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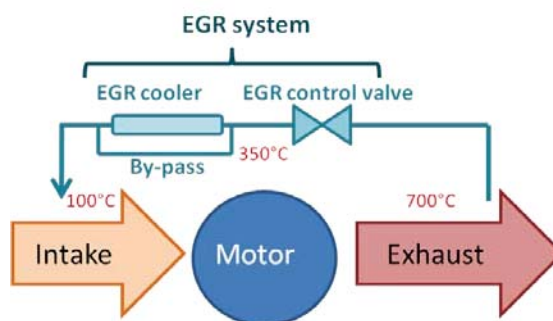
## IMPLEMENTATION OF AN EXPERIMENTAL PILOT REPRODUCING THE FOULING OF THE EXHAUST GAS RECIRCULATION SYSTEM IN DIESEL ENGINES

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**Abstract:** The European emission standards EURO 5 and EURO 6 define more stringent acceptable limits for exhaust emissions of new vehicles. The Exhaust Gas Recirculation (EGR) system is a partial but essential solution for lowering the emission of nitrogen oxides and soot particulates. Yet, due to a more intensive use than in the past, the fouling of the EGR system is increased. Ensuring the reliability of the EGR system becomes a main challenge. In partnership with PSA Peugeot Citroën, we designed an experimental setup that mimics an operating EGR system. Its distinctive features are (1) its ability to reproduce precisely the operating conditions and (2) its ability to measure the temperature field on the heat exchanger surface with an Infra Red camera for detecting in real time the evolution of the fouling deposit based on its thermal resistance. Numerical codes are used in conjunction with this experimental setup to determine the evolution of the fouling thickness from its thermal resistance.

### 1. INTRODUCTION

The European emission standards EURO 5 and EURO 6 define more stringent acceptable limits for exhaust emissions of new vehicles [1]. The Exhaust Gas Recirculation (EGR) system is a partial but essential solution for lowering the emission of nitrogen oxides (NOx) and soot particles. The EGR is made up of a control valve and a heat exchanger (Figure 1). By re-circulating a fraction of the engine exhaust gas back to the engine cylinders, the EGR leads to a better NOx/soot particle compromise after combustion. This strategy of pollution reduction is completed downstream by (1) a particle filter removing the residual soot and (2) a selective catalytic reduction system for



**Figure 1:** Exhaust Gas Recirculation (EGR) system

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NOx. Yet, respect for new European emission standards results in a more intensive use of the EGR system than in the past. Mention for example the increased EGR rate, the extension of use of the EGR to more "heavy" operating points (higher speed and torque level due to smaller engine sizes), wider and harder test cycles for engine certification...

This more systematic use of the EGR system increases its fouling. New factors also contribute to fouling: mention the higher fraction of bio-fuels which is now added to bio-diesels or the evolution of motor technologies towards a lower fuel consumption (hybrid engine, "Stop and Start" systems). The impact of this whole set of factors upon the formation and the evolution of the fouling deposit must be considered, since failures in the EGR can happen in case of fouling: blocking of the control valve, reduction of the heat exchanger thermal efficiency...

For car manufacturers, ensuring the reliability of the EGR system becomes a main challenge. Understanding the formation and evolution of the fouling deposit requires (1) a good knowledge of the intervening coupled physical / chemical mechanisms and (2) a good control of the operating conditions towards identifying the most relevant factors with respect to fouling. Mention for example the organic matter / soot particulates ratio of the exhaust gas mixture, the engine's operating range (which determines at least partially the thermal gradients involved in the EGR heat exchanger), the EGR rate... It is also important to anticipate eventual the impact upon the fouling deposit of the evolution of the fuel (increase of the bio fuel rate) of the multiplicity of the fuels placed on the market. All this explains why car manufacturers need data on fouling in order to build more realistic predictive fouling laws (deposit type, thickness) covering the variability of the situations raised by the EURO 5 and 6 context.

