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Experimental investigation on n-decane plasma cracking in an atmospheric-pressure argon environment

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Abstract

In order to solve the problem of the difficulty of igniting and steadily propagating a continuous rotating detonation engine when using liquid hydrocarbon fuel, an experiment was carried out using a dielectric barrier discharge excited by a nanosecond power supply to crack n-decane, the single alternative fuel to aviation kerosene, in a pre-heated argon environment. By changing the voltages and the discharge frequencies, the concentrations of different components as well as a number of different species were acquired. The generating mechanism of olefins and alkanes together with their competition mechanism were acquired. The influence of the voltage on isomer products was also analyzed. The results demonstrate that the bond energy distribution and the species generating condition are the main factors affecting the formation of the products. With the increasing of voltage and discharge frequency, small molecule olefins, large molecular olefins, large molecular alkanes, small molecular alkanes, and hydrogen were detected, and in turn, their concentrations were also increased except for ethylene; what is more, when the voltage was increased over 8.5 kV, the n-butene converted to trans-butene, and the n-pentene converted to isoamylene.

Keywords: dielectric barrier discharge, plasma cracking, n-decane, isomer

(Some figures may appear in colour only in the online journal)

1. Introduction

With the development of the aviation industry, it has become much more difficult to improve the isobaric combustion efficiency of the conventional turbine aircraft engine [1]. However, due to the higher efficient thermal cycling [2], detonation combustion has become a research hotspot. Compared with the earlier pulse detonation engine, the continuous rotating detonation engine (CRDE) is a gradually developed new concept aircraft engine based on the previous pulse detonation engine. CRDE can maintain continuous work with only one ignition and in a wide range of Mach number as well as produce more stable thrust [3]. So far, the most common fuel used for detonation of a CRDE is gaseous fuel, such as hydrogen, ethylene, methane, etc. But they are so dangerous that it is hard to carried them on an aircraft. Therefore, the detonation of liquid fuel (such as aviation kerosene) is an important step in the engineering application of continuous rotating detonation engine. However, compared with gaseous fuel, liquid fuel is more difficult to detonate. The reason is that the proper working of a CRDE is very sensitive to the test conditions. For example, poor fuel mixing, improper fuel injection rate, low chemical reaction rate, the establishment of a boundary layer as well as the heat

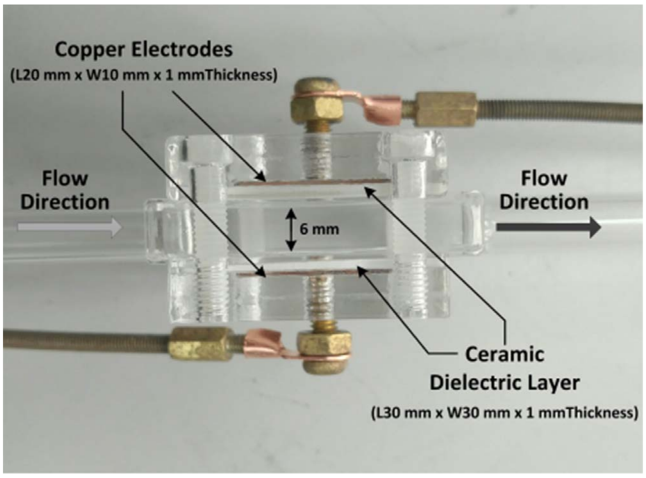


Figure 1. DBD cracking reactor.

conduction of the wall and other factors, all can lead to the quench of the rotating detonation wave [4]. In order to ignite the CRDE fueled with kerosene, the most common method is to add hydrogen [5] or oxygen [6] into the fuel. Another novel way is cracking kerosene large molecule hydrocarbon fuel into more active small molecule fuel, by which the rate of successful ignition can be greatly increased.

The traditional thermal and catalytic cracking of hydrocarbons and their kinetics have been well studied [7–9]. For example, a list of several hundred free-radical reactions which occur during the low temperature (700–850 K) pyrolysis of small n-alkane molecules was assembled and a set of reliable, self-consistent Arrhenius rate parameters was assigned by Allara and Shaw [10]. The criteria for the development of complex and non-regular kinetic schemes (up to thousands of reactions involved) for hydrocarbons pyrolysis reactors have been presented by Dente *et al* [11]. The product distribution and the kinetics of the thermal cracking of kerosene under the conditions that are close to those used in industrial operation have been investigated by Van Camp *et al* [12]. Ghashghaee *et al* [13, 14] used a set of first-principles mathematical models to implement multivariable optimization in olefin production to get about 1.4% increased profitability and have also done a lot work on an efficient two-step thermal upgrading process for converting extra heavy fuel oil to light olefins and fuels. What is more, a rigorous but practical reaction mechanism for an industrial naphtha cracker model was developed by Joo *et al* [15]. Feedstock characterization and prediction of product yields for industrial naphtha crackers on the basis of laboratory and bench-scale pyrolysis have been discussed by Szepesy [16]. However, conventional thermal and catalytic cracking have many disadvantages, such as high cracking temperature, slow temperature rise [17], catalyst failure, and reaction interruption [18], respectively. It is proven that plasma can produce active particles, such as ions, high energy electrons, radicals, and so on, which are capable of breaking chemical bonds in large molecule hydrocarbons and transform them into small hydrocarbons. What is more, using plasma to crack fuel cannot only improve the reaction rate, but can also change the chemical reaction path and decrease the energy consumption [19]. Therefore, using plasma to crack large molecule fuel has been of great interest recently.

