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ASSESSMENT OF FOULING FORMED BY THERMO-OXIDATIVE DEGRADATION OF JET FUEL IN TURBULENT FLOW IN A CAPILLARY TUBE

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Abstract.
Jet fuel is used as cooling fluid in the propulsion system of jet aircrafts. The repetition of heating and cooling cycles may cause oxidative thermal degradation of the fuel, leading to the formation of insoluble deposits at the surface of pipes and orifices. In the High Reynolds Thermal Stability test (HiReTS test), a turbulent fuel flow in a capillary tube is heated, leading to thermal degradation and deposition of insoluble solid deposits at the tube surface. The amount of fouling formed during a fixed test time is used as an index to measure the thermal stability of the fuel sample. In this work, a direct method for measuring the amount of fouling is proposed using a gravimetric method combined with chemical removal. The method is applied to thermally stable and unstable fuel samples. The results are compared to the indirect measurements and a correlation is found between the mass of fouling and its thermal resistance. The method developed will allow for the estimation of the kinetics of formation of solid deposits in an accelerated degradation test, contributing for the development of fuels and fuel systems.

Keywords: *Fouling, Aviation kerosene, Stability of jet fuel, Fuel system.*

1. INTRODUCTION

The aviation kerosene (QAV) is used as the main coolant in the jet aircraft engine from now on. It flows out of the fuel storage tank, and after absorbing heat, a part of the fuel passes through the injector and is burned and the other part is recirculated back to the tank. Thus, when the QAV is subjected to a sufficiently high temperature in the heat exchange, hydrocarbon molecules, oxygen and impurities react. These reactions occur by the temperature increase and with the presence of oxygen cause the degradation of the QAV, consequently, solid and insoluble deposits are created. These insoluble materials accumulate to form deposits in components of the aircraft, especially in the heated zones (Mielczarek, 2015). Thermo-oxidative degradation strongly depends on chemical and physical factors. The physical aspects that influence are: time, temperature, pressure, mass flow, Reynolds number, characteristics of the deposit formed, and roughness. The chemical aspects are related to the composition of the fuel and chemical species that react with the fuel. They are: Hydrocarbon type, heteroatomic species, treatment processes, and metal surfaces. Thus, as the thermo-oxidative degradation of QAV is closely linked to its chemical composition, when trying to introduce a more sustainable and renewable fuel source, such as aviation bio-kerosene, its thermal stability needs to be studied and approved.

The deposits have a complex composition, presenting carbon, hydrogen, oxygen, sulfur and nitrogen. They are made of all insoluble materials in the fuel. A phenolic resin binds them together and gives the deposit the adhesive characteristic. Sulfur and nitrogen appear to be imprisoned in the deposit as insoluble salts. Phenols also have antioxidant properties, inhibiting free radical oxidation. This creates an anomalous relation between the rate of oxidation and the formation of deposits, as fuels with less stability oxidize more slowly and those without phenols oxidize more quickly and have less potential for deposit formation. (Naegeli, 1997).

The formation of insoluble solid deposits is complex and depends on several factors, (Taylor, 1979) proposed a mechanism for the formation of the general deposit. Initially, there is an aviation kerosene, with its characteristics and properties. It is subjected to the influence of the test environment and suffers oxidation. This kerosene starts to form deposit precursors in its composition, promoting the process of degradation and formation of soluble deposits. There is incorporation of insoluble materials and formation of insoluble deposits. Finally, deposition occurs from the agglomeration of insoluble deposits that are taken to the inner walls, encrusting and growing.

Tests on real scales would take a long time before the effects of instability could be studied. And that way, they would consume high amounts of fuel. HiReTS is a quantitative test for determining thermal stability on a small scale, in which the test uses a turbulent flow, thus being more faithful to the real system. It was developed in the 1990s by the Universities of Sheffield and Leeds in partnership with Shell (Wilson *et al.*, 2010). When compared to full scale tests, HiReTS uses a smaller amount of fuel and in a shorter time. A 125 min complete test consumes 5 liters of fuel. Compared to JFTOT, which uses laminar flow, in addition to turbulent flow to better describe the conditions of the original system, HiReTS also provides a quantitative analysis, while JFTOT provides a qualitative analysis and a color scale Brown *et al.* (2005).