In this contribution, we first present the experimental setup that we designed in partnership with PSA Peugeot Citroën in order to "mimic" the EGR system in working conditions. Stress is put on its distinctive features: (1) the ability to monitor/reproduce very precisely (all) the operating conditions, (2) the ability to measure the temperature field on the heat exchanger surface with an infra-red (IR) camera in order to detect *in real time* the appearance of the fouling deposit and the evolution of its thickness based on its thermal resistance. The experimental data that are being currently collected shall be later used to refine a fouling law. Then, we discuss how the formation of deposits that are similar to those obtained at PSA Peugeot Citroën during engine test bench cycles allows validating our experimental setup. We show that gas mixtures including or not CO<sub>2</sub>/CO lead to almost similar deposits from a chemical viewpoint. This permits to simplify the gas mixture injected in the heat exchanger and to enhance safety. On the other hand, our experimental setup reveals the impact of an additional factor, the degree of ageing of the vegetable oil methyl ester entering in the composition of the gas mixture. After presenting the objectives of an ongoing experimental design, we last discuss the first trends observed when monitoring/tracking the deposit formation via the Infra-Red camera.

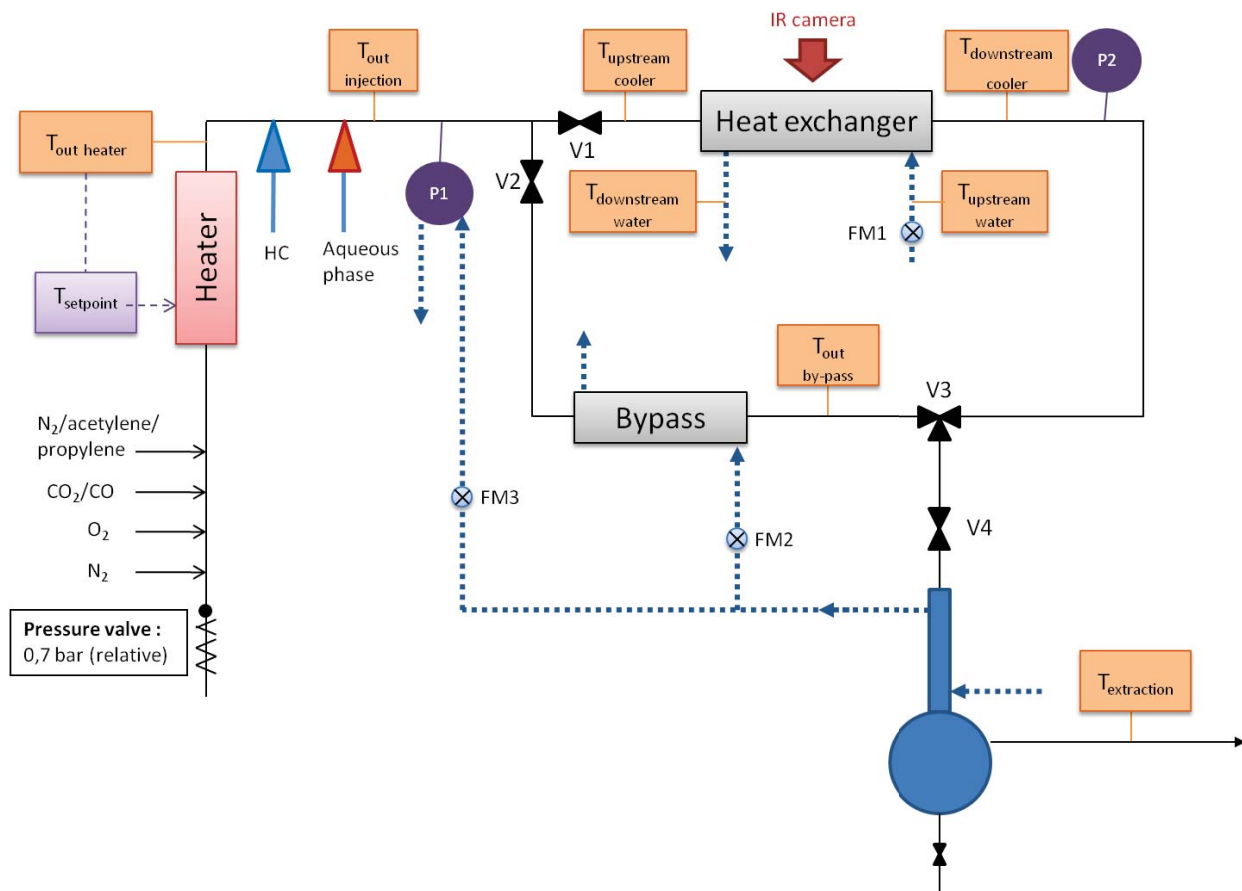
## 2. MATERIALS AND METHODS

Our experimental setup is designed to be a simplified representation of a working EGR system (see Figure 2). It allows cooling a flowing mixture of hot gases (initially at around 350°C) whose composition is representative of a diesel engine exhaust gas (N<sub>2</sub>, CO/CO<sub>2</sub>, acetylene/propylene, unburned or partially oxidized hydrocarbons, burned hydrocarbons due to the presence of biofuel, water and sulphuric acid). Cooling is assured by a homemade heat exchanger (see Figure 3a). Our set-up allows a better control of the operating conditions (gas temperature at the input/output of the heat exchanger,

