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Fuel composition effects on transportation fuel cell reforming

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Abstract

This work examines the effect of various hydrocarbons on fuel processor light-off and reforming. Major hydrocarbon fuel constituents, such as aliphatic compounds, napthanes, and aromatics have been compared with the fuel processing performance of blended fuel components and reformulated gasoline to examine synergistic or detrimental effects the fuel components have in a real fuel blend.

Short chained aliphatic hydrocarbons tend to have favorable light-off and reforming characteristics for catalytic autothermal reforming compared with longer-chained and aromatic components. Oxygenated hydrocarbons have lower light-off requirements than do pure hydrocarbons. Gas phase oxidation favors higher cetane # fuels, which tend to be longer chained hydrocarbons. Energy consumption during the start-up process shows a large fuel effect. Methanol and dimethylether (DME) show lower start-up energy demands for the fuel processor start-up than do high temperature reforming hydrocarbon fuels such as methane, gasoline and ethanol. Aromatics and longer chained hydrocarbons show a higher tendency for carbon formation, increasing the amount of carbon formed during the light-off phase while the addition of oxygenates tends to lower the carbon formed during the start-up process.

Keywords: Fuel processing, fuel cells; Hydrogen production, fuel composition, carbon formation

1. Introduction

Fuel cells have attracted a significant amount of attention for transportation applications because of their high efficiency for conversion of fuel to electricity. To speed implementation of fuel cells into the transportation sector, on-board hydrocarbon reforming is required to enable the earliest commercial entry. The choice of fuel is likely to be different for differing fuel cell applications; primary motive and auxiliary power. Auxiliary electrical power production most likely will require the fuel cell system to operate on the fuel already available on-board the vehicle, typically diesel fuel. For primary motive power, the fuel is more likely gasoline, or a modified/optimized gasoline-like hydrocarbon.

Due to stringent requirements for start-up, volume, efficiency and capabilities for transients, most developers of automotive applications have concentrated efforts on ATR (auto-thermal reforming) for hydrogen production. The partial oxidation reaction for a generic aliphatic hydrocarbon is shown in Eq. (1) and is highly exothermic:

$$C_nH_{(2n+2)} + (m/2)O_2 \rightarrow mCO + C_{(n-m)}H_{2(n-m)} + H_2 \quad (1)$$

The steam reforming reaction (written for a steam to carbon ratio of one or S/C = 1) is shown in Eq. (2) and is highly endothermic:

$$C_nH_{(2n+2)} + nH_2O \rightarrow nCO + (2n+1)H_2 \quad (2)$$

For maximum efficiency, the partial oxidation exotherm is coupled and balanced with the steam reforming endotherm. The H_2O content is minimized for the highest reforming efficiency, however minimal water content also adversely affects the reforming kinetics and carbon equilibrium. Typically oxygen-to-carbon ratios (O/C) of ~0.7–0.8 are used with steam-to-carbon ratios (S/C) of ~1.0–2.5 in the ATR.

The start-up of fuel processors for transportation applications is one challenging area requiring development for commercialization. Major issues with the start-up of a fuel processor include the time for start-up, the energy

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consumed during the start-up process, start-up without stored water onboard the vehicle, carbon formation during the light-off process and the complexity of the system used for the fuel processor start-up. These start-up challenges can be affected by the fuel composition used for the reformer. A major effect that the fuel composition can have during the start-up of the fuel processor is the relative propensity for light-off in the partial oxidation section or ATR, or the catalyst temperature which is required to achieve light-off. Light-off is the temperature at which the hydrocarbon oxidation reaction exotherm becomes self-sustaining such that the temperature increases for complete depletion of reactants (in this case the depletion of oxygen). To examine the fuel effect on fuel processor light-off, individual fuel components were examined, including blends of pure fuel components and real fuel mixtures. Measurements were made to define the fuel composition effect during start-up including the catalyst temperature required for reactor light-off and the tendency of fuel components for forming carbon during the light-off process. Carbon formation for various fuel components and fuel blends has been measured during vaporization, reforming and during the light-off process.

