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Pyrolysis of Supercritical Endothermic Fuel: Evaluation for Active Cooling Instrumentation.

Nicolas GASCOIN^{a*}, Philippe GILLARD^a, Stéphane BERNARD^a, Emeric DANIAU^b, Marc BOUCHEZ^b

^aLaboratoire Energétique, Explosion, Structure
63, avenue de Lattre de Tassigny - 18020 Bourges Cedex

^bMBDA France
8 rue Le Brix - 18000 Bourges

Abstract

Hypersonic flight is expected to be achieved in the coming years by use of Supersonic Combustion RAMJET (SCRAMJET). One of the main issues is the thermal management of the overall vehicle and more specifically the cooling of the engine. In order to simulate the behaviour of an actively cooled SCRAMJET by use of supercritical endothermic fuel, a one-dimensional transient numerical model has been developed with heat and mass transfer, fluid mechanics and detailed pyrolysis chemistry. A dedicated experimental test bench is now available since 2006 to study supercritical fuel pyrolysis under stationnary and transient conditions. It aims to provide understanding of coupled phenomena, validation data for the numerical code and evaluation of onboard and real-time measurement methods for industrial use. A brief overwiew of the numerical code and a presentation of the experimental bench are proposed in this paper. Experimental results are discussed and a comparison is provided between numerical and experimental data. Discrepancies are shown to be lower than few percent in term of molar chemical compositions. This is due to uncertainties on experimental temperature measurement and to 2-D effects, which are not taken into account by the modelling. The numerical code appears to be of great importance to access to unmeasured data. Experimental and numerical tools are proved to be efficient to test future measurement methods under extreme conditions, specially at supercritical state.

Keywords: endothermic fuel, supercritical state, pyrolysis

1. Introduction

Hypersonic flight is expected to be achieved with SCRAMJET engine. Because the total temperature of external air reaches temperatures as high as 4950 K for example at Mach 12, even composite materials could not withstand such large heat load. Thus, an active cooling system has to be used. Furthermore, the time allocated to the combustion in the engine is about 10⁻³ s. These two points lead to use the fuel to cool down the engine's wall and then to burn it in the combustion chamber (C.C.). Fuel is injected in a composite channel which surrounds the engine. When heated above 800 K, the fuel is pyrolysed and thanks to its endothermic behaviour, it ensures the active cooling of the hot walls. This pyrolysis produces lighter hydrocarbons species, which are easier to ignite. It is important to note that the expected high pressure in the cooling loop (>3 MPa) causes the fluid to become supercritical in the channel. The principle of SCRAMJET technology, its advantages, and the interest to use hydrocarbon fuel have been fully studied in the literature (Powell et al., 2001). Sicard et al. (2006) present an industrially oriented bench related to this field of research but it is not suitable to test measurement methods for the SCRAMJET control. Modelling is another feasible method for conducting engineering studies and for furthering research related to this topic. As both the pyrolysis and the combustion chemistry are never treated in the literrature, coupled phenomena are not considered and the relationship between fuel composition and SCRAMJET thrust cannot be taken into account. The need of a specific tool considering the overall vehicle, its cooling as its thrust, is thus evident to study the entire coupled phenomena

^{*} Nicolas.Gascoin@bourges.univ-orleans.fr

involved in the SCRAMJET's cooling. The present work falls within the framework of the COMPARER project (a French acronym for COntrol and Measure of PArameters in a REacting stReam). It aims at understanding the pyrolysis of endothermic hydrocarbon fuel under supercritical states with complementary numerical and experimental approachs. This work should help to respond to the industrial need, which is related to mass flow rate measurement and burning capacity determination.

A stationnary study has been first conducted before working on the transient system behaviour. This is a relatively innovating study because pyrolysis under supercritical states are rarely conducted and because numerical simulations are used to furnish information which are not measured during the experiments. A numerical code, called RESPIRE (a French acronym for SCRAMJET Cooling with Endothermic Fuel, Transient Reactor Programming), has been specifically developed. It considers a complete hypersonic vehicle with active cooling. It allows for studying under transient conditions, the coupling between the pyrolysis inside the cooling channel and the combustion inside the engine, so as to the thermal consequences on the engine and the thrust of the vehicle. The cooling channel can be studied alone to simulate a chemical reactor, such as an experimental bench. RESPIRE could treat several fluid type but the main one is n-dodecane, for which a full pyrolysis mechanism (Dahm et al., 2004) is used (1185 reactions and 153 species, designed by the French laboratory DCPR). The purpose of the developed model is mainly to understand the relationships between phenomena. The aim is to determine temperatures to a tenth of a degree and the chemical composition at a mole fraction of about 1 %.

