Nickel-catalyzed autothermal reforming of jet fuel surrogates: *n*-Dodecane, tetralin, and their mixture

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Abstract

The potential of a nickel-ceria-zirconia based catalyst was examined for autothermal reforming of *n*-dodecane, tetralin and their mixture into a hydrogen-rich product stream suitable for high-temperature fuel cells. *n*-Dodecane and tetralin were chosen as representative compounds for alkanes and bicyclic compounds in jet fuel. It was possible to reach conversions greater than 90% for both components, as long as sufficiently high oxygen concentrations were maintained in the feed. Tetralin gave larger yields of reforming products than *n*-dodecane. During the transient start-up phase of the reactor, large temperature excursions were observed, suggesting that the reaction starts with complete combustion of fuel, giving way to autothermal reforming after a few minutes on stream. These high-temperature excursions during reactor start-up are large enough to change the catalyst surface areas. Interestingly, the mixture of tetralin and *n*-dodecane did not behave as a linear combination of the two pure components, but showed reforming characteristics similar to pure tetralin. The non-linear behavior of the mixture provides a caveat that investigations of single component model compounds may not be able to capture the reforming behavior of more complex fuel mixtures.
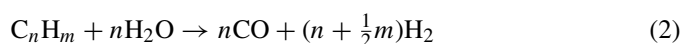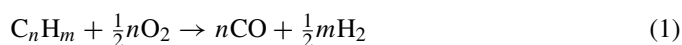© 2006 Elsevier B.V. All rights reserved.

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1. Introduction

Jet fuel is a critical transportation fuel that is primarily used in commercial aircraft and military vehicles. The United States consumes over 50 million gallons of jet fuel a day [1]. The term "jet fuel" encompasses kerosene-based liquid hydrocarbon mixtures that are used to power turbine engines. Jet fuel is heavier than gasoline, but lighter than diesel. There are many different types of jet fuel: JET A, JET A-1, JP-5, and JP-8. The key differences between them are fuel additives and distillation fractions. Jet fuel chemistry is challenging to study experimentally since the composition of commercial fuels is variable with geography and season. There is great interest in using jet fuel to power solid oxide fuel cell (SOFC) auxiliary power units (APU) on-board of vehicles. An auxiliary power unit is a device that is used to provide additional electrical power to a vehicle, particular when the vehicle is not in motion. Auxiliary power units displace engine idling and thus reduce fuel consumption. To use jet fuel for this

purpose requires the development of an on-board catalytic fuel processor capable of converting fuel into a hydrogen and carbon monoxide-rich reformate.

Current reforming technologies have been developed mainly for stationary applications involving the conversion of light hydrocarbons. In fact, the two principal processes for hydrogen production in industry are steam reforming (SR) and partial oxidation (POX) of natural gas. Autothermal reforming (ATR) is the combination of steam reforming and partial oxidation. It is used in large stationary applications, where the construction of an oxygen plant is economical. The general reactions for hydrocarbon reforming are shown below:

$$C_nH_m + \tfrac{1}{2}nO_2 \rightarrow nCO + \tfrac{1}{2}mH_2 \tag{1}$$

$$C_nH_m + nH_2O \rightarrow nCO + (n + \tfrac{1}{2}m)H_2 \tag{2}$$

$$CO + H_2O \leftrightarrow CO_2 + H_2 \tag{3}$$

For onboard applications some attempt has been made at gasoline reforming for PEM fuel cells [2,3]. This approach was abandoned by the DOE in a no-go decision because of the complex reactor systems needed to convert gasoline

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into high purity hydrogen feeds required for PEM fuel cells [4]. Nevertheless, investigating on-board diesel and jet fuel reforming for use in SOFC-APUs has merit because solid oxide fuel cells can use both hydrogen and carbon monoxide as a feed, thus greatly simplifying the fuel processing system.