The method uses Joule effect to heat the test section. An electrical voltage is applied to a capillary tube of constant cross section, which ensures uniform heat flow condition Mielczarek (2015). Heating is controlled through the fuel outlet temperature which must be kept constant throughout the test (Gadsby, 2017). As the incrustation grows inside the test tube, there is an increase in thermal conduction resistance, because the deposits form a layer of thermal insulation that makes it difficult to transfer heat from the tube to the flowing fuel. As the fuel temperature at the exit of the test section must be kept constant, the outside tube temperature increases. The increase in temperature over time is measured using an optical pyrometer, in pre-established positions (Khodabandeh and Frederick, 2005).

Thereby, the main purpose of this work is to measure the solid waste deposition mass in high Reynolds number in degradation test as a function of analysis time and relate it to the HN generated in the test. Also, stabilize a profile of the solid deposit layer inside the capillary tubes as a function of the test parameters and operating time.

2. MATERIALS AND METHODS

2.1 Fuel

The fuel used to study the degradation inside the test capillary is a QAV sent to the HiReTS project in 2019 and was stored at ambient temperature until the period of testing, in 2021. At the Table 1 the main physicochemical characteristics of the evaluated kerosene are described.

Table 1. Fuel description

Experimental Tests	**Methods**		Kerosene 2010-006930-48
Density (20ºC), kg/m³	D4052		**815.9**
Distillation, ºC	D86	P.I.E.	**153,6**
		10 %v/v	**175.0**
		50 %v/v	**206.4**
		90 %v/v	**252.1**
		P.F.E.	**277.1**
Total Sulfur, mg/kg	D5453		**51**
Mercaptide Sulfur, mg/kg	D3227		**<2**
Sat + Olefins, %m/m	HPLC		**78.6**
Monoaromatics, %m/m			**19.4**
Diaromatics, %m/m			**1.9**
Polyaromatics, %m/m			**0.1**
Total Aromatics, %m/m			**21.4**
Aromatics, %v/v	D1319		**18.1**
Olefins, %v/v			**0.9**
Saturated, %v/v			**81.0**

2.2 HiReTS Experimental Bench

The experimental bench used was created based on the 60000-0 SETA HiReTS TESTER equipment (ASTM-D6811-02, 2007). Some modifications were made from the original system, such as add fuel recirculation, replace the acquisition and control system with a more modern one, change the fuel pump, replace the optical pyrometer for an infrared thermography camera, and include a line for extraction of the sample.

In the main test line, the fuel is pumped at a constant flow rate of 35 mL/min, from the sample vessel to the test capillary section. At the test section, the capillary was heated by Joule heating, and the temperature was measured by the infrared camera. After the fuel leaves the capillary at an increased temperature, it flows through a cooling system with a single heat exchanger pass. The backpressure valve is set at 20 bar in order to guarantee the system pressure. After passing through the back pressure valve, the fuel passes through the rotameter, where it is possible to check the system flow. Finally, it arrives at the waste vessel. If the recirculation valve is open, fuel returns to the sample vessel for continuous testing.

The capillary tube is connected to a power source and heated by Joule heating promoting the degradation of the QAV. The temperature of the fuel in the exit of the capillary tube is measured by a PT100 thermo-resistor. The power of the Joule heating is controlled to achieve a set-point temperature of 290 °C in the fuel flow at the outlet of the capillary tube. For this work the original tube of the equipment 60000-0 SETA HiReTS TESTER was used, (ASTM-D6811-02, 2007). The capillary tube is made of 316 stainless steel, 152 mm length, 1.66 mm outside diameter, and 0.26 mm inside diameter. It has a black paint coating on the center of the tube, in a range of 112 mm, leaving only 20 mm uncoated at both extremities which it is connected to the busbars.

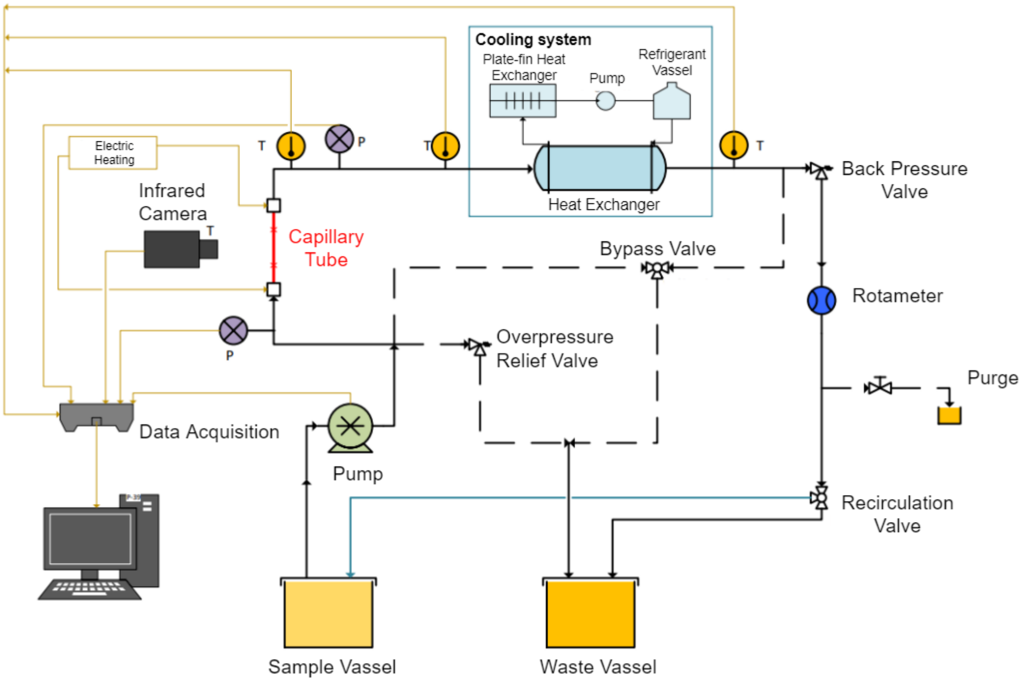


Figure 1. Test equipment process diagram, modified HiReTS bench.

2.3 HiReTS Experimental Procedure

All tests used the same fuel sample from the same gallon and were accomplished under the same test conditions, with variation only in the test duration, which were 15 min, 30 min, 45 min, and 55 min. The time tests were defined based on the temperature measured by the infrared camera and compared with the analysis made by (Gadsby, 2017) about the relation between the temperature and the thickness of the deposit formed in the capillary tube. Although the standard test suggests an analysis time of 125 min, in times longer than 55 min a thermo-oxidative degradation was observed so severe that it blocked the capillary tube, raising the system to maximum pressure. Thus for QAV evaluated it was not possible to use longer times.

For each test was used 700mL of QAV and a set of capillary tube with unique washers and nuts installed in the equipment. The system flow was set to 35000 μL/min and the QAV inlet temperature in the capillary tube was gradually heated and the test started when this temperature was 290 ºC, kept constant throughout the test. At the end of the test, the used capillary tube and the insoluble deposits adhered to the inner wall were identified and separated for further evaluation of the deposits. For each test performed in the HiReTS, a capillary sample with deposits inside was obtained, each one was identified with numbers, according to the nomenclature described in Table 2 corresponding to the test time.

Table 2. Capillary nomenclature with test time.

Capillary	Test time
C1	15 min
C2	30 min
C3	45 min
C4	55 min
C5	30 min
C6	Without test

Given the temperature measurements along the capillary tube positions as a function of the test time, it is possible to generate a three-dimensional graph. Thus, from the graph it is possible to develop an index that measures the thermal stability of fuel, it is called HiReTS Number or Total HiReTS Number (HN). The value registered is the sum of the differences between the temperature measured at certain positions on the tube surface at the end of the test and the minimum temperature measured during the test, as described in equation (1). In general, the temperature at the beginning of the tube at the beginning of the test (Wilson *et al.*, 2010).

$$\mathrm{HN} = \sum_{n=1}^{i} (\Delta T_{final} - \Delta T_{min}) \tag{1}$$

2.4 Insoluble Deposit Characterization

To characterize the deposit, two measurement approaches were defined after the test in the HiReTS equipment. Measuring the mass in an electronic microbalance and evaluate the thickness of the deposit formed inside the capillary by interferometry and optical microscope.