2. Experimental

The fuel reforming (both oxidation and steam reforming) reactions were performed over noble metal based catalysts (platinum and rhodium). The catalysts are supported on a substrate of reticulated YSZ (Yttria Stabilized Zirconia) foam. For gasoline reforming, the gasoline, or gasoline component, is vaporized, then mixed in vapor phase with steam. The fuel/water mixture is then mixed with air, preheated and introduced to the reforming catalyst. Carbon formation during light-off was measured by monitoring via in situ laser absorption measurements and by physical collection of particulates downstream of the reactor. Post-characterization of catalysts has also been conducted by TGA (thermal gravimetric analysis) and evaluating the catalyst mass increase to determine the accumulation of carbon on the catalyst surface. The reforming reactor which operated adiabatically is shown in Fig. 1, showing the laser optics used. The oxidation catalyst is typically preheated by dry nitrogen to about 325 °C so that light-off quickly occurs when the fuel/air mixture is introduced. The operating pressure of the system was 15 psig.

The analysis of the product composition stream was conducted by various analytical techniques, specifically laser scattering for carbon formation, gas chromatography for quantitative gas analysis, NDIR (non-dispersive infrared) for real time carbon monoxide and carbon dioxide measurements, paramagnetic oxygen and GC/MS (gas chromatography/mass spectrometry). Main fuel components that were tested included aliphatic (straight-chained and branched chained), aromatic, napthenic and olefinic. Oxygenated fuels and sulfur-containing hydrocarbon components were also examined. Other 'real' fuel streams that were tested include hydrotreated naptha, reformulated gasoline, kerosene, de-odorized kerosene, low sulfur diesel, diesel fuel and bio-diesels.

Light-off measurements were conducted in an isothermal micro-scale reactor. Lower light-off temperatures require less preheating, potentially reduce start-up time and possibly reduce start-up energy. The measurements were made using Pt/Rh catalysts supported on YSZ reticulated foams, which were ground and sieved to a particle size of 595–1000 μm. The light-off of different fuels and fuel components was measured by introducing the fuel/air/steam mixture to the reactor catalyst at a O/C = 0.9, S/C = 1.0, and an initial temperature of 125 °C. A space velocity of about 11,000 h^{-1} was used. After the feed reached the catalyst bed, the temperature of the catalyst bed was ramped linearly at 4 °C/min. The catalyst bed temperature and the outlet composition were measured in real time by thermocouples and a mass spectrometer to observe when oxygen consumption started, and hydrogen, carbon monoxide and carbon dioxide were generated. An example light-off for *iso*-octane with 20% pentane is shown in Fig. 2. At about

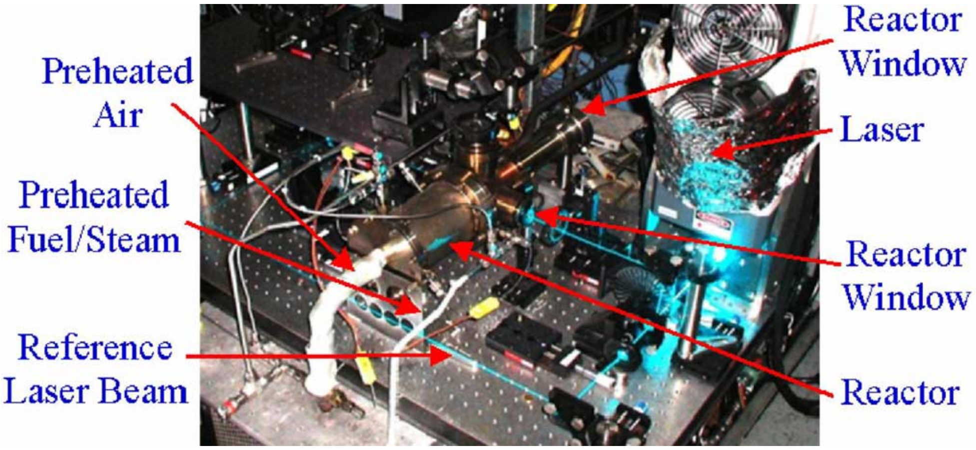

Fig. 1. Catalytic auto-thermal reformer with catalyst observation windows and Argon Ion laser to measure carbon formation via laser extinction.

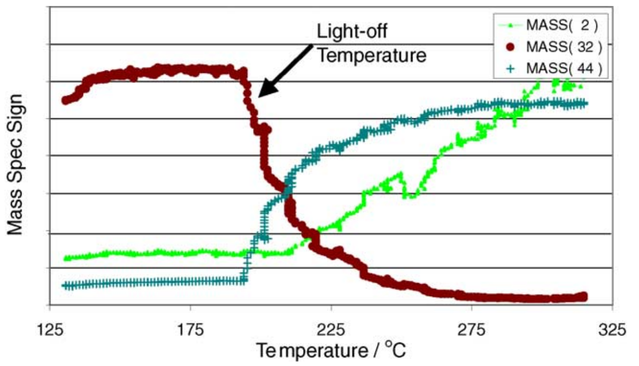

Fig. 2. Reformer light-off with *iso*-octane and 20% pentane at O/C = 0.9 and S/C = 1.0.

