breakthrough. On the other hand, purely homogeneous operation is a less effective means to transfer heat to the reactor walls and is susceptible to blowout. Increasing the limiting dimension increases the rate of homogenous reactions and numerical results have shown that combined heterogeneous-homogeneous (HH) reactions offers greater stability to blowout and extinction than purely homogeneous or heterogeneous reactions. Moreover, peak temperatures in HH combustion are lower than those encountered in purely homogeneous combustion, an important factor in increasing reactor and catalyst lifetimes while limiting the production of pollutants. It follows that HH combustion may provide a long, stable and complete reaction zone at temperatures ideal for the clean and efficient operation of TPV devices over a long lifetime. In this work, the HH combustion of lean dodecane/air mixtures in a parallel plate microreactor is examined. Control over the relative rates of homogeneous and heterogeneous reactions is achieved through varying the gap distance between the platinum-coated reactor walls. Conditions that provided high wall temperatures with mild gradients—suitable for TPV reactors—are identified and examined on the basis of relative homogeneous and heterogeneous reaction rates and heat transfer rates.

1. Introduction

Liquid hydrocarbon fuel based energy conversion devices are a promising alternative to replace the current favorite portable power source, lithium ion batteries, because of their superior energy density and ease of refueling. Efforts to generate electricity from small scale fuel powered energy conversion devices have typically resulted in <5% efficiency in converting chemical energy to electrical power. Several recent reviews have addressed the difficulties in achieving reasonable conversion efficiency for both mechanical and solid state devices (mainly thermoelectric based technology) [1-4]. For mechanical power conversion devices, (ie. microturbines) the increased relevance of friction losses at the small scale make it difficult to achieve efficient operation. For solid state devices, most efforts have focused on the use of thermoelectric (TEs) but advances in TEs have not been able to reach the level of efficiency and temperature difference that theory has promised and commercially available TEs are still limited to approximately the same conversion efficiency levels that they were decades ago.

While frictional losses in mechanical devices remain a barrier to their adoption and advances in TEs are yet to come, thermophotovoltaics (TPVs) offer an alternative that is especially promising in light of some of the advances made in photovoltaics and spectral control recently. It is believed that, with sufficient focus, the bandgap of photovoltaics can be

reduced so that it absorbs well into the infrared spectrum (~0.5 eV, 1240 nm) with efficiencies of 30% or higher. In addition to the hopes placed on photovoltaics, it is believed that novel optical materials for selective emitters and high temperature filters can be integrated with combustion devices so that the energy radiated from their surfaces is targeted to match the absorption spectra of the photovoltaic. Finally, since the beginning of the combustion micropower field about 15 years ago, there have been important advances in the areas of fuel vaporization and waste energy recirculation. One recent analysis has shown that, by achieving reasonable levels of photovoltaic, emitter, and heat recirculation efficiency, small scale TPV devices can be expected to produce ~10W electrical with 10% efficiency, a goal that would far surpass Li ion battery energy density [5].

Regardless of the energy conversion technology used (TE or TPV), the ideal platform for generating heat are similar. Both platforms will operate at targeted and elevated temperatures with TPV generally being higher in order to radiate a sufficient amount of energy. Both platforms would recycle as much heat as possible, using it to vaporize fuel and preheat incoming reactants. Similarly, in both cases, significant pressure drops—through heat exchangers and through fuel delivery—must be avoided as the parasitic energy losses (friction) are large. Also important for a personal device is that it allows essentially no fuel, CO, or other harmful pollutants (ie NO_x) to exit the reactor. Finally, one of the most important and difficult to control characteristics of a highly efficient device is that they approach an isothermal operating condition. Achieving mild temperature gradients during operation is important because hot spots can damage critical materials such as reactor walls, catalyst and TECs.

To date, most solid state energy conversion devices have focused on available TE modules limiting them to ~200-500 °C. Many of these devices have achieved high thermal efficiency by utilizing “excess enthalpy” reactors. However, efficient thermal-to-electric energy conversion will require higher operating temperatures (800 to >1000 °C) [5]. Reaching high temperatures limits reactor size. In order to estimate the size of a TPV heat source, an idealized square parallel plate reactor radiating energy to vacuum with 1mm thick SiC walls ($k = 100 \text{ W/m/K}$), a gap of 1mm and an internal heat transfer coefficient $h=100 \text{ W/m}^2/\text{K}$ [2] burning a stoichiometric mixture of CH₄/air ($T_{in}=750 \text{ K}$) with an enthalpy input rate of 98.6 W was examined (see table 1). The results show that the smallest reactors reach the highest temperatures, but the amount of heat radiated is less than their larger counterparts because a larger portion of energy is lost to the exhaust. Based on this, we estimate that with perfect spectral control (optical emitters and filters) and near perfect heat recuperation, the optimal size footprint of the reactor is ~3x3 cm. Without spectral control the optimal size will be smaller ~2x2 cm. Regardless, for a reactor with an enthalpy input rate of about 100 W, the reactor footprint will be on the order 2x2 cm and the total radiative surface area will be ~10 cm² (after accounting for both sides). A reactor of this size will require a flow velocity (assuming a gap of 1mm) of ~400 cm/s.

Reactor size (cm)	T _{max} Wall (K)	Radiation $\lambda>0.5\text{eV}$ (W)	Total Radiation (W)	$\eta_{\lambda>0.5 \text{ eV}}$ (%)	η (%) Ideal Spectral control
1.3x1.3	1300	17.6	53.6	17.8	54.4
1.5x1.5	1250	18.5	61.3	18.7	62.2
2x2	1170	18.8	77.2	19.1	78.3
2.5x2.5	1090	16.9	87.3	17.2	88.5
3x3	1030	14.3	94	14.5	95.3

Table 1. Energy delivery and losses and peak wall temperatures for a square parallel plate reactor. The large reactors effectively spread heat, resulting in a significant reduction of in band radiation flux. For small reactors, transferring heat to the walls is limited by size and more heat leaves through the exhaust. The balance of power is lost to the exhaust.

The most effective route to achieving stable and complete combustion with mild temperature gradients at high temperatures and flow velocities is not clear. From the perspective of heat transfer, catalytically oxidizing the fuel would be ideal since the heat transfer occurs directly at the wall. However, it is not likely that a fuel can fully react at the high flow velocities and short distance (and therefore short residence times) associated with a surface dominated reaction and as a result, CO or fuel breakthrough will occur. At the other extreme, a purely homogenous reaction (i.e. a premixed flame stabilized at the burner inlet, will fully react all the fuel but this may also lead to problems with stability, material failure due to extreme temperatures and temperature gradients as well as NO_x production. Furthermore, delivering heat from hot gas to the reactor/emitter walls may not be the most ideal route, causing much of the heat energy to be sent to the heat recuperator prematurely.

Combining the two approaches by using a reactor that operates with significant amounts of gas phase and surface reactions is an approach that may utilize the best characteristics of each. This class of hybrid hetero-/homogeneous reactions (HH) has been shown to be more stable than either purely heterogeneous or purely homogeneous reactors [6]

over a wide range of heat losses and inlet flow velocities. The reasons underlying the increased stability are attributed to the fact under conditions of high heat loss and high inlet velocities, a purely heterogeneous reactor will not provide sufficient time to react the fuel whereas, when significant amounts of gas phase reactions are added, the incoming unburned gasses are able to fully react. The increased heat flux to the reactor walls further preheats the upstream reactants, increasing the rates of both surface and gas phase reactions and stabilizing the flame against blowout.

As research gears toward milder combustion temperatures and more fuel-lean operation to lower emissions and increase efficiencies [7] the milder and lengthened reaction zones achievable through HH provide hope for variety of technologies. In addition to their increased stability, the hybrid HH reactor offers additional benefits in terms of overall reduced temperature gradients [8]. By spreading the heat loss over the length of the reactor, it is possible to approach a more isothermal operation. Creating an environment with a milder temperature gradient (and therefore a lengthened reaction zone) and lower maximum temperatures in the gas phase results in lower peak wall temperatures which is important for lengthening the lifetime of both the reactor and its catalytic walls and also less NO_x production. In the following sections, we examine how confinement on the order of quenching distance promotes HH operation using a surrogate for a practical fuel source (JP8) and how HH operation can provide the high temperature conditions with mild gradients that TPV requires.

