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Evaluation of in-line viscosity measurement sensors in gravure printing

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Abstract

For the stabilisation of printing quality, gravure printing machines are normally equipped with viscosity measurement systems. Recently two newly developed viscosity measuring systems, a microelectromechanical tuning fork sensor and an acoustic wave sensor, were introduced to the market. Those systems compete with the traditional rotary viscometer and the dropping body measurement. A system comparison of the different systems were implemented and performed at a Rotomec MW 60 rotogravure press. The aim was to find a system for in-line viscosity measuring of printing inks, which is as accurately as possible and does need minimal cleaning effort. Four experiments were conducted to evaluate the different viscosity measuring instruments: accuracy of the solvent concentration measurement, equipment capability, temperature behaviour in ink and influencing factors of viscosity measurement in the printing process. The results show that the acoustic wave sensor and the rotary viscometer are suitable for the viscosity measurement in gravure printing.

Keywords: viscosity sensors, tuning fork sensor, acoustic wave sensor, rotary viscosimeter

1. Introduction and background

For the stabilisation of printing quality, gravure printing machines are normally equipped with viscosity measurement systems. Recently, two newly developed sensor systems were introduced to the market, a microelectromechanical tuning fork sensor and an acoustic wave sensor. Those systems compete with the traditional rotary viscometer and the dropping body measurement. With regard to the technical equipment of the machine, a system comparison of the different measurement systems were implemented at a Rotomec MW 60 rotogravure press.

For the examination of the different sensors, four tests have been designed, investigating their accuracy, behaviour during the printing process, temperature behaviour in ink, and the behaviour in reaction to external disturbing factors. Based on these tests, recommendations were derived.

Most commonly used viscosity parameters are the kinematic viscosity ν and the dynamic viscosity η (Equation 1). Dynamic viscosity η is measured in $Pa \cdot s = kg \cdot m^{-1} \cdot s^{-1}$, whereas kinematic viscosity ν is measured in $m^2 \cdot s^{-1}$. The conversion is via the the density ϱ.

$$\eta = \frac{\nu}{\varrho} \quad [1]$$

In gravure printing, efflux cups are most common and with these, viscosity is measured in (efflux cup) seconds. Calibration curves between (efflux cup) seconds and the kinematic viscosity ν are defined, e.g. according to DIN 53211 (1978).

Acoustic wave sensors measure the product of dynamic viscosity and the density of the measurement sample, i.e. the kinematic viscosity, as the measurement of an acoustic wave sensor is based on the acoustic impedance (Equation 2).

$$Z = (\omega \cdot \varrho \cdot \eta)^{1/2} \quad [2]$$

where ω represents the angular frequency $\omega = 2 \cdot \pi \cdot f$.

To measure the viscosity, the resonator, an electrostrictive quartz crystal plate, is put in contact with the fluid. The resonator is driven by the electrode at the bottom surface (Figure 1) and moves sinusoidally perpendicular to the sensor surface with the frequency ω and the amplitude U. The frequency is defined by the construction of the sensor, whereas the amplitude depends on the strength of the applied electrical signal. A certain layer of the fluid is hydrodynamically coupled with the sensor surface; its thickness depends on the viscosity.

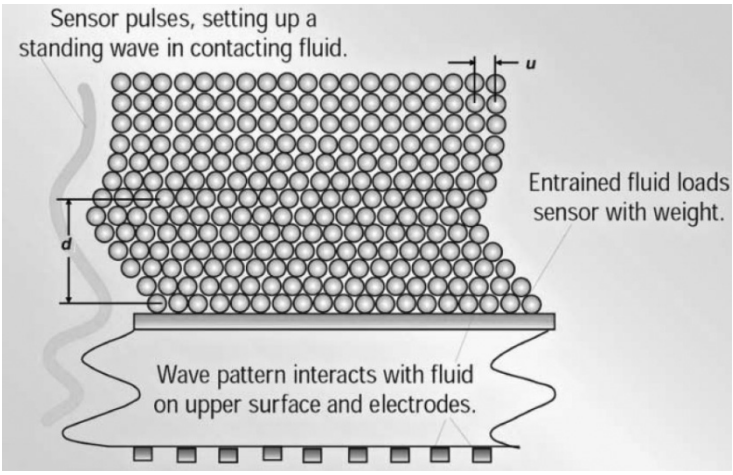

Figure 1: Cross section of acoustic wave sensor (Durdag. 2007)

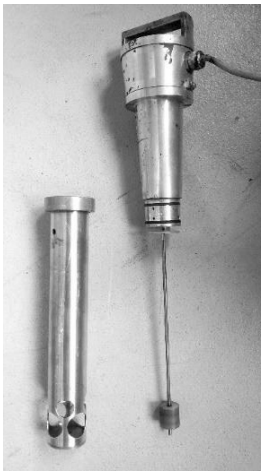

Figure 2: Rotary viscometer with cylindrical test block

The penetration depth d of the wave into the fluid is dependent on the frequency, the viscosity and the density of the fluid (Equation 3):

$$d = \left(\frac{2 \cdot \eta}{\omega \cdot \varrho} \right)^{1/2} \quad [3]$$

The acoustic viscosity is measured through the power consumption of the quartz resonator that emits ultrasonic waves into the measurement fluