

A simplified kinetic model for isothermal catalytic ignition

Propane/air mixture on platinum wire

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Abstract The ignition of catalytic combustion of the stoichiometric propane/air mixture on an isothermally heated platinum wire in different experimental conditions of total pressure and wire temperature is studied and discussed on the basis of a simplified kinetic model. The platinum wire is heated electrically with a specially designed power supply, which ensures a quasi-rectangular profile of its temperature. The ignition process is monitored by measuring the input power required to maintain a constant temperature of the wire during an exothermic catalytic reaction. The difference between the input powers recorded in air and in a fuel/air mixture, for the same wire temperature and gas total pressure, allows the elimination of the heat transferred to surroundings and conversion of the results into the catalytic reaction rate r_R versus time curves of S-shaped form, illustrating the transition from kinetic to diffusion regime. The curve can be used to evaluate the ignition delay, as reported previously and also to fit different models to the data. The quasi-exponential increase of the isothermal reaction rate during the early stages of the process can be rationalized on the basis of a simplified kinetic model implying the multiplication of the adsorbed active intermediates. The adopted hypotheses allow the derivation of an analytical solution for the catalytic reaction rate before and during the ignition process, without diffusion limitations.

Keywords Isothermal catalytic ignition · Propane/air mixture · Platinum wire · Multiplication of surface active intermediates

Introduction

The ignition and subsequent combustion of a fuel/air mixture by hot solid surfaces, either inert or catalytic, is a subject of great interest from both practical and theoretical points of view [1]. Several important fields have been thoroughly explored: the assessment of explosion risks in flammable gaseous mixtures [2–4], the catalytic combustion of fuel/air mixtures for chemical energy conversion into heat or work with lower pollutants emissions [5–7], complete catalytic oxidation of volatile organic compounds [8–11] and emission control of automotive exhaust gases in catalytic converters [12] etc. At lower temperatures, the presence of a solid catalyst in a fuel/air mixture triggers a surface reaction which can subsequently become stationary [13–15]. At higher temperatures, the surface active species can diffuse into the adjacent gas phase resulting in a heterogeneous–homogeneous combustion followed, under specific circumstances, by the ignition of the homogeneous gas phase combustion that occurs at much higher temperatures [13, 16–18]. Since the combustion reaction is highly exothermic, the ignition is associated with a rapid temperature increase which gives rise to a self-acceleration of the process (positive feedback). Consequently, a large number of thermal runaway models for ignition have been elaborated and discussed offering the theoretical basis for many practical problems. Within these models, the ignition is considered to be a sudden transition from kinetic to mass transfer control of the overall process [13, 19] owing to the exponential increase of the reaction rate with temperature.

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If the ignition occurs in isothermal conditions, with a constant temperature gradient between the solid catalyst and the surrounding gas, the existence of a self-acceleration process cannot be attributed to the reaction exothermicity; only the multiplication (branching) of the active intermediates on the catalyst surface can account for the self-acceleration of the process and consequently for ignition. The rapid heat release during a catalytic combustion reaction generally impedes the attainment of an isothermal regime. However, when the working temperature of the catalyst is obtained through Joule heating from an external power supply, the evolved reaction heat can be compensated by a corresponding decrease of the input power, thus maintaining a constant temperature. The associated experimental technique is based on a step-wise perturbation of the platinum wire temperature followed by an electronically regulated isothermal regime. The analysis of the feeding current during this constant temperature regime allows the measurement of the reaction rates during the entire range of the process. Several results have already been published regarding the catalytic ignition and combustion of alkane/air mixtures using isothermally heated platinum wires [20–25]. In these conditions, the variation in time of the catalytic reaction rate has an S-shaped form, similar to the well-known conversion versus temperature variation (light-off curve) [26]. The recorded curves for the stoichiometric propane/air mixture have been used to measure the ignition delays and the steady-state reaction rates and to discuss their variation with pressure and temperature [25]. In this article, a new set of experiments has been carried out to determine the reaction rates during the early stages of the catalytic ignition, at different pressures and temperatures, for the stoichiometric propane/air mixture occurring in isothermal conditions on platinum wire. The results are rationalized on the basis of a simple kinetic model implying the multiplication of the surface active intermediates and resulting in a simple analytical solution for the overall reaction rate, similar to the equation describing the isothermal branched chain explosion in homogeneous systems [27]. The physical significance and the variation with the wire temperature of the involved parameters are discussed.