Aerodyne Rockedyne DE Inc [4] has developed a new concept, a plasma CRDE. Plasma transient generators excited by a nanosecond pulse power supply were installed in outer and inner cylinders. Through the cracking of kerosene by plasma, the chemical reaction rate and detonation performance are greatly improved. It is also good for the maintenance of the detonation wave. It shows that using plasma to crack large molecule kerosene into more active small molecule fuel is an important way to improve the ignition and wave propagation of the CRDE.

Anikin *et al* [20] experimentally investigated the slow oxidation of n-butane and n-decane in lean mixtures with oxygen under the action of a nanosecond pulsed discharge in a 20 cm long quartz tube. It was found that under the same experimental conditions in lean mixtures with oxygen, the times for the n-decane and n-butane oxidation are equal. Under the background of ignition in a sub-combustion ramjet, Song *et al* [21, 22] researched the mechanism of microsecond pulse discharge cracking n-decane in an argon environment at normal temperature. According to the products, the cracking mechanisms as well as parts of reaction paths were inferred. It was found that specific energy consumption has a selectivity influence on the production of hydrogen.

A typical volume dielectric barrier discharge (DBD) flow cracking reactor, which is excited by a nanosecond pulse power supply, is adopted in this paper. The single surrogate component of aviation kerosene, n-decane [23], was cracked in this experiment. The experiment was conducted at a preheated temperature of 670 K. It is expected to provide a parameter basis and possible mechanism for plasma cracking aviation kerosene, aiming to improve the performance of ignition and the stability of wave propagation.

2. Experimental device and method

The experimental system consists of a gas supply device, fuel pre-evaporation device, DBD cracking reactor, nanosecond pulse power supply, discharge, and voltage collecting device, and gaseous production analysis device (shows in figure 2). The DBD cracking reactor (shown in figure 1) adopts a parallel plate volume DBD. At the center of the reactor, there is a square quartz glass tube with a wall thickness of 1 mm ($8 \times 12 \times 6\ \mathrm{mm}^3$). Two ceramic dielectric layers ($30 \times 30\ \mathrm{mm}^2$) are placed on the outer side of the upper and lower walls of the quartz tube, on the outer side of which, there are two copper electrodes ($20 \times 10\ \mathrm{mm}^2$) placed to press against the dielectric layers. Both of them are tightly fixed by two quartz shells. The copper electrodes are connected to the high-voltage and ground terminals of the nanosecond power supply, respectively. The voltage of the pulse power supply can be adjusted, ranging from 0–12 kV. The frequency of this power supply can also be adjusted from 200 Hz–5 kHz, which is triggered by a DG535 digital delay pulse generator. It can achieve precise control to the frequency. The discharge voltage and current waves are collected by a P6015A high

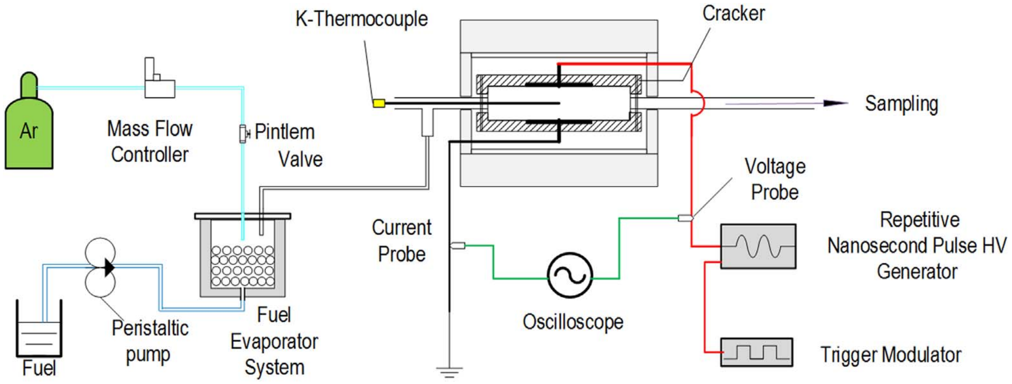


Figure 2. Schematic of the experimental system.

voltage probe and TCP0030A current probe, respectively, and they are recorded by an MIDO3024 oscilloscope. The experiment adopts three voltages, 7.5, 8.5, and 9.5 kV. Under each voltage, there are six experimental cracking frequencies, 1, 1.25, 1.5, 1.75, 2, and 2.25 kHz.

In this experiment, using a peristaltic pump (BT300M) n-decane is pumped into a stainless electric heating pre-evaporator which is full of small stainless balls. The feed flow is controlled at 0.169 ml $\min^{-1}$, the heating temperature of the pre-evaporator is maintained at 200 °C. Argon (with a purity of 99.999%) is blown into the pre-evaporator at the flow rate of 980 sccm controlled by a mass flow controller (DNS-MFC-400A). Argon is used as a carrier gas to blow n-decane into the cracking reactor. The residence time of n-decane vapor in the reactor is 34.56 ms. The DBD cracking reactor is heated by a tube furnace, where the temperature can be maintained at 670 K. (At that temperature, only 230 ppm C_2H_4 is detected, due to the thermal cracking, which plays a minor role in n-decane cracking compared with plasma.) The cracking environment temperature is measured by an axially movable K-type thermocouple. After the fuel vapor is processed by plasma, the cracking production is analyzed by a gas chromatograph (Agilent 7980B). The gas chromatograph adopts a three-channel refinery gas program. After being separated by molecular-sieve capillary columns, C_1–C_5 hydrocarbon compounds will be tested by a flame ionization detector channel and the hydrogen will be specifically detected by a thermal conductivity detector channel.

The power P(W) of the nanosecond discharge injected into a DBD cracking reactor can be obtained by formula (1).

$$P = f\sum_i \left(\frac{V_{i+1}+V_i}{2}\right)\left(\frac{I_{i+1}+I_i}{2}\right)(t_{i+1}-t_i). \quad (1)$$

In the formula, I_i and I_{i+1} represent the current at t_i time and t_{i+1} time. V_i and V_{i+1} represent the voltage at t_i time and t_{i+1} time, respectively. The current as well as the voltage data can be recorded by an oscilloscope. f (kHz) represents pulse frequency.

Energy density $ED(\mathrm{J}\cdot\mathrm{L}^{-1})$ represents the energy injected into the unit volume flow gas, which can be calculated by formula (2).

$$ED = P/F. \quad (2)$$

In the above formula, F represents gas flow, and its unit is $\mathrm{L}\cdot\mathrm{s}^{-1}$.

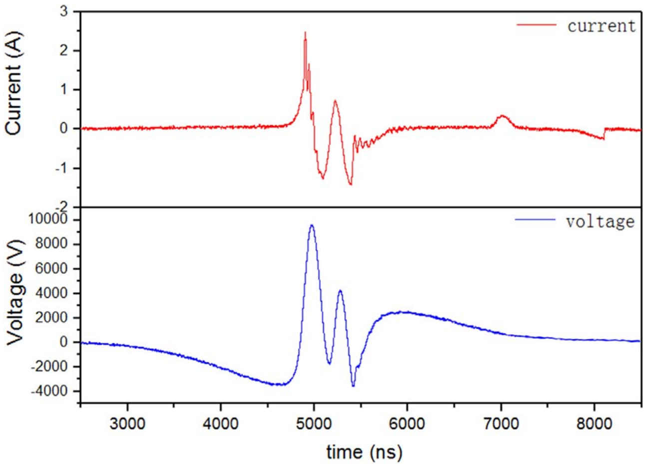


Figure 3. Typical voltage-current waveform of discharge.

3. Experimental results and analysis

3.1. Discharge characteristics

The typical discharge waveform of a nanosecond pulse power supply is shown in figure 3. The highest voltage is 9.5 kV, the maximum current is 2.5 A, and the frequency is 1 kHz. From the figure, we can see that the voltage wave consists of two pulses. Among the two pulses, the peak voltage of first pulse is 9.5 kV, and its rising edge and its falling edge are 200 and 210 ns, respectively. Following the first pulse, the second pulse appears with a significant decrease, of only 4.26 kV. its rising edge and falling edge are 120 and 140 ns, respectively.

Two current pulses appear, along with the appearance of two voltage pulses. The voltage waveform and current waveform are similar to capacitive oscillating discharge waves, because the structure of this flow reactor can be equivalent to a capacitor with a resistor. It can be found that the peak current of the first group is 2.5 A and the second peak current decreases to 0.73 A.

As shown in figure 4, the energy density increases with the rise of the voltage and the frequency. What is more, with the rise of the voltage, the current as well as the energy density rising rate increases. From the figure, it can be found that increasing the load voltage is a more efficient way to

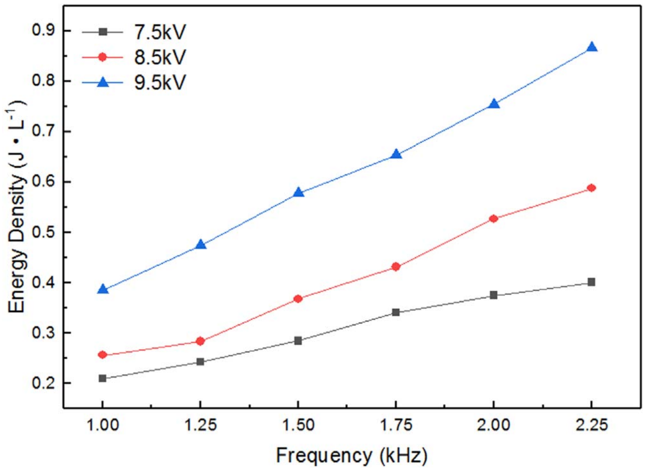


Figure 4. The relationship between injected energy density and current-voltage of discharge.

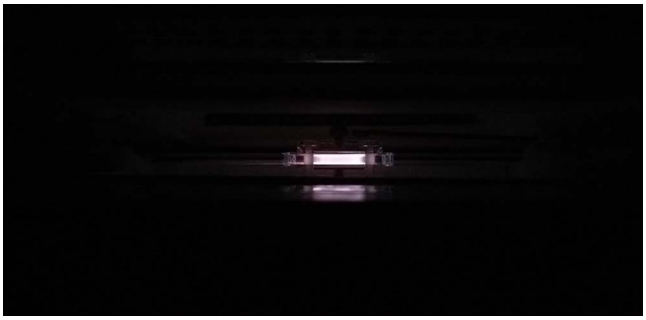


Figure 5. Image of the discharge of n-decane vapor in an argon environment.

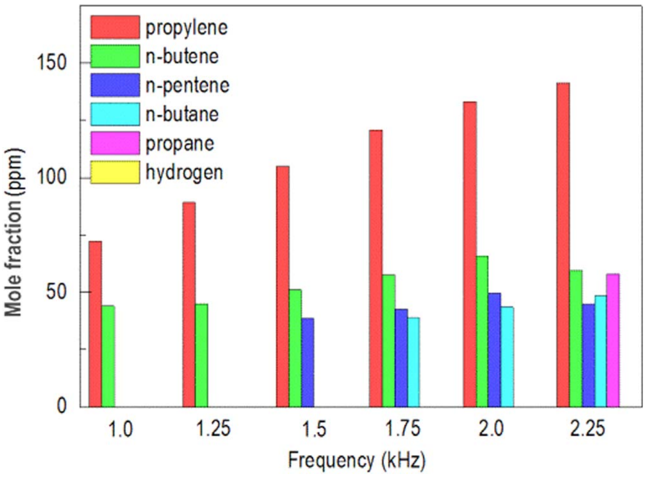


Figure 6. The concentrations of products in different discharge frequencies under the voltage of 7.5 kV.

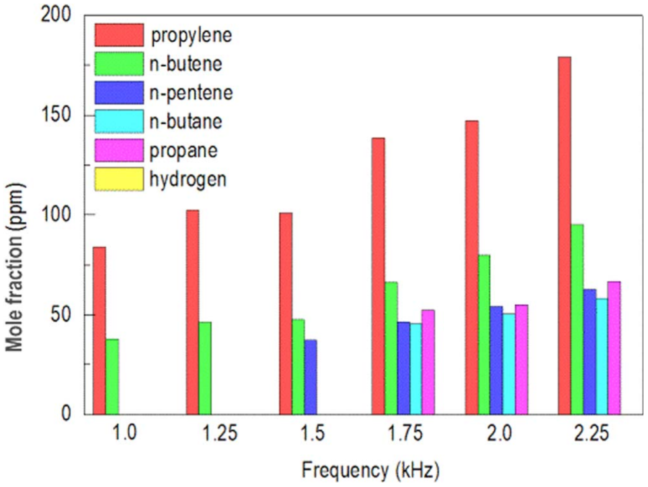


Figure 7. The concentrations of products in different discharge frequencies under the voltage of 8.5 kV.

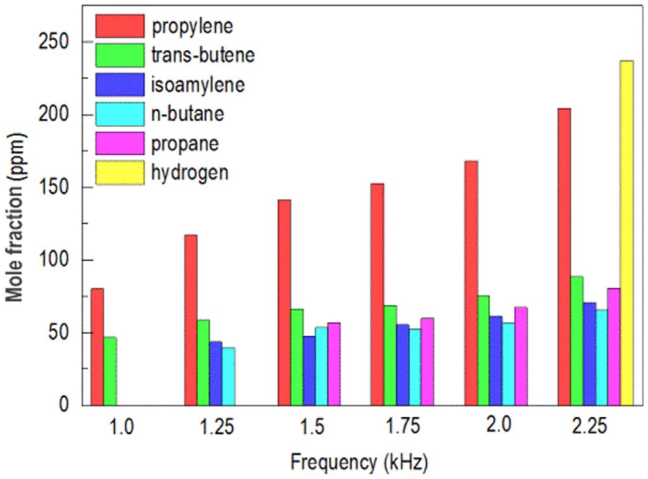


Figure 8. The concentrations of products in different discharge frequencies under the voltage of 9.5 kV.

