Abstract—This paper reports on studies obtained from RF-driven atmospheric-pressure plasma-jet excitation of methane and ethane. Differentiation with other works is achieved in that others have considered hydrocarbon decomposition at either low pressure or high temperature. In our experiments, we can clarify the effect of pure-plasma treatment of hydrocarbons, as opposed to the thermal effect of gas heating that results in pyrolysis. Gas-chromatography analysis was used to detect and quantify the main decomposition products. Kinetic modeling of the pertinent chemistry was performed by dividing the reactive system in two main parts: a plasma region where the electron impact processes leading to decomposition are considered and a postplasma region where recombination of nonstable species occurs. A reasonable qualitative agreement between the experimentally measured by-product concentrations and the calculations was achieved. It is observed that our proposed recombination mechanism correctly predicts ethane and ethylene formation from a CH4 discharge and methane, ethylene, propane, and acetylene formation from the C2H6 discharge. By means of calculations, the main role of radicals in the pertinent hydrocarbon chemistry under nonthermal plasma conditions is confirmed.

Index Terms—Atmospheric-pressure plasma jet (APPJ), electron-impact process, plasma-assisted combustion, recombination model.

I. INTRODUCTION

INTEREST in nonthermal plasmas as a tool to alter combustion properties of hydrocarbons has increased in the last several years. Some nonthermal-plasma technologies have been developed to pretreat fuels just prior to combustion. A nonthermal plasma signifies less waste of energy in terms of heat deposited in the whole gas (air + fuel), because the energetic electrons are responsible for starting the cracking reactions of hydrocarbon molecules [1].

Some works in the field of plasma-assisted combustion have been carried out using a dielectric-barrier-discharge reactor to activate pure hydrocarbons and hydrocarbon/air mixtures with very promising results [2], [3]. But it is of paramount importance to better understand the chemical-activation mechanisms involved in these novel techniques to achieve practical applications.

In the past several years at the Los Alamos National Laboratory, a new nonthermal plasma source called the atmospheric-pressure plasma jet (APPJ), was developed as a system for a number of applications, such as materials processing and surface decontamination [4], [5]. The APPJ discharge is essentially different from other atmospheric-pressure plasma sources, because it is characterized by a stable, volumetric, and homogeneous discharge, which fills the volume between the electrodes [6]—attributes that make it a potential technique to study and model the basic nonthermal plasma triggered hydrocarbon chemistry.

Some of the characteristics of the APPJ have been already studied [5]. It produces a homogeneous discharge at atmospheric pressure using 13.56-MHz RF power, utilizing a predominate fraction of helium feed gas to stabilize the discharge, and consequently, it is characterized by a relatively low breakdown field of ~1 kV/cm. The APPJ operates without any dielectric-eleectrode cover yet is free of filaments, streamers, and arcing. The gas temperature of the discharge is typically between 50 °C and 300 °C, so thermal effects on hydrocarbons can be easily separated from the study of electron-driven processes [4].

While passing through the APPJ, the feed gas becomes excited, dissociated, or ionized by electron impact, producing an effluent stream of highly reactive chemical species. In this paper, the reaction-species production is estimated by means of the ELENDIF code [7], which is capable of calculating the electron-energy distribution function and convolving it with the cross sections of different electron-impact processes, giving, as a result, parameters such as the electron drift velocity and specific electron-impact rate coefficients that can be used to estimate the decomposition product concentrations per deposited plasma-energy density, expressed as a G-value (i.e., product yield in molecules per 100-eV deposited energy). For example, for the following reaction:

\[ e + AB \xrightarrow{k_1} A + B + e \]

\( k_1 \) represents the specific rate coefficient for the stated process. Now, the G-value can be defined for each different product of the reaction

\[ G_A = \frac{n_A}{\varepsilon_d} = \frac{k_1}{\pi} \int_0^t n_e n_{AB} dt = \frac{k_1 n_{AB}}{q v_d E} \]

where \( n_A \) is the concentration of the A species, \( \varepsilon_d \) is the plasma energy density, \( q \) is the charge on an electron, \( n_e \) is the electron concentration, \( v_d \) is the electron drift velocity, and \( E \) is the electric field.