195 °C, the oxygen signal (*m*/*e* = 32) starts decreasing, and the CO_2 signal (*m*/*e* = 44) start increasing, indicating the hydrocarbon oxidation is beginning. Hydrogen (*m*/*e* = 2) production starts at a slightly higher temperature of 210 °C. As the temperature increases, full oxygen conversion occurs at approximately 275 °C. The light-off of an adiabatic reactor occurs somewhat differently, in that once the oxidation starts, the reaction exotherm raises the catalyst temperature, thus the light-off occurs without additional heat input. Light-off measurements were conducted to observe carbon formation for different fuel components in the adiabatic reactor shown in Fig. 1. The catalyst was preheated to about 325 °C, before fuel and air were introduced at an O/C = 1.0 and for various S/C ratios. Once the fuel/steam/air mixture is introduced, light-off occurs quickly, with the reformer outlet temperature quickly rising to its normal operating temperature. After 30 s of operation the reactor fuel and air were shut off, while a nitrogen purge was initiated, and the carbon that was produced was quantified.

3. Results and discussion

3.1. Hydrocarbon effect on light-off

The light-off temperature for catalytic partial oxidation was measured for individual fuel components and fuel blends to define different fuel component effects on the light-off. Various hydrocarbon chemical functionalities were included to help define the chemical impact on the light-off temperature. The various functionalities include straight-chained aliphatic, branched chained aliphatic, aromatic, olefinic, oxygenated and sulfur containing hydrocarbons. *Iso*-octane (2,2,4 trimethyl pentane—branched C_8) is often used as a simulant for gasoline fuel, and is thus used for comparison purposes in this study. Fig. 3 shows the light-off of straight-chained hydrocarbons, from *n*-pentane to *n*-decane, *iso*-octane and mixtures of *iso*-octane with pentane and *iso*-octane with hexane at 80% *iso*-octane. As the carbon length of the straight-chained molecules is increased, the light-off temperature increases. However, the light-off temperature of all of the straight-chained hydrocarbons up to C_{10} is lower than the light-off temperature of *iso*-octane (C_8). As *iso*-octane is mixed with the straight-chained hydrocarbons, the light-off temperature is reduced in comparison to 100% *iso*-octane. This indicates that the light-off temperature is a combination of the hydrocarbon constituent properties, and not just a function of one component or the other. The light-off temperature does not seem to correspond to the fuel component C–H or C–C bond dissociation energy. For example, the bond dissociation energy of straight-chained hydrocarbon C–C bonds decreases as the hydrocarbon chain length increases, however the light-off temperature increases with increasing chain length. The same is true for aliphatic hydrocarbon C–H bond dissociation energies, [1] thus there does not seem to be a correlation between the hydrocarbon bond dissociation energies and the fuel light-off temperature.

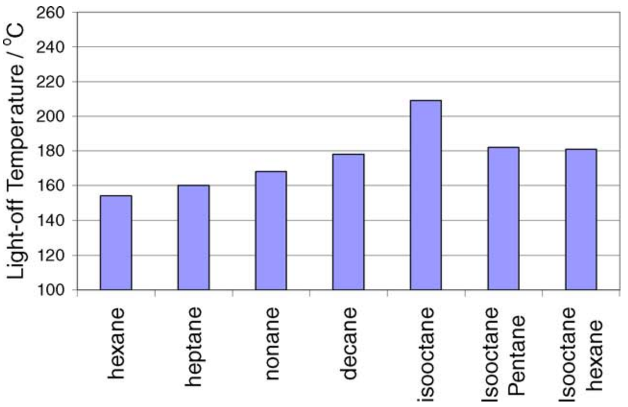

Fig. 3. Reformer light-off with various aliphatic hydrocarbons (*n*-hexane, *n*-heptane, *n*-nonane, *n*-decane, *iso*-octane, *iso*-octane + 20% *n*-pentane and *iso*-octane + 20% *n*-hexane).