Table 1. C–C and C–H binding energies in n-decane.

Bond in n-decane	E (kJ · mol^{-1})
C1–C2	343.13
C2–C3	329.22
C3–C4	330.96
C4–C5	331.81
C5–C6	331.65
C1–H1	416.72
C2–H2	399.80
C3–H3	400.45
C4–H4	400.16
C5–H5	400.06

improve the energy density without the limitation of power supply. On the other hand, changing the frequency can achieve accurate control of the injected energy density.

In this reactor, the plasma region (volume is about 1200 mm^3) generated by the DBD discharge is composed of a large number of uniform filamentary pulse discharges. Figure 5 shows the discharge of n-decane in an argon atmosphere. When the load voltage is increased, the electric field strength will increase, and more filament discharge will be generated in the unit space. When the discharge frequency is increased, the number of discharges per unit of time will increase. When the argon flow rate is decreased, the residence

Table 2. Cell size data for gaseous fuels.

	Initial pressure (kPa)	Initial temperature (K)	Equivalence ratio	Cell width (mm)
H_2	100–101.452	293–300	0.9721–1.0233	8.0–15.1
CH_4	101.3	293–298.15	1	279.6–349.5
C_2H_2	101.3	293–298.15	1–1.3242	4.6–9.2
C_2H_4	92.5–101.3	293–298.15	0.999 537–1.070 638	19.5–33.8
C_2H_6	92.5–101.3	293–298	0.993 25–1.0958	50.0–66.1
C_3H_8	92.5–101.3	293–298.15	1–1.2925	46.0–75.2
C_4H_{10}	101.3	293	1.0088	75.1

Table 3. Critical initiation energy data for gaseous fuels.

	Initial pressure (kPa)	Initial temperature (K)	Equivalence ratio	Critical initiation energy (J)
H_2	101.3	293	1–1.190 51	4248.8–9040.0
CH_4	101.3	293	1	88 658 800.0
C_2H_2	101.3	293	1–1.0871	4332.0–6780.0
C_2H_4	101.3	293	0.9866–1.0844	55 596.0–60 568.0
C_2H_6	101.3	293	1.0765–1.157	77 368.8–90 137.8
C_3H_8	101.3	293	0.993 08–1.124	215 192.7–283 114.7
C_4H_{10}	101.3	293	1.0169–1.13	196 362.4–275 824.0

time of the n-decane in the cracking reactor will increase. All of them can raise the energy density injected into the fuel flow, and help accomplish cracking more fully.

3.2. Production analysis

N-decane is cracked at the heating temperature of 670 K, where the gas participating in the reaction is more chemically active, the discharge channel is easier to form, and the cracking reaction is more complete. Figures 6–8 show the cracking production of n-decane except for ethylene. Because the concentration of ethylene is much higher than other species, it is not listed in the comparison. From the figures, we can draw the conclusion that as the discharge voltage and the discharge frequency rise, the concentration of the cracking production as well as the number of the species increases. The reaction mechanism is that when the load voltage is increased, the electric field strength of the discharge gap increases, and the increased breaking bond energy of high-energy electron and the excited metastable state argon atom make the cracking reaction more sufficient. When increasing the discharge frequency, the discharge pulse interval is shortened, and some short-lived intermediate products are further deeply cracked, and the chemical reaction chain is extended [21], resulting in an increase in product concentration and species number. According to the appearance sequence of cracking products as the discharge voltage and discharge frequency increase, the easy-to-difficult rank of cracking products is $C_2H_4 > C_3H_6 > C_4H_8 > C_5H_{10} > C_4H_{10} > C_3H_8 > H_2$.

From the above graphs, we can draw the conclusion that the first cracking products are small molecular olefins, followed by large molecular olefins. According to the bond energy distribution in the n-decane molecule [24] (shows in table 1), it can be known that the β–C–C bond between the second and third carbon atoms has the lowest bond energy. So, at the low discharge voltage and low discharge frequency, high energy electrons and excited metastable argon atoms collide n-decane atoms, resulting in β dissociation of the carbon chain to produce ethyl. Then, under the attack of H atoms and other free radicals, the dehydrogenation of ethyl groups produces small molecule ethylene. With the discharge voltage and the discharge frequency raised, the γ–C–C bond breaks, which has a higher bind energy, producing larger molecular propylene. According to this rule, larger molecular olefins will be produced with the increase of voltage as well as frequency. In a similar way, after the beginning of the cracking reaction, β dissociation and γ dissociation can also happen in free radicals to produce ethylene, propylene, and so on. What is more, the smaller molecule the olefins are, the wider resource and the larger concentration the olefins will have.