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Then, the concentration of produced species $A$ can be estimated as well

$$n_A = G_A \varepsilon_d.$$  

The species produced by electron impact become part of the gas in the plasma region, and after they leave the reactor, these species react and are consumed in a variety of reactions. This subsequent interaction of stable and nonstable species is described by a chemical mechanism tested in the KINEMA chemical kinetics code [8].

In the end, the species densities obtained by means of the calculations are compared with the experimental results to validate our proposed decomposition mechanism.

II. EXPERIMENTAL METHODS

A capacitively coupled coaxial-cylinder APPJ was used for our experiments (Fig. 1). It consists of two coaxial electrodes, having an interelectrode gap of 16 mm, through which the feed gas flows. The inner electrode is powered by a radio-frequency source. The grounded outer electrode has a water-jacket cooling system. An air-cooled 13.56-MHz 2-kW RF Generator, manufactured by RF Plasma Products Inc., is coupled to the APPJ reactor through an impedance-matching system by RF Plasma Products (Model AM-5N). Fig. 2 shows the front view of the discharge in the mixture of He and CH$_4$.

Previous works have described the APPJ system in more detail [6], [9], [10]. At the end of the reactor, there is a glass tube used to join the APPJ effluent to a gas-collecting system (a Tedlar bag).

The gas products from the reactor were analyzed using a commercial Varian CP-3800 gas chromatograph (GC) equipped with a 3800 flame-ionization detector (Varian Analytical Instruments). The GC is fitted with a packed column, 1.8 m × 1/8" × 2 mm, packed with Haysep T, 80/100 mesh particle size (Varian Analytical Instruments). Helium was used as a carrier gas at a flow rate of 30 ml/min (constant flow). The analysis was carried out at isothermal conditions at $T = 60 \degree C$. Samples were directly taken from the Tedlar bag attached to the inlet of the GC multivalve system, the gas being drawn in by a 1/4-hp Duty Master A/C Motor (vacuum pump) connected to the outlet port of the multivalve line. In this system, samples are drawn at 1.2 ml/min for 2 min through the valve line before the line is opened. After that, this valve is opened and closed after 1 min. A 250-µl sample, which is the loop-volume capacity, was then delivered to the column. A personal computer is connected to the GC to control, acquire, and process the chromatographic data. For data acquisition, Star Chromatography Workstation (Varian) software is used.

An addition of up to 10% of hydrocarbon gas to the helium allowed the APPJ to operate with a stable homogeneous discharge. The total flow was kept between 15 and 20 lpm. Experiments intended to observe the power effect on hydrocarbon decomposition were carried out with a 6.4% methane discharge and a 4.9% ethane discharge. Samples of the exhaust gas at different power values were collected and analyzed by GC.

The hydrocarbon-concentration effect was tested in values ranging from 0% to 10% for both methane and ethane, and the decomposition products were measured. These experiments were carried out at constant power of 150 and 110 W for methane and ethane, respectively.

III. EXPERIMENTAL RESULTS

The methane data set included a background-gas sample for which the discharge was off and three more measurements with the discharge turned on at 120, 150, and 180 W of plasma power. The major decomposition products of the methane/helium discharge were ethane ($C_2H_6$) and ethylene ($C_2H_4$). The product concentrations can be plotted as a function of the energy density delivered to the reactor (the power divided by the gas-flow rate), and it can be seen that the ethane and ethylene concentrations steadily increase as the delivered energy density increases over the tested range from 335.66 up to 629.37 J/L, which was the maximum energy density tested (Fig. 3).
The ethane data obtained also included a background taken with the discharge off. Three more data sets were taken using energy density values of 237.62, 336.63, and 435.64 J/L. With the reactor turned on, the chromatograms of the treated samples showed methane, ethylene, propane (C₃H₈), and acetylene (C₂H₂) as the main products resulting from ethane decomposition. There is a visible dependence on the by-product (methane, acetylene, ethylene, and propane) concentrations on the power delivered to the reactor. For all these compounds, the relation appears to be linear in the studied energy density range (Fig. 4).

Using the ELENDIF Boltzmann-equation solver, the rate coefficients for the electron-impact processes involving methane and ethane were calculated for a wide range of reduced electric-field values (1–50 Td). These calculations were done for discharges in a 6.4% CH₄ field values (1–50 Td). The calculations show higher values of rate coefficients for dissociation processes than for the ionization ones, if we just consider processes that generate a structural change than dissociation to develop a first model of hydrocarbon decomposition for our plasma (Table I). In the case of ethane, the dissociation rate coefficients exceed by five or six orders of magnitude the ionization coefficient values for the E/N range calculated. At the specific experimental reduced electric field of 2 Td, ionization represents only 0.00015% of the dissociation in terms of rate-coefficient values. Therefore, for ethane, the exclusion of the ionization component of the hydrocarbon electron-impact effect is even more reasonable for simplifying the decomposition model (Table II). These calculations can suggest that, for higher molecular-weight hydrocarbons exposed to similar plasma conditions, dissociation could be the dominant reaction. Thermochemically, it can be explained in terms of the nature of the existing bonds. As the molecular weight of a hydrocarbon increases, the number of C–C bonds also increases. The strength of the C–C bonds is less than the strength of a C–H bond (435 kJ/mole for C–H in methane and 368 kJ/mole for C–C in ethane [11]), and it appears understandable, therefore, that, with the existence of weaker bonds, dissociation will be even more expected to occur under these electron-energy conditions. The preferential rupture of C–C bonds to produce radicals in high molecular-weight hydrocarbons has been observed for high-temperature decomposition (pyrolysis) for many years [12], [13].

A decomposition model can therefore be developed considering only dissociation reactions and subsequent chemistry that takes into account only recombination and other series of reactions involving radicals and neutral stable species. Regardless of the specific chemical mechanism proposed for a theoretical prediction of the final species produced in the APPJ, the kinetic model needs initial concentrations of some of the species. These species are the dissociation products coming from the electron-impact reactions, and because the input hydrocarbon concentrations and also the dissociation rate coefficients are known by the

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**TABLE I**

<table>
<thead>
<tr>
<th>E/N (Td)</th>
<th>Vibrational excitation</th>
<th>Dissociation</th>
<th>Dissociative attachment</th>
<th>Ionization</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.26 × 10⁻⁸</td>
<td>1.90 × 10⁻¹³</td>
<td>5.60 × 10⁻¹⁶</td>
<td>4.80 × 10⁻¹³</td>
</tr>
<tr>
<td>2</td>
<td>1.37 × 10⁻⁹</td>
<td>1.90 × 10⁻¹³</td>
<td>1.24 × 10⁻¹⁵</td>
<td>4.80 × 10⁻¹²</td>
</tr>
<tr>
<td>10</td>
<td>5.90 × 10⁻⁸</td>
<td>1.70 × 10⁻¹¹</td>
<td>7.80 × 10⁻¹⁴</td>
<td>4.60 × 10⁻¹⁴</td>
</tr>
<tr>
<td>20</td>
<td>1.08 × 10⁻⁸</td>
<td>4.61 × 10⁻¹⁰</td>
<td>7.44 × 10⁻¹³</td>
<td>1.90 × 10⁻¹¹</td>
</tr>
<tr>
<td>50</td>
<td>1.59 × 10⁻⁸</td>
<td>4.13 × 10⁻⁸</td>
<td>1.94 × 10⁻¹²</td>
<td>6.90 × 10⁻¹⁰</td>
</tr>
</tbody>
</table>

Unit [= cm³/(molecule · s)]

**TABLE II**

<table>
<thead>
<tr>
<th>E/N (Td)</th>
<th>Dissociation</th>
<th>Ionization</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.66 × 10⁻¹¹</td>
<td>9.47 × 10⁻¹⁶</td>
</tr>
<tr>
<td>2</td>
<td>1.67 × 10⁻¹⁰</td>
<td>9.47 × 10⁻¹⁶</td>
</tr>
<tr>
<td>10</td>
<td>3.99 × 10⁻¹⁰</td>
<td>9.47 × 10⁻¹⁴</td>
</tr>
<tr>
<td>20</td>
<td>5.47 × 10⁻¹⁰</td>
<td>1.08 × 10⁻¹³</td>
</tr>
<tr>
<td>50</td>
<td>1.30 × 10⁻⁹</td>
<td>1.30 × 10⁻¹¹</td>
</tr>
</tbody>
</table>

Unit [= cm³/(molecule · s)]
ELENDIF calculations, then the amount of radicals produced can be estimated.

For methane, dissociation is represented as follows:

\[ e + CH_4 \xrightarrow{k_1} \cdot CH_3 + \cdot H + e, \]

where the G-value or the generation efficiency of the chemically active species, as explained before, for CH\(_3\) and H in this case, was calculated to be \(2.36 \times 10^{16}\) molecule/J. The required parameters to perform this calculation are shown in Table III. \(k_{\text{dis}}\) and \(k_{\text{totaldis}}\) are the dissociation rate coefficients for CH\(_4\)/He and C\(_2\)H\(_6\)/He plasma, respectively, calculated with ELENDIF; \(q\) is the electron-charge value, and \(v_d\) is the electron drift velocity (calculated with ELENDIF), both values used in the formula to calculate G-values; E/N is the APPJ reduced electric-field value, and \(\epsilon_{\text{d}}\) is the energy density used to calculate the density of electron-impact-reaction-produced species.

Ethane decomposition at low electron energies can follow two different pathways, depending on the type of bond that is being cleaved

\[ e + C_2H_6 \xrightarrow{k_2} \cdot CH_3 + \cdot CH_3 + e \]

\[ e + C_2H_6 \xrightarrow{k_3} \cdot C_2H_5 + \cdot H + e. \]

The radicals formed after the simple dissociation of C\(_2\)H\(_6\) considered in this paper are \(-CH_3\) for a C–C bond dissociation and \(-CH_2\) and \(-H\) for a C–H bond dissociation. However, the electron-impact cross-sectional data available for ethane dissociation is just the total value, an immediate calculation of each type of radical produced cannot be done. Based on the difference between the bond energies, an estimate of the relative quantity of dissociated bonds can be done, and then, the G-value for each type of radical can be calculated (Table IV). It is assumed that only the relative bond strength defines the bond-dissociation extent and, due to the relatively low electron energy, just one C–H bond can be dissociated per C\(_2\)H\(_6\) molecule.

As stated before, the KINEMA kinetics code was tested for a set of reactions compiled from the NIST Chemical Kinetics Database [14], using as input data their associated Arrhenius parameters and the species concentrations calculated in the plasma region. The reactions included in the model were chosen as aforementioned, considering those which include the species with the higher concentrations (both neutral radicals and molecules) and the higher rate coefficients. The chemical species included in each of the models are presented in Table V. In the first reactive system (CH\(_4\)/He), a total of 14 chemical species are considered, and the species He, -H, -CH\(_3\), and CH\(_4\) are introduced as reaction products. In the second system (C\(_2\)H\(_6\)/He), a total of 15 chemical species (the same ones considered for the first case plus -C\(_2\)H\(_2\)) are considered, and the species He, -H, -CH\(_3\), -C\(_2\)H\(_5\), and C\(_2\)H\(_6\) are introduced as reaction products.

The kinetic model includes the reaction mechanisms resulting in the formation of all hydrocarbons not heavier than propane and the direct recombination of hydrogen radicals into hydrogen molecules. The base gas of the discharge, helium, is included as a third-body collision partner. The presence of a third body is particularly important to satisfy the momentum conservation law when light radicals, such as hydrogen and methyl, are involved in the recombination, because these species do not have bonds, or a sufficient amount of them, for kinetic energy to be easily dissipated.