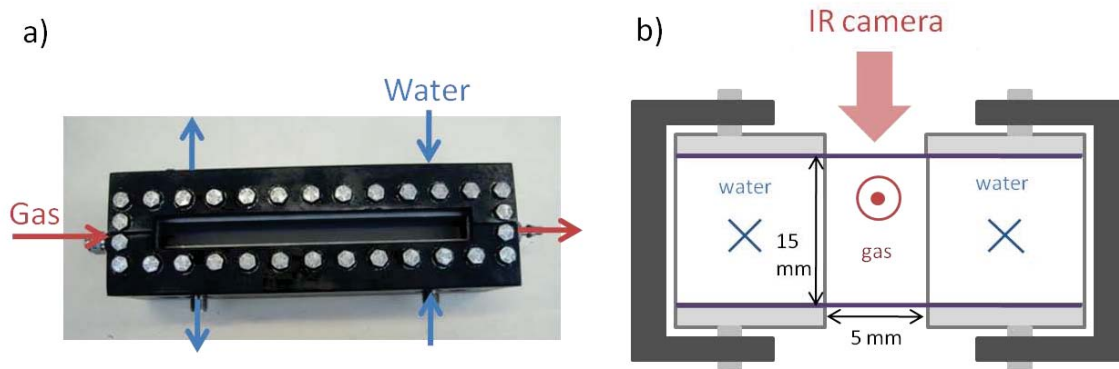
pressure, mass flow and gas mixture composition) compared to a conventional engine test bench. Figure 4 shows temperature measurements at different locations in the experimental setup and highlights the good control of experimental conditions. The possibility of varying the operating conditions allows running a complete experimental design in order to find the most relevant factors for the fouling deposit formation/evolution. In particular we investigate the impact of temperature and flow rate variations at the entrance of the heat exchanger as well as the influence of the gas composition.

Another advantage of our experimental set up is the monitoring, versus time, of the temperature field at the top of the heat exchanger with an IR camera (see Figure 3b). The surface temperatures that are collected are used to identify numerically the local heat transfer resistance induced by the fouling. This shall ultimately allow to obtain the thickness of the deposit (inverse methods). Surface temperature data are also related to bulk temperature within the heat exchanger.