2.4.1 Determination of deposit mass

Before evaluating the deposits formed inside the capillary tubes, the black paint coated was removed. After removing the external paint, the capillary tube was washed with distilled water and absolute ethanol three times and dried in an oven for one hour at a temperature of 100 °C. This step is performed in order to remove the remaining QAV from the test along with deposits not adhered to the capillary wall. To facilitate the measurement of the mass of deposits inside the tube, they were sectioned into four sections of 28 mm each. Each section was considered a sample for mass measurement, identified with letters, in addition to the capillary number previously specified. Figure 2 represents each part of the section created according to the direction of fluid flow.

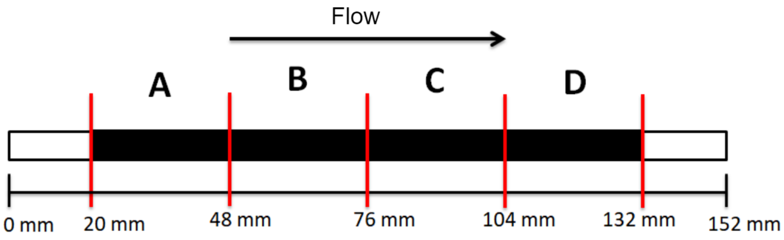


Figure 2. Test capillary and samples created for analysis.

The mass deposited was obtained in an electronic microbalance, for each section, by the difference between the tube before and after the deposit removal. The removal was performed according to the methodology proposed by (Pei and Hou, 2016), where the capillary tubes were subjected to an ultrasonic bath for 2 hours, immersed in a solution of $KMnO_4$ (125 mg/L). After this period, the samples were washed with distilled water and ethanol and dried in an oven at 100 ºC for 1 hour.

2.4.2 Characterization of the test capillary tube and the deposit

The characterization will be implemented in the same region of temperature analysis in the HiReTS experimental bench, which corresponds to section D of the capillary. The C2 test, which obtained as a previous result the greatest number of deposit mass, was replicated with the same experimental procedure and the same test time, obtaining the C5 capillary. The same procedure for mass determination was executed, even before the first mass measurement on the microbalance, for capillary C5 and for an unused capillary C6. Thus, three samples were obtained, C2D, C5D, and C6D that correspond to capillary 2, 5, and 6 in section D to be analyzed.

The samples (C2D, C5D, and C6D) were embedded in resin without allowing resin penetration inside the capillary and sanded. After sanding, the samples were cleaned with distilled water, dried at ambient temperature for 48 hours, and stored for later image analysis.

After embedding, the three samples were taken for analysis by optical interferometry in Quickvision Apex 302 and analysis in optical microscopy in the Dino Lite equipment with 200 times scale.

In addition to the three samples described above, a capillary section used in a two-hour standard test with the same fuel a year before the analyzes of this work was also embedded. It was called zero capillary. For this inlay, there was a penetration of the resin inside the ends of the capillary. Two samples were created, one with a cross-section and the other longitudinal. These samples were visualized in a Leica DM4000 M optical microscope and in an optical interferometer.

3. RESULTS AND DISCUSSION

3.1 Results after testing on the HiReTS experimental bench

The calculated values for the HN are shown in the Table 3. It was expected that from C1 to C4 the HN would gradually increase in value, and in C5 it would be a value close to C2. Because the test time is longer and the degradation is greater over time. The HN value for the C2 test proved to be extremely higher when compared to C5. In the C2 test, the fuel would already present instability regarding thermal stress, since the HN is greater than 1000 Emens *et al.* (2004).

The temperature values used in the calculation of HN are those of the D section of the capillary. Thus, the HN value does not account for deposits formed in the central region of the capillary, as well as deposit detachments that may occur during the HiReTS test. In the literature, HN values > 1000 for the standard 120 min test indicate that the sample is unstable. In the test performed in 30 minutes, the HN found was greater than 1000, indicating the high instability of the sample used.

The repeatability of the HiReTS tests reported in the literature decreases for low HN values, characteristic of thermally stable samples Brown *et al.* (2005). During this study, in which a sample with low stability was used, the repeatability of the tests was not obtained, also reflected in the dispersion observed in the HiReTS numbers found. Possible causes for the variability are the variation in internal roughness from one capillary to another and difficulties in controlling the tests due to the formation of deposits, causing obstructions and pressure variations.

Table 3. Calculation of the Number of HiReTS.

Capillary	HN
C1	193
C2	1129
C3	387
C4	976
C5	259
C6	-

Figure 3 shows the result of the HiReTS test for a time of 45 minutes. It is possible to notice a behavior similar to the standard test in the literature, the temperature difference is noticed at all points, and it is slightly larger at the outflow. Some variations in the temperature difference can be seen throughout the test, with some decreases at different times. Which may be an indication of detachment from the deposit. When displacing the deposit, the "capillary + deposit" thickness decreases, which modifies the resistance and consequently the measured external temperature. In this way, the outside temperature decreases and the difference with the lowest temperature during the test becomes smaller. However, in general, the temperature difference grows with the testing time. Thus, it is possible to indicate that in the C3 test there was no strong interference of the capillary and the formation of deposits increased with the flow. Thus, an increase in mass of sections from A to D is expected.

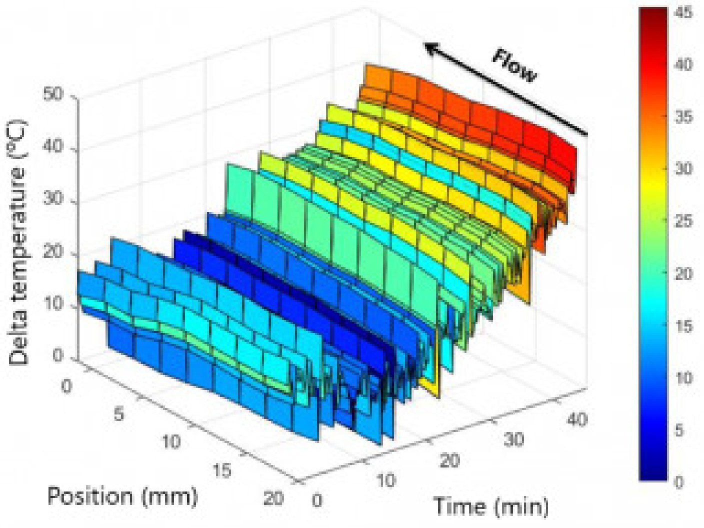


Figure 3. Temperature difference as a function of position along the tube and test time for sample C3.

3.2 Determination of deposit mass

The measured mass values for the evaluated samples are shown in Figure 4. Each letter of the label is related to the respective section of the capillary shown in Figure 2. The section of capillary tubes was necessary for the upper limit of the mass set not exceed the limit of the micro-balance, but that the measuring range of the balance could still detect the mass measurement without difficulties and large errors. The measurement error considered, which can be measured, was the micro-balance error of 0.002 mg. The measurement error is smaller than the size of the symbol that shows the measurement value obtained in the graph. However, as seen before, not all tests on the HiReTS equipment had good repeatability, which can directly affect the mass measurement.

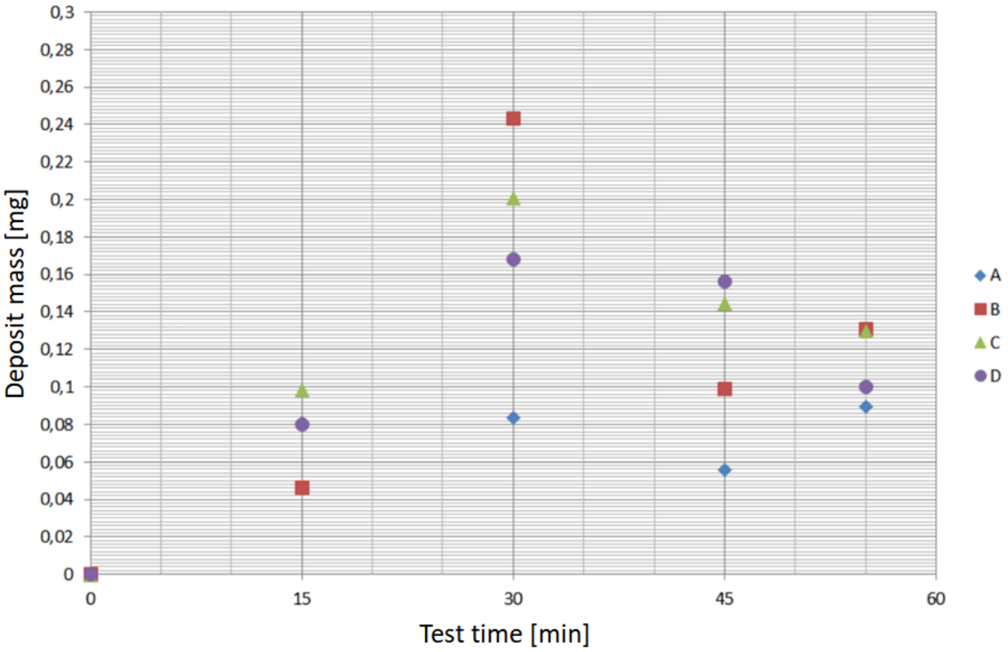


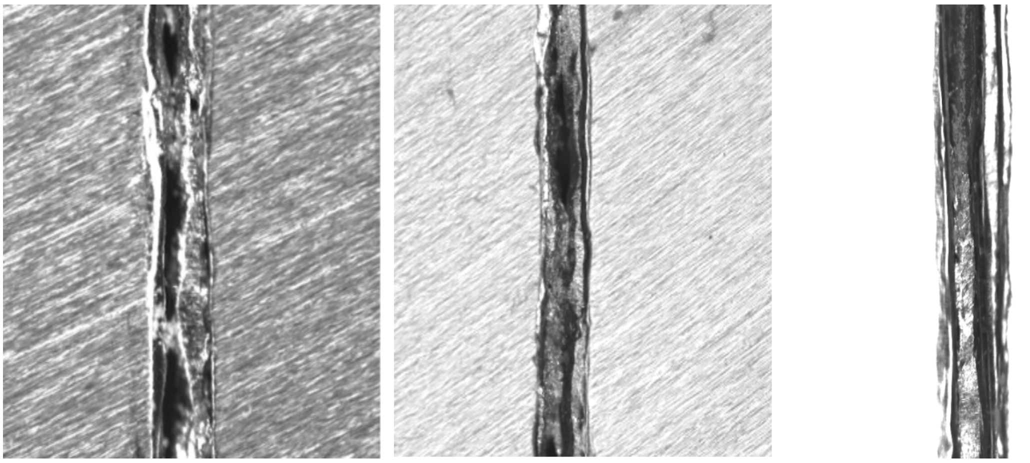
Figure 4. Determination of deposit mass by test time for each capillary section with all points on the same graph.

The graph in Figure 4 when observing the test time axis presents four curves with the accentuated growth of deposit at the beginning of the capillary and potentially a limitation of oxygen transport to the boundary layer close to the wall in the posterior regions, or deposit displacement by mechanical action of the flow.

3.3 Test Capillary Imaging Evaluation

In this step, it was intended to analyze by image three samples C2, C5, and C6 in the same test section. C2 is a section of the capillary tube after the entire mass analysis procedure, C5 is a copy of the C2 test, with the same thermal stress parameters but without removing the deposit from its interior, and C6 that is an unused capillary. The purpose of evaluating the new capillary tube was to visualize and measure the internal tube wall roughness. In addition to using it for comparison with the clean capillary in a potassium permanganate solution and an ultrasound bath, in order to check whether the deposit has been completely removed. With the section that did not remove the encrustation, the objective was to visualize the deposit and measure its thickness.

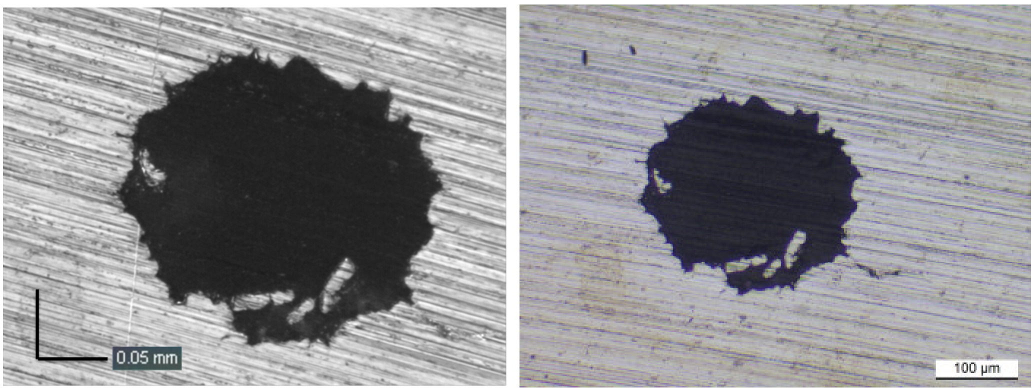
Figure 5 shows the images generated for the three samples $C2D$,$C5D$ and $C6D$ by the optical interferometer. From these samples, it was not possible to measure the roughness of the capillary inner wall through the method, nor to detect the presence of the thickness of the deposit. As shown in the same figure, because of the very irregular characteristic of the inner surface of the capillary wall, the images formed by the light are confused and similar to each other, not allowing the identification in the image of the portion referring to the capillary wall or to the deposits. It was expected that when placed for measurement side by side, there could be a comparison between the capillary with deposits ($C5D$), new ($C6D$) and clean ($C2D$) by the methodology used to measure the mass. One of the hypotheses of the non-visualization and non-measurement of deposits by the interferometer may be the low time of the analyzed test, Pei *et al.* (2018) only characterized the deposits in an electron microscopy image after 60 minutes of testing, time in which the formation was already much denser. Thus, even with deposition, it is still not measurable by the methods applied in this work.