Oxygenated hydrocarbons show a lower light-off temperature, both in mixtures with *iso*-octane and in reformulated gasoline (RFG). Fig. 4 shows the light-off

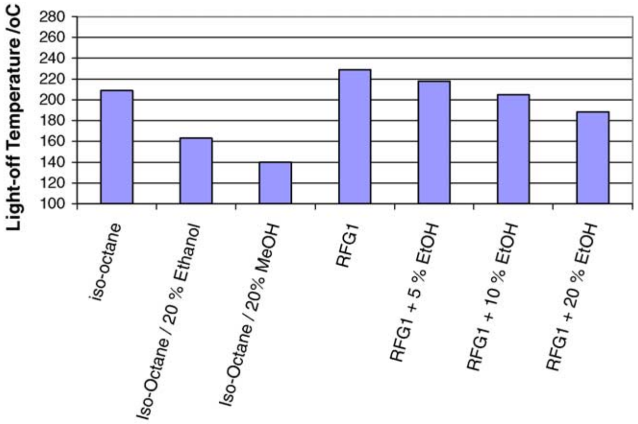

Fig. 4. Reformer light-off with various oxygenated hydrocarbons and fuels (*iso*-octane, *iso*-octane + 20% ethanol, *iso*-octane + 20% methanol, RFG – reformulated gasoline, RFG + 5% ethanol, RFG + 10% ethanol and RFG + 20% ethanol).

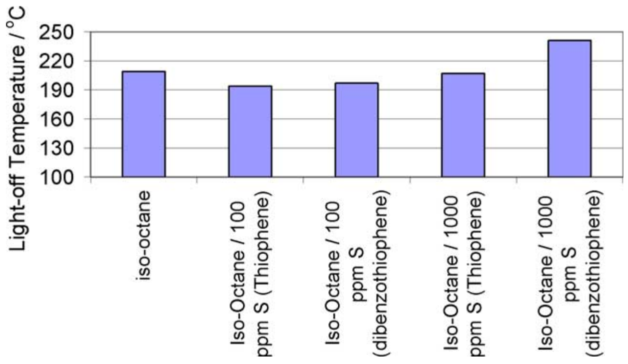

Fig. 5. Reformer light-off with *iso*-octane and *iso*-octane with 100 ppm thiophene, 100 ppm dibenzothiophene, 1000 ppm thiophene and 1000 ppm dibenzothiophene (ppm by weight of S).

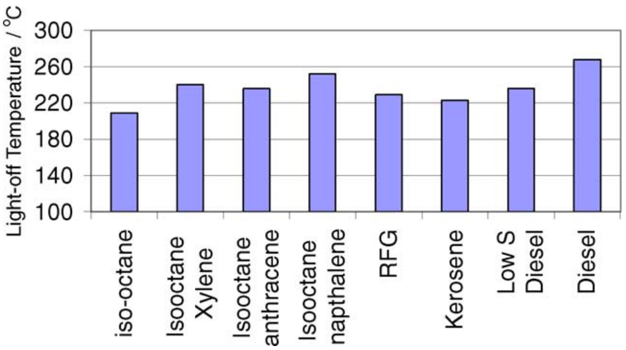

Fig. 6. Reformer light-off with various aromatic hydrocarbons and fuels (*iso*-octane, *iso*-octane + 20% xylene, *iso*-octane + 5% anthracene, *iso*-octane + 5% naphthalene, RFG – reformulated gasoline, kerosene, Low sulfur diesel fuel, and commercial diesel fuel).

temperatures for *iso*-octane mixed with 20% ethanol, *iso*-octane mixed with 20% methanol and RFG mixed with concentrations of ethanol varying from 5 to 20%. In all cases, the mixing of oxygenated compounds reduces the light-off temperature, with the greater reduction happening with methanol in comparison with ethanol. The higher the concentration of the oxygenate, the more the reduction in the light-off temperature.

A ubiquitous impurity in hydrocarbon fuels is sulfur, which has been shown to be detrimental to the reforming of hydrocarbons [2]. To examine the effect of sulfur and sulfur compounds on the light-off of reformers, thiophene and dibenzothiophene were added to *iso*-octane at levels of 100 ppm and 1000 ppm. These results are shown in Fig. 5. The sulfur at levels of 100 ppm for both thiophene and dibenzothiophene is not detrimental to the light-off of the reformer, in fact these compounds decreased the light-off temperature of the catalyst by 10–15 °C. However, when the compounds are added at a concentration of 1000 ppm, the favorable effect disappears with thiophene, and dibenzothiophene increases the light-off temperature by over 30 °C.