2. Methods

The reactor used in this study was designed after [9]. It consists of two stainless steel plates separated by an interchangeable stainless steel gasket. Welded to the top plate are stainless steel tubes, one for reactant inlet and one for exhaust outlet. Inside the reactor chamber, fine stainless steel screens (250 μm opening) are placed immediately before and immediately after the reactor section in order to straighten flow and limit wide distributions of residence times “jetting” as the flow develops. In the middle, the reaction zone consists of catalyst coated alumina inserts separated by thin strips of compressed alumina insulation. The length of the reaction zone is 5cm, the width is 1cm and the height is governed by the thickness of the gasket chosen. The assembly is held together by a series of screws around the circumference of the reactor. Inside, the compressed alumina insulation maintains a tight fit between the catalytic inserts and the stainless steel walls. The snug fit ensures that the pressure drop through the compressed insulation is greater than that along the length of the reactor, and therefore, flow is guided through the reactor.

The catalytic surfaces are solid thin amorphous alumina (15x50x0.65 mm) coated with a layer of γ -alumina which is impregnated with platinum. Developing the catalyst begins with doctor blading a layer of Boehmite ($\text{AlO}(\text{OH})$) dispersed in water onto the solid alumina strip. Ordinary scotch tape is used as a cast. The cast Boehmite layer is allowed to dry at standard conditions for 1 hour before it is baked for a period of 5 hrs at 700 $^\circ\text{C}$. This step serves to drive off any residual moisture in the solution, to oxidize the organic viscosity modifier and to transform the Boehmite into γ -alumina. Next, the inserts are masked and ~0.2 ml of chloroplatinic acid (Aldrich, 8 wt. % in H_2O) are delivered by pipette onto the surface. The solution dries at room temperature, the mask is removed and the loaded inserts are once again baked for a period of 5 hours at 700 $^\circ\text{C}$ in order to reduce the chloroplatinic acid into platinum. Each insert is loaded with approximately 8 mg of platinum.

Industrial grade dry air (Airgas) is used as the oxidizer and dodecane (Aldrich) is delivered by a syringe pump into heated air. The air flow rate is controlled by a pressure controller upstream of a choked nozzle. The air is heated by passing it through a coil wrapped in heating tape. Dodecane is injected at 150 $^\circ\text{C}$, a temperature slightly above its saturation temperature for stoichiometric mixtures. Process temperature is controlled by feedback from an inline thermocouple. The reactor assembly is sandwiched between two 5 cm thick needled silica mats ($k = 0.12 \text{ W/m/K}$) wrapped in aluminum tape.

Once the air stream is brought up to operating temperature, fuel delivery commences. In order to light off the reactor, the upper insulating layer is temporarily removed and a butane torch is used to heat the reactor to a point where autothermal operation is possible. Six K type thermocouples are welded along the center of the bottom plate of the reactor and are used to monitor temperature along the length of the reactor. The thermocouples are placed in 1 cm intervals and they coincide with the location of the catalytic insert. Flow control and data acquisition are controlled using a LabVIEW interface. Three equivalence ratios ($\phi=0.5,0.7,0.9$) and three gap heights (0.5, 1.0, 2.0 mm) were tested with volumetric flow rates varying (from 1.5 to 3 SLPM). This corresponds to enthalpy input rates from 43 to 156 W and inlet velocities (nominally taken to be 423 K, the temperature of the reactor inlet and probably significantly lower than the mean velocity at the inlet of the catalytic section) from 180 to 1410 cm/s. The conditions chosen are enthalpy input rates, flow rates, and gap distances that are representative of values that would be present in a small scale portable power converter. Details of the operating conditions can be found in Table 2.

