NEW IN-LINE VISCOSITY MEASUREMENT FOR VERY HIGH VISCOSITY PRODUCTS APPLICATION TO PLASTIC AND ELASTOMER EXTRUDER

Dr. Luc K. Bellière
Sofraser
Villemandeur, France

KEYWORDS

VISCOSITY, VISCOMETER, INJECTION, EXTRUSION, POLYMERS, PLASTICS, cP, mPa.s

ABSTRACT

Numerous materials such as polymers are solids under standard conditions but pasty in process conditions. In a laboratory at ambient temperature and low shear rates, these products can have viscosities comprised between 10^3 Pa.s and 10^{12} Pa.s. These polymers, plastics or elastomers must have the capacity to flow in the process and have viscosities inferior to 10^3 Pa.s in order to be extruded and shaped. This is made possible with the effects of temperature and shear rate on the properties of these Non-Newtonian products. Typical products concerned are polymers for extrusion and injection.

The plastic extrusion process applies to liquid and melted polymers. It starts with the material being fed to the extruder, melted and then pushed through the system by a rotating screw. The liquid polymer then goes into a die where it is extruded under high pressure according to a molding which will give a specific shape to the final part. Adjustments of the ingredients in the polymer, melting temperature, extrusion speed and pressure are many of the parameters influencing the final quality of the extruded polymer. Viscosity is a physical property of these non-Newtonian polymers and is correlated to intrinsic viscosity, melt flow index and others. In-line viscosity control of the melted polymer before the extrusion die is the ideal solution to obtain precious information in order to set up and adjust the optimum extrusion parameters, and consequently realize process savings in material, labor, time and money.

In this paper will be reviewed the state of the art of available technologies for viscosity measurement of very high viscosities in extrusion processes. Since none of the existing

solutions provide in-line systems including a viscosity sensor, we will describe the idea of using the vibrating principle at resonance frequency, a renowned principle in viscosity measurement, inside the extrusion pipe. This paper will then describe the design of the sensor, its integration in extrusion dies and provide results on very high viscosity fluids. Finally, we will consider potential applications of this technology in very high viscosities petroleum products (asphalts, bitumens, residues…).

INTRODUCTION

Extrusion and injection processes are some of the most common methods used to give shape to polymers and process plastics into finished products. The operation of extruders requires heavy use of rheology principles and analysis, as temperature, pressure and composition will influence greatly the efficiency of such equipments. One of the properties within rheology which also impacts operations of extruders is viscosity. Unfortunately, there are a limited number of equipments capable of accurately measuring viscosity in extrusion conditions and none of them are in-line with the extruder.

The work described in this paper stems from a collaborative project realized on the thematic of the multi-parametric analysis of elastomers mixing and extrusion processes [1]. This project involved different companies whose global objective was to develop a monitoring system capable of early detection of eventual dysfunctions during fabrication processes and for production equipments.

The company has focused its research work on the development of an in-line viscous material characterization system based on the vibrating technology at resonance frequency that could be dedicated to the production, extrusion and injection of polymers. The challenge was to conceive the first in-line viscosity sensor for very high viscosities and demonstrate the pertinence of the vibrating technology at resonance frequency for materials other than liquids.

1- STATE OF THE ART OF VERY HIGH VISCOSITIES PRODUCTS MEASUREMENT SOLUTIONS IN PROCESS

In 1981, Sofraser created and patented the first vibrating viscometer using the vibrating technology at resonance frequency (Patent N° FR 2 544 496) [2].

Today, vibrating viscometers are recognized worldwide as the optimal solution to measure viscosity in process. The sensor response time is close to zero, and the viscosity information (stability or variation) is available continuously. This allows the control of processes even in the presence of transitory phenomenon or rapid disturbances. The absence of moving parts avoids wear, guarantees no drift in time and no maintenance.

The vibrating viscometer at resonance frequency is a sensor working at a rather high shear rate to reduce measurement fluctuations due to fluid speed or flow rate when the product is pseudo-plastic or shear-thinning and, is perfectly adapted to process measurements. This process viscometer is able to measure viscosities up to one million centipoises (cP). However from five 500,000 cP onward, the value is linearized.

For extruders, there are rheological methods to determine the viscosity of the extruded product by measuring a pressure difference. However these methods cannot be used in a production environment.

In extrusion processes, when viscosity exceeds that range, industrials are forced to use different measurement techniques based on the capillary principle. The technology used by capillary systems measures the pressure difference between the inlet and the outlet of the capillary (Figure 1). They have the advantage of being compact, are fragile and prone to fouling.