After the production of large molecular olefins, large molecular saturated alkanes are gradually detected. Then with the further increase of discharge voltage and frequency, small molecular saturated alkanes generate. Besides, as the resource of small alkanes is wider, the concentration of propane detected is higher than that of n-butane. From the perspective of thermodynamics, the n-decane being cracked to alkanes is an exothermic reaction, while it being cracked to olefins is an endothermic reaction [25]. It is because the cracking reaction is conducted in a 670 K pre-heated environment which is conducive to an endothermic reaction that the experiment prefers to generate small molecular unsaturated olefins. This also confirms the conclusion in the literature that the high temperature is selective for olefins [26], which makes sense for detonation propagation [27, 28]. Therefore, the large molecular alkanes and small molecular alkanes are detected after the olefins. As the discharge becomes stronger, the free H atoms increase. They can attack other free radicals and take

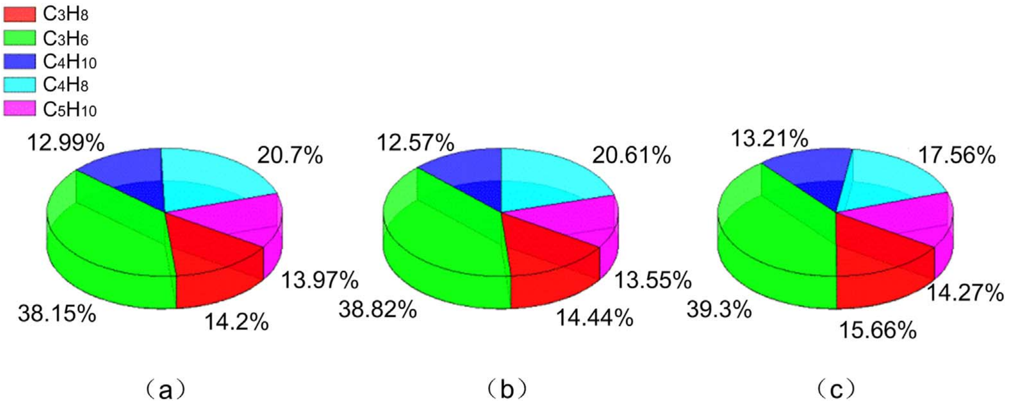


Figure 9. The different percentages of cracking products (except for ethylene) under different cracking conditions. (a) 8.5 kV, 2 kHz; (b) 8.5 kV, 2.25 kHz; (c) 9.5 kV, 2 kHz.

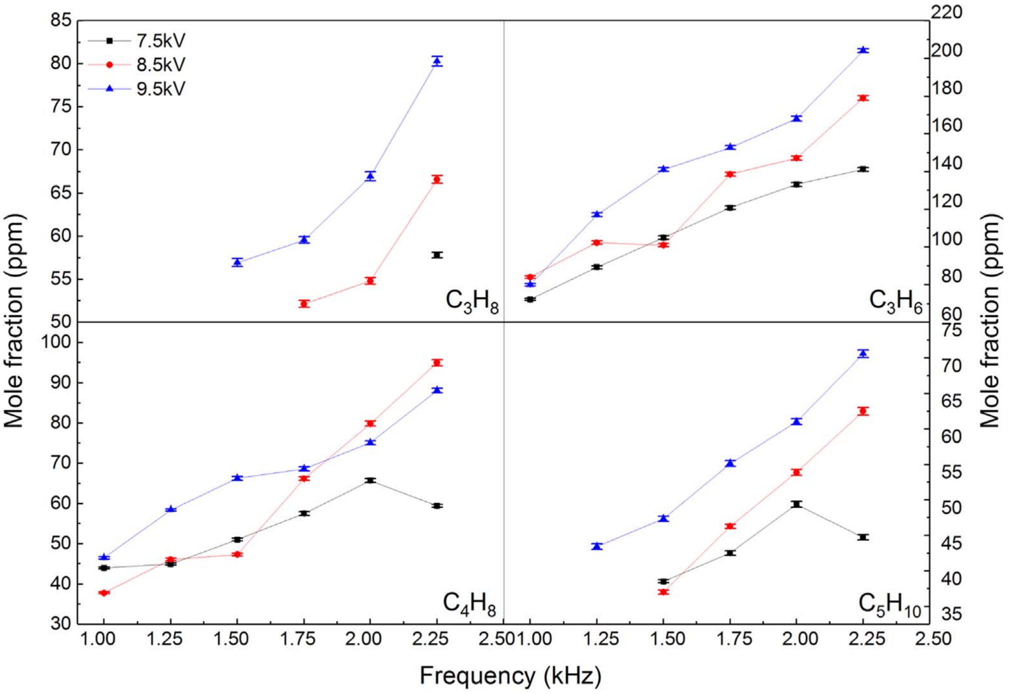


Figure 10. The production of C_3H_6, C_4H_8, C_5H_{10}, and C_3H_8 under different experimental conditions.

hydrogen abstraction. Finally, the H can be released as hydrogen which is greatly sensitive for a detonation wave.