Then, an experimental test bench has been realised to serve as a validation apparatus for RESPIRE and also as a comprehension tool. This bench allows for studying fuel pyrolysis under stationnary and transient conditions. It is notably composed of a fuel tank, a pump, a furnace, a Gas Chromatograph and a Mass Spectrometer, Maximum operating parameters are about 1800 K, 80 bars and 0.6 g,s⁻¹ inside the chemical reactor. Working conditions are controlled to conduct parametric studies about pyrolysis under various conditions, particularly under supercritical state. Coupling experimental and numerical approachs is hoped to improve knowledges about pyrolysis in order to identify one or two specific parameters to provide a future measurement method for the engine's controle. The test bench is briefly described in a previous paper (Gascoin et al., 2006a) and some experimental results are proposed to illustrate the complexity of fuel composition after pyrolysis. The fuel choose for this study is the n-dodecane because of its representativity of aeronautic kerosene, because it is a pur component easier to study and because of the detailled pyrolysis mechanism, available for the numerical study. The purpose of this paper is firstly to give a brief overview of the RESPIRE code and a full description of the test bench. Secondly, experimental results are proposed and studied in the light of numerical informations obtain thanks to the RESPIRE simulations. A feedback is obtain thanks to experimental data to give a supplementary validation of the modelling. It will be underline in this study how pyrolysis conditions, obtained with the test bench, will allow to evaluate measurement methods for industrial applications.

2. RESPIRE : modelling and validation

The modelling related to the combustion chamber and to the overall vehicle is not presented in this paper as it is not necessary for the pyrolysis study. The cooling channel, represented by a chemical reactor, can be computed as a stand-alone part. Because RESPIRE is a 1-D code, all variables are constant on a cross-section of the fluid or of the material. The fluid is considered to be homogeneous on each slice along the flow. It is treated as an average single phase flow but possibly multi-species. Equations are written in transient state and the resolving method is a finite differences one. Partial derivative equations are discretized in space with centred explicit scheme and then solved in time with explicit scheme. The spatial step is of the order of 5.10⁻³ m and the time step is chosen in behalf of the flow velocity expected in the channel so as to keep a particle inside a given spatial step between two time steps. Energy, continuity and momentum equations are solved inside the cooling channel and effusion through the walls can be investigated, as the coke deposit along the channel. Transport of chemical species and species balance are considered. All the details about the modelling equations are precisely and entirely given in previous work (Gascoin et al., 2006b and 2006c). A full n-dodecane pyrolysis mechanism (Dahm et al., 2004) is used (1185 reactions and 153 species). This model is the minimum one which is able to successfully predict the thermal decomposition. Most of the radical species, which have minimal

concentrations in the mixture, play a determining role in the production and consumption of all other species. Mixtures have never been found to be at thermodynamic state because of residence times allowed for the pyrolysis, roughly lower than 20 s. Chemical composition cannot be given by data tables because the pyrolysis is fully controlled by the working conditions. This acts to consider detailed chemistry mechanism in relationship to thermal and hydraulic calculations.

A full validation of thermal, hydraulic and chemical aspects has been conducted under transient and stationnary conditions with analytical, numerical and experimental cases. A previous work (Gascoin et al., 2006a) furnish a chemical validation under stationnary conditions as a validation of the transient thermal and hydraulic behaviour of RESPIRE. Good agreement is found on temperature, compared to the Computationnal Fluid Dynamics software CFD-ACE, which has been validated and tested by Dufour and Bouchez (2002). Discrepancies appear to be lower than ten degree, which are attributed to 2-D effects in the channel and to low Reynolds number flow. Thermal and hydraulic time evolutions are reasonable and quantitative results are adequate for a 1-D model, used as predimensioning tool. Computed chemical compositions are found to be of the same order as validation data (1 % molar fraction discrepancies). Calculations in the RESPIRE code of fluid properties under various conditions, particularly in supercritical state, have been presented and validated in previous work (Daniau et al., 2004). Considering real pyrolysed mixture properties, and not the one of a perfect pure fluid, can decrease fluid temperature at the cooling channel outlet of about 20 %. This is due to the heat capacity which varies until a factor 2. This results in a completely different fluid composition and combustion, which justifies its consideration. Transient conduction through channel walls has been investigated using steady state and transient heat flux conditions (Gascoin et al. 2005a). Less than a 2 % variation on wall temperature, compared to analytical data, has been found with steady state heat flux. The method used to compute the chemical fluid composition has been validated. Compared to an experimental test with NORPAR fuel, the n-