Catalysts for onboard jet fuel reforming have a myriad of design requirements. They must be active for both partial oxidation and steam reforming reactions. They must be able to prevent coking during operation at low steam to carbon ratios. They must not sinter at the high temperatures associated with partial oxidation. They must be able to start-up and shut down without complicated regeneration procedures. They must be tolerant to some amount of sulfur. They must be flexible to the variability in fuel composition. The concept of heavy liquid hydrocarbon reforming is not new and was researched during the 1980s [5]. The literature shows that since then great progress has been made towards reforming of hydrocarbons larger than C_8 [6–16]. However, there are still many open questions regarding the influence of individual fuel components on the reforming behavior of commercial fuels. The catalytic systems described are usually precious metal or nickel supported on oxide supports. There seems to be a consensus that the reforming of hydrocarbons typical for jet fuel is possible, but difficult because of problems associated with carbon formation and sulfur poisoning. Consequently, the literature shows a strong interest in catalytic supports with oxygen storage capacity, particularly mixed ceria-oxides, as a means to reduce coke formation [17]. It has been suggested that oxygen in the support plays an important role in the reforming of the liquid hydrocarbons, possibly by making the catalyst more resistant to coking by oxidizing surface carbon species. However, the detailed mechanisms for liquid hydrocarbon reforming, carbon deposition, and coke formation remain open to debate. One school of thought is that coke forms by the polymerization of large hydrocarbons into arrays of polycyclics. It is believed that aromatics are detrimental to reforming because the compounds act as precursors for the formation of the polycyclic networks. On nickel catalysts, an additional route for carbon deposition needs to be considered, namely the growth of carbon filaments [18]. Some attempts have been made to understand how the chemical structure of fuels influences coking behavior, but a clear picture does not currently exist.

In previous work in our laboratories, we found that a $Ni/Ce_{0.75}Zr_{0.25}O_2$ catalyst was very effective in autothermal reforming of isooctane and toluene [2]. Encouraged by the work on isooctane reforming, the feasibility of using this $Ni/Ce_{0.75}Zr_{0.25}O_2$ catalyst was explored for autothermal reforming of jet fuel constituents. The influence of the oxygen to carbon ratio on hydrocarbon conversion was briefly examined. The behavior of jet fuel paraffins and bicyclic aromatics was compared in terms of conversion and apparent deactivation. This paper is intended to be an exploratory study of fuel component behavior with the future goal of developing a more detailed understanding of the relationship between molecular structure and reforming behavior. The ultimate goal is the development of a robust jet fuel autothermal reforming catalyst.

2. Experimental

2.1. Materials

The hydrocarbons found in jet fuel can be grouped by their chemical structure into the following classes: paraffins, olefins, cyclic paraffins, aromatics, and bicyclic compounds [19]. It is from these classes of hydrocarbons that pure model compounds can be selected to represent key characteristics of jet fuel. This initial study was limited to *n*-dodecane and tetralin, which represent the paraffinic and bicyclic characteristics of jet fuel. Dodecane has a number of carbon atoms similar to the average molecular formula of jet fuel ($C_{11}H_{21}$). It is thought to be a good single component representative of jet fuel as a whole. Tetralin is suspected to be a coke precursor because of its similarity to graphene. Both tetralin and dodecane were purchased from Sigma–Aldrich at >99% purity.

Catalyst supports were prepared by co-precipitation of precursor salts. Appropriate quantities of $Ce(NO_3)_3 \cdot 6H_2O$ and $ZrOCl \cdot 8H_2O$ were dissolved in stirred de-ionized water. A solution of 4 M NH_4OH was added drop wise by an HPLC pump while stirring to precipitate the solution. The precipitate slurry was aged overnight while stirring, and then recovered by vacuum filtration. The filter cake was washed twice with de-ionized water, and then dried overnight in an oven at 110 °C. The resulting solid was crushed and calcined in air at 800 °C for 1 h.