Experimental method

The basic diagram of the electronic circuit, designed to obtain a quasi-rectangular temperature profile of a platinum wire, was described in several previous articles [20–25, 28]. Practically, it consists in a Wheatstone bridge. A platinum wire (99.99% from Aldrich), 0.1 mm in diameter and 4.5 cm long, having the resistance R_w , is an arm of this bridge. It is connected in series with a standard resistor of known

resistance R_{st} . A potentiometer forms the other two arms of the bridge which is fed from a dc power supply through a series transistor. The feeding voltage is divided between the bridge and transistor. When the bridge is equilibrated, the temperature dependent R_w is established by R_{st} and the ratio of the two arms of potentiometer. Any unbalance of the bridge, induced by certain endothermic or exothermic processes occurring on the platinum wire, is detected by an integrated circuit which readjusts the voltage across the bridge through the series transistor, maintaining a constant R_w and consequently its prescribed temperature. A capacitor, charged at an adequate voltage from an additional dc source, is discharged at the beginning of the heating process to obtain a quasi-rectangular temperature profile of the wire with a rise time of about 2 ms. The variation of the feeding current through the wire can be followed by measuring the voltage drop U_{st} across the standard resistor using a Tektronix TDS 210 oscilloscope for digital recording of transient processes. The platinum wire is assembled in the centre of a cylindrical test cell of 9 cm diameter and height. The test cell is evacuated and filled with air or with gaseous fuel/air mixture of known composition at pressures between 20 and 100 kPa. The gaseous mixture containing 4.02% propane in air was prepared in a stainless steel cylinder at maximum 500 kPa total pressure by partial pressure method and used 24 h after mixing.

The relevant input power on R_w is given by:

$$P_w = R_w \cdot U_{st}^2 / R_{st}^2 \quad (1)$$

It can be calculated from the known R_{st} and measured R_w and U_{st} in air or in a fuel/air mixture. If an exothermic reaction occurs on the wire when this is immersed in a fuel/air mixture, the corresponding input power at the same temperature and total pressure is lower than in air. The difference represents, to a very good approximation, the reaction heat flow rate, dQ_r/dt :

$$dQ_r/dt = (R_w/R_{st}^2) \cdot [(U_{st}^2)_{air} - (U_{st}^2)_{mixture}] \quad (2)$$

The first term in the right hand side represents the heat transferred to the feeding and measuring conductors and to the surrounding gas through convection, conduction and radiation in air. The second term represents the same property measured in the fuel/air mixture. Since the fuel concentration is very low, it is assumed that the transferred heat is the same in air and in the fuel/air mixture for the same wire temperature and total gas pressure. The reaction heat flow rate dQ_r/dt is related to the catalytic reaction rate r_R through:

$$r_R = (dQ_r/dt) / (\Delta^c H_T^0 \cdot S_w) \quad (3)$$

where $\Delta^c H_T^0$ is the standard heat of combustion and S_w the wire surface. Other details on the experimental setup and procedures can be found in the foregoing references.

Results and discussion

A typical result of the voltage drop U_{st} versus time recording in air and in the 4.02% propane/air mixture is illustrated in Fig. 1. In the same figure, the reaction rate variation in time of the surface catalytic reaction calculated according to (2) and (3) is given.

The sudden transition of the reaction rate from lower to higher values marks the ignition process, followed by steady-state combustion. This transition is characterized by an ignition period τ_i , dependent on the mixture composition, total pressure and wire temperature. A critical ignition temperature T_i delimitates the non-ignitable from ignitable systems. At lower temperatures ($T < T_i$), even if small exothermic effects are detected, the ignition (transition) does not occur. At higher temperatures ($T \gg T_i$), the induction periods are of the same order of magnitude with the rise (≈ 2 ms) time of the quasi-rectangular temperature profile of the platinum wire and cannot be measured using this technique. Consequently, the measurements have been carried out within the temperature range 479–555 K.

The S-shaped form of the reaction rate variation in time illustrated in Fig. 1 is common for all C_2 – C_5 studied alkanes [20–25] and suggests a self-acceleration process subsequently limited by the mass transfer, occurring in isothermal conditions.

The results obtained from our present work for the stoichiometric propane/air mixture will be discussed on the basis of a simple model, implying a multiplication of the surface active intermediates. The model is based on several simplifying hypotheses regarding the reactant diffusion, adsorption and surface reaction. According to literature data, the oxygen is dissociatively adsorbed on the

platinum surface even at normal temperature [29]. At working temperatures ($T_w \geq 479$ K), it can be assumed that the platinum surface is preponderantly covered with adsorbed oxygen atoms, with fractional coverage θ_{ox} . The combustion is initiated by the chemisorption of alkane which is also considered a dissociative process in many pertinent works [30, 31]. If the fractional coverage of each reaction product is negligible, the fraction of vacant sites available for propane is $\theta_0 = (1 - \theta_{ox})$. The comparison of the reaction rates at $t = \tau_i$ (approximately $1 \times 10^{-2} \text{ mol m}^{-2} \text{ s}^{-1}$ for highest temperatures, as can be checked from the data given later in Table 1) with lowest fuel collision rates during the ignition period (approximately $20 \text{ mol m}^{-2} \text{ s}^{-1}$ calculated as $p_F/(2\pi M_F RT)^{1/2}$, where p_F and M_F are the fuel partial pressure and molar mass, respectively) indicates that the overall process is not diffusion limited. Consequently, the reactant concentrations within the diffusion layer are the same with the bulk values. To compare the surface concentrations $C_{S,j}$ with the initial volume concentrations $C_{V,j}^0$ within the adsorption

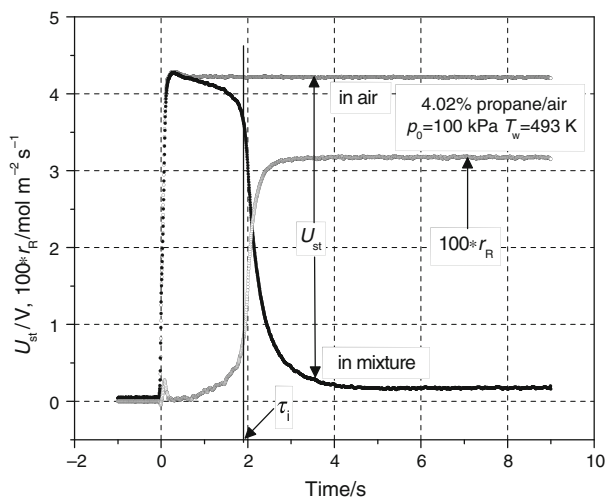


Fig. 1 Variation in time of U_{st} and of the reaction rate r_R for the stoichiometric propane/air mixture ($R_{st} = 5 \Omega$, $\Delta^c H_T^0 = 2043.15 \text{ kJ/mol}$, $S_w = 1.41 \times 10^{-5} \text{ m}^2$)

Table 1 Ignition periods and fitting parameters of Eq. 12 at various pressures and temperatures

p_0/kPa	T_w/K	τ_i/s	$1000\cdot a/\text{mol m}^{-2} \text{ s}^{-1}$	$100\cdot b/\text{mol m}^{-2} \text{ s}^{-1}$	c/s^{-1}
101	479	4.85	0.387	3.22E-06	2.49
101	493	1.94	0.754	2.70E-04	4.22
101	506	1.09	0.601	3.71E-03	5.31
101	521	0.69	0.801	6.61E-03	7.20
101	538	0.36	0.655	5.13E-02	8.62
101	551	0.21	0.821	15.3E-02	9.99
80	494	2.06	0.458	0.426E-04	4.61
80	508	1.15	0.117	3.77E-03	4.80
80	534	0.37	0.133	7.43E-02	7.26
80	554	0.20	0.102	15.4E-02	9.92
60	496	2.0	0.436	0.608E-04	4.69
60	506	1.1	0.243	0.497E-02	4.55
60	524	0.65	-0.188	3.63E-02	5.29
60	536	0.34	-0.283	10.8E-02	6.81
60	551	0.18	-0.641	19.4E-02	9.11
40	494	1.8	0.739	2.99E-04	4.17
40	509	1.05	0.367	6.96E-03	4.38
40	520	0.60	0.687	4.18E-02	5.10
40	539	0.32	0.199	11.8E-02	6.15
40	553	0.15	1.39	20.4E-02	8.66
20	494	0.98	3.35	1.26E-03	5.61
20	507	0.82	0.912	9.96E-03	4.03
20	523	0.39	4.62	7.15E-02	5.61
20	543	0.20	4.84	0.278	5.58
20	555	0.14	4.00	0.493	6.19

layer, having different dimensions, it is useful to introduce the thickness of the adsorption layer, ϕ [20], assumed to be the same for all adsorbed species. Consequently, one can approximate: $C_{S,F} \approx C_{V,F}^0 \cdot \phi$, $C_{S,O_2} \approx C_{V,O_2}^0 \cdot \phi$.

The kinetic model takes into account the following steps:

- oxygen adsorption–desorption



- fuel adsorption (k_a^*)



- intermediate multiplication (k_m^*)



- overall surface reaction (k_r^*)



where \otimes is a free catalytic site, O is an adsorbed oxygen atom and X is an adsorbed reactive intermediate. To obtain an analytical form for the overall reaction rate, a major simplification was adopted: all the intermediates have similar reactivity and consequently lose their identities and can be described by the same common symbol X. The agreement between the model predictions and the experimental results might confirm to which extent this simplification is acceptable or not. Step (4) is considered quasi-equilibrated. Step (5) gives two active intermediates assumed to have similar reactivity (initially, one is the adsorbed hydrogen and the other is an adsorbed alkyl radical). In the step (6), representing a multiplication process, each active intermediate reacts with the excess adsorbed oxygen to generate two active intermediates of similar reactivity. The intermediate multiplication is considered an elementary step and its rate is expressed accordingly [32]. In the step (7), the active intermediate reacts with the adsorbed oxygen to form the reaction products. To account for the conservation of the free catalytic sites and to obtain the overall stoichiometric equation ($C_3H_8 + 5O_2 = \text{Products}$), the steps 4, 5, 6 and 7 should be multiplied with 5, 1, 4 and 6, respectively. The quasi-equilibrated step (4) generates a quasi-constant concentration of adsorbed atomic oxygen, O. Its surface concentration can be calculated from $C_{S,O} = K_{ad,O_2}^{1/2} \cdot (C_{V,O_2}^0)^{1/2} \cdot \theta_0$. Since for excess adsorbed oxygen θ_0 is given approximately by $\theta_0 \approx 1 / (1 + K_{ad,O_2}^{1/2} \cdot C_{V,O_2}^{1/2})$ [33] the adsorbed atomic oxygen concentration can be expressed simpler by a Freundlich-type relationship $C_{S,O} = \beta \cdot (C_{V,O_2}^0)^m$ [33], with m and β being empirical

constants. The total variation of the surface active intermediate concentration is consequently given by:

$$\begin{aligned} dC_{S,X}/dt &= 2 \cdot k_a^* \cdot \theta_0^2 \cdot C_{V,F}^0 \cdot \phi + (k_m^* - k_r^*) \cdot \beta \\ &\quad \cdot (C_{V,O_2}^0)^m \cdot C_{S,X} \\ &= k_a \cdot C_{V,F}^0 \cdot \phi + (k_m - k_r) \cdot C_{S,X} \end{aligned} \quad (8)$$

where k_a includes $2 \cdot \theta_0^2$ while k_m and k_r include $\beta \cdot (C_{V,O_2}^0)^m$.

Integration of this equation from $C_{S,X} = 0$ at $t = 0$, to $C_{S,X}$ at time t gives:

$$C_{S,X} = [k_a \cdot C_{V,F}^0 \cdot \phi / (k_m - k_r)] \cdot (\exp((k_m - k_r) \cdot t) - 1). \quad (9)$$

The reaction rate is given by:

$$\begin{aligned} r_R &= k_r \cdot C_{S,X} \\ &= [k_a \cdot k_r \cdot C_{V,F}^0 \cdot \phi / (k_m - k_r)] \\ &\quad \cdot (\exp((k_m - k_r) \cdot t) - 1). \end{aligned} \quad (10)$$

The working relationship will be used in the form:

$$r_R = b \cdot (\exp(c \cdot t) - 1) \quad (11)$$

where the parameters b and c have obvious significances. To account for the unavoidable shifts of experimental data, an adjustable correction term, a , of the reaction rate should be additionally introduced in Eq. 11, resulting:

$$r_R = a + b \cdot (\exp(c \cdot t) - 1). \quad (12)$$

The exponential form of the obtained equation is similar to the equation describing the isothermal branched chain explosion in homogeneous systems [27] with the difference that the rate of fuel adsorption is used instead the rate of chain generation and the rate of surface reaction is used instead the rate for the chain termination. It is a useful tool associated with small-scale tests [34] for predicting the hazards of combustible/air mixtures.

This equation describes the variation in time of the catalytic reaction rate from the beginning of the process up to the inflexion point where the mass transfer limitations become significant. Equation 12 can be fitted on the experimental data to check the validity of the model. An example is illustrated in Fig. 2, for the data given in Fig. 1.

Taking into account the simplifications adopted for the proposed model, the fit can be considered satisfactory.

Similar measurements have been performed for various total pressures and wire temperatures. The results are given in Table 1.

The induction period, τ_i , has been previously discussed and its temperature and pressure dependencies reported on the basis of the single step reaction approximation [24]:

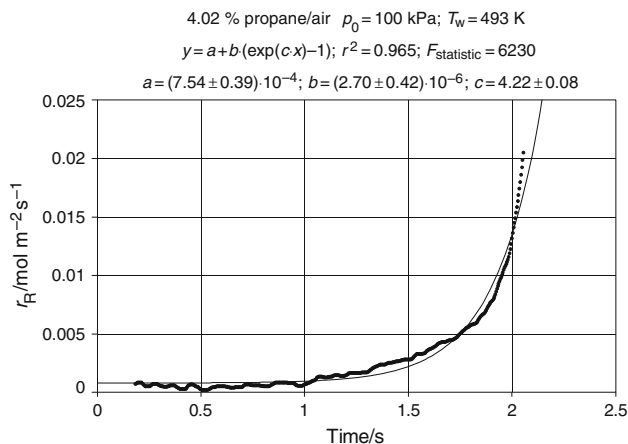


Fig. 2 Fitting of Eq. 12 on the r_R versus time data for the stoichiometric propane/air mixture

$$\tau_i = A \cdot (p_0/p_{\text{std}})^{-n} \cdot \exp(E_a/RT_w) \tag{13}$$

where A is a proportionality constant, p_{std} the standard pressure (101 kPa), n the overall reaction order, R the universal gas constant and E_a the overall activation energy. The regression analysis, $\ln(\tau_i)$ against $1/T_w$, of the data in Table 1 resulted in the activation energies provided in Table 2.