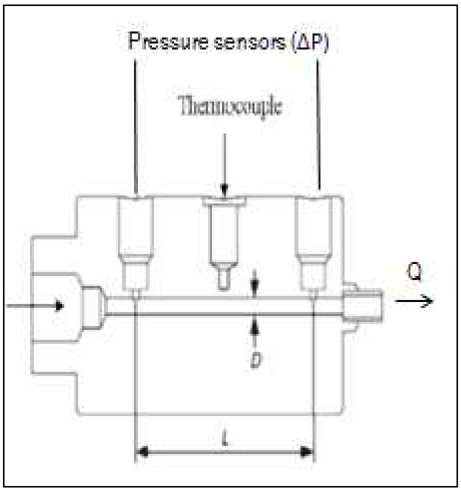

FIGURE 1. SCHEMATIC PRINCIPLE OF THE CAPILLARY SENSOR

In those systems, dynamic viscosity is calculated according to the following expression.

$$Dynamic\ viscosity = \frac{{}^{\Delta P}/_{L} \cdot {}^{D}/_{4}}{{}^{Q}/_{2\pi DR^3}}$$

Where:

η = Dynamic viscosity (Pa.s)

P = Pressure (N/m)

L = Length between two pressure sensors (m)

D = Capillary tube diameter (m)

Q = Flow rate (m^3/s)

Other systems based on the same capillary technology are available on the market to measure the viscosity of highly viscous products in extrusion or injection processes. They are online systems, meaning that they use a derivation from the main line. The product used for the derivation may or may not return to the process [2].

Only high-value systems (from 50+ K€ / $70K+) provide satisfactory results and are merely used by plastic or recycled plastic manufacturers. These systems are also widely used by manufacturers having regular modifications of their recipes. From these considerations, it seems that only the vibrating technology offers an alternative answer to this very high viscosity measurement market with a direct solution in the stream.

2- DESIGN OF A SPECIFIC PROCESS VISCOMETER FOR VERY HIGH VISCOSITIES AND INTEGRATION IN EXISTING INSTALLATIONS

2.1 SCIENTIFIC AND TECHNOLOGICAL REQUIREMENTS

The considerations that have been taken into account before proceeding to the design of a new sensor were the following:

- Developing a sensor resistant to the very high viscosities, pressure and temperature conditions used in polymer extruders and mixing tanks while maintaining sufficient sensitivity to the viscosity of the material. Conditions required the ability to reach pressures up to 300 bar / 4530 psi and temperature up to 250 °C / 480 °F.

- Developing a sensor for direct insertion inside the main pipe stream with the vibrating rod parallel to the flow of the product. Practically speaking, given the viscosities targeted in this project, it was nearly impossible to consider having the vibrating rod of the sensor perpendicular to the flow, as the flow rate would have too much influence on the measurement.

The main technological obstacle was to design a sensor robust enough to be installed inside the extruder or mixing tank while maintaining the sensor's sensitivity, repeatability and the thermal drift correction.

Once identified, the main requirements were transformed into a designing and experimentation plan which will be described in the next sections.

2.2 PRESENTATION OF THE VIBRATING TECHNOLOGY USED

The principle of the vibrating viscometer at resonance frequency is simple. The active part of the sensor is a vibrating rod held in oscillation at its resonance frequency. The vibration amplitude of this movement varies according to the viscosity of the product in which the rod is immersed.

The motion of the rod is created by a magnet fixed on the rod and placed in front of a coil driven by an alternative current. Another magnet attached to the rod induces a current in a separate coil which is an image of the motion of the rod (Figure 2). The resulting voltage amplitude is an image of the viscosity (Figure 3).