Calculations were conducted at constant temperature, taking this value from the measured APPJ output-gas temperature, which was the same for the plasma-region calculations, 393 K, and it is assumed to remain constant. Neither a radial or axial temperature profile along the reaction length nor a radial species density profile was considered. Then, using the assumption of a well-mixed system, our method was to stay focused on the main objective: to give a first insight into the chemical pathways followed by the hydrocarbons under atmospheric-pressure and low-temperature plasma conditions.
The complete kinetic mechanism for the CH$_4$/He recombination reactions is compiled in Table VI (where mechanism I corresponds to CH$_4$ recombination reactions, mechanism II corresponds to C$_2$H$_6$ recombination reactions, and $A$, $n$, and $E_a$ are the Arrhenius parameters used to calculate the rate constants). The main results of the kinetic simulation are shown in Fig. 5. In this figure are shown the concentration profiles of the stable product molecules. Overall, the main products produced during the postplasma-species interactions are H$_2$, C$_2$H$_4$, and C$_2$H$_6$. Ethane is predicted as the major product from methane decomposition, and its concentration shows a very rapid increase, more than that for hydrogen and ethylene, species that show a parallel formation. Eventually, hydrogen reaches a concentration close to that of ethane, and the ethylene concentration remains about one order of magnitude below these values.

In Fig. 6, the methane and the ·CH$_3$ and ·H radical concentration evolution profiles resulting from the computation are shown. Because methane initially has the higher concentration relative to helium, it does not show a significant change. Over the computation time, ·H and ·CH$_3$ radicals decrease, giving rise to the recombination products. The rapid decrease in ·CH$_3$ concentration matches the rapid formation of C$_2$H$_6$, suggesting that the direct recombination (assisted by a third body as aforementioned) of these radicals might be one of the dominant reactions.

The final gas composition differs from other previous CH$_4$ RF-driven plasma decomposition works in terms of experiments and calculations. In those previous works, where pure methane is used as an input gas and the pressures are about 10$^{-1}$ torr, sometimes H$_2$ and C$_2$H$_6$ are obtained as major products, as in this paper, followed by ethylene, acetylene, and propane as minor products. However, all species are in different concentration orders of magnitude, depending on the specific conditions. The overall qualitative composition agrees with those previous works, except for the presence of propane as a final product. A strong dependence of the final composition on the pressure, temperature, and the energy density is then suggested [15].

The complete C$_2$H$_6$/He kinetic model is compiled in Table VI. For this case, the calculations show methane, ethylene, propane, and acetylene as the most important products resulting from decomposition. As shown in Fig. 7, the CH$_4$ density increases since the early stages of calculation and stays relatively constant over the whole reaction time. According to the kinetic calculation, ethylene formation also occurs rapidly...
and maintains its concentration for almost all the time displayed. Ethylene and propane show lower concentrations, and their formation also appears to be slower, probably because they come from other species that might need more time to be produced in the chain of reactions. Moreover, according to the proposed mechanism, C₂H₂ formation requires the existence of the radical C₂H₃ in most of the reactions that lead to it, and C₃H₈ requires the previous formation of C₃H₇.

As in the CH₄/He case, in the C₂H₆/He discharge, the feed hydrocarbon ethane is the chemical compound with the next highest concentration relative to helium. Even when it participates in many of the considered reactions, its initial density remains without noticeable change after the recombination reactions occur. The ·CH₃ radical concentration decreases slowly as compared to that of the ·H radical and compared with its quick decrease in the CH₄/He calculation (Fig. 8). The exact reasons for this difference are difficult to explain but it is hypothesized here as a very strong dependence of the final gas composition and the favored reactions on the initial concentration of reactants.

The comparison between the densities of the decomposition products measured by GC and the densities obtained by means of the kinetic calculations are shown as follows. The results correspond to cases of 6.4% CH₄ and 4.9% C₂H₆ as initial hydrocarbon concentrations (Table VII).