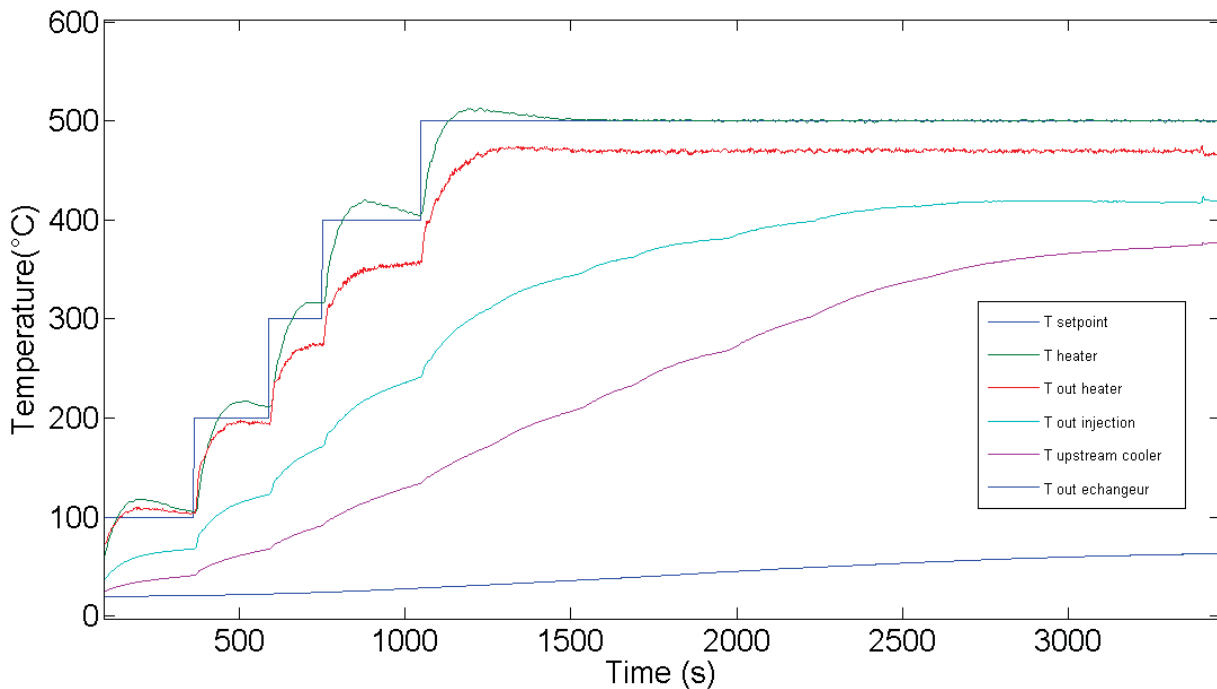
Our heat exchanger pulls apart in order to permit the characterization of the deposit (SEM, Raman spectroscopy) without damaging the latter. Part of the deposit is also analysed by Thermo Gravimetric Analysis (TGA) and combined Pyrolysis/Gas Chromatography/Mass Spectroscopy (PY/GC/MS). These analytical devices allow validating the experimental setup and help identifying the main chemical processes (oxidation and/or nitro-oxidation and/or polymerisation) involved in the formation/evolution of the deposit.



**Figure 2:** The experimental set-up



**Figure 3:** The heat exchanger geometry: a) view of the top and b) cross section



**Figure 4:** Heating module of the experimental set-up: gas temperature monitoring

### 3. RESULTS AND DISCUSSION

#### 3.1. The validation of the experimental set-up

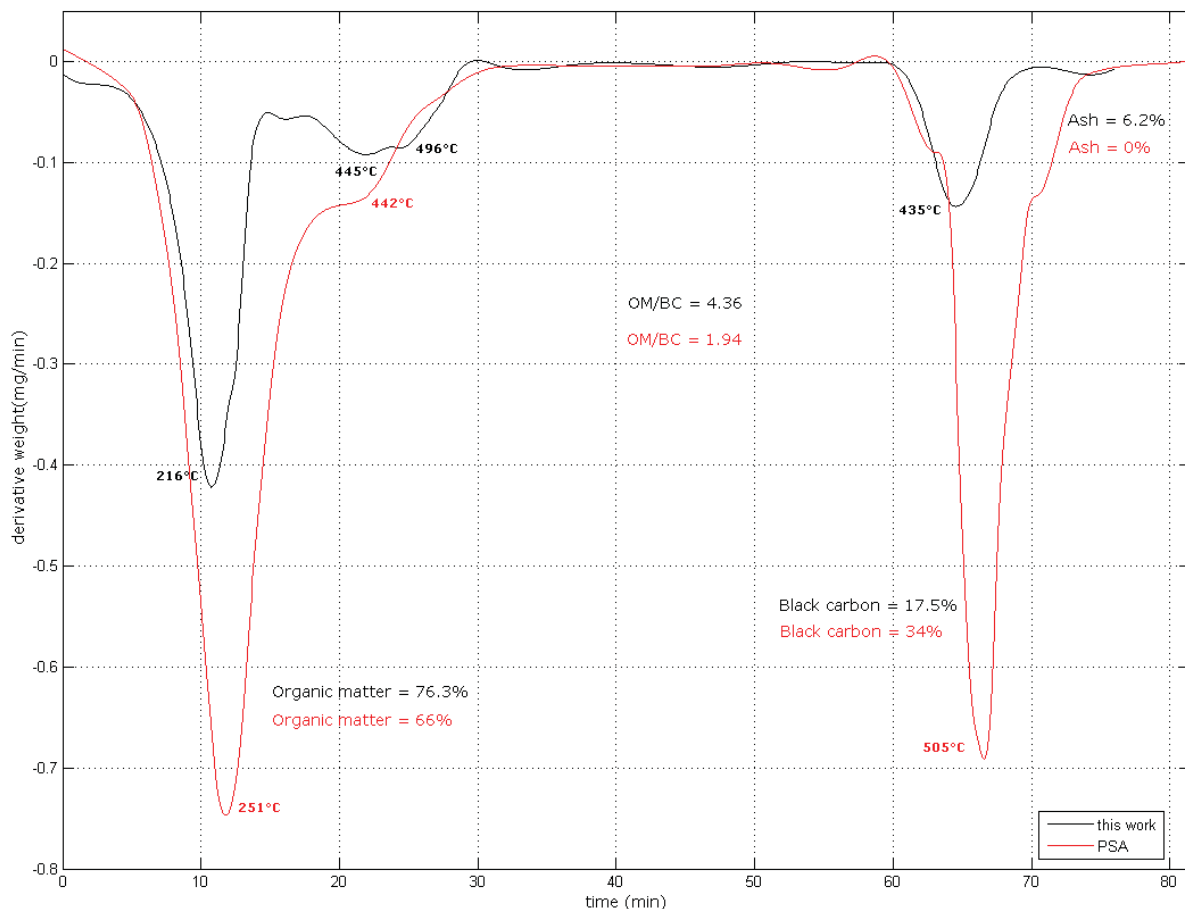
Validating the experimental set-up is a crucial point. A first requirement is to obtain a "big enough" deposit to make possible its subsequent chemical analysis. This was possible through 2 day long experiments wherein the effective operating time is 7 hours (every experiment includes a series of 30 to 60 minutes of operation followed by an interruption of the same duration; at mid term point, the experiment is stopped overnight). The validation is based on a quantitative comparison (observations, weight, organic matter/carbon black ratio and chemical composition) between our own fouling and those obtained at PSA Peugeot Citroën during engine test bench cycles.