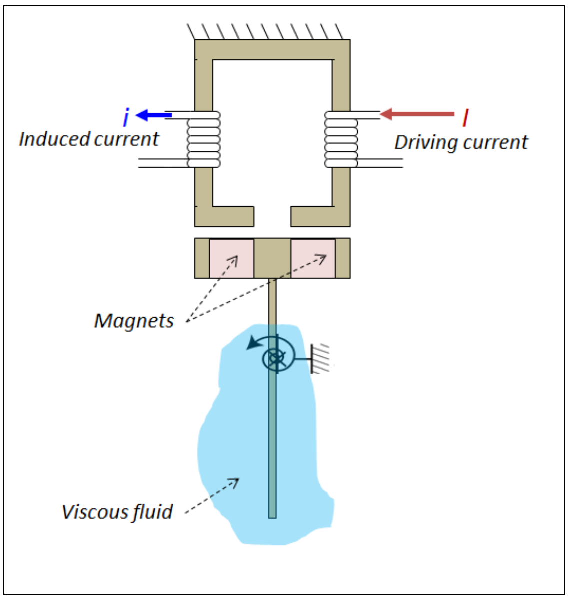

FIGURE 2. PRINCIPLE OF THE VIBRATING VISCOSITY SENSOR AT RESONANCE FREQUENCY

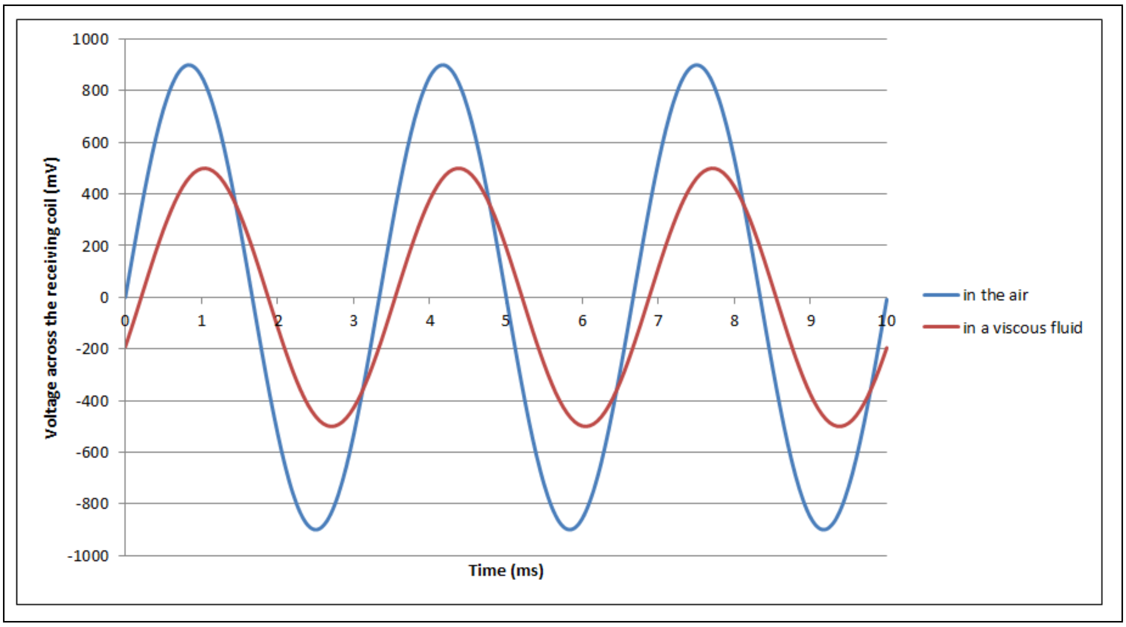

FIGURE 3. AMPLITUDE DIFFERENCE IN AIR AND IN VISCOUS FLUID

During calibration, the amplitude of the vibration is correlated to the viscosity of the product by comparing the vibration in the air (maximum vibration) and in the viscous fluid (Figure. 3), thus providing a reliable, repeatable and continuous viscosity measurement. This principle is described in Patents FR 2 911 188 B1 [3] and FR 2 921 726 B1 [4]

2.3 VISCOSITY SENSOR DESIGN ADAPTATION AND INTEGRATION INTO THE EXTRUDER

As previously commented, one of the constraints of the in-line measurement in the extruder was also to position the vibrating element parallel to the flow.

For standard installations of the viscosity sensor on process pipes, one of the most common mounting is the pipe elbow mounting. It is the recommended mounting as it provides an optimum flow circulation parallel to the sensing zone of the vibrating rod (Figure. 4).

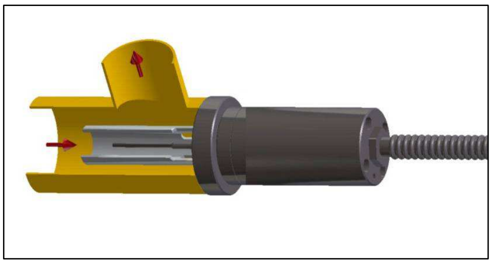

FIGURE 4. USUAL PIPE ANGLE MOUNTING FOR VISCOSITY SENSOR

This typical mounting is not possible on the targeted systems as extrusion and injection systems do not present any angle.
A special straight-line mounting of the viscosity sensor could be imagined with a special protecting tube design for the rod. This would ensure correct fluid circulation, as illustrated on the simulation of the Figure 5.

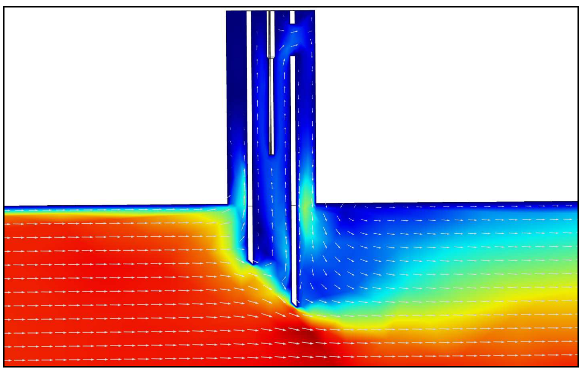

FIGURE 5. SPECIAL STRAIGHTLINE MOUNTING FOR VISCOSITY SENSOR

Although this mounting could be of interest for the extruder, it has a limited viscosity range and would not allow for efficient vibration of the sensing element in very high viscosities.

This is the reason why a new geometry design of the sensor had to be created to be able to place the equipment in line, directly in the stream of the extruder.