Catalysts were synthesized via wet impregnation of the mixed oxide support with a nickel solution. Appropriate quantities of $Ni(NO_3)_2 \cdot 6H_2O$ were dissolved in water, then added drop wise to the support powder until it was saturated. The saturated support was then allowed to dry. The addition of nickel solution and drying procedure was repeated until the desired nickel loading was achieved. Following the impregnation, the catalyst was calcined in air at 600 °C for 1 h. Catalyst particles were then crushed and sieved to a size of 40–80 mesh. All catalysts used in this work were prepared as nominally 10% nickel by weight, calculated as the weight of nickel divided by the weight of the nickel and the support.

2.2. Experimental apparatus

The reactor consisted of a 43 cm long quartz tube with 1.27 cm o.d. and 1.0 cm i.d. Powder catalyst samples were packed between two plugs of quartz wool inside the reactor tube. The typical catalyst weight was 400–700 mg. Inconel sheathed K-type thermocouples were placed in both the downstream and upstream side of the catalyst bed. The downstream thermocouple was placed in direct contact with the backside face of the catalyst bed. The quartz tube was then placed inside a thermostat-controlled horizontal tube furnace whose temperature was maintained constant within ±1 °C. The flows of air and N_2 were metered into the system by MKS mass flow controllers. The flows of fuel and water were metered into the system by an Isco syringe pump and an Instech peristaltic pump, respectively. All three components (fuel, air, water) were mixed in a

heated cross which exited into a 3 m long section of heat traced 6.35 mm o.d. stainless steel tubing that served as a vaporizer. A thermocouple located under the insulation of the vaporizer showed a temperature of approximately 209 °C, but the readout was sensitive to thermocouple placement suggesting higher temperatures inside the vaporizer tube. In order to determine that the fuel was fully vaporized the mass balance of the fuel through a blank reactor tube held at 250 °C was performed. The blank run showed complete vaporization of the fuel with better than 99% closure of the mass balance. From the vaporizer the reactant mixture passed into a 21 cm long preheating section of the quartz tube reactor, raising the temperature of the feed stream to 550 °C prior to reaching the catalyst bed. The reactor effluent passed through an ice trap to knock out any condensable liquids before entering the gas analysis train. The gaseous reactor effluent concentrations were determined by an online Varian gas chromatograph equipped with two TCDs, which were used for permanent gas analysis, and one FID for heavier hydrocarbon analysis. The condenser bottoms were analyzed with an Agilent GC-MS for product identification. Concentrations of gaseous product compounds were determined from calibration curves and the conversion X was determined from the following equations:

$$X = \frac{\text{Carbon in gas products}}{\text{Carbon in fuel}} = \frac{F_{\text{total out}}(y_{\text{CO}} + y_{\text{CO}_2} + y_{\text{CH}_4})}{n F_{\text{fuel in}}} \tag{4}$$

$$F_{\text{total out}} = \frac{F_{\text{N}_2 \text{ in}}}{y_{\text{N}_2 \text{ out}}} \tag{5}$$

In Eq. (4), n represents the number of carbon atoms in the fuel molecule investigated. F is the molar flow rate in moles s^{-1}, and y the mole fraction of carbon containing products, both determined on a dry basis.

2.3. Start-up and shutdown procedure

Initially, the catalyst bed and reactor were heated under flowing nitrogen to a temperature of 550 °C. The flow of nitrogen was ceased and then steam and air were brought on-stream. The flow of steam and air was given approximately 10 min to reach steady-state before the fuel was brought on stream to initiate the reaction. Once the reaction started, the furnace remained on to pre-heat the reactants to a feed temperature of 550 °C. None of the catalysts were pretreated with hydrogen before reaction because in previous work the catalysts were found to auto-reduce in situ. The typical duration of an experimental run was 4 h, after which the reaction was extinguished by turning off the air and fuel simultaneously. Nitrogen was then brought on-stream and the steam was turned off. The reactor was cooled under N_2 flow.