3. Fuel Pyrolysis Experimental Test Bench

The aim of the experimental bench is to provide a detailled comprehension of fuel pyrolysis in conditions near the one identified for the engine. It should furnish validation data for the RESPIRE code and allow to test several measurement methods to be suitable for on-board and real-time industrial use. The conception of the experimental apparatus has been based on the engine's working. The cooling channel is represented by a chemical reactor, the thermal aspect of the combustion chamber by a furnace and the combustion of pyrolysis products by a bunsen burner. Predimensionning calculations have been performed to define the study parameters, such as the furnace length, the temperature profil along the furnace, the inner diameter of the chemical reactor, the mass flow rate. This is an important step because it guarantees a better agreement with the optimal experimental bench. This approach is rarely encountered in the field of research to be mentionned. Calculations have been conducted thanks to the CFD software Fluent and to the code NANCYNETIK, which has been used for previous studies (Daniau et al., 2004).

dodecane pyrolysis gives relatively good agreement, typically a 3 % variation in terms of mole fraction. Further validations of fluid temperature have been presented in previous work (Gascoin et al., 2005b).

The simplified test bench is represented by the figure 1. The dodecane is injected in the chemical reactor thanks to a pump limited to a maximum working pressure of 10 MPa and to a maximum mass flow rate of 0,6 g.s⁻¹ with dodecane at ambiant conditions. Gradients of mass flow rate can be studied to investigate transient behaviour of the system. Pressure fluctuations are limited thanks to an absorber. An electrovalve is used to control the injection in the process. The chemical reactors used for the present study are made in stainless steel 316 L. Some tests have been conducted with standard steel and with Titanium. Thus the maximum operating temperature is limited at about 1200 K for the stainless steel. The furnace, in which is inserted the reactor, allows for reaching a maximum temperature of roughly 1850 K. Temperature is measured thanks to a dedicated thermocouple located in the middle of the furnace and it is controlled thanks to a regulator. Various transient temperature profiles can be realised to study transient phenomena behaviour. Pressure is regulated through an automatic valve composed of severals components. This valve is controlled so as to vary the operating pressure in transient conditions. A Coriolis mass flowmeter is avalaible at the reactor inlet to measure transient mass flow rate and density. At the chemical reactor outlet, a cooling apparatus has been specifically designed to decrease the pyrolysed fluid temperature

from a maximum of about 1600 K to 300 K. After the regulation pressure valve, the pressure is near the atmospheric one and pyrolysed mixture is collected in a dedicated phase separator laying on a mass balance to obtain density of liquid phase. The gas phase is evacuated thanks to a burner. No combustion study are presently conducted. Gas and liquid sampling are possible, so as to inline gas analysis.

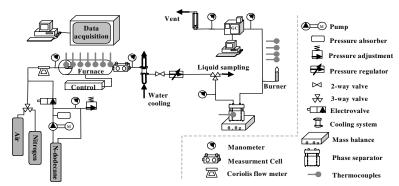


Figure 1. Simplified sketch of the fuel pyrolysis test bench.

All these equipments, working under the conditions identified above, allow to take into account the phenomena involved in the SCRAMJET cooling. A specific instrumentation has been settled to characterise phenomena and furnish suitable data for our purpose. A Gas Chromatograph (VARIAN, Star 3800) equiped with 10 sampling loops is used to analyse the gas samples at ambiant conditions, thanks to a WCOT column. The TCD sensor is used to quantify hydrogen whereas the Flame Ionisation Detector is used for hydrocarbons. The latest is calibrated for species from CH₄ to C₃H₈. For hydrocarbons, liquids at ambiant conditions, a coupling Gas Chromatograph - Mass Spectrometer (AGILENT, 6850 - 5973) is used with automated injection. The pyrolysis products compositions are studied in behalf of study parameters. These ones are obtained thanks to about forty simulatenous measures (temperature, pressure, mass flow rate) and to an acquisition apparatus. During the entire day of test, these data are acquired typically every 10 s for stationnary studies. Thermocouples, K and C type, are conceived in the laboratory. A specific cell, made in stainless steel 316L, has been designed to test measurement methods for industrial need and to observe the pyrolysed fluid at the furnace outlet. It is equiped by five diamond windows and it is instrumented. To conduct experiments under stationnay conditions, the furnace temperature, the mass flow rate and the pressure are fixed. After stabilisation, gas and liquid samples are collected. Furnace temperature is then increased to another desired process temperature. At the end of the day, chemical composition of pyrolysis products is avalaible for various temperature at given fixed conditions of pressure and flow rate. The n-dodecane is used for the present study (purity of 99 %).