The figures are in fair agreement with the data previously reported [25]: 99.9, 92.6, 58.9 and 56.3 kJ/mol, at 100, 70, 50, and 30 kPa, respectively. The quality of the fit is indicated by the determination coefficient r^2 and $F_{\text{statistic}}$. The error limits of the parameters are given for a 95% confidence level for all reported data. The dispersion of the parameters must be associated with the well-known difficulties in maintaining a reproducible surface structure of platinum during the high temperature combustion reactions. According to the present proposed model, the temperature dependence of the ignition period resulted from Eq. 11:

$$\tau_i = (1/c) \cdot \ln(1 + r_{R,\tau}/b) \tag{14}$$

where $r_{R,\tau}$ is the reaction rate corresponding to the ignition period, is more complex than indicated by the single step reaction approximation, due to the temperature variation of all involved parameters.

The temperature dependencies of parameters b , c , $(b \cdot c)$ and $r_{R,0.1}$ (the reaction rate at the same time $t = 0.1$ s), are also given in Table 2. All of them exhibit an Arrhenius type variation with temperature. The significance of the resulted activation energies for b and c parameters is quite complex and highly dependent on the model structure and the lumping procedure. It can be seen that the product $(b \cdot c)$ has a simpler significance than b :

$$\begin{aligned} b \cdot c &= k_a \cdot k_r \cdot C_{V,F}^0 \cdot \phi \\ &= 2 \cdot \theta_0^2 \cdot k_a^* \cdot k_r^* \cdot \beta \cdot (C_{V,O_2}^0)^m \cdot C_{V,F}^0 \cdot \phi. \end{aligned} \tag{15}$$

Table 2 Temperature dependence of the kinetic parameters from the regression $\ln(y)$ against $1/T_w$

y	p_0/kPa	$E_a/\text{kJ mol}^{-1}$	r^2	$F_{\text{statistic}}$
τ_i	100	91.3 ± 4.3	0.991	458
	80	89.9 ± 3.1	0.998	862
	60	96.3 ± 5.7	0.989	281
	40	94.1 ± 5.3	0.991	321
	20	76.8 ± 5.7	0.984	179
b	100	307 ± 41	0.935	57
	80	299 ± 74	0.891	16
	60	308 ± 72	0.858	18
	40	249 ± 49	0.894	25
	20	221 ± 23	0.968	90
c	100	40.0 ± 4.4	0.955	85
	80	30.3 ± 4.3	0.961	50
	60	28.0 ± 6.1	0.877	21
	40	27.0 ± 5.3	0.893	25
	$b \cdot c$	100	344 ± 46	0.933
80		329 ± 70	0.918	22
60		336 ± 70	0.894	25
40		276 ± 45	0.926	38
20		218 ± 26	0.960	72
$r_{R,0.1}$	100	55 ± 12	0.838	21

Its temperature dependence is given essentially by $\theta_0^2 \cdot k_a^* \cdot k_r^*$ indicating that the associated activation energy is also a composite parameter.

The activation energy evaluated from the regression $\ln(r_{R,0.1})$ versus $1/T_w$, at a time lower than the shortest ignition period, when for all temperatures the reaction is controlled kinetically, is also given for comparison. It indicates that the self-acceleration mechanism through active centres multiplication is additionally enhanced by the self-heating in the non-isothermal systems [35].

The proposed model differs essentially from other models reported in literature [36, 37] through the intermediate multiplication steps which are implicit in the detailed kinetic treatment of non-isothermal catalytic combustion of simpler fuels [30, 31]. The results presented here certify the existence of the isothermal branched chain explosion for a heterogeneous catalytic combustion reaction, easier to achieve than a similar homogeneous combustion reaction, where the temperature control is more difficult.

Conclusions

One can conclude that the multiplication of the active centres on the catalytic surface plays a significant role

during the induction period. For isothermal conditions, when the thermal acceleration of the reaction rate is absent, the multiplication (branching) of the adsorbed active intermediates represents the only positive feed-back which can explain the catalytic ignition considered as a sudden transition from a kinetic to a diffusion-controlled process. Even if this role was found for isothermal conditions according to a very simple model, it is likely to be also present during the non-isothermal catalytic ignition. In these cases, when many chemical species and chemical reactions are implied, only numerical solutions are possible, even for simpler systems like H_2 /air or CH_4 /air catalytic combustion.

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