COMBINING TCV & CORRELATION, THE KEY TO IMPROVE PROCESS MONITORING

Mr. Stéphane MILLET/Mr. Kevin VANCAYZEELE/Dr. Philippe BURG/ Dr. Luc BELLIERE

Sofraser, Villemandeur, France

KEYWORDS

CONTINUOUS MEASUREMENTS - CORRELATION - EXPERIMENTAL DATA PROCESSING - IN LINE VISCOSITY MEASUREMENT – POLYMERIZATION - PROCESS MONITORING - REFERENCE TEMPERATURE – TEMPERATURE COMPENSATED VISCOSITY - VISCOSITY – VISCOMETER

ABSTRACT

Viscosity is one of the physical characteristics that defines fluid like liquids, gels and pastes. Depending on the process, product viscosity is either a key specification, indirectly related to another key characteristic or impacting the efficiency of the process that uses or processes it. Viscosity is essential to determine the quality of the products and/or the efficiency of the processes. The manufacturer will be able to characterize the product and adjust process parameters to optimize production and control the product's quality, for instance.

Sampling and laboratory measurement remain the industry standard practice for viscosity and quality controls. Laboratory measurements are usually performed at low shear rates and at ambient temperature. Depending on whether or not the viscosity is in the correct range, the manufacturing team may have to adjust process parameters. It is a time-consuming process with significant response time that may not be fully

representative of the product's viscosity while running through a line or process and its variations with time. The increase of automation and technological improvement led to the development of inline viscometers.

In-line viscosity measurements enable continuous measurement and offer real-time viscosity readings at process temperature. Due to the operating principles of the instruments and differences in operating conditions, in-line viscosity measurements are generally performed at high shear rates, while sampling is usually carried out at low shear rates. What's more, in most cases, the two instruments are operated at different temperatures and non-constant temperature in the case of on-line measurement.

It is essential to take into account all the parameters that may influence the measure of viscosity when proceeding to their comparison. Implementing an inline process viscometer may be seen as a challenging task especially for non-Newtonian fluids where the shear rate from the process and the instrument combined have an impact on the viscosity. Therefore, for Non-Newtonian products, results of viscosity measurements performed in laboratory will give different results with an inline viscometer for the same sample and at the same temperature.

A solution to approach this challenge is to calculate the Temperature Compensated Viscosity (TCV) and combine it with a correlation. The TCV will provide the operator with a viscosity value at a unique/chosen reference temperature. Additionally, combining TCV with a correlation will provide the customer with a measurement that will be close to the one performed under laboratory conditions. Indeed, the correlation will take into account the non-Newtonian characteristic of a fluid. Thanks to this solution the viscosity provided by the inline viscometer will match with the viscosity provided by the sampling and laboratory viscometer and allow for an easier process control. Combining TCV and correlation provides the end user with stable and repeatable measurement which are fundamental for Quality Control (QC) and process monitoring in general.

This solution is helping the industry save costs on sampling and laboratory measurements as the instruments continuously display real time viscosity. Consequently, allowing the manufacturer to reach the end-point of a reaction/process faster and in safe conditions. Finally, it allows the end-user to avoid viscosity overshoots and prevents any damage to the process installation.

The paper explains in detail how combining TCV calculation and correlation significantly improves industrial processes, especially for the manufacturing or handling of non-Newtonian fluids such as polymers, personal care products and water-based glue.

The first stage of the methodology is to record the process viscometer and reference instrument data. The process viscometer provides continuous data while reference measurements are discrete datapoints.

The second stage is to determine the relationship between both sets of results, the best mathematical model and its parameters. The model and parameters are then implemented in the viscosity processor. The validity of the model and its parameters

are confirmed by a second measuring campaign. After this validation step, the process viscometer is displaying information very close to the reference instrument.

This method can be used in many industries such as oil and gas, chemical, packaging, food and beverages, body care and cosmetics. It applies to continuous processes operated in steady state as well as to batch processes for process control and/or quality control. Finally, the reference measurement may be a viscosity measurement or another functional property.

INTRODUCTION

Viscosity is one of the physical characteristics that defines fluid products like liquids, gels, pastes, emulsions, solutions or suspensions. Depending on the process, product viscosity is either a key specification, indirectly related to another key characteristic or impacting the efficiency of the process that uses or processes it. For many applications, viscosity is essential to determine the quality of the products and/or the efficiency of the processes. The manufacturer or user will be able to characterize the product and adjust process parameters to optimize production and control the product's quality for instance.

Sampling and laboratory measurement remains the industry's standard practice for quality controls (QC). Laboratory viscosity measurements are mainly performed using rotational, capillary, falling ball, flow cup measuring principles, delivering a viscosity measurement at low shear rate and most of time at ambient temperature. Depending on whether or not the viscosity is in the correct range, the manufacturing team may have to adjust process parameters. It is a time-consuming process with significant response time that may not be fully representative of the product's viscosity while running through a line or process and its variations with time.

The increase of automation and technological improvement led to the development of inline viscometers providing instantaneous and continuous viscosity measurements at process conditions.

Nevertheless, inline viscosity is measured at process temperature and shear rate. Indeed, as a consequence of in-pipe flow and mixing in reactors, products are submitted to velocity gradients and associated shear rates. The total shear rate is defined as the process shear rate added to the shear rate induced by the viscometer. As a consequence of differing measurement conditions between laboratory and process, the measured value differs too.

The impact of temperature on measurement could be managed with a viscosity analyzer at reference temperature. They provide a viscosity measurement at a constant temperature that may be the same as the reference temperature. This type of analyzer and the working principles have been detailed in a previous paper [1].

This solution is usually recommended when the process temperature is very high and different from the reference temperature. Analyzer solutions are complex and expensive because of the sample conditioning system that requires more engineering and needs to be implemented. Additionally, this is an investment that may be uneasy to justify. Whether it is for the viscometer used in this study, the impact of shear rate on measurement may be seen as a challenge for product manufacturers when measuring in-line viscosity of non-Newtonian products as the viscosity value differs from the reference measurement, making it more difficult to correlate.

An alternative solution for this type of applications and products is described in this part.

The paper explains in detail how combining TCV calculation and correlation (to a reference/Laboratory measurement) significantly improves industrial processes, especially for the manufacturing or handling of non-Newtonian fluids Polymers, Personal Care products and Water Based Glue for instance. The method detailed in the paper has been experimented and proven in many industries.

EXPERIMENTAL

1. The process viscometer

The viscometer used in this study has been patented in 1981 (Patent N° FR 2 911 188) [2] and the working principle is the vibrating type at resonance frequency.

The vibrating viscometer at resonance frequency is a sensor working at a high shear rate. As a consequence, pseudo-plastic or shear-thinning fluids are not affected by varying flow rate reducing measurement fluctuations. The process viscometer used in this study is the MIVI viscometer. It is able to measure viscosities up to one million centipoises (cP).

The active part of the sensor is composed of a vibrating rod held in oscillation at a resonance frequency by driving magnets. When the rod is immersed into a viscous material, the amplitude of the vibration is dampened. The vibration’s amplitude and its frequency vary according to the viscosity and the density of the product in which the rod is immersed. The sensor’s receiving coil detects the response and the signal is converted into a viscosity value through the electronics. The factory calibration is performed with certified viscosity standards.

The motion of the rod is created by a magnet fixed on the rod and placed in front of a coil driven by an alternating 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 1). The resulting voltage amplitude is an image of the viscosity.

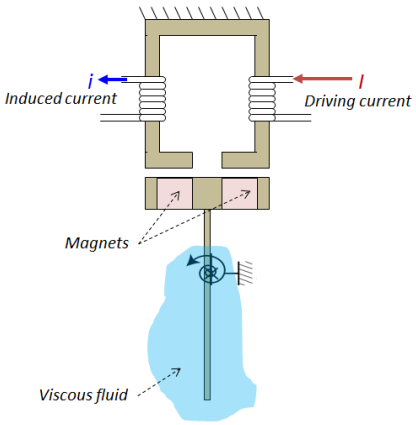

Figure 1. PRINCIPLE OF THE VIBRATING VISCOSITY SENSOR AT RESONANCE FREQUENCY.

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. 2), thus providing a reliable, repeatable and continuous viscosity measurement. This principle is described in Patents FR 2 911 826 [3] and FR 13 62 507 [4].
The viscometer can be equipped with an optional Pt100 temperature probe (temperature range up to 250C/482F. For the purpose of the trials and applications described in the article, the temperature probe is required because temperature is one of the key parameters.

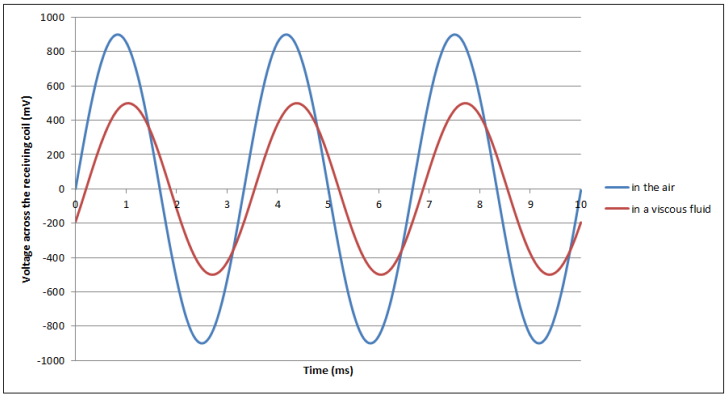

Figure 2. AMPLITUDE DIFFERENCE IN AIR AND IN VISCOUS FLUID.

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 guarantees no drift in time and no maintenance required.

2. The process

A proper installation is required in order to get reliable and useful viscosity and temperature readings. Whether it is a continuous or batch process, the viscometer installation location needs to be chosen wisely and a global understanding of the process and product is usually needed. There is a wide diversity of processes ranging from batch to continuous whether through inline and/or on reactor installations. The viscometer can be installed in various ways at different locations throughout the process. The sensor is equipped with a flow damper that helps provide a stable and laminar flow at the measurement point. Figure 3 summarizes the main industrial process configurations and viscometer mounting options.

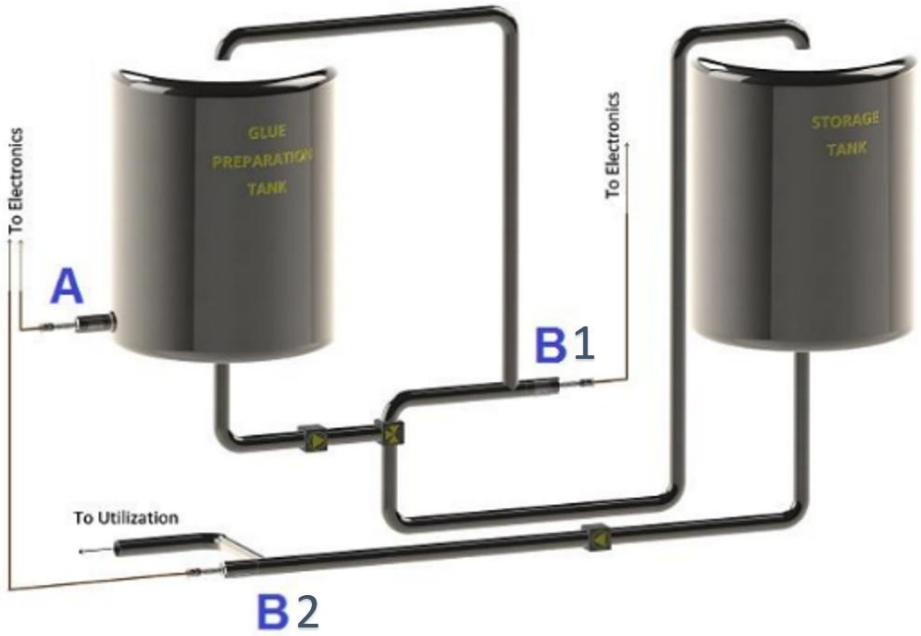

Figure 3. VISCOMETER MOUNTING SOLUTIONS

"**A**" shows the viscosity sensor installed on a mixing tank that is operated in batch mode in the application used for this example. The viscometer may be installed directly on a tank using the proprietary mounting flange welded on the wall of the reactor or tanks to a small insertion tube installed on a flanged nozzle. On-tank installations are generally preferred for batch processes for which Viscosity, Temperature (among other variables) change over time. At the start of a batch, the operator fills the tank with raw ingredients. While temperature and mixing occurs, the product's viscosity changes along with time due to the advance of chemical reaction, cooking, hydration and dissolution... The batch usually ends when viscosity is within the right/correct range. The product is then sent to the next step of the process (storage tank or application).

"**B1**" shows the viscometer installed on a pipe that is in a recirculation loop for this example. According to the size of the process and the piping the viscometer may be installed using a proprietary flow cell, an on-pipe angle mounting or a small immersion tube fixed on a flanged T. The recirculation loop is dedicated to renew the product and homogenously mix it during the batch. The loop can also be the place to install

measuring instruments. The instrumentation loop is generally punctually activated at the end of the batch for Quality Control. It is possible to install the viscometer on any type of loop using the on pipe mounting.

"**B2**" also shows the viscometer installed on a pipe. In the application described in this example it is operated in continuous mode and product is pumped from the storage tank to the place of use or packaging. Continuous processes parameters including the product's Viscosity and Temperature are generally constant over time. It is the case for most storage tanks, in which the product is stable. Inline viscosity monitoring is critical for polymerization control for instance, where over-polymerization may damage the installation or the product. The viscometer is easy to install on a manufacturer's existing process connection flange, measuring chamber or customized solutions such as flanged nozzle or an immersion tube.

3. The Reference Measurement

The reference measurement is the one used by the manufacturer to characterize the product they produce or use. In the vast majority of cases, the Reference Measurement refers to the type of measurement that is being used for Quality Control purposes in laboratory conditions after sampling.

Below is a non-exhaustive list of laboratory measurements / at line equipment:

- Viscosity / Viscometer
- Flow Curve / Rheometer
- Consistency / Consistometer
- Concentration / Titrator
- Dry extract, moisture content / weight loss, Hg evaporation
- Flow time / Flow Cups

In the case of the application described in the paper, the reference measurement is a lab viscometer working at low shear rate and at controlled temperature of 25°C / 77°F which is referred as the reference temperature (T_R).

4. Data recording

Obtaining a representative set of data is essential to build a reliable and useful correlation. During this stage the manufacturer follows the “usual” production schedule and records all the data and process variables including Viscosity, Temperature and raw signals provided by the instrument. There are multiple ways that allows an easy recording. The main recording solutions are the following:

- Directly on the viscometer’s HMI processor by inserting a SD card and activating the recording
- Using the RS485 output connected to the Viscometer software or connected to the process DCS/Processor
- Using the 4/20 mA outputs of the viscometer HMI processor.

The recording procedure is the same whether it is for batches or continuous processes. It is essential to note all events (ingredient added, product changes, particularities) and to keep using the reference instrument in order to build the correlation. Figure 4 is an example of viscosity and temperature recording for a batch process.

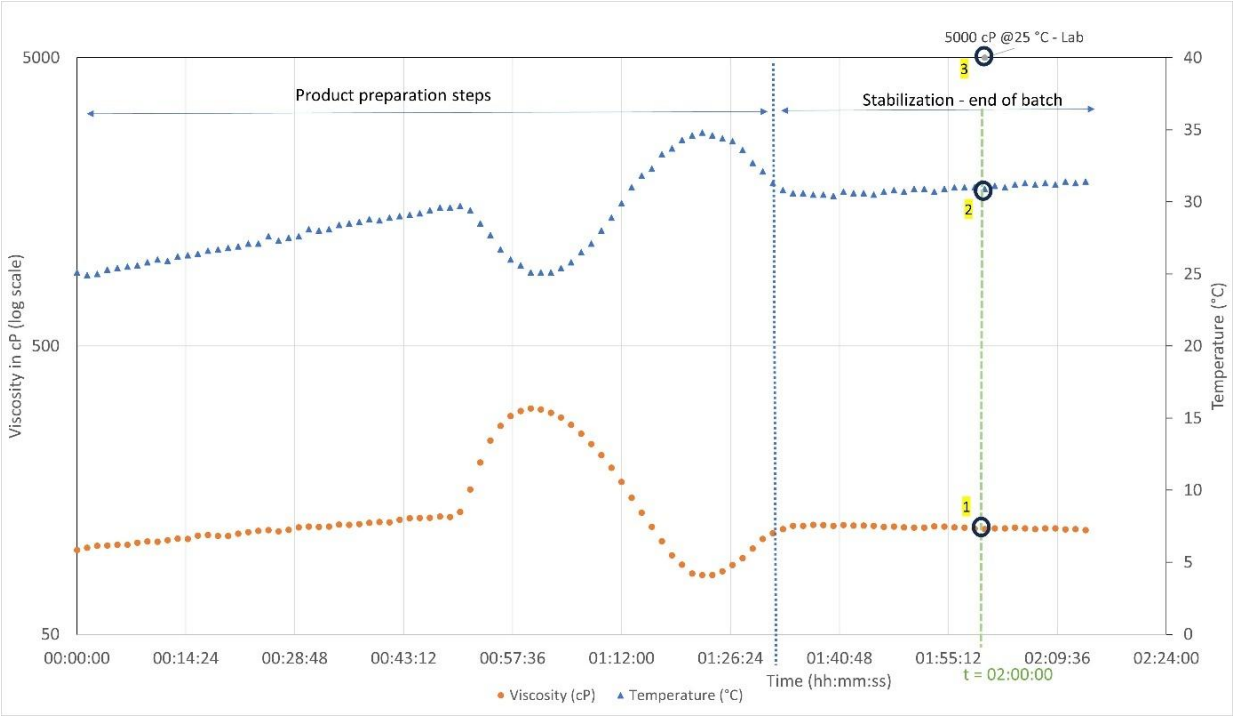

Figure 4. Example of data recording

During a batch manufacturing, multiple variables may affect the product’s viscosity:

- Adding an ingredient
- The advancement of a chemical reaction
- Temperature change
- The global shear rate applied to the fluid.

However, at the end of a batch, the product’s properties generally tend to stabilize and the viscosity becomes a function of Temperature and shear rate that are maintained constant. This is usually when sampling is performed and an operator take a sample

and check the viscosity using the reference method, providing the reference viscosity value "**3**". It is important to note the time at which the sample was taken, so that this value can be compared to the viscosity measured by the process viscometer "**2**" as well as its temperature "**1**".

RESULTS & DISCUSSION

If we take the example of a batch process, when viscosity stabilizes, the following points are obtained from Figure 4:

η_P – the Viscometer dynamic viscosity "**1**"

T_P – the Process Temperature "**2**"

η_{LAB} – the Reference measurement at Reference Temperature "**3**".

Repeating the operation for a representative number of batches will allow for a better analysis. In the case of the application described, the results of a total of 10 batches have been pooled in Figure 5.

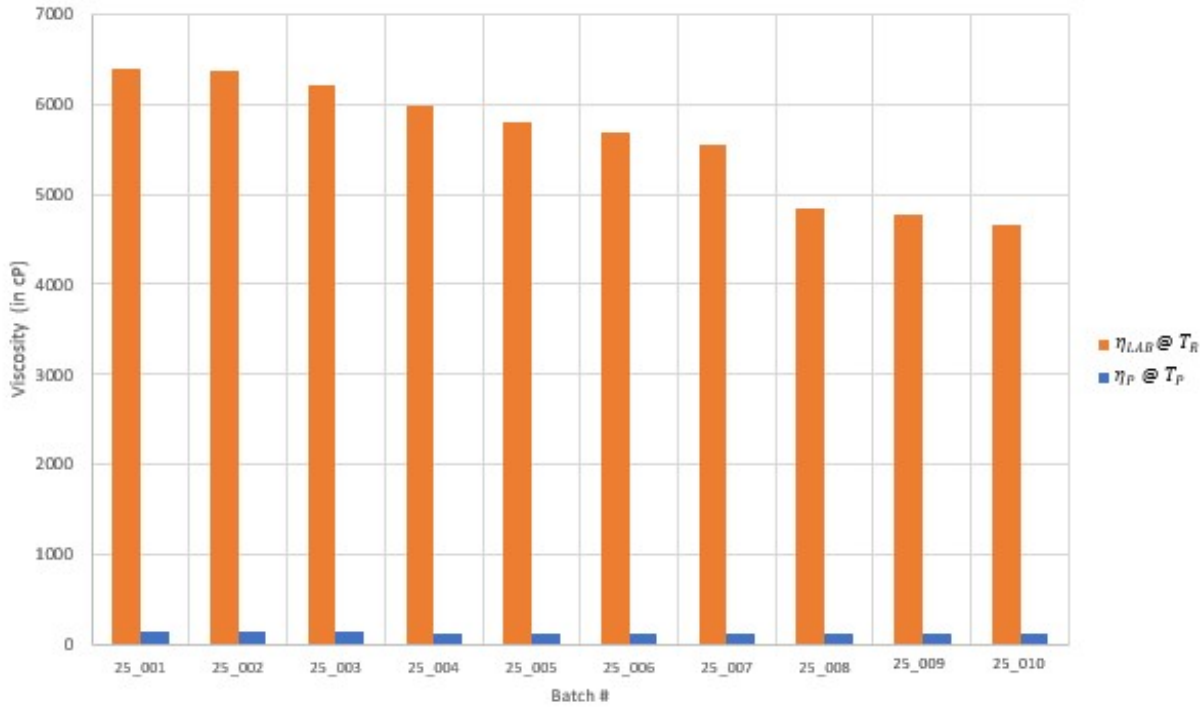

Figure 5. PROCESS vs LAB VISCOSITY VALUES

The graph shows the viscosity at laboratory reference temperature ($\boldsymbol{\eta_{LAB}}$) and the viscosity at process temperature ($\boldsymbol{\eta_P}$) of 10 batches; points are sorted by decreasing laboratory viscosity values. The difference of viscosities between two measurements is mostly due to the differing temperature and shear rate conditions. Indeed, laboratory measurement is generally performed a constant temperature and at low shear rate whereas process measurements see varying temperature and a higher shear rate.

Figure 6 shows the same data in viscosity versus temperature scale.