2.4. Packed bed experiments

A constant flow rate of dodecane or tetralin was used in each set of experiments; the flow rates were chosen so that the total carbon molar flow rate was constant between the fuels

(0.026 mol C min^{-1}). The space velocity was held constant at 225,000 h^{-1} by varying the amount of catalyst in the reactor to compensate for varying flow rates associated with different oxygen to carbon ratios. Fresh catalyst samples were used at each experimental condition.

3. Discussion and results

A typical temperature history of an autothermal reforming experiment is depicted below in Fig. 1. As soon as fuel was added to the feed stream, the temperature downstream from the catalyst bed immediately increased beyond the feed temperature. For approximately the first minute of operation, the catalyst was exposed to a temperature spike greater than 1100 °C, followed by a gradual settling to a steady-state temperature where the downstream temperature was about 200 °C higher than the upstream temperature. The magnitude of the initial temperature spike suggests that highly exothermic combustion of fuel is taking place during the transient start-up phase, giving way to autothermal reforming after a few minutes. Catalytic performance data were collected during steady state operation. However, even then occasional smaller temperature fluctuations were measured by the downstream thermocouple.

There is evidence that the high temperature spikes during start-up caused sintering of the catalyst support. Catalysts surface areas were analyzed pre- and post-reaction by single point nitrogen BET. The surface area of the fresh catalyst was 27 $m^2 g^{-1}$. Surface areas of the catalysts post reaction ranged from 0.7 to 6.4 $m^2 g^{-1}$. The decrease in surface area correlates with the magnitude and duration of the temperature spike. In general, the post reaction surface areas decreased as the oxygen to carbon ratio increased. This trend makes intuitive sense because higher reaction temperatures are associated with greater degrees of exothermic oxidation. The uncontrolled sintering of the support during transient start-up is a source of concern, as it introduces additional complexity into the interpretation of reaction data.

Two pure components, *n*-dodecane and tetralin, and a 50:50 molar mixture of the two were reacted over a Ni/$Ce_{0.75}Zr_{0.25}O_2$ catalyst at varying oxygen to carbon ratios. The atomic oxygen
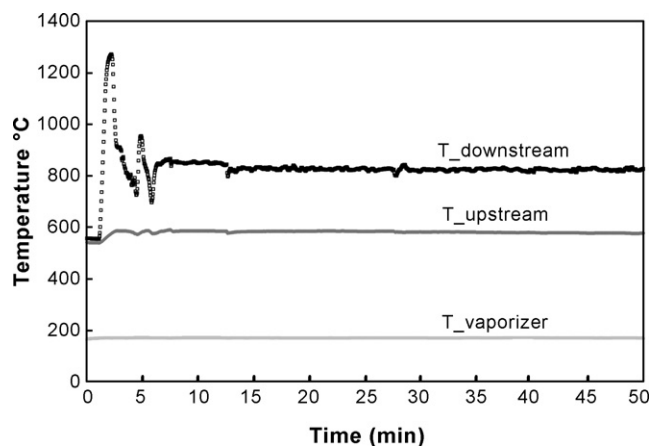