Current commercial gasoline and diesel fuels have a typical content of aromatic compounds on the order of 20%. Aromatic components have been shown to slow the reforming kinetics and to increase carbon formation [3]. Fig. 6 shows the light-off measurements of *iso*-octane, and *iso*-octane mixed with xylene, anthracene and naphthalene. The light-off temperatures of reformulated gasoline (RFG), kerosene, low sulfur diesel fuel and a commercial diesel fuel are also shown. Addition of the aromatic compounds all increased the light-off temperature of *iso*-octane. Also, all of the fuel blends showed higher light-off temperatures than *iso*-octane, which is probably a combined function of the aromatic content and long chained hydrocarbons.

Hydrogen production via homogeneous partial oxidation is a fundamentally different methodology compared with catalytic partial oxidation. Although most developers have not favored this approach, it does have potential for ease in reformer start-up, as the fuel air mixture does not require catalyst heating. At least two homogeneous oxidation systems have been developed for hydrogen production for fuel cells [4–6]. For reactor ignition, a spark is generally used to initiate the combustion reaction. The operation of homogeneous oxidation has shown that the oxidation reaction prefers fuels with longer-chained hydrocarbons which correlate to high cetane # fuels [7]. Similar effects have been observed for the partial oxidation fuel ignition. For homogenous reaction tests, the same preheat as the adiabatic partial oxidation reactor was used for the ignition. The air/fuel ratio was changed to observe the limits for which the reactor ignition occurred. *Iso*-Octane requires a high air/fuel ratio (O/C = 1.2), while RFG (gasoline) ignition occurred at a lower air/fuel ratio (O/C = 1.0).

3.2. Special fuels light-off (hydrogen, methanol, and DME)

The purpose of the on-board reforming is to provide hydrogen for the fuel cell. One method to help with the fuel processor light-off is to store some of the produced hydrogen for subsequent reformer light-off. Once the partial oxidation reaction is at temperature, the fuel can be changed to the normal on-board fuel. The advantage of using hydrogen for the partial oxidation light-off is that light-off occurs at room temperature. This is shown in Fig. 7. The initial temperature rise occurs concurrently with the addition of air without significant lag time. The time at which the reactor reaches operating temperature is mainly a function of the air/hydrogen feed rates. In this test, 80 SLPM (standard liters per minute) of hydrogen and air were fed, with the hydrogen flow started first, followed by air 4 s later with the nominal operation temperature of the reactor reached within about 17 s of starting the hydrogen, or about 13 s of starting the air.

Methanol is another special case when reforming of fuels is considered. It does not contain any C–C bonds, and tends to adsorb on the catalyst surface via the oxygen from the alcohol. As such, it avoids the formation of methylene species, and thus the reforming of methanol occurs at relatively lower temperatures (∼220–270 °C). Methanol is also potentially readily available on-board a vehicle as it is a

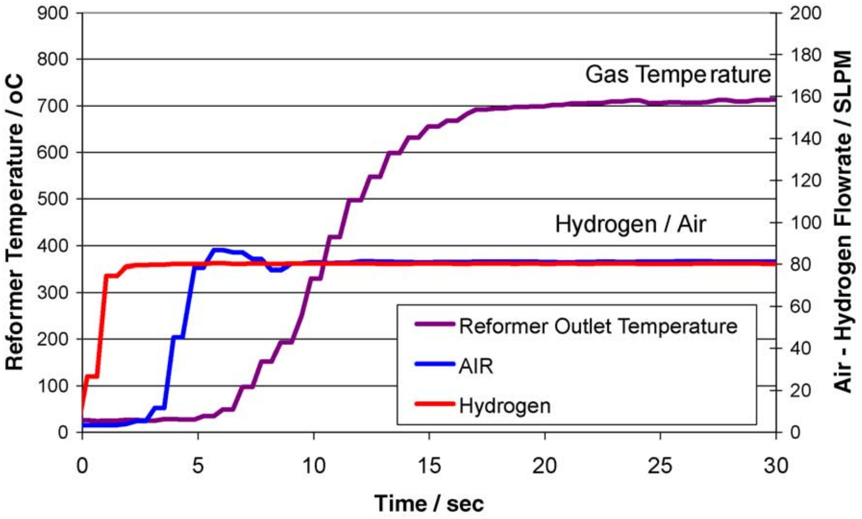

Fig. 7. Partial oxidation reactor light-off with hydrogen/air. Initial temperature was approximately 25 °C.

main ingredient in most windshield wiper fluids. The light-off of methanol shows that it also has catalytic ignition at low temperatures. Fig. 8 shows a reformer light-off at room temperature with methanol. For this test, a 1.2 water/methanol mixture was used, with the air feed being slowly ramped up from an O/MeOH ratio of 0–2. As the air/methanol ratio was increased, light-off of the mixture occurred at about an O/MeOH ratio of 1.45, while the air/fuel feed temperature was maintained at 13 °C. This demonstrates the ability to use a water/methanol mixture to light-off a reformer without any catalyst preheating.