In order to install it in the harsh environment of the extruder, the actual sensor design has been developed with a new geometry impacting its global external shape. The external shape has been designed to facilitate the fluid's flow inside the pipe, such as illustrated in Figure 6 (Demand of patent FR13 63 507 deposited) [5].

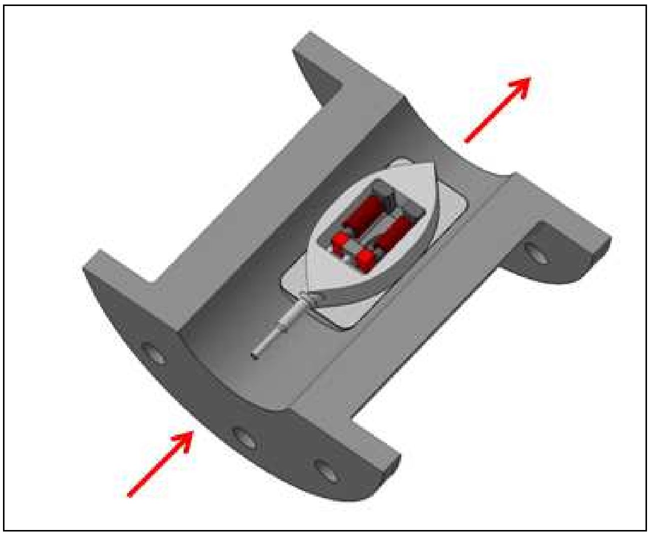

FIGURE 6. SHAPE OF THE NEW VISCOSITY SENSOR FOR EXTRUDER

The sensor occupies minimum space inside the pipe (see Figure 7) and its shape does not alter the flow of the fluid.

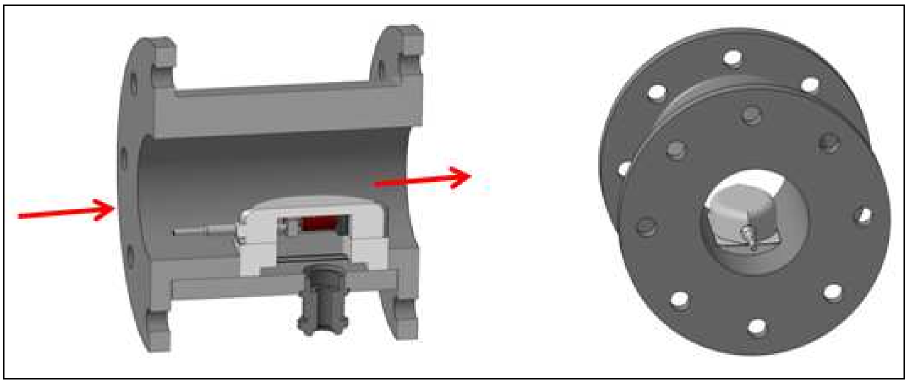

FIGURE 7. NEW VISCOSITY SENSOR FOR EXTRUDER

In order to be integrated on the extruder, the complete sensor is fixed with a connecting piece customized to the extruder's dimensions which ensures ease-of-insertion before the extrusion die (Figure 8). As it is attached with a clamp, the viscosity sensor can easily be mounted and dismounted from the extruder. A prototype has been designed to be implemented inside an extruder with a 60 mm diameter, as illustrated in below Figure 9.