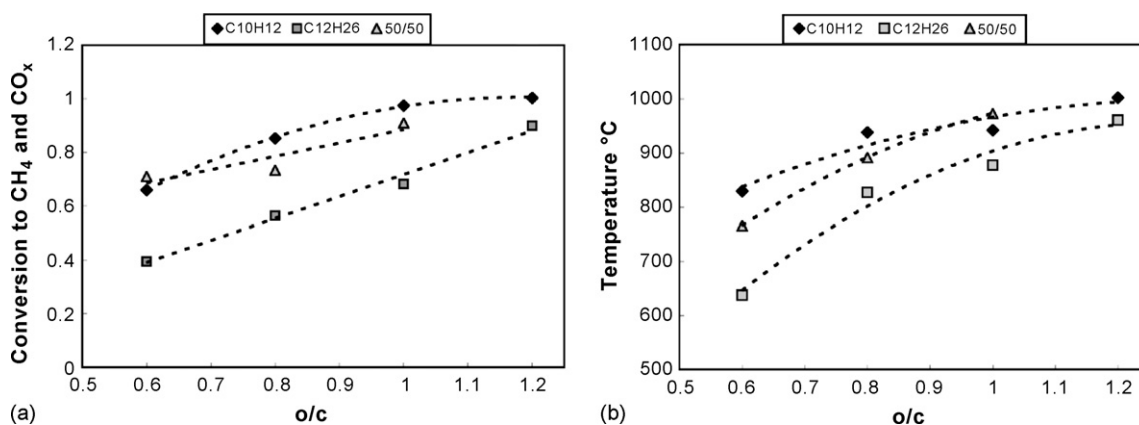Fig. 1. Typical experimental time-temperature trajectory.

Fig. 2. (a and b) Dodecane, tetralin, and 50/50 mixture conversion and downstream temperature: feed temperature = 550 °C, H_2O/C = 2.0, O/C = 0.6–1.2, GHSV = 225,000 h^{-1} ±10%.

to carbon ratios explored ranged from 0.6 to 1.2, while the steam to carbon ratio was held constant at H_2O/C = 2.0. More than 95% of the O_2 was converted during all of the experiments and the reactor effluent contained less than 1 mol% O_2. Each data point reported here represents the average of measurements collected over approximately a 4 h time period. No significant deactivation was observed during the duration of the experiments. An increase in pressure drop across the catalyst bed is often an indicator of coke formation; this was not observed during any of the experiments.

A comparison between the conversion of fuel to reforming products and the downstream temperatures for the three feeds are shown in Fig. 2. For all three feeds, increasing the oxygen to carbon ratio (O/C) raised both the conversion as well as the downstream temperature. As the oxygen to carbon ratio increased from 0.6 to 1.2, the conversion of tetralin increased asymptotically towards unity, while the conversion of *n*-dodecane increased linearly but did not reach completion. More interestingly, for the range of oxygen to carbon ratios explored, tetralin had a greater conversion to CO_x and CH_4 than *n*-dodecane. This observation implies that for a given set of reaction conditions it is easier to reform tetralin than *n*-dodecane. This observation seems counterintuitive because the stability of the aromatic ring in tetralin would tend to hinder reforming reactions. The literature has shown that aromatic compounds have higher activation energies for steam reforming than alkanes [17]. When interpreting autothermal reforming reaction data, the influence of reaction temperature cannot be neglected. Since autothermal reforming does not take place isothermally, fuel components at identical experimental conditions may not experience identical reaction temperature profiles inside the catalyst bed. Simulations in ASPEN[TM] have shown that tetralin has a higher adiabatic equilibrium reaction temperature than *n*-dodecane. Figs. 2a and b show that while the conversion of tetralin is greater than dodecane, so is the downstream temperature for tetralin reforming. A possible explanation of why tetralin has a greater conversion than *n*-dodecane is that tetralin is reacting at a higher temperature than *n*-dodecane. While these experiments provide information about the bulk behavior of the

model compounds for a given experimental condition, they do not provide insight into the relative reaction rates of the two compounds.

When the conversions and reaction temperatures of the mixture are compared to the pure components an interesting result is observed (Fig. 2). The reforming behavior of the mixture does not appear to be a linear combination of the two compounds. Both the downstream temperature and conversion were closer to tetralin than *n*-dodecane.