A similar fuel to methanol, but without some of the toxicity issues that methanol presents, is dimethylether (DME). Similar to methanol, DME does not have C–C bonds, will absorb via the O onto catalytic surfaces and thus steam reforming can occur at relatively low temperatures. A steam reforming comparison of DME and methanol is shown in Fig. 9, showing that while a higher temperature is required for full conversion of DME compared with methanol, the steam reforming reaction still proceeds at low temperatures (350 °C) compared with hydrocarbons such as gasoline. DME is a vapor at atmospheric pressure, so it has an advantage for partial oxidation reactor light-off in that it could be fed as a vapor for the initial light-off.

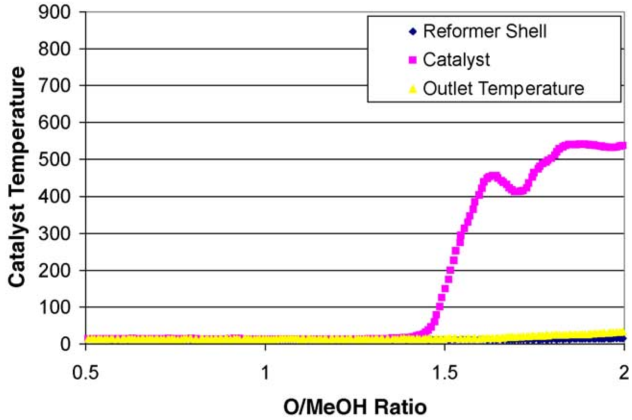

Fig. 8. Light-off of a 1.2 water/methanol mixture with air from 13 °C. Air flowrate was slowly increased with the catalyst temperature monitored to observe light-off.

3.3. Carbon formation during light-off

Carbon formation is recognized to be a potential limiter of fuel processor ATR durability. Equilibrium defines fuel processor operating conditions during normal operation to avoid issues with carbon formation. Equilibrium calculations can identify proper operating conditions to prevent carbon formation, however, during the start-up of the fuel processor, avoiding non-zero carbon equilibrium is difficult. This is shown in Fig. 10. The curve is the calculated carbon equilibrium disappearance temperature, i.e. the temperature above which carbon formation is no longer predicted by equilibrium for specified conditions. As a partial oxidation/reforming system is started under fuel rich conditions, we can see from Fig. 10 that carbon equilibrium is predicted during the reformer light-off for standard ATR steam contents (i.e. S/C < 2.5). At start-up of the fuel processor,

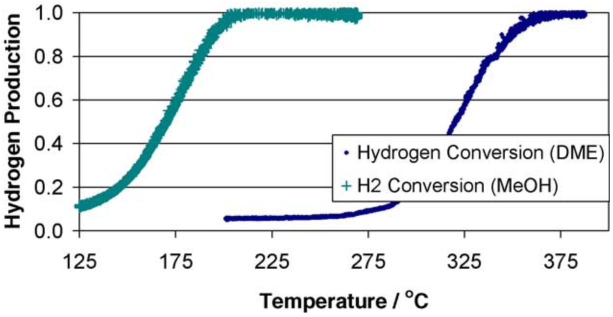

Fig. 9. Steam reforming comparison of methanol and DME over a Cu/ZnO catalyst.

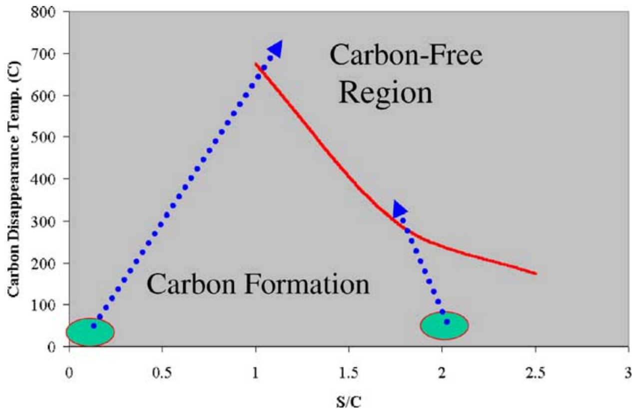

Fig. 10. Carbon formation equilibrium modeling of the carbon disappearance temperature vs. steam/carbon ratio.

water availability is also questionable due to issues with storing water on-board a vehicle (due to freezing conditions).