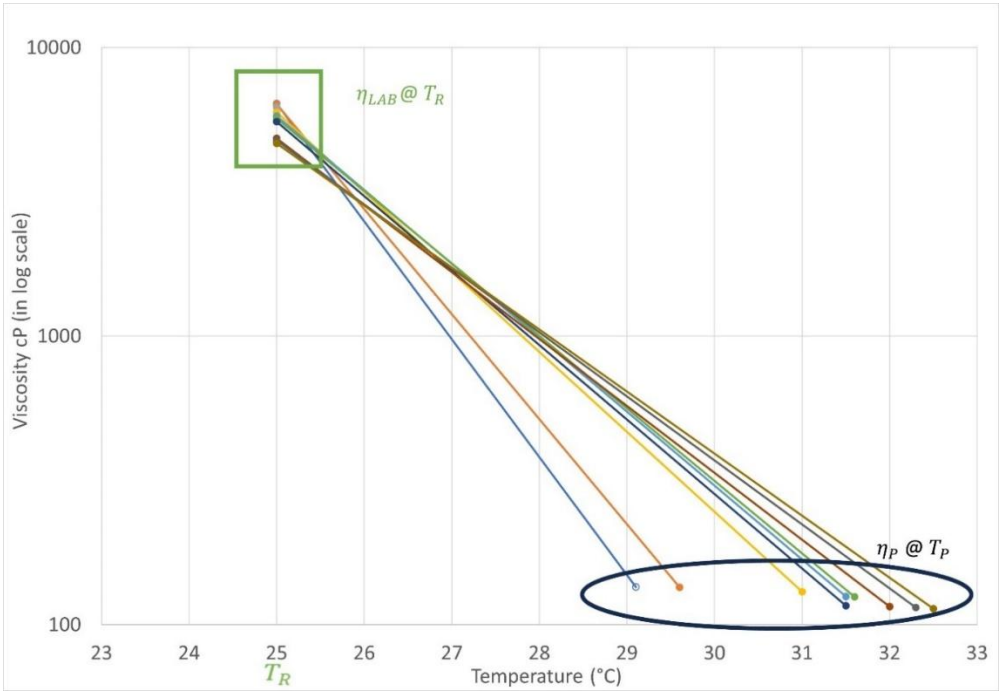

Figure 6. 10 batches - Data recording

After the initial batch recording steps, comes the calculations and transformation to get the TCV at reference temperature and then perform a correlation.

The first transformation step is illustrated Figure 7. The “TCV“ transformation is explained below “**1**”.

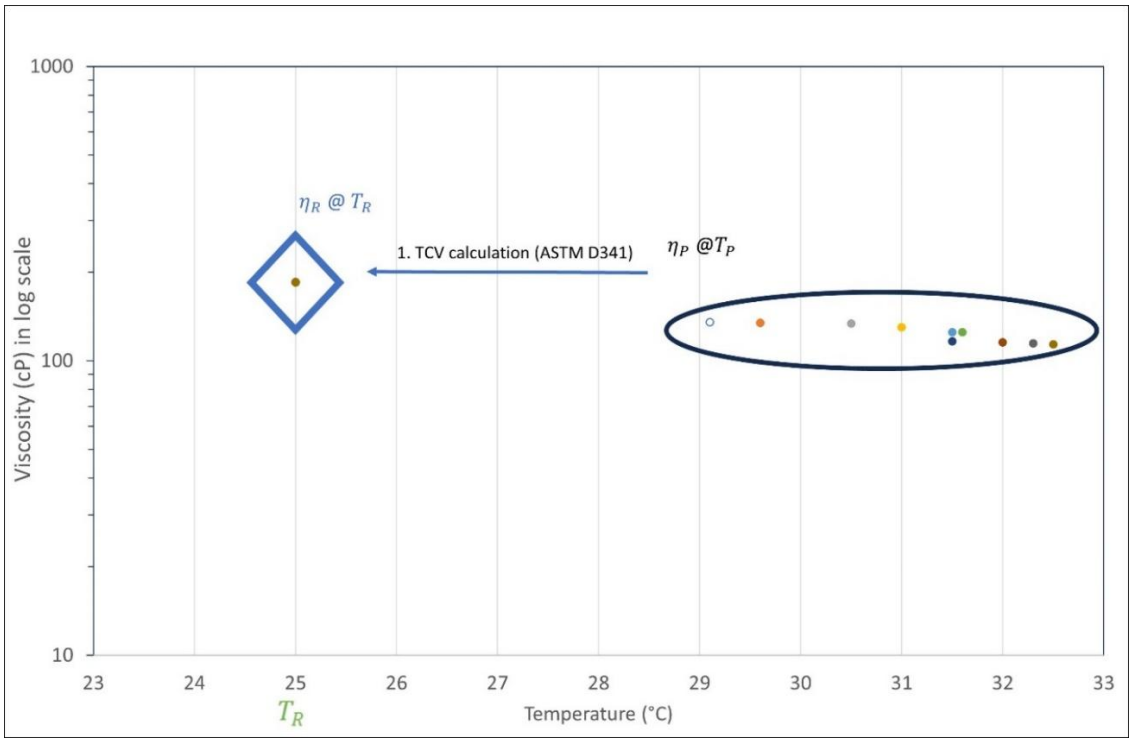

Figure 7. Data Processing – Methodology / Step 1

Step 1 - The methodology starts by compensating the effect of temperature on viscosity by calculating the Temperature Compensated Viscosity (TCV) at a Reference temperature. In most cases, the temperature at which the laboratory viscometer is operated. In order to calculate the TCV, a mathematical expression describing the influence of temperature on Viscosity is necessary. Below are a few examples of temperature compensation equations that can be found in the literature:

$\eta_R = X_1.\eta_P.(\frac{T_P}{T_R})^{X_2}$ Power Law (1)

$\eta_R = X_1.\eta_P.e^{X_2.(T_P - T_R)}$ Exponential (2)

$\eta_R = \eta_P.[1 + X_1.(T_P - T_R) + X_2.(T_P - T_R)^2]$ Polynomial (3)

Where X_1, X_2 are compensation coefficients.

The equation works with both dynamic or kinematic viscosity as they are related one to the other with density (4).

η = ρ* ν (4)

Where ν = kinematic viscosity (cSt)

The HMI processor that is used with the Viscometer is based on the simplified equation (5) described in the ASTM D341 standard [5] that has been validated for petrol-based products but can also be applied to other industries and products.

$log.\log(\eta + 0.7) = A + B.logT$ (5)

Where η = Dynamic Viscosity (cP)

T = Temperature (K)

A & B = Coefficients

Equation (5) provides information on viscosity at any chosen temperature using A, B coefficients determined by experimentation within a defined temperature range. The assumption is that the curves (in log scale) for products that have similar properties are straight lines, parallel one to another. It is then possible to calculate a viscosity value at any temperature thanks to the viscosity measured in process conditions, the process temperature and only the B coefficient that is the slope of the lines. After a few mathematical transformations, (5) becomes (6), the compensation equation.

$\eta_R = -0.7 + 10^{\log(\eta_P + 0.7) * 10^{B.[\log(\frac{T_R}{T_P})]}}$ (6)

Where η_R = Dynamic Viscosity at Reference Temperature (cP)

η_P = Dynamic Viscosity at Process Temperature (cP)

T_R = Reference Temperature (K)

T_P = Process Temperature (K)

The first stage of data processing is transforming the viscosity at process temperature and process shear rate (η_P @T_P; $\dot{\gamma}_P$) to the viscosity at reference temperature and same shear rate (η_R @ T_R; $\dot{\gamma}_P$). As a result, all viscosity values are expressed in relation to the same temperature.

Step 2 – The purpose is to compensate the effect of shear rate on the measurement by transforming the viscosity at reference temperature and process shear rate (η_R @ T_R; $\dot{\gamma}_P$) to the viscosity at same temperature but at the shear rate used by the laboratory viscometer (η_R @ T_R; $\dot{\gamma}_{LAB}$).

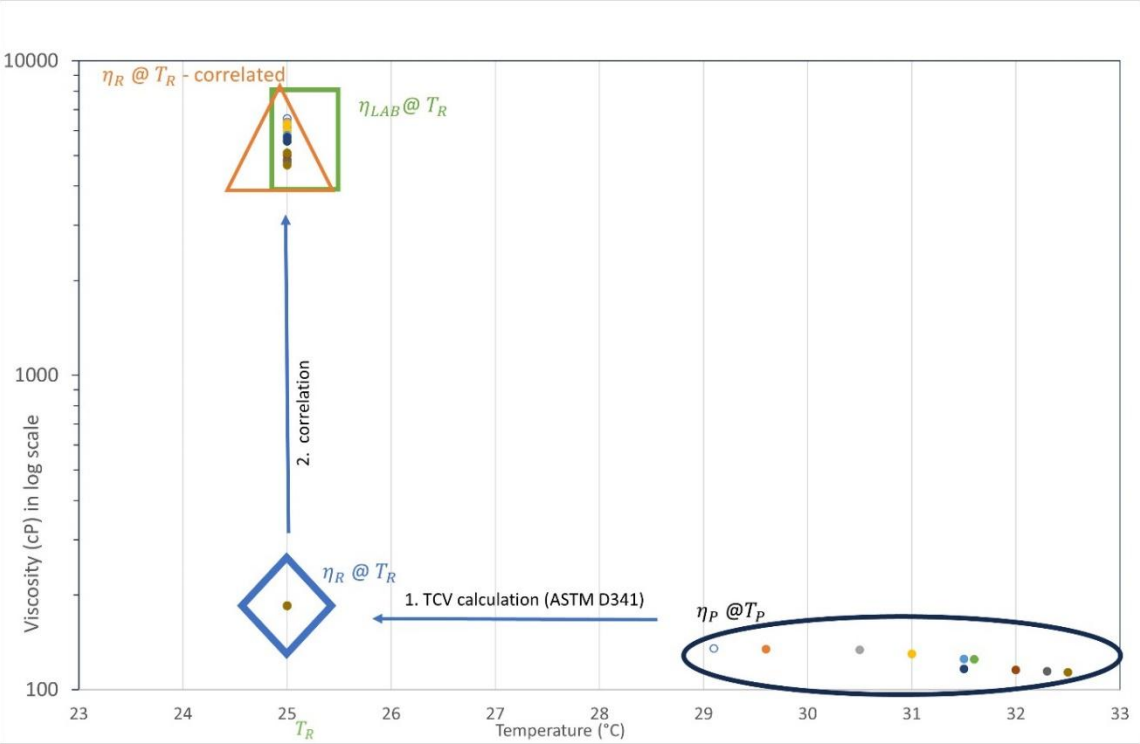

Figure 8. Data Processing - Methodology - step 1 & 2

The calculated viscosity after temperature compensation (**step 1**) and correlation (**step 2**) are compared to the viscosity values measured by the laboratory viscometer (η_{LAB} @ T_R). Figure 8 shows the correlation between the two set of values.