The foiling deposit in Figure 5 is obtained with the following operating conditions: a gas mass flow of 1g/s, a gas mixture temperature at the entrance of the cooler of 350°C and a cooling water at 16°C. The gas mixture flows through the heat exchanger during 7 cycles of 60 minutes operation followed by 60 minutes of interruption and with a long break overnight. The deposits that are formed (Fig. 5) have brown to black *matte* colour. From that point of view they differ slightly those gloss black obtained during engine test bench cycles. This difference is easily explained by the fact that the latter include engine oil.



**Figure 5:** Our deposit

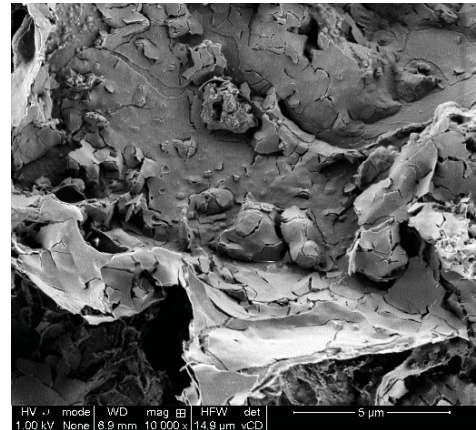
A fraction of each of our deposits is systematically analysed by Thermo Gravimetric Analysis (TGA). We compare our thermograms to some obtained at PSA Peugeot Citroën on deposits formed during engine test bench cycles (Figure 6). They look similar. The peak at 505°C is less important in our case, since we form only soot precursors. The reason for this is that we do not include any soot particles in our heated gas mixture whereas engine exhaust gas include some. The Thermo Gravimetric Analysis also enables us to calculate the percentage of organic matter/black carbon (weight % OM/BC). This ratio is a macroscopic characteristic of the deposit chemistry. Note surprisingly, we obtain a higher OM/BC ration on our deposits due to the absence of soot particulates in the flowing mixture, whence a smaller proportion of black carbon. These results validate our experimental set-up.



**Figure 6:** Thermo Gravimetric Analyse of our deposit (in black) – Comparison with a PSA Peugeot Citroën deposit (in red)

### 3.2. Experimental designs and deposit analyses

Towards simplifying the experimental design to be run subsequently, and enhancing safety, we looked at the impact of CO<sub>2</sub> and CO on the fouling deposit formation. Deposits (e.g. Fig. 7) obtained with and without CO<sub>2</sub>/CO have been analysed by Thermo Gravimetric Analysis (TGA) and combined Pyrolysis/Gas Chromatography/Mass Spectroscopy (PY/GC/MS). The chromatograms look generally similar with and without CO<sub>2</sub>/CO, to within a few nuances. Inclusion of CO<sub>2</sub>/CO in the gas mixture leads to slightly less formation of black carbon (BC) (see %BC smaller in Table 1) and to more reactive graphitic sheets including oxygen atoms (lower T°C BC). We also note that the presence of CO<sub>2</sub>/CO, leads to a smaller polymer quantity (in weight) and the polymers are longer than those obtained without CO<sub>2</sub>/CO (the temperature of the third peak of organic matter (T°C OM) is 510°C instead of 455°C).