Quantification of carbon formed during reformer light-off was conducted for a number of fuel compositions at S/C = 0.0, and S/C = 0.5. Fig. 11 shows the results of these carbon measurements, and indicates that carbon was made during start-up for all fuels and conditions tested. For *iso*-octane, adding water during start-up suppresses carbon formation, however carbon is still formed in smaller quantities. Addition of ethanol to *iso*-octane or RFG also suppresses carbon formation. The addition of 20% ethanol decreases carbon formation by about 40% for RFG, and by 80% for *iso*-octane. Aromatic fuel components show higher carbon formation during start-up. Normal steady-state operation at similar conditions (O/C = 0.75, S/C = 1.0) shows no measurable carbon formation over short periods of operation (∼1 h).

Post analysis has been conducted on the supported Pt/Rh catalysts used in this study to quantify adsorbed carbon on the catalyst surface. Both TGA and weight gain measurements of the reforming catalysts indicate little carbon remains adhered to the catalysts [10]. However, this appears to be in contrast with diesel reforming measurements made on Pt/Al_2O_3 catalysts, where carbon has been observed on the catalyst surface, [11] and for auto-thermal reforming with nickel catalysts [12].

The amount of carbon formed during start-up was about ∼0.5–3% of the carbon from fuel over the 30 s start-up period. Durability targets are for 5000 h of operation, with the number of start-up cycles ranging from 4000 to 10,000. Over 10,000 start-up cycles forms on the order of 2.5 kg of carbon for RFG gasoline. This amount of carbon is significant in comparison with the available fuel processor system volume. This quantity of carbon most likely would clog available pore volume, valves, tubing and downstream catalysts. Thus for a durable system, the carbon formed during start-up needs to be either reduced significantly (as it could by the use of hydrogen, or the addition of ethanol), or regular maintenance will be required.

3.4. Start-up energy

The start-up energy requirement is the energy required to bring the fuel processor system from ambient temperature to an operating temperature where fuel cell quality hydrogen can be produced. The likely method to generate heat for the fuel processor operation is to combust the on-board fuel that is used to generate the hydrogen-rich fuel cell feed. To further compare the effect of fuels on starting up the fuel processor, we have modeled the start-up energy as a function of fuel. The fuel processor start-up energy requirement using various fuels was calculated relative to a gasoline fuel processor meeting the 2010 DOE target technology [8]. The fuel processor for this study was modeled as an ATR (autothermal reformer), HTS (high temperature water gas shift), LTS (low temperature water gas shift) and PrOx

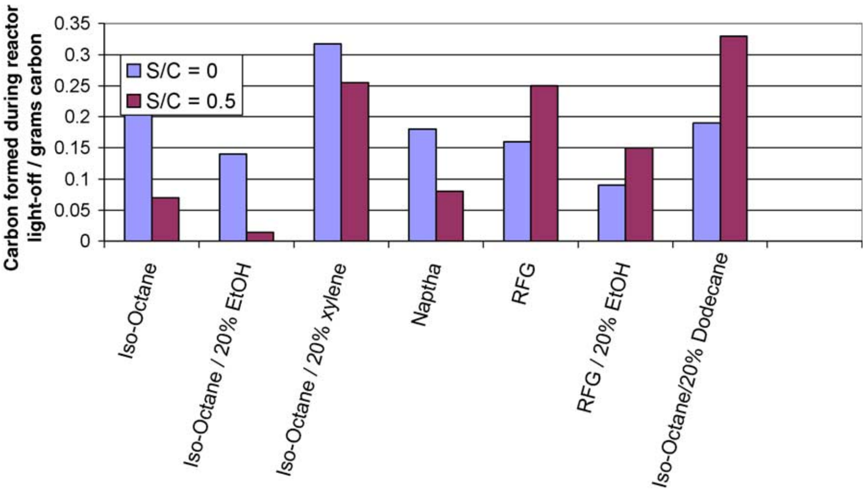

Fig. 11. Carbon formed during the start-up of adiabatic partial oxidation reactor for various fuel components and mixtures at steam/carbon ratios of 0.0 and 0.5.