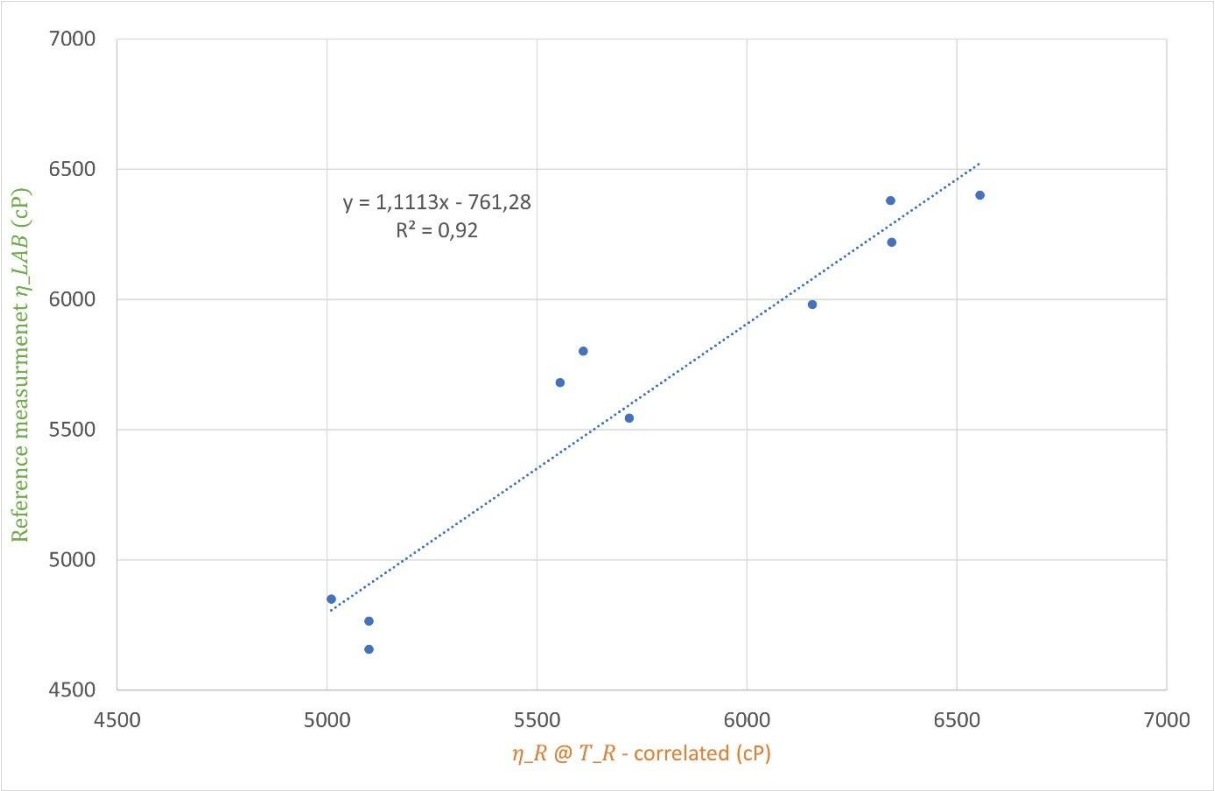

Figure 9 Lab vs Transformed Process Viscosities

In addition to the data processing steps the TCV and correlation parameters are set in the Viscometer's HMI processor. From now on, the TCV and correlation transformations are directly applied to the viscosity measured in process condition and the result is directly comparable to the viscosity measured by the laboratory viscometer. The results of the methodology are product dependent. A change of product behavior requires an update of the parameters.

The methodology may also be applied to other products that have a behavior similar to the one that has been studied. If the influence of temperature and the influence of shear rate are similar, then the TCV and correlation parameters are applicable. This assumption is validated after a set of trials on these news products. If the process is used to manufacture numerous products with different behaviors, they are classified into groups and for each group a set of parameters is determined and stored in the processor.

CONCLUSION

The solution described in the article has been implemented by many Sofraser viscometers users. It is a good alternative to the use of a viscosity analyzers at reference temperature, which is are complex and requires a higher investment. However, analyzer but does not provide a complete solution. The ideal solution allows the inline process viscometer to deliver an information that is directly comparable to the reference values. In addition to taking the effect of temperature into account, the methodology also considers the difference of shear rate conditions. The solution can also be transposed to other physical quantities correlated with viscosity, thus extending its scope of application.

REFERENCES

[1] Belliere, L.K., et al., “New analyzer for viscosity measurement at reference temperature”, ISA 60th Analysis Division, Galveston, TX, USA, 2015

[2] FR2 911 188, Sofraser, “Procédé et système mettant en oeuvre un élément oscillant pour déterminer les caractéristiques physiques d’un produit”, 1985

[3] FR2 921 826, Sofraser, “Method and system for determining the viscosity of a product”.

[4] FR 13 62 507, Sofraser, “Système et procédé de mesure en ligne de la viscosité d’un produit”

[5] ASTM D341-09 (2015): “Standard practice for viscosity – Temperature charts for liquid petroleum products”