4. Experimental pyrolysis results and supplementary numerical explanation

4.1. Characterisation of the test bench

Operating conditions presented in this paper are in the range of 1 MPa to 6 MPa, of 620 K to 1220 K, of 0,05 g.s⁻¹ to 0,1 g.s⁻¹; for an internal reactor diameter of 4,5.10⁻³ m. Residence time is not experimentally available but it can be obtain by means of RESPIRE simulations (Figure 2). The fluid temperature first increases in the first third of the furnace, then it is approximately constant before decreasing in the last third. This is due to the furnace temperature profile which is maximum at the center and which decreases at the border. For a global residence time of about 100 s, the time allocated to the pur n-dodecane heating is about 75 s and the one corresponding to its downstream cooling is 15 s. This cooling is due to the furnace temperature profile and will be explained in the following section. The pyrolysis appears during a residence time equal to 10 % of the global one inside the chemical reactor. This is a characteristic time for the chemical process, which cannot be measured experimentally. This clearly underlines the necessity to simulate each experiment to obtain such important data. It is particularly true for transient study. Despite residence times higher than in the SCRAMJET's cooling channel, calculations at thermodynamic equilibrium, with the HSC commercial software, have demonstrated that this one is not obtained inside the chemical reactor. This point proves the need of chemical kinetic to study such reactive flow.

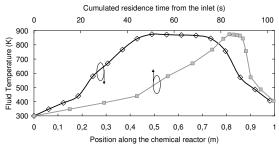


Figure 2. RESPIRE calculations of fluid temperature (0,05 g.s⁻¹, 5 MPa, 4,5.10⁻³ m diameter).

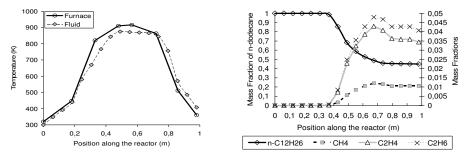


Figure 3. RESPIRE simulation of a given test case (0,05 g.s⁻¹, 5 MPa, 4,5 mm reactor diameter) using experimental furnace temperature as boundary conditions: fluid temperature (left), pyrolysis products composition (right).

An example of furnace temperature profile is given (Figure 3, left). This one is not exactly centered but it is shifted to the outlet because of the fluid flow inside the chemical reactor. The thermal gradients at the inlet and outlet do not have consequences on the pyrolysis process as n-dodecane does not pyrolyse under 700 K to 800 K. The most important part for the pyrolysis is the central one, where the maximum temperature is measured. In order to characterize stationnary experimental tests, this maximum temperature measured inside the furnace is supposed to be the maximum one reached by the fluid. This has been confirmed thanks to RESPIRE calculations (Figure 3). Maximum temperature discrepancies between the fluid and the furnace, for all the various tested conditions, are about 50 K, i.e. less than 5 % of temperature (Figure 3, left). Furthermore, the maximum fluid temperature corresponds to the maximum pyrolysis rate for the n-dodecane (Figure 3, right). Few variations are noticed on the chemical composition after the maximum reached fluid temperature. This allows for taking the maximum furnace temperature as a characterisation parameter of the pyrolysis, as it is representative of the pyrolysis products analysed experimentally. Nevertheless, it has been determined, by use of 2-D calculations with CFD-ACE software, that thermal radial gradients inside the chemical reactor can reached 40 K for a turbulent fluid flow. For low Reynolds number flow, hydraulic 2-D effects have been observed inside the chemical reactor and thermal gradients are thus waited. These gradients play an important role on the temperature distribution inside the fluid and thus on its pyrolysis. This point cannot be studied neither numerically nor experimentally because these tools are roughly 1-D. But using turbulent fluid flow or single phase flow may decrease the effect of these gradients. As the n-dodecane pyrolysis starts at a temperature around 700 K to 800 K, depending on residence time and pressure; a working pressure higher than 1,8 MPa corresponds, for pur n-dodecane, to a transition from the liquid phase to the supercritical state around 658 K. For lower pressure, a coexistence of liquid and gas phases appears. It is interesting to obtain supercritical pyrolysis as the fluid can be treated as a single phase flow, even for low Reynolds number where a stratification of phases could appear with gas and liquid. This is a good point for RESPIRE, as it considers constant values in a cross section of the reactor, and also for the process because it is more convenient to study. Thanks to a dedicated observing cell, located at the furnace outlet and equiped with diamond windows, the pyrolysed dodecane has been studied. Near the critical point of pyrolysed mixture, fluid becomes green and then very dark. We have even observed conditions with "mirror" effect for which we can see ourself in the fluid. This is due to optical fluid properties modification near the critical point. Our observations are consistent with the one of Oag et al. (2004).