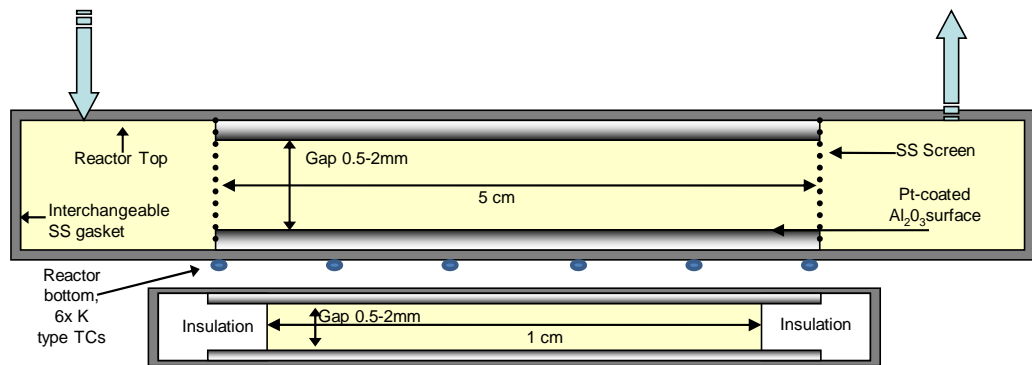


Figure 1. (Top) a lengthwise view of the reactor. Unburned gasses enter the parallel plate reactor and are passed through a fine SS screen at the entry of the 5 cm long catalytic section. Reacted gas is passed through another SS screen at the exit of the catalytic section. (Bottom) a cross-section of the width of the reactor. The reacting volume lies between the vertical boundaries created by the catalytic inserts and the horizontal boundaries created by the alumina insulation. The insulation is compressed and acts to maintain a gap between the catalytic walls and a barrier to flow out to the gasket, which controls the gap distance.

ϕ	\dot{V} (SLPM)	\dot{H} (W)	V_{in} (cm/s) gap = 0.5mm	V_{in} (cm/s) gap = 1mm	V_{in} (cm/s) gap = 2mm
0.9	3	156	1410	710	350
0.7	3	121	1410	710	350
0.5	3	87	1410	710	350
0.9	2.5	130	1180	590	290
0.7	2.5	101	1180	590	290
0.5	2.5	72	1180	590	290
0.9	2	104	940	470	240
0.7	2	81	940	470	240
0.5	2	58	940	470	240
0.9	1.5	78	710	350	180
0.7	1.5	61	710	350	180
0.5	1.5	43	710	350	180

Table 2. Summary of the reactor conditions in this study. The parameters chosen are representative of values expected to be present in a TPV based portable power conversion device.

3. Results and Discussion

The results examined here are based primarily on the steady state location-dependent temperature of the reactor, though some transient behavior is worth mentioning. Transients in the system are quite long, on the order of 1000 seconds. The longest transient is that associated with reactor startup (not shown). Upon heating the outer wall of the reactor, hot spots are created on the platinum, surface bound oxygen is desorbed and the oxidation of fuel begins. Despite being well insulated, it is believed that the slow transients are due to heat transfer in the axial direction of the catalyst. The axial spread of heat is slowed by heat lost due to preheating the incoming reactants as they are transported through the reactor. In each case, a wall temperature of 250 °C was sufficient to autothermally operate even the coolest conditions in this study ($\phi = 0.5$, $\dot{V} = 1.5$, $\dot{H} = 43$ W). For the purposes of this study, steady state was taken to be when all of the location dependent temperature readings reached a standard deviation lower than 1.5% of signal for a period of two minutes. The results are shown in figure 2.