Comparisons between the product yields of H_2, CO, CO_2 and CH_4 for the three feeds are presented in Fig. 3. The yield of H_2 was calculated by dividing the effluent atomic molar flow rate of H by the feed atomic molar flow rate of H in the fuel. The yield of carbon containing products was calculated by dividing the effluent atomic molar flow rate of C in the product by the feed atomic flow rate of C in the fuel. When comparing the behavior of the two pure components, the yields of H_2, CO, and CO_2 are always larger for tetralin than *n*-dodecane. This is logical given that tetralin also has a higher conversion to reforming products. The opposite trend, however, is seen for the yield of CH_4, with *n*-dodecane producing more CH_4 than tetralin. The amount of CH_4 produced by tetralin was at least an order of magnitude less than *n*-dodecane and was below the accurate detection threshold of our instrument. For all practical purposes the yield of CH_4 from tetralin was zero. It seems logical that tetralin would produce far less CH_4 than *n*-dodecane because tetralin has a high degree of unsaturation. This trend in CH_4 is in agreement with ASPEN[TM] equilibrium calculations between the two pure components.

With increasing oxygen to carbon ratios, the CO_2 yields show very similar trends for both tetralin and dodecane, with the yields asymptotically increasing. The yield of CO trends positively with the oxygen to carbon ratio for both tetralin and *n*-dodecane. The most striking differences between the two pure components can be seen in the yield of H_2. A steady increase in H_2 yield was observed for *n*-dodecane when the oxygen to carbon ratio was increased from 0.6 to 1.2. In comparison, the tetralin data showed that there was an optimal hydrogen yield at an oxygen to carbon ratio of 1.0. The decrease in H_2 yield at an oxygen to
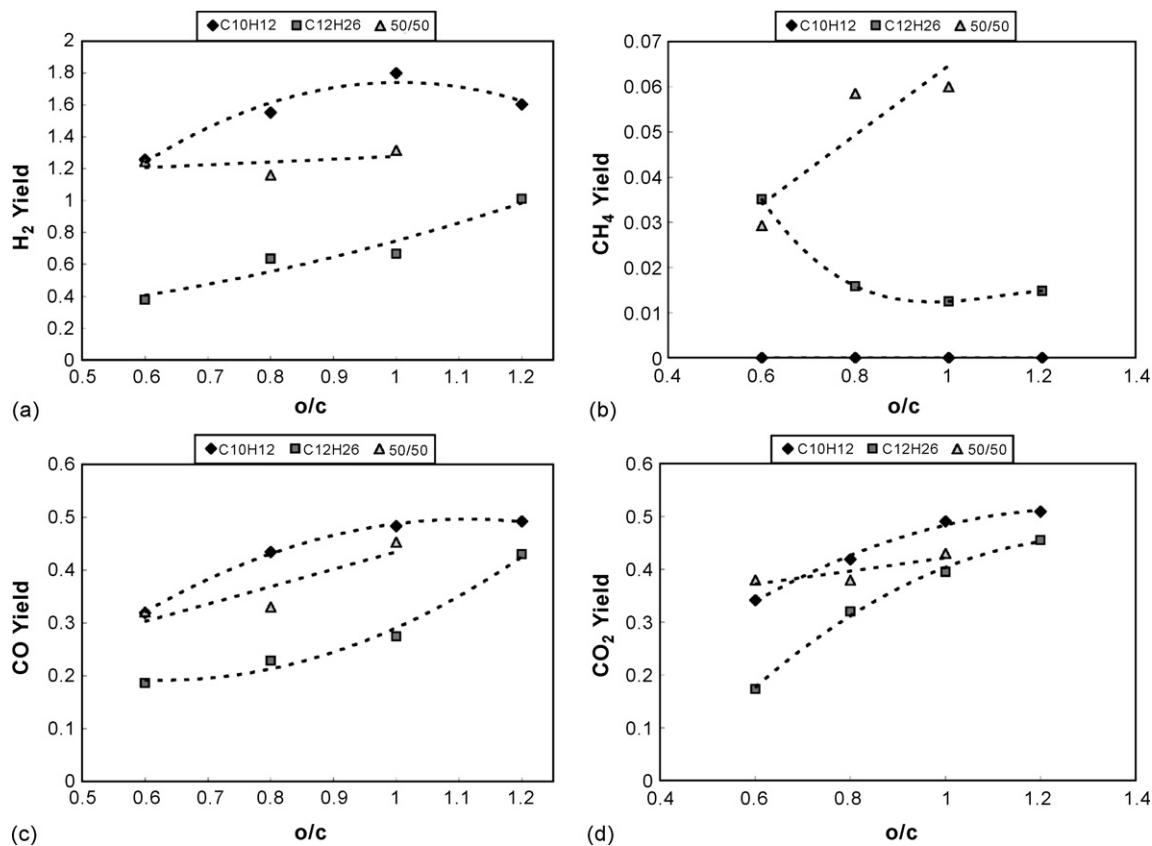
Fig. 3. (a–d) Dodecane, tetralin, and 50/50 mixture product yields: feed temperature = 550 °C, $H_2O/C = 2.0$, O/C = 0.6–1.2, GHSV = 225,000 h^{-1} ±10%.