(a) Interferometry imaging in the C2D capillary. (b) Interferometry imaging in the C5D capillary. (c) Interferometry imaging in the C6D capillary.

Figure 5. Result of optical interferometry on samples $C2D, C5DeC6D$.

When observing by optical interferometry the cross section of the zero capillary, shown in Figure 6, it was possible to visualize a deposit layer in some parts of the capillary wall. Using the equipment's own image processing, the thickness of 0.065 mm and also the diameter of the capillary of 0.24 mm were measured. The same samples observed under the optical microscope reveal that the capillary diameter can be approximated by an ellipse with minimum and maximum diameters of 0.241 mm and 0.254 mm, respectively. However, the microscope image reveals that the thickness of the deposit is in the order of micrometers, therefore, 10 times smaller than the value measured by interferometry. Thus, it is concluded that measurement by interferometry was not effective for the capillaries used.

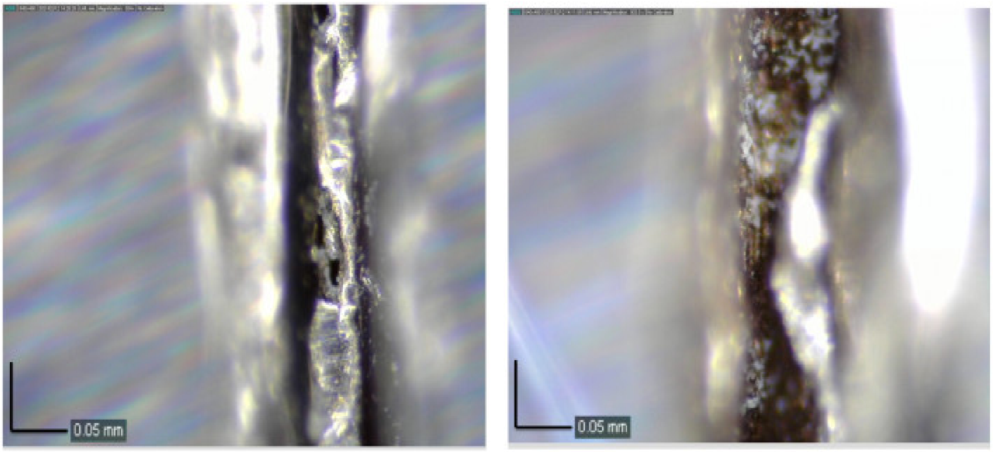


(a) Optical interferometry image of the capillary cross section.

(b) Optical microscopy of the cross-section of the capillary, 200x amplification.

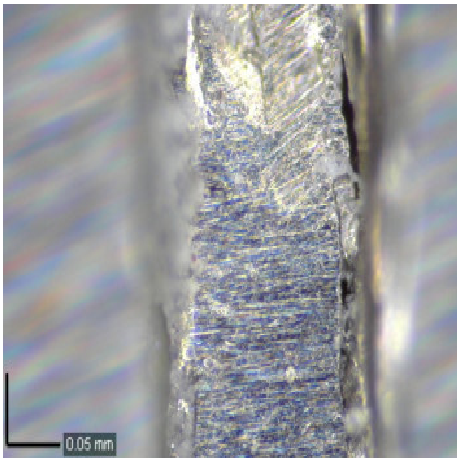
Figure 6. Measurement of deposit thickness by optical interferometry and optical microscopy from the capillary cross section for the C0 test.