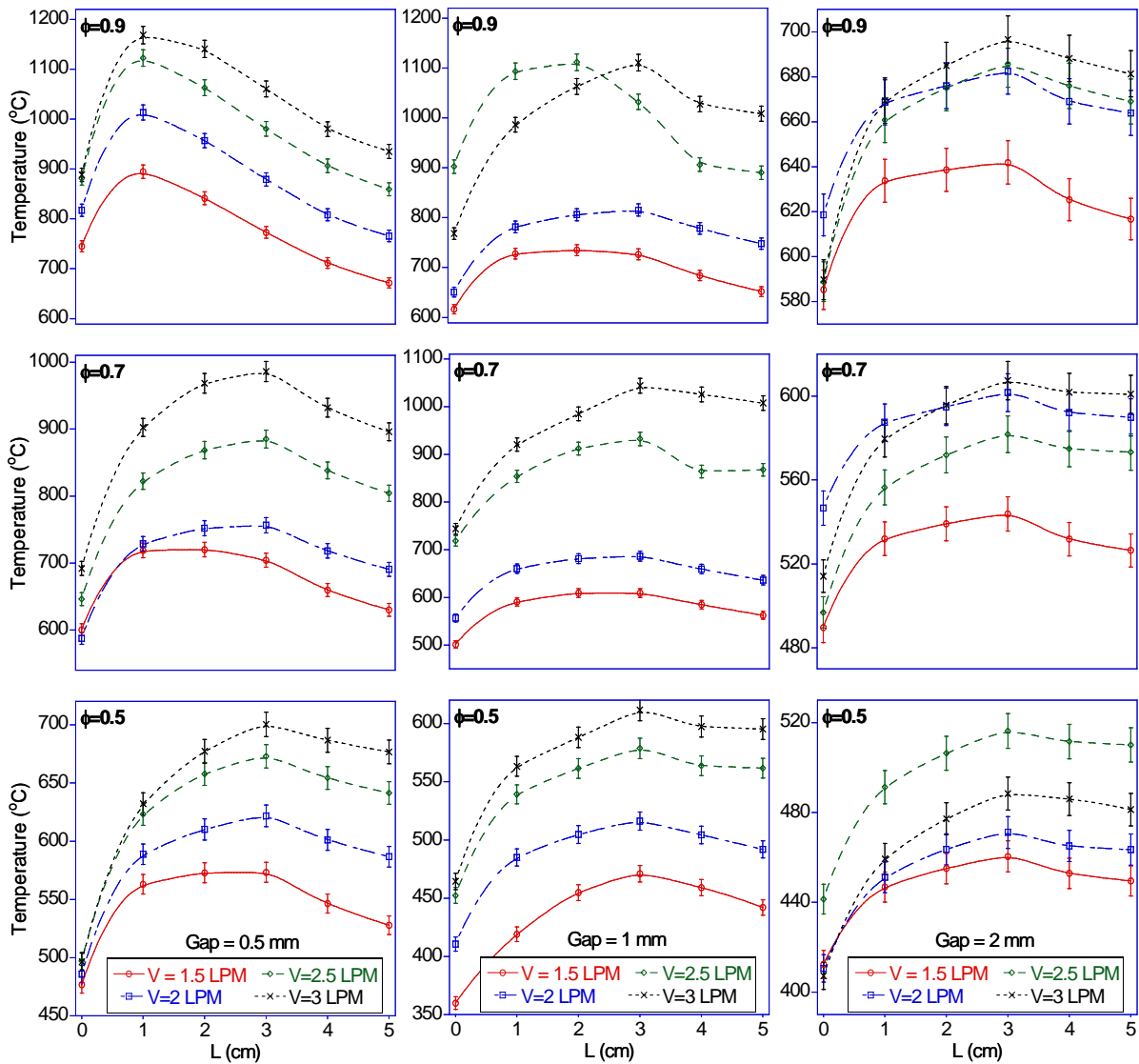


Figure 2. Temperatures along the center of the wall as a function of distance along the catalyst. From left to right, the columns represent 0.5, 1.0 and 2.0 mm gaps, respectively.

In order to understand the phenomena governing the heat transfer and reactions in HH reactors, it is important to understand the coupling of the exothermic reactions and heat transfer as they relate to flow velocity and gap size and the temperatures at which catalytic and gas phase reactions will dominate. As gap size decreases, both heat transfer from the wall to the gas and mass transfer from the bulk gas to the walls will increase. Increased mass transfer to the catalytic walls, in turn, leads to the coupling of higher wall temperature and faster reaction rates. Surface reactions of hydrocarbons can be sustained at temperatures much lower than gas phase reactions (~ 250 °C for the low heat loss conditions in this study). Due to the larger activation energy associated with gas phase reactions and the fact that radicals are scavenged by high surface area walls, gas phase reactions have a negligible effect at low and intermediate temperatures. A computational investigation into this effect, examining the coupling of gas and surface reactions of H_2 /air in an annular microreactor found that gas phase reactions contribute little to the overall conversion below ~ 750 °C [10]. In the limit of diminishing gap distance, it would seem as if the combination of heat transfer from the reactions at

the wall surface to the gas would be sufficient to sustain a flame. However, as the unburned gas travels down the length of the reactor, it loses fuel, radicals and heat to the walls and even though there is a high rate of heat transfer from the wall to the gas, the net effect may act to either stabilize or inhibit flames. Meanwhile, the heating will cause gas expansion and increase the velocity through the reactor, limiting its total residence time. Further complicating this increase in velocity magnitude is that the velocity profile will change as well and there is a possibility that unreacted gas traveling through the center of the reactor will “jet” through without reacting. As gap distance increases, the rate of heat transfer decreases (the heat transfer coefficient may be estimated by $h = \text{Nu}k/g$, where $\text{Nu} \sim 4$, k is the conductivity of the gas and g is the gap size). This leads to less preheating impact on the unburned gasses and lower mass transfer rates. Additionally the surface area to volume ratio will decrease. Therefore, the impact of wall reactions will have less effect on the overall fuel conversion rate at larger gas sizes. The net effect may be to promote gas phase chemistry if there is sufficient preheating and radical concentration or to inhibit it if there is insufficient preheating.