**Figure 7:** Scanning Electron Microscopy (SEM) image of our deposit

Trial	% OM (polymer)	%BC	OM/BC	T°C OM	T°C BC
With CO <sub>2</sub> /CO	72.8% (17%)	11.4%	3.4	87, 206, 510	431
Without CO <sub>2</sub> /CO	71.8% (30%)	22.5%	3.2	95, 205, 455	457

**Table 1:** TGA results of the trials with and without CO<sub>2</sub>/CO

PY/GC/MS analyses show that we have the same quantity of carboxylates in all deposits, with or without CO<sub>2</sub>/CO in the gas mixture. This may suggest that the carboxylic acid is formed by ester acid hydrolysis ( $\text{RCOOR}' + \text{H}_2\text{O} \rightarrow \text{RCOOH} + \text{R}'\text{OH}$ ). In any case, the small impact of CO<sub>2</sub>/CO enables us to remove these components from our gas mixture.

Our gas mixture contains some vegetable oil methyl ester to take into account the biofuel that is now added in fuels. Our ester has been analysed by PY/GC/MS to know precisely its chemical composition. It is composed essentially of methyl linoleate (C<sub>18</sub>H<sub>32</sub>O<sub>2</sub>) and methyl oleate (C<sub>18</sub>H<sub>34</sub>O<sub>2</sub>) in equivalent proportions. The methyl linoleate has two carbon-to-carbon double bonds whereas the methyl oleate has only one. That is why the linoleate has a more important oxidation rate than the oleate [2, 3]. The deposit mass observed in our successive experiments increases over time. This may suggest that the ageing impact the deposit mass. Experiments run with an aged ester (10 days at 50°C) give a more important deposit. Work by McCormick et al. [3] shows that the deposit formation is strongly impacted by the ester oxidability. When the ester oxidizes, it forms longer molecules due to polymerization reactions in solution, which favours deposit formation and seems to agree with our results. Additional experiments are currently run to confirm this trend. Note that the issue of the impact of ester ageing is an important one for car manufacturers deposit formation, since more and more biofuels are introduced in fuels. These biofuels can have different chemical compositions depending on the plants used to make biofuels e.g., rapeseed in France, soybean in the USA).

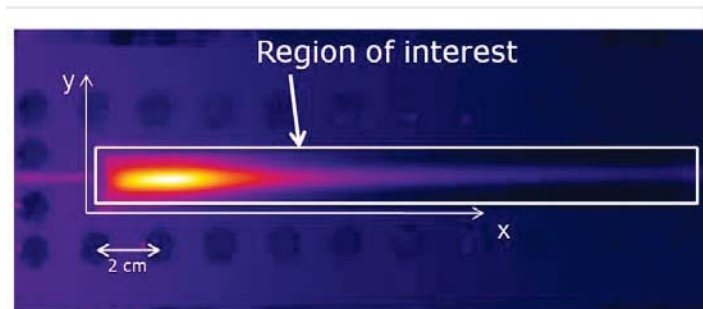
Two experimental designs are currently run to determine more precisely the EGR systems fouling factors. The first experimental design concerns the operating conditions (mass flow rate, heating temperature, cooling temperature...) in order to cover the variability of engine operating points. The other experimental design varies the chemical

composition of the gas mixture in order to assess which products favor the deposit formation.