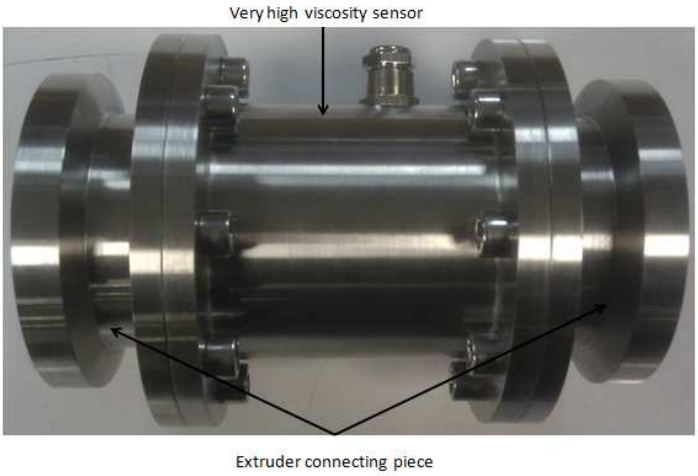

FIGURE 8. NEW VISCOSITY SENSOR FOR EXTRUDER WITH CONNECTING PIECE

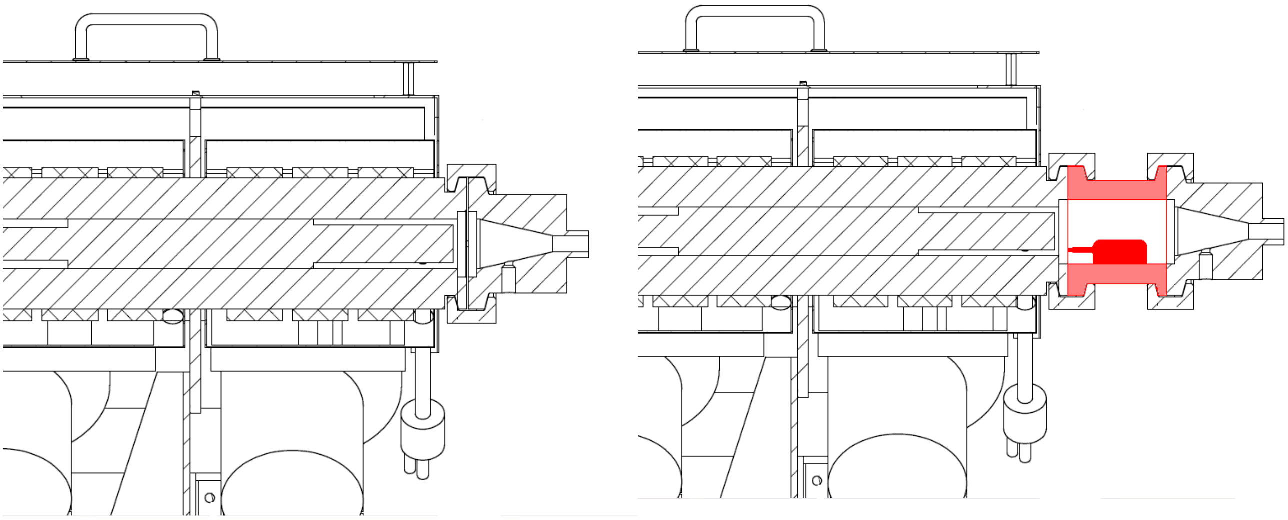

FIGURE 9. ILLUSTRATION VERY HIGH VISCOSITY SENSOR MOUNTING ON EXTRUDER

The connecting piece presents the advantage of being customizable, and thus fit multiple diameters on the extruder where it needs to be installed. Positioned right before the extrusion die and integrating a heating system, the connecting piece maintains the temperature of the very high viscosity fluid and measures its viscosity seconds before the extrusion.

3- RESULTS

3.1 CALIBRATION OF THE VERY HIGH VISCOSITY SENSOR

Once the very high viscosity sensor prototype had been created, it needed to be calibrated.

The vibrating rod was immersed in different calibration standard oils whose viscosity was precisely known at different temperatures. The response of the sensor was recorded and allowed establishing a relationship between the product viscosity and the sensor response.

The sensor was to be used at a temperature around 200°C / 400 °F but the behavior of the viscosity sensor is not identical at 200 °C / 400 °F than at ambient temperature. To correct for this drift, the response of the sensor wand heat cycles were performed in a stove to account for the effects of temperature.

In order to reduce the influence of temperature on the measurement and provide a better sensitivity to the measurement, it was chosen to drive the sensor with voltage rather than current. 3D fits were conducted for both current drive (Figure 10) and tension drive (Figure 11).

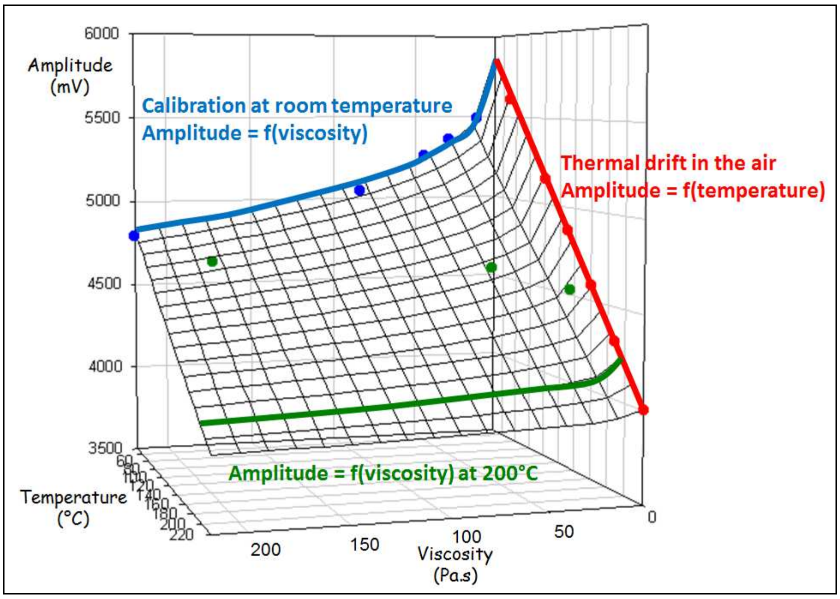

FIGURE 10. 3D FIT WITH CURRENT DRIVE

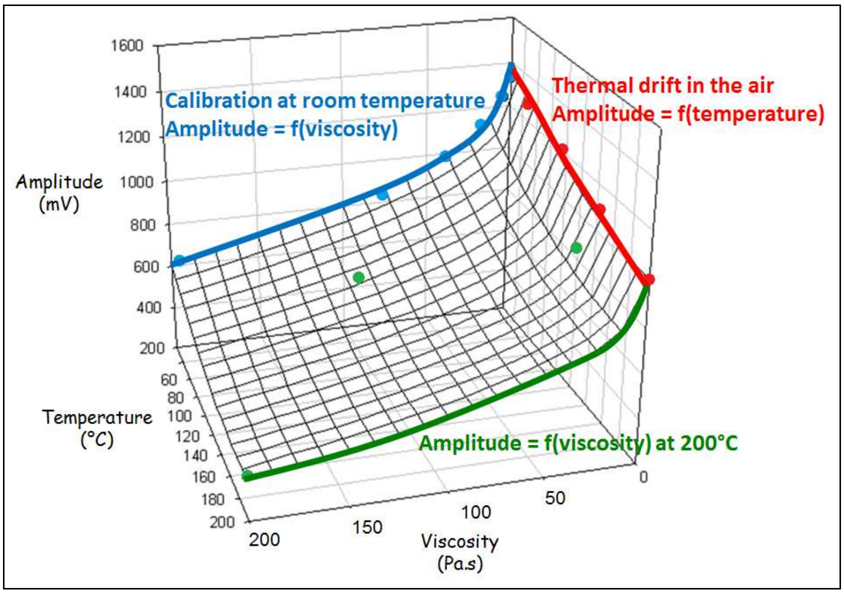

FIGURE 11. 3D FIT WITH VOLTAGE DRIVE

As the working range of the sensor is higher than the viscosity standards used, the last step of the calibration included a trial on the extruder with one reference product in order to be able to plot a final 3D fit. In this case the reference product A is a low density polyethylene (Figure 12).

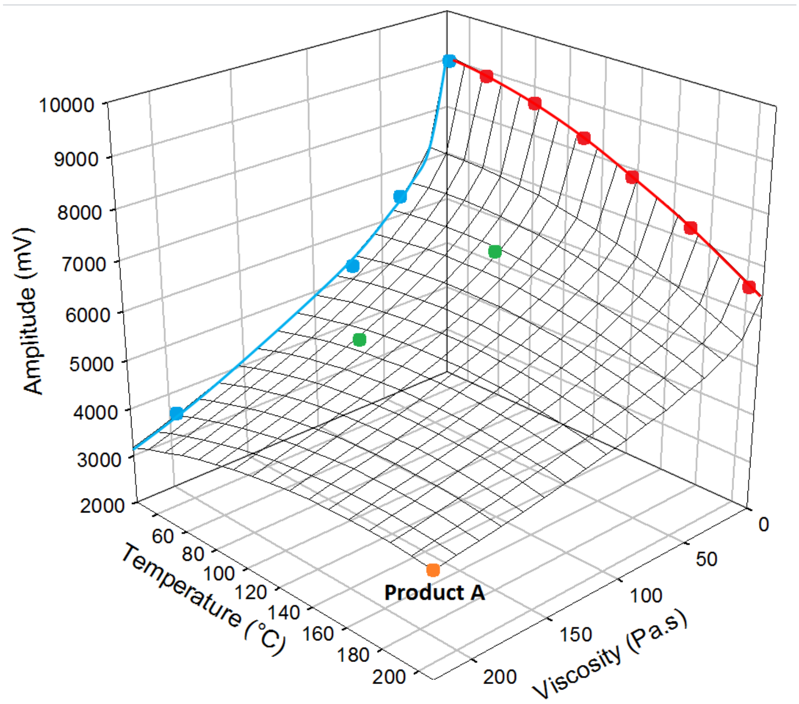

FIGURE 12. FINAL FIT 3D WITH REFERENCE PRODUCT TESTING

After establishing the final calibration curve, it was possible to validate the use of the very high viscosity sensor on the extruder with three different products.

3.2 RESULTS ON EXTRUDER WITH THREE VERY HIGH VISCOSITY PRODUCTS

The trials were performed on three different products B, C and D at constant temperature for with various extrusion speeds. The products used for calibration and trials are the following:

Product A: Low density polyethylene

Product B: Thermoplastic elastomer of confidential composition

Product C: Thermoplastic elastomer including a mixture of polypropylene and SEBS (Polystyrene-b-polyethylene butylenes-b-polystyrene)

Product D: Thermoplastic elastomer including a mixture of polypropylene and EPDM (Ethylene-Propylene-Diene Monomer)

The installation was done as described in the previous chapter (Figure 13), before the extrusion die of the extruder.