The same three samples $C2D$,$C5D$ and $C6D$ were also analyzed using the optical microscope Dino Lite. This equipment has a high amplification, up to 200x, and it is possible to view the image in color. Because it's a small, handheld microscope, it's easier to look at the entire sample with it, looking for patterns in each one. Figure 7 illustrates the results of images obtained by him.



(a) Optical microscopy image obtained from the Dino Lite of the C2D Capillary.

(b) Optical microscopy image obtained from the Dino Lite Capillary C5D.



(c) Optical microscopy image obtained from the Dino Lite Capillary C6D.

Figure 7. Optical microscopy of the inner wall in capillaries showing the capillary without deposits in (a) after removal, deposit formation in (b) and an unused capillary in (c).

In these three images, it is possible to compare each capillary sample of the three proposed situations. Figure 7 (c) identifies the unused capillary (C6D), which has no signs of deposition in its interior. Figure 7 (a) corresponds to the capillary that was submitted to 30 minutes of testing and had its deposits removed using the methodology discussed above. This image is very similar to the one found in C6D, it has some dark spots, however they are also found in the C6D capillary, which indicates that they are not deposits. Confirmation that the dark spots are not in fact deposits was analyzed using Figure 7 (b), which corresponds to the 30-minute test with deposits, and many spots of completely different pattern and color are identified in it.

In Figure 7 (b) it is possible to identify the deposit and its color. In this type of equipment, it was not possible to measure the deposit thickness. However, it can be seen that the deposition is very similar to that proposed by the JFTOT methodology, with a classification between 3 and 4, a deposit with a light to dark tan, which by the JFTOT methodology would indicate an unapproved fuel because it is thermally unstable ASTM (2006). Another pattern that can be observed from Figure 7 (b), is the inhomogeneity of the wall deposit, which does not form a film on the entire surface of the wall, but only in some points.

4. CONCLUSIONS

This work focused on the determination of deposit mass using the HiReTS method, used to quantify the thermal stability of aviation fuels. The HiReTS method presents a quantitative result in terms of the number of HiReTS (HN). The objective of this work was to apply methods for determining the mass and thickness of the deposit layer that can be associated with the standard HiReTS method.

The mass measurement method was applied to determine the deposition mass over time and capillary length. The gravimetric method of mass determination is limited by the sensitivity of the microbalance used. An initial estimate recommended cutting the capillary into 4 longitudinal sections, a procedure that has proven effective. The image-based method, using interferometry and optical imaging, was used to characterize the cross-section of the capillary and to estimate the thickness of the deposit. In the tests analyzed, samples of fuel with low thermal stability were used, in order to evidence the formation of deposits.

From the analysis of the results obtained during the work, it can be concluded that:

- The gravimetric method of mass measurement proved to be effective for the tests performed. The measurement uncertainty is less than 1% of the measured deposit mass values in 28 mm long sections (1/4 of the capillary length);

- The image analysis of a capillary, after removal of the deposit formed in a test, shows the same characteristics as a new capillary, showing that the deposit removal by chemical pickling is effective;

- The image of the inner surface of the capillary after the 30-minute test shows the presence of insoluble deposits. The image is similar to the JFTOT method, showing a light to dark tan color, in classification 3 and 4. According to the JFTOT methodology, this color would indicate a thermally unstable fuel. In the situation analyzed, HN = 259 was found;

- It was not possible to find values for the thickness of the deposit by the optical interferometry method applied in this work for tests with time up to 55 min. A possible hypothesis is the existence of irregularities in the inner wall of the capillary, which reflect light in an extremely irregular way, not allowing measurement by the interferometer.

4.1 Future Works

- Using a capillary with a larger internal diameter, keeping the same Reynolds number through the mass flow;

- Choose commercial tubes with lower internal roughness to allow analysis with an optical interferometer. One possibility is the capillary tubes used in liquid chromatography equipment, which have less internal roughness;

- Improve the control system in order to eliminate problems that could affect test repeatability;

- Use scanning electron microscopy to measure deposit thickness.

5. ACKNOWLEDGEMENTS

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