carbon ratio greater than 1.0 is most likely caused by either the reaction of H_2 with excess O_2 to form H_2O or the formation CO_2 from excess O_2 followed by the reverse water gas shift reaction. The tetralin showed H_2 yields that are greater than 1.0, while the dodecane had H_2 yields less than 1.0. H_2 yields greater than 1.0 imply that there is either significant steam reforming or water gas shift present during the reaction and that the conversion is relatively high. The yields of H_2 for dodecane are probably below 1.0 because of its relatively low conversion.

The product yields of the 50%/50% molar mixture are particular interesting because they deviate from the two pure components. At an oxygen to carbon ratio of 0.6 the H_2 yield is very similar to that of pure tetralin. As the oxygen to carbon ratio is increased from 0.6 to 1.0, the H_2 yield remains nearly constant. This behavior is very different than the two pure components that generally show increasing H_2 yields with increasing oxygen to carbon ratios. This same trend is mirrored in the CO_2 yield. The yield of CO behaves more as expected with yield having a strong dependency on the oxygen to carbon ratio and with a behavior similar to pure tetralin as observed earlier in the conversion to reforming products. The yield of CH_4 for the mixture shows an increase in methane production over either of the single components. It is clear that the mixture is behaving in a very non-linear way. The source of this behavior still remains unclear. One speculative explanation for this behavior is that the different reaction temperature profile of the mixture causes

the differences in H_2, CO_2, and CH_4 yields. At low oxygen to carbon ratios there is the opportunity for the greatest H_2 yields because a large portion of conversion must come from steam reforming. As the oxygen to carbon ratio increases there is more opportunity for partial or even complete oxidation at the expense of steam reforming. These two competing influences may cause the H_2 yield to remain constant as the oxygen to carbon ratio is changed.

For SOFC applications, the two most important metrics of fuel processor performance are the actual H_2 and CO flow rates, because these reformer products are consumed in the SOFC to generate electricity. Fig. 4a shows the hydrogen flow rates achieved during autothermal reforming. All of these reactions were performed at identical carbon feed flow rates, thus the flow rates can be compared on the basis of equivalent carbon atoms fed. At low oxygen to carbon ratios, the mixture clearly shows a synergistic effect, producing substantially more hydrogen than the pure components. However, the CO flow rate for the mixture is somewhere between the flow rates observed for the pure components, lying somewhat closer to that of tetralin (Fig. 4b).