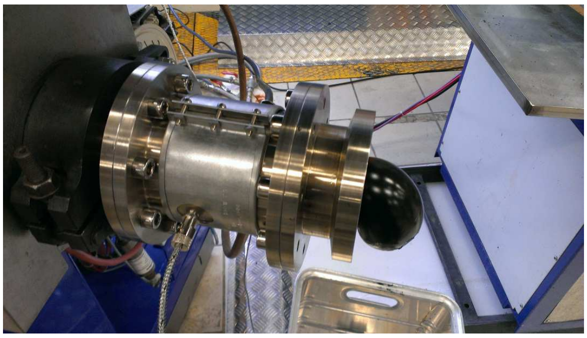

FIGURE 13. VISCOSITY SENSOR AND CONNECTING PIECE INSTALLATION ON EXTRUDER

The results obtained on the three products provide viscosity and temperature information on the materials (Figure 14).

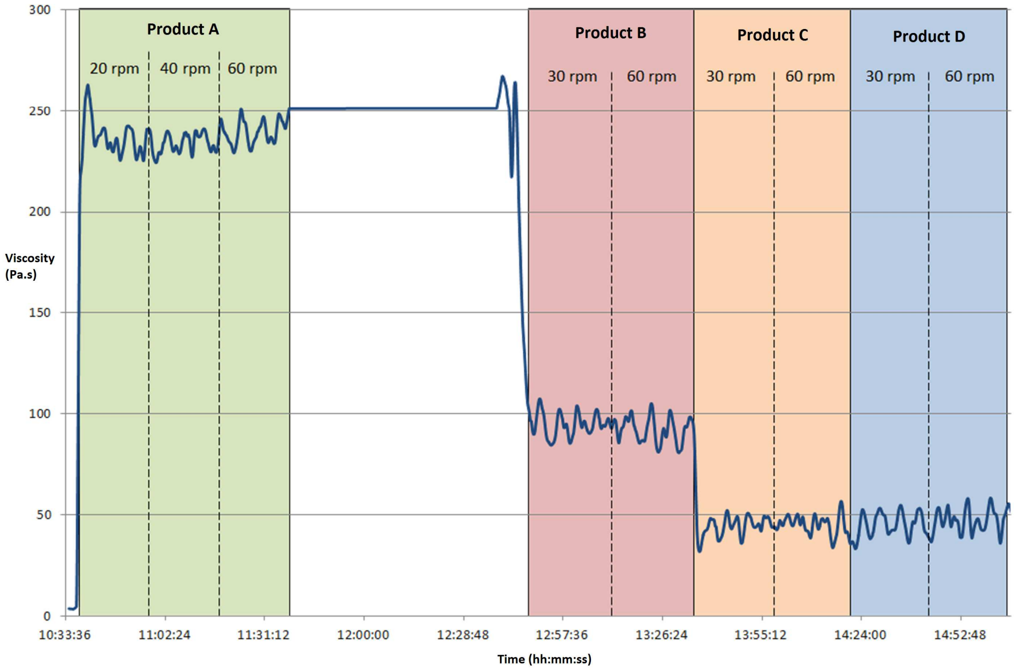

FIGURE 14. VISCOSITY RESULTS IN TIME FOR PRODUCTS A, B, C AND D.

Products B and C are very well differentiated by the viscosity sensor and products C and D have a similar viscosity. The results obtained with the in-line very high viscosity sensor were then compared to a laboratory rheometer. The results of the laboratory rheometer are following (Figure. 15).