Analyzing the results of a highly confined reactor is hampered by the limited information available. This information is essentially limited to reactor wall temperature and a species analysis at the exhaust outlet since it is not feasible to sample gas or perform optical experiments in such small geometries at this time. Ideally, numerical modeling with detailed kinetics can give insight into the phenomena governing the reactions in these closed systems. Indeed this has been done to investigate the effects of gas phase chemistry in the stability of these reactors. Of course, detailed kinetic modeling can only be performed on simple fuels since surface reaction mechanisms of complex fuels (C3 and larger) are limited. One way to estimate the onset of gas phase reactions is to look for high localized temperatures. Since the heat release zone of a flame is thin, a large localized spike in temperature is one possible indication of a flame. It should be noted that there may be conditions under which there are flames but there is no evidence of an extreme temperature gradient (mild combustion). A detailed analysis of the contributions of gas phase and surface chemistry would require more concrete evidence (numerical modeling or in situ diagnostics) than are available at this time. Nevertheless, abrupt jumps in temperatures and the movement of temperature extremes indicate some gas phase chemistry in the microreactor.

As expected, as fuel input increases (increasing ϕ) and flow rates remain constant, the reactor temperature increases under every condition examined here. In general, the reactor temperatures are directly related to the rate of enthalpy input (\dot{V}), with higher enthalpy values resulting in higher reactor temperatures. This can be readily explained by the fact that the reactor is well insulated. While higher temperatures will result in greater heat losses, the increase in enthalpy input more than offsets this heat loss. This behavior is apparent in all cases tested for the reactors with 0.5 and 1 mm gaps (see figure 2). The reactor with a 2 mm gap deviates from this behavior at each equivalence ratio for flow rates above 2 SLPM. Under these conditions, the contribution of heat lost to incoming reactants, low heat transfer from the wall to the bulk gas and lower residence time act to slow the surface reactions, resulting in lower wall temperatures.

At the smaller gap sizes, the coupling between increased mass transport to the walls and increased heat transfer from the wall to the gas overcomes the effects of decreased residence time and the maximum wall temperatures are higher in nearly every case as the gap decreases. An interesting behavior occurs in the gap between conditions in which the maximum wall temperature ranges between 700 and 900 °C. In this gap there is little data available because there is a marked jump in temperature at the reactor wall. This jump is significant because of its severity and also because this temperature range ~ 750 °C is where significant amounts of gas phase can occur. Based on this, it is believed that a large portion to the heat release above 700 °C is due to gas phase reactions. In addition to the large jump in steady state temperature, it can be seen that there is a severe jump in transient temperatures in this region. While the focus of this research is not to examine transient behavior, it should be noted that there is a discontinuity in the derivative of temperature when moving from conditions that appear to be dominated by gas phase reactions to those dominated by surface reactions. Figure 3 shows the temperature and its time derivative history of the maximum wall temperature as a function of time as the reactor transitions from a steady state with high maximum temperature to one of low maximum temperature. After lowering the equivalence ratio from 0.7 to 0.5, the maximum wall temperature gradually drops over a period of approximately 150 seconds, then there is a very rapid drop in maximum temperature followed by a long period of more gradual cooling for ~ 1000 sec. This sudden temperature drop suggests flame extinction. Finally, while thermocouple data is limited to 1 cm intervals and it is not possible to tell the exact location of the temperature extremes, it is clear that as flow rate increases maximum temperatures for conditions that suggest a significant portion of gas phase reactions moves downstream. This is consistent with the understanding of a catalytically stabilized thermal (CST) combustion operation, in which a flame is stabilized downstream of a catalyst section that is used to preheat reactants [10,11].