4.2. Experimental results: understanding, validation and exploitation

Experimental results of pyrolysis are given (Figure 4) for the following test conditions: 1 MPa, 0.05 g.s⁻¹, 4,5.10⁻³ m stainless steel reactor diameter. The conversion rate passes from 20 % to 95 % in a temperature range of about 200 K. Pyrolysis of n-dodecane (Figure 4, left) produces intermediate hydrocarbons with 7 to 10 carbon atoms. This should be accompanied by a production of lighter hydrocarbons, gaseous under ambiant conditions, but this is not experimentally confirmed (Figure 4, right). No gaseous products are detected before 900 K, whereas pyrolysis starts around 700 K. It is attributed to low production rate of such species at moderate temperature. This conducts to have low gaseous pressure which disables the gas phase analysis because sufficient pressure is needed to bring chemical products to the Gas Chromatograph. This points is not dramatic as it corresponds to a gaseous products mass fraction lower than few percents. The experimental test bench aims at studying working conditions with high gaseification. The gaseification is defined at ambiant condition as the ratio between gaseous products mass and pyrolysis products mass. A relationship between the gaseification rate and the Hydrogen/Carbon ratio appears. H/C ratio greater than 0,19 are representative of pyrolysis products which are gaseous at ambiant conditions. Thus, information about this ratio could give further information about the production of light species. Despite the fact that coking appears at temperature around 900 K, it can be noticed that no acetylene is found in the gaseous pyrolysis products (Figure 4, right). This is confirmed by the numerical simulations. Coke formation has been macroscopicly studied and characterised but it is not fully presented in this paper. To have an idea of coking activity during pyrolysis, a typical stainless steel reactor, with an internal diameter of 4,5.10⁻³ m, is completely jammed by coke after five hours at 900 K with 0.05 g.s⁻¹. We have identified that H/C ratio gives information about pyrolysis. It is equal to 0,18 for pur n-dodecane and it increases as the process temperature increases. This ratio is characteristic of compounds. It is equal to 0,166 for alcenes and it varies between 0,34 for methane to 0,166 for heavy hydrocarbons. It could be an interesting tool to predict carbon deposit since the coke formation should be accompanied by a higher H/C ratio at the reactor outlet.

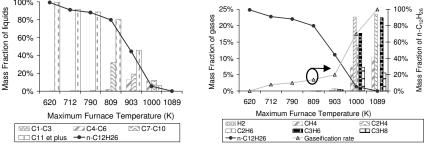
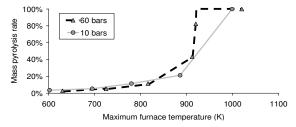


Figure 4. Experimental mass fractions of pyrolysis compounds: liquid (left) and gaseous (right) phase.

It is interesting to observe the quantity of gaseous products, thus the gaseification rate, increasing with temperature process (Figure 4, right). This is due to the formation of allways lighter species at higher temperature. A relationship between gazeification and pyrolysis rate has been outlined during the experimental tests but this may depends on operating conditions as gliding may occur between gas phase and liquid phase inside the process, after the furnace outlet. This relationship could be interesting for the ground industrial process as it could give information about pyrolysis rate without chemical analysis.



 $Figure\ 5.\ Influence\ of\ subcritical\ and\ supercritical\ conditions\ on\ experimental\ pyrolysis\ rate.$

A parametric study has been conducted on various process parameters, such as temperature, pressure, mass flow rate, reactor diameter and chemical nature of reator materials. This has permitted, thanks to