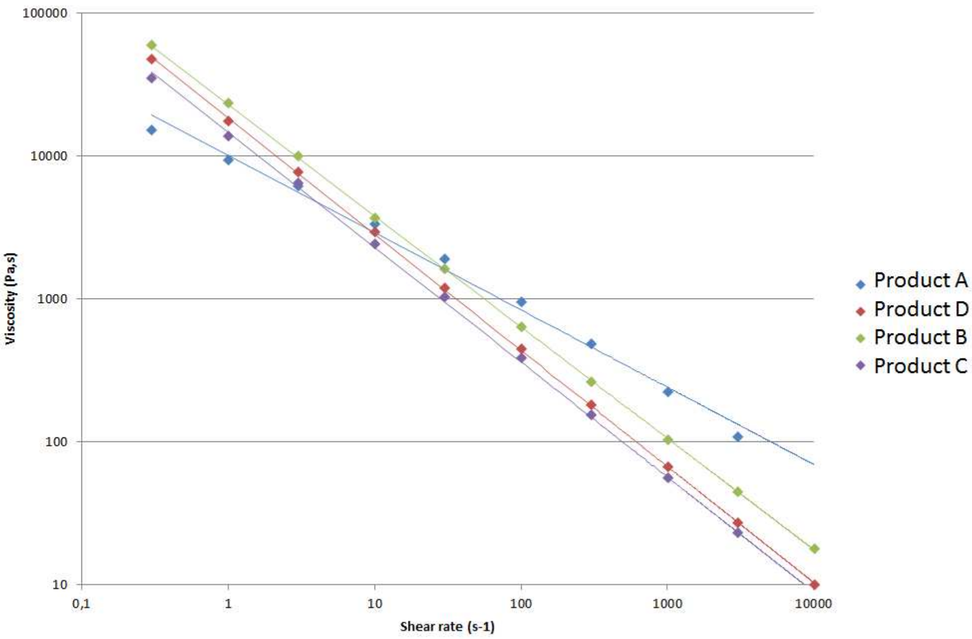

FIGURE 15. PRODUCTS A, B, C AND D VISCOSITY VS SHEAR RATE ON LABORATORY RHEOMETER

The vibrating viscometer at resonance frequency measures viscosity at a shear rate around 1000 s^{-1}, nevertheless, we made a large shear rate sweep with the rheometer to characterize the products at hand. On the results above, it seems that the products measured at a viscosity between 50 and 80 Pa.s at 1000 s^{-1} have a viscosity between 1000 and 10 000 Pa.s at near 0 shear rate. The comparison between the in-line viscometer and the laboratory capillary rheometer are given in the following graph (see Figure 16).

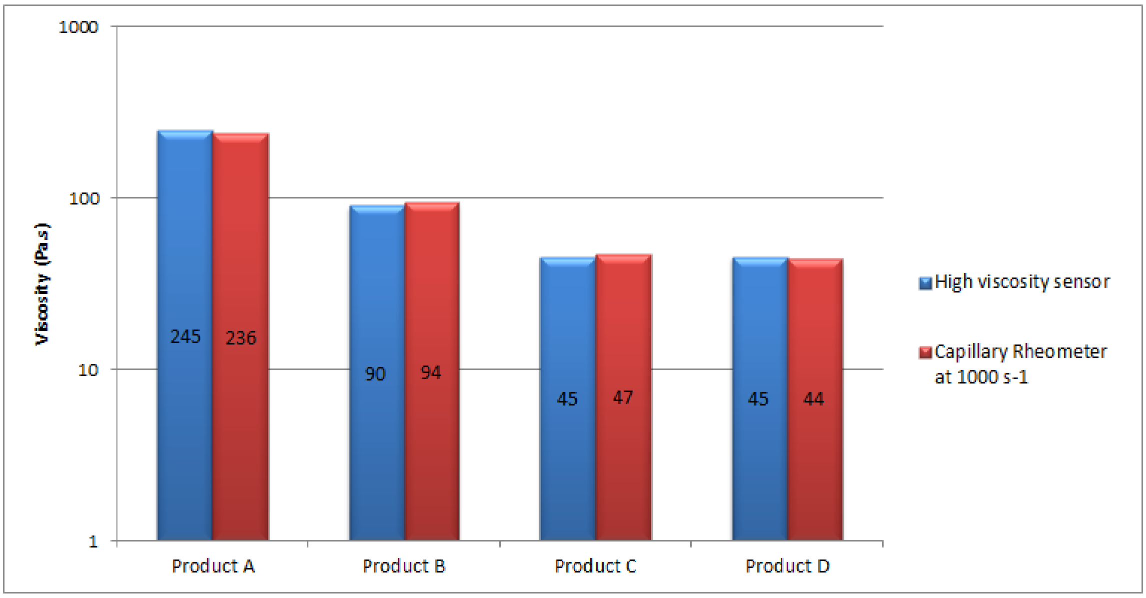

FIGURE 16. COMPARATIVE RESULTS WITH IN-LINE VISCOMETER AND OFFLINE CAPILLARY RHEOMETER

The in-line viscometer displayed measurements very close to what was measured by the rheometer with an error lower than 5%. The drift can be explained by a potential uncertainty on the exact shear rate in the extruder, due to the impact of the screw and flow variations. These results are extremely positive for the in-line process viscosity measurement of very high viscosity fluids in the extrusion stream.

CONCLUSIONS

The in-line viscometer designed for very high viscosity products measurement during extrusion processes reached its goal of being easy to integrate to extruders and provide reliable and continuous viscosity information during the extrusion process. Thus the viscometer allows for precise characterization of the product viscosity and allows for potential adjustments of speed, material and/or temperature depending on viscosity variation.

This principle is easily transposable to many other extrusion and injections processes, in the area of plastics, polymers, elastomers and even for petroleum products such as asphalts, bitumens or heavy residues.

Those results demonstrate the pertinence of the use of the vibrating technology at resonance frequency in the context of very high viscosity products. This new design has a pending patent and brings a new innovation in very high viscosity products measurement to today's market.

REFERENCES

[1] LAMPE²: L'Analyse Multiparamétrique des Procédés de mélangeage et d'Extrusion des Elastomères

[2] Goettfert, MBR / MBR-TD, Rheometer without melt return into the process, RTR / RTS-TD, Realtime rheometer with melt return and bypass

[3] FR2 911 188, Sofraser, "Procédé et système mettant en oeuvre un élément oscillant pour déterminer les caractéristiques physiques d'un produit".

[4] FR2 921 826, Sofraser, "Method and system for determining the viscosity of a product".

[5] FR 13 62 507, Sofraser, "Système et procédé de mesure en ligne de la viscosité d'un produit"