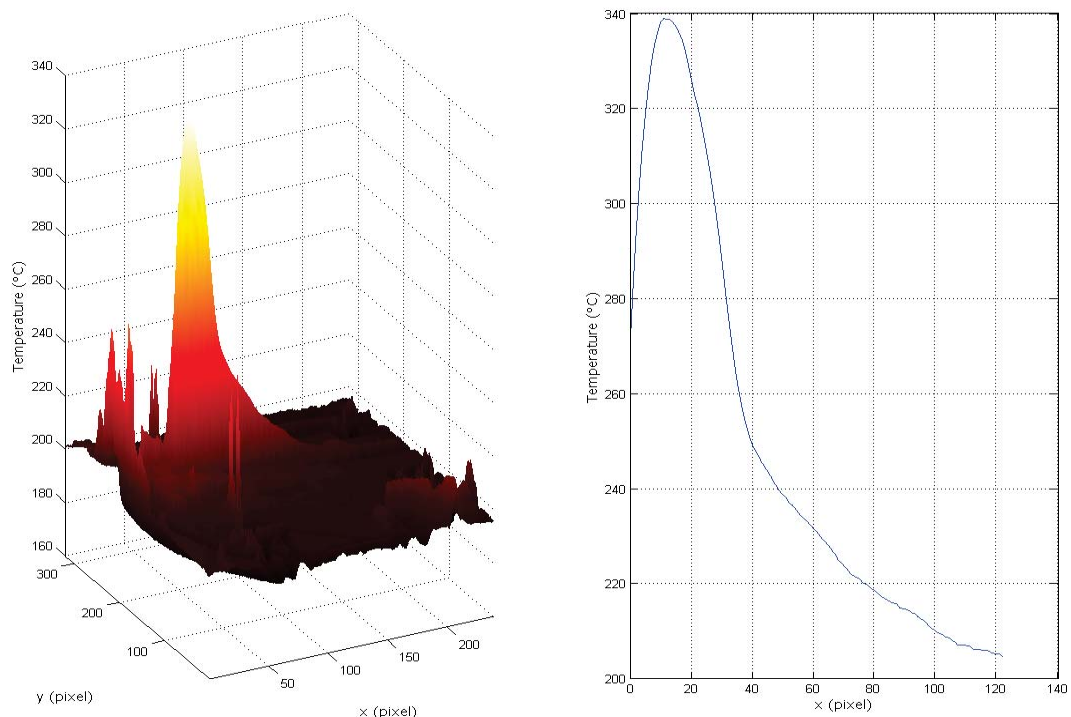
### 3.3. The monitoring of the deposit thickness evolution

The online monitoring of the evolution of deposit is useful in a lot of subject of studies. Different methods for the detection of wax or deposit have been developed [4, 5]: electric resistance, radiography, heat transfer techniques... In our case, we would like to monitor the evolution of our deposit with an infra-red camera. This technique has already been used to study temperature fields [6, 7]. This method requires specific heat exchanger geometry (Figure 3b). It is important to have a thin stainless steel layer which has a thermal resistance similar to the deposit thermal resistance. Indeed a too big layer thermal resistance doesn't allow seeing the variations of temperatures linked to the deposit.

The temperature fields are filmed on the top of the heat exchanger (Figure 8). Temperature profiles are obtained as a function of the length and the width (Figure 9). The increase of the deposit thickness changes the thermal resistance and decreases the temperature on the surface of the heat exchanger. Knowing the surface temperature without deposit, it will be possible to find out the deposit thermal resistance by inverse method. The thickness profile will be the same as the resistance profile, supposing that the fouling thermal conductivity is constant ( $R_d = e_d/k_d$ ).



**Figure 8:** Temperature field on the top of the heat exchanger



**Figure 9:** Temperature profiles: on the left, the temperature versus the length and the width and on the right the mean temperature versus the length



#### 4. CONCLUSION

Our experimental set-up has been built in order to better understand the fouling phenomena in diesel motor EGR systems. The ability of controlling the flow, the temperature and the chemical composition of the gas mixture (representative of a motor exhaust gas) is advantageous compared to conventional engine test benches where these parameters cannot be known precisely and where repeatability is quite difficult to realize. This experimental set-up shall provide a better understanding of the fouling process. Data that collected shall help to write new fouling laws accounting for the variability of the operating conditions raised by the Euro 5 and 6 context. These laws shall be ultimately plugged into in numerical codes for the design of EGR system architecture. The use of an Infra-Red camera to deduce (from the acquired temperature fields) the deposit thickness evolution in real time is definitely a distinctive feature of our approach.

We observe that (1) the biofuel composition (that differs amongst countries) and (2) the storage/ageing of the biofuel both impact the deposit formation. This observation, which has been somewhat overlooked up to now, shall be re-placed within the existing (huge) literature on the stability of biofuels. The experimental designs on operating conditions and gas mixture chemical composition will enable us to quantify the impact of the different experimental parameters.

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