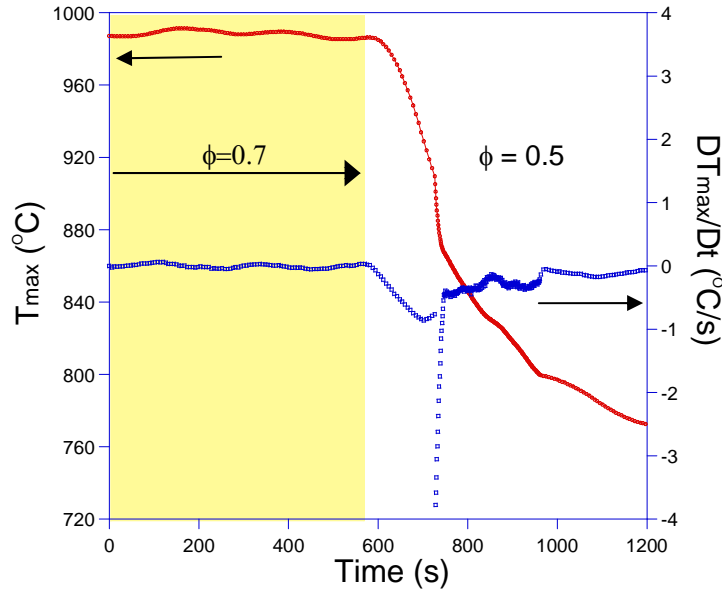


Figure 3. Transient response of changing the equivalence ratio in a reactor (gap = 1 mm) from conditions that support gas phase reactions to conditions that do not. The initial response to the decrease in enthalpy input is gradual for a period of about 150 sec. The sudden drop in maximum wall temperature suggests the gas phase reactions are extinguished.

The goal of this research is to show that reactor temperatures and their gradients can be controlled by tuning the rates of surface and gas phase reactions in order to generate conditions amenable to TPV power conversion devices. As discussed previously, the target for TPV reactors is to reach 1000 °C or greater with minimal temperature gradients. Table 3 summarizes the condition that meet the simplest of the criteria, a maximum wall temperature >1000 °C. In addition to maximum wall temperature, temperature averages and deviations are quantified. Average wall temperatures are defined as

$$T_{avg} = \int_0^L T dx / L$$

and a mean deviation from this average is normalized by the maximum wall temperature using

$$NMD = \frac{\int_0^L |T - T_{avg}| dx}{LT_{max}}$$

where 0 and L are the locations of the beginning and the end of the catalytic section, respectively [6]. By this measure, it appears the conditions that clearly favor a significant amount of both heterogeneous and homogeneous reactions (1 mm) result in the high and uniform conditions favored by TPV. It should be noted that the most uniform temperature conditions in terms of NMD, occur at low temperatures (not shown). This is to be expected for surface dominated reactions where there is no coupling between abrupt temperature jumps and chemistry (flames). While HH operation provides temperature characteristics that are ideal for TPV under the well insulated conditions in this study, it remains to be seen how similar temperatures and gradients can be achieved with large heat losses from the walls to a TPV

gap (mm)	ϕ	\dot{V} (SLPM)	V_{in} (cm/s)	\dot{H} (W)	T_{avg} (°C)	T_{max} (°C)	NMD
1	0.9	3	710	156	1020	1111	1.49
1	0.9	2.5	590	130	1012	1112	1.63
1	0.7	3	710	121	975	1044	1.76
0.5	0.9	2.5	1180	130	995	1122	4.61
0.5	0.9	3	1410	156	1060	1168	4.62
0.5	0.9	2	940	104	897	1013	5.35

Table 3. Summary of the conditions favorable to TPV operation. In terms of minimizing temperature gradients, the conditions that show strong evidence of vigorous gas phase reaction also have the lowest NMD.

4. Conclusions

HH operation of reactors shows promise to deliver stable and complete combustion of fuels with mild temperature gradients and lower peak gas temperatures. The result is that peak gas temperatures will be lower, radiation delivery to the TPV will be more efficient and fuel and harmful species including CO and NO_x will not escape the reactor. While a clear and quantitative analysis of the exact phenomena at play inside the reactor cannot be known without detailed in situ analysis or numerical modeling, there is strong evidence of a vigorous gas phase reaction under conditions of moderate confinement and these conditions appear to be the most promising to deliver high and uniform temperatures to a TPV.

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