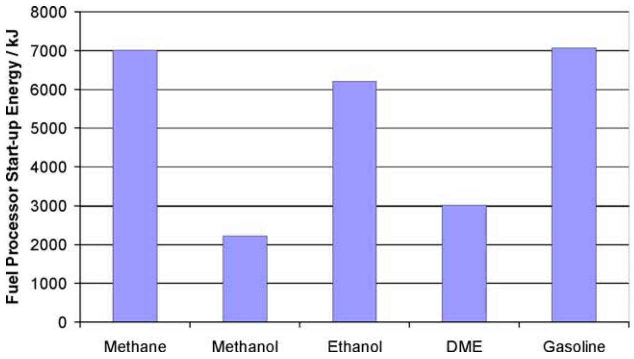

Fig. 12. Modeled energy consumption for various fuel feedstocks to achieve fuel processing system operating temperature.

(preferential oxidation of carbon monoxide) reactors. A gasoline fuel processor reactor was scaled on a volume basis to meet the DOE 2010 technical targets for fuel-flexible fuel processors, then modified for proper configuration as a function of the modeled fuel. The start-up energy was calculated for the fuels: gasoline, natural gas, ethanol, methanol and DME.

The energy consumption for the fuel processor start-up is shown in Fig. 12 as a function of fuel. Gasoline, methane and ethanol have similar energy requirements for the start-up of their associated fuel processors, while methanol and DME have significantly lower energy requirements. Although ethanol has a lower light-off temperature, it has a similar energy requirement to gasoline as the energy requirement is calculated to raise the temperature of the ATR, HTS, LTS and PrOx reactors to their operating temperatures, and these fuel have similar requirements for reactor volumes. Since methanol and DME have much lower operating ATR temperatures, and do not require HTS reactors, they thus have much lower start-up energy requirements.

Correlating the fuel processor start-up energy demand as a function of modeled fuels to overall efficiency of the fuel cell system indicates that a large reduction from the system steady-state efficiency will be observed for typical driving conditions. For example, if the steady-state system efficiency is assumed to be 44% for the gasoline system, we model that for a standard driving schedule the reduction due to the start-up energy consumption in efficiency is about 7%, thus the realized system efficiency when start-up energy consumption is included is 37%. The methanol system shows a lower reduction from steady-state efficiency due to the lower start-up energy consumption. From this, we can conclude that the start-up energy requirement can have a significant impact on the system efficiency and fuel consumption and will be a factor determining if on-board reformers are to be commercially realized for the transportation market. Further details on the modeling of the fuel effect on fuel processor start-up energy can be found in a separate publication [9].

4. Conclusions

Fuel composition is important to the on-board production of hydrogen for transportation fuel cell systems. The on-board reforming of existing fuels already present in the infrastructure can help speed the commercialization of fuel cell technology. Fuel composition can be modified for more optimal reforming characteristics, and it will be easier to modify the current fuel infrastructure (i.e. addition of oxygenates, removal of sulfur and aromatics) than it will be to develop a new infrastructure based on hydrogen.

Catalytic oxidation and reforming requires catalyst pre-heating before the oxidation reaction becomes self-sustaining (lights-off). The fuel composition affects the light-off of the reformer. Aliphatic hydrocarbons tend to have more favorable light-off and reforming characteristics compared with aromatic hydrocarbons. Shorter chained aliphatic compounds have lower light-off temperatures than longer chained hydrocarbons, and straight-chained species lower than similar length branched-chain. The addition of oxygenated compounds reduces the light-off temperature.

Homogeneous oxidation favors longer chained hydrocarbons, which correlates to high cetane # fuels. Other methods of light-off which occur at low temperatures include using hydrogen (which could be produced on-board and stored for subsequent start-ups), or using methanol as a start-up fuel.

Chemical equilibrium favors the formation of carbon during the light-off phase of partial oxidation reactors. Monitoring of carbon formation during the light-off of the reactor showed that at least some carbon was formed for all conditions and species tested. Aromatics and longer chained hydrocarbons show a higher tendency for carbon formation, while the addition of oxygenates tends to lower the carbon formed during the start-up process.

The magnitude of the fuel processor start-up energy demand was determined to be lower for methanol and DME compared with simulated gasoline, methane and ethanol. The efficiency penalties incurred due to start-up can have a large impact on the overall system efficiency for short drive-cycles.

Acknowledgments

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