Post-reaction analysis was performed on the liquid compounds recovered from the ice trap using GC-MS. The liquid condensate from tetralin autothermal reforming contained mainly tetralin, along with small quantities of naphthalene, indene, 2-methylindene, and 1,2-dihydro-naphthalene. It appears that the nickel catalyst was capable of dehydro-
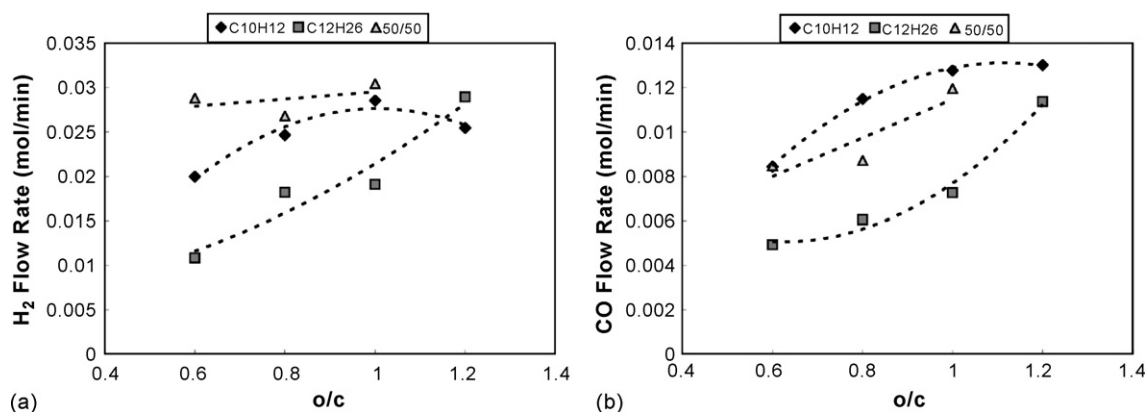
Fig. 4. (a and b) Dodecane, tetralin, and 50/50 mixture product flow rates: feed temperature = 550 °C, $H_2O/C = 2.0$, O/C = 0.6–1.2, GHSV = 225,000 h^{-1} ±10%.

genation of tetralin. For the design of a commercial fuel processor, the formation of naphthalene could be problematic because it will solidify at moderate temperatures, possibly fouling the downstream equipment. The liquid condensate from *n*-dodecane autothermal reforming contained: *n*-dodecane, 4-dodecene, 1-undecene, and oxygenated compounds like tetrahydro-2-isopentyl-5-propyl furan. The oxygenate compounds were difficult to identify on the GC-MS because of co-elution. The majority of the liquid product was unconverted *n*-dodecane, and all other compounds were formed in only small quantities. Although the conversion of both model compounds was relatively high, under many of the reaction conditions studied the water condensate in the cold trap was impure and not suitable for an SOFC.

4. Conclusions

Nickel-ceria-zirconia catalysts give relatively high conversions for the autothermal reforming of tetralin and dodecane even at space velocities greater than 200,000 h^{-1}, without significant deactivation. Small quantities of dehydrogenation products (naphthalene, indene) were observed in tetralin autothermal reforming. The aromatic compound tetralin has a greater conversion for any given oxygen to carbon ratio than dodecane, with maximum H_2 yield reached at oxygen to carbon ratio of 1.0. In the range of oxygen to carbon ratios explored, dodecane does not show such a maximum, but gives a steady increase in H_2 yield with increasing oxygen to carbon ratio.

The conversion of a mixture of dodecane and tetralin to reforming products does not seem to be a linear combination of the two individual components and is closer to the behavior of pure tetralin. The hydrogen yield of the mixture remained constant over the range of oxygen to carbon ratios explored. However, at low oxygen to carbon ratios, the mixture shows a synergistic effect, producing substantially higher hydrogen flow rates than the pure components. It remains to be seen to what extent such synergistic effects exist when additional components present in commercial fuels are examined. The non-linear behavior of mixtures has important consequences for the design of a

fuel processor for commercial fuels. Specifically, simple single component model compound studies may not be able to capture the true behavior of a complicated mixture over a range of operating conditions. Further work will be needed to find optimal operating conditions and materials for a jet fuel processor, with special attention paid to gaining better control over the temperature excursions during start-up.

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