The overall numbers show a good qualitative agreement between the experiments and the kinetic calculations. Although, for some of the species, the difference reaches one order of magnitude, the species concentrations show the same trends, and we conclude that the qualitative prediction of the species concentrations can be correctly achieved by means of the model. For the CH₄/He case, the main observed products were ethane and ethylene for experiments as well as for the calculations. For the C₂H₆/He discharge, the experiments and the calculations lead to the production of methane, ethylene, propane, and acetylene as the main species. Molecular hydrogen is also an expected product species because ·H recombination and many other reactions (Table VI) lead to its production. However, because of technical difficulties, the molecular hydrogen concentration could not be measured by GC, neither for the methane discharge nor for the ethane discharge (He is the predominant gas species and, unfortunately, has a thermal conductivity very close to that of H₂, thereby precluding the accurate detection of H₂ by the GC’s TCD (Thermal Conductivity Detector). For the methane case, the calculated hydrogen density had a value of 7.39 × 10¹⁵ cm⁻³, and for the ethane case, this concentration had a value of 5.38 × 10¹³ cm⁻³. Further investigation in terms of the GC method, or some other quantification method, will have to demonstrate the predicted hydrogen production.

Even when an exact match of the species concentrations cannot be accomplished, the species concentrations and their concentration sequence can be very well estimated, and the main purpose of the model is then achieved. The differences shown are reasonable considering that the calculations, as with any other model, have assumptions that define its own limitations, and the estimation of the species concentrations is intended for the unique conditions of low temperature and atmospheric pressure.

### IV. Conclusion

The basic experimental results showed that, under the nonthermal and atmospheric-pressure conditions of the APPJ, the end products arising from parent molecule decomposition in a helium-diluted methane discharge are ethane and ethylene, and for the helium-diluted ethane discharge, the main products are methane, ethylene, propane, and acetylene.

The electron-impact study showed that the main decomposition mechanism of hydrocarbon molecules is based on dissociation reactions that lead to the formation of radicals. The calculations demonstrated that ionization processes for hydrocarbons have much lower rate coefficients, compared to those
for dissociation. Therefore, with the intention of explaining the observed products, ionization reactions can be neglected in understanding the behavior of the hydrocarbons in the APPJ and in explaining the final plasma composition. It was then observed that a purely plasma-based decomposition can be achieved and is differentiated from the thermal effect observed in other works.

Experimentally, it was observed that, with an increase in the plasma power, higher decomposition levels of methane and ethane can be achieved at constant initial concentrations. This increase shows a linear trend for all the products for studied energy-density levels ranging from 335.66 to 629.37 J/L for the CH4/He discharge and from 237.62 to 435.64 J/L for the C2H6/He discharge. This result was explained in terms of the energy available for electrons to cause more dissociation in the parent hydrocarbon and, as a consequence, to achieve higher concentrations of the recombination products.

The comparison between the experimentally measured by-product concentrations and those calculated based on the kinetic model was presented. There was good agreement between these two obtained results in qualitative terms. Both led to the production of the same compounds in the same relative concentrations, and an overall reasonable estimate of the real concentrations was obtained with the kinetic model. The assumptions were seen to be reasonable and allowed the development of a simple and consistent model. For the specifically studied treatment of methane and ethane by the APPJ, a study of the plasma and the postplasma phenomena by separate means were performed at either low pressure or high temperature.

Further basic investigations in terms of hydrocarbon chemistry under nonthermal plasma conditions are necessary to understand the fundamental chemical-decomposition processes important for plasma-assisted combustion, which involves mainly production of radicals and their recombination products, which together have so far demonstrated the known combustion-enhancing effect.

REFERENCES


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Dr. Rosocha has presented in many invited talks and served as a Session Chair, Organizer, and Committee Member for several major international conferences and workshops, including organizing the First International Workshop on PAC in 2003. He has served as a Referee for several journals and was an Associate Editor for nonthermal plasmas for the Journal of Advanced Oxidation Technologies and is currently a Guest Editor for this special issue of the IEEE TRANSACTIONS ON PLASMA SCIENCE on Plasma-Assisted Combustion. He is currently a member of the American Physical Society. He has previously been a member of the International Ozone Association, Sigma Pi Sigma, and Phi Beta Kappa.

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