It is known that a rotating detonation wave is very sensitive to the gaseous fuel. Different fuel corresponds to different cell sizes of detonation wave as well as different initiation energy [29] (shown in tables 2 and 3). From figure 9, it can be found that, not only the species number and the products concentration are affected by the discharge voltages and frequencies, but the percentage of different products varies under different conditions (discharge voltages and frequencies). From this point of view, plasma can regulate the different percentage of cracking products, fueled by which a rotating detonation engine will have different performance (such as different wave speed, pressure, successful rates of ignition) [30].

As shown in figure 10, according to the results of the analysis of n-decane cracking products by gas chromatography, it can be found that the concentration of gaseous products rises with the increased discharge voltage and frequency, except for ethylene. While as shown in figure 11, at a certain voltage, the concentration of ethylene rises with the increase of discharge frequency in the initial stage. When the concentration peaks, it gradually decreases with the frequency increases. What is more, when the cracking frequency is low, the concentration of ethylene increases as the voltage rises. When the frequency is high, the concentration of ethylene decreases as the voltage increases. Meanwhile, it is found that the change in the concentration of the ethylene has a significant relationship with the appearance of butane. When butane is detected in the product, ethylene production reaches its maximum, and as the production of butane increases, the ethylene production begins to decrease. The reason is that the formation of ethylene is mainly affected by the β cracking of alkyl radicals and the rearrangement of alkyl radicals at position one (1-alkyl) [31]. When

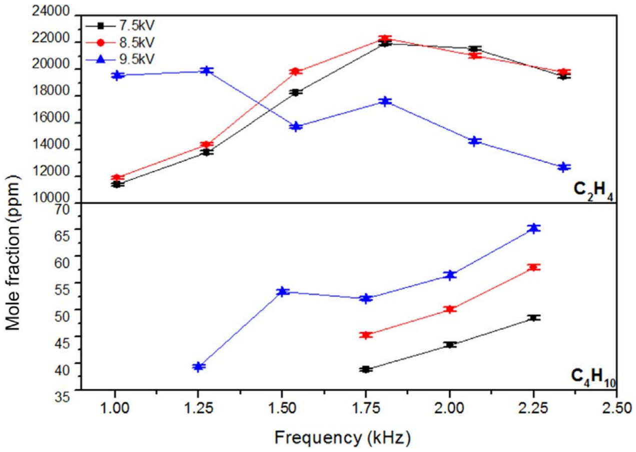


Figure 11. The production of C_2H_4 and C_4H_{10} under different experimental conditions.

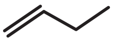
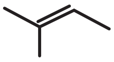
the cracking frequency is low, the former reaction plays the leading role, resulting in the increase of the ethylene concentration as the voltage rises. While when the cracking frequency is high, the latter reaction significantly affects the ethylene production, leading to the decrease of ethylene concentration. Meanwhile, C_1–C_3 alkanes are often formed by the hydrogen abstraction reaction of C_1–C_3 radicals. Alkanes above C_4 are often generated by a combined reaction of free radicals [32]. When the butane begins to appear in the products, it will consume double C_2 radicals. With the concentration of butane increasing, it competitively consumes the radicals which should be used to produce ethylene. When the rate of free radical consumption is greater than the rate of ethylene formation, it will lead to the decrease of the ethylene concentration.

According to the analysis based on the cracking species, it is found that there isomers appear under different voltage strengths. When the load voltage is below 8.5 kV, it is observed from the chromatography that the structure of C_4H_8 produced by plasma cracking is all n-butene (the first row in table 4), and the structure of C_5H_{10} is all n-pentene (the third row in table 4). When the load voltage is over 8.5 kV, the C=C double carbon bond of butane occurs as the the positional isomerization. Most of the butane products detected are trans-butene. As for pentene, a skeletal dissimilation reaction occurs, and the linear olefins convert to branched olefins. All the pentene products detected are 2-methyl-2-butene (isoamylene). This shows that raising the load voltage is more conducive to generate trans-butene and isoamylene for C_4 and C_5 products. The load voltage has a selective effect on the isomers of some products. This confirms that plasma may have great advantages compared with the traditional catalyst in isomer conversion.

According to the above analysis of detected product species and their concentration variation, parts of the possible reaction pathways in n-decane-Ar plasma are demonstrated as follows.