design of experiments, to show the first importance of temperature and residence time on pyrolysis rate. Parameters effects are not easy to analyse because their fluctuations are accompanied by various other ones. For example, an increase in mass flow rate raises the convective exchange between the fluid and the reactor but it decreases the residence time. Finally, the pyrolysis decreases. The pressure effect is easier to understand but two phenomena are thus involved (Figure 5). A pressure increase corresponds to a residence time increase through the one of density, for a given mass flow rate. And it is accompanied by a higher collision number between molecules, which favours the chemical reactions too. Finally, it is difficult to know which contribution increases the pyrolysis rate. Nevertheless, it has been observed that pressure has an influence mainly in the subcritical domain. Pyrolysis under supercritical state show results almost independent from pressure. To give better characterisation of experiments, a mass balance has been performed for the experimental test cases, on liquid and gas phase and for hydrogen and carbon mass. Discrepancies are lower than 1 %, which is sufficient for our purpose. This allows for studying effusion through porous reactor walls for future experiments. Indeed, the injected mass flow rate for the SCRAMJET engine is expected to be slightly lower than the one pumped from the tank because of film cooling through porous walls. This point needs to be studied because it influences the combustion and thrust. Experimental results have also served to validate the RESPIRE code (Figure 6). The main differences are obtained for lighter hydrocarbons, which concentration is numerically higher. This can be attributed to the code, which does not consider thermal and hydraulic gradient in a cross section. But it can also be due to experimental measurement, as the gas phase might be underestimated. The Gas Chromatograph used to identify and quantify gaseous species is not calibrated for four carbon atoms species and beyond. These compounds can be badly taken into account if they are gaseous at the ambiant conditions. Depending on daily ambiant temperature and pressure, some species can be experimentally taken into account into the gaseous phase or into the liquid one, whereas computations consider a fixed temperature of 298 K and a fixed pressure of 0,1 MPa, to determine the phase of these various compounds. Finally, due to uncertainties on experimental temperature measurement, discrepancies are totally acceptable as a 200 K temperature gradient can correspond to a variation of pyrolysis rate of 100 %. Numerous conducted validations have proved RESPIRE to give sufficiently good results for our purpose through the entire range of pyrolysis rate, from 0 % to 100 %.

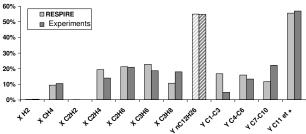


Figure 6. Gaseous Molar Fraction in the gas phase and Compounds Mass Fractions in the pyrolysis products: Experimental analysis for a given test case (773 K, 0,05 g.s⁻¹, 5 MPa, 4,5 mm reactor diameter).

5. CONCLUSIONS

A one-dimensional transient model has been programmed in order to simulate the SCRAMJET active cooling and to determine the mixture composition at the cooling channel outlet. A complete detailed mechanism with 1185 reactions and 153 species has been implemented. Equations have been presented in previous works and the main possible validations at this time, depending on the available data, have been conducted. RESPIRE is thus quantitatively validated. It is suitable to simulate the experiment conducted thanks to the dedicted test bench. RESPIRE should help to build a cooling strategy and to test measurement methods for industrial need. The COMPARER test bench has been fully described in this paper. Operating conditions so as specificities of some components have been evaluated thanks to a numerical approach in order to ensure its reliability. The experiment is suitable to study the stationnary and transient behaviour of hydrocarbon pyrolysis, possibly at supercritical state, under extreme conditions (8 MPa, 1850 K, 0,6 g.s⁻¹). Chemical composition of pyrolysis products have been investigated

experimentaly thanks to a Gas Chromatograph and a Mass Spectrometer. Various operating parameters (temperature, pressure, mass flow rate, geometric configuration) were tested. A parametric study has shown the prior importance of temperature and residence time on the pyrolysis. Pressure effect appears to be negligeable under supercritical state, whereas it can play a role at subcritical conditions. Some relationship have been identified between gazeification rate, pyrolysis rate, Hydrogen/Carbon ratio and coke deposit. The experimental and numerical tools allows for taking into account the relationships between coupled phenomena, such as fluid mechanics, heat transfer, chemical kinetics. They consider a hydrocarbon fuel, without dilution, in a reacting flow with a varying temperature profile, which is far from an ideal PSR configuration. The RESPIRE code demonstrates an important capacity to give information about unmeasured experimental data, such as the time dedicated to pyrolysis and the fluid temperature inside the process. As a feedback, experimental results have been used to complete the validation set of the code. Both numerical and experimental tools have shown an interesting complementarity and adequation with the need for the COMPARER project. They will permit to test measuring methods on the pyrolysed fuel, such as Infra-Red spectroscopy or flow meter based on pressure loss. The dedicated experimental test bench can provide further validation data on chemical aspects and on the dynamic response of inlet time variations. Furthermore, an experimental combustion study is expected to be conducted in 2007 on premixed and diffusion flames for the pyrolysed fluid. These data should bring additional validation cases. Pyrolysis studies under transient conditions have already been, and are still, conducted.

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