As figure 12 shows, the beginning decomposition of n-decane consists of three main pathways. Since the β–C–C bond has lower bond dissociation energy compared with γ–C–C bond and α–C–C bond, pathways ② and ③ only play minor roles in the decomposition of n-decane. In products, alkenes are mainly produced from the β–C–C scission reactions of alkyl, which are also the dominant decomposition pathways of alkyl radicals [33]. It can also be found that alkanes above C_4 are often generated by the combination reaction of free alkyl radicals. C_1–C_3 alkanes are often formed by the hydrogen abstraction reaction of C_1–C_3 radicals. Generating alkanes will competitively consume alkyl radicals which could be used to produce alkenes. Besides, the heating environment is conducive to the production of alkenes. What is more, the β–C–H scission of alkanes could produce 2-alkyl, which will lead to the C=C bond position isomerism (for example, n-butene and trans-butene). The combination reaction between CH_3 and 2-alkyl will lead to C–C bone isomerism (for example, n-pentene and 2-methyl-2-butene). The H-abstraction reaction by H atom will lead to the formation of hydrogen [34].

Table 4. Structural formula of isomers in cracking productions.

Cracking product	Structural formula
N-butene	
Trans-butene	
N-pentene	
2-methyl-2-butene	

4. Conclusions

In conclusion, with the increase of discharge voltage and discharge frequency, on the one hand, the concentration of gaseous cracking products increases, except for ethylene. On the other hand, the species of the products increase, too. According to the differences in the distribution of n-decane bond energy and the differences in product formation conditions, it is found that plasma cracking hydrocarbon compounds in a preheated environment are more conducive to the formation of small molecular olefins. The formation of ethylene is mainly affected by the β cracking of alkyl radicals and the rearrangement of alkyl radicals at position one. When the cracking frequency is low, the former reaction plays the leading role, resulting in the increase of the ethylene concentration as the voltage rises. While when the cracking frequency is high, the latter reaction significantly affects the ethylene production, leading to the decrease of ethylene concentration. What is more, C_1–C_3 alkanes are often formed by the hydrogen abstraction reaction of C_1–C_3 radicals. Alkanes above C_4 are often generated by a combined reaction of free radicals. The increase of butane will competitively consume the C_2 radicals, leading to the decrease of ethylene. With the increase of load voltage, cracking products will become isomers conversion. For example, the cracking

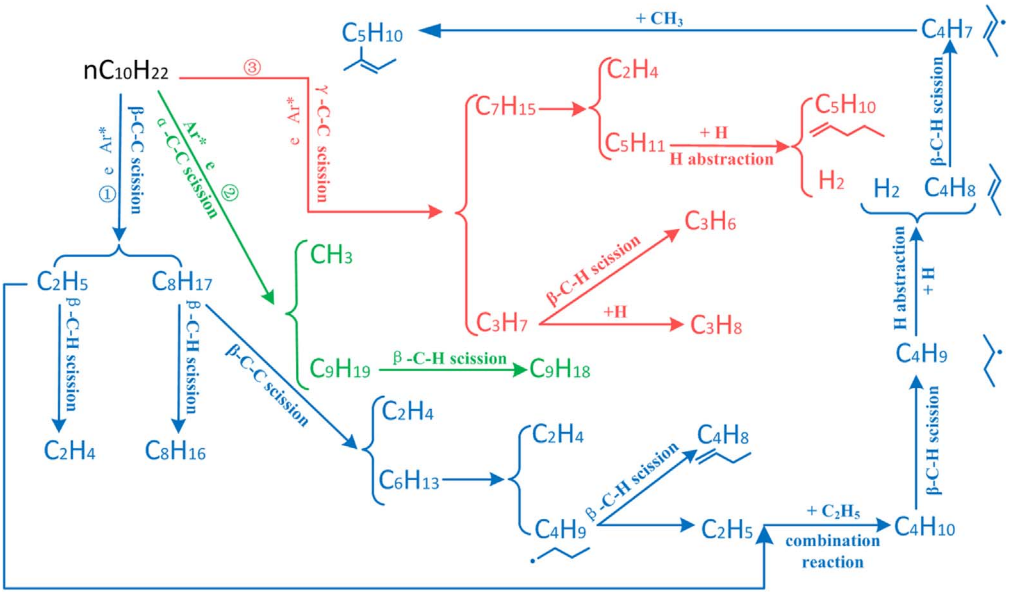


Figure 12. Reaction pathways in n-decane-Ar plasma.

products n-butene and n-pentene in low voltage will become trans-butene and 2-methyl-2-butene respectively in high voltage respectively.

According to the influence of discharge voltage and discharge frequency on DBD cracking products, it is believed that better control and formulation of the proportion of active gaseous products according to demand is greatly helpful to realize the successful initiation of liquid hydrocarbon fuel for CRDE.

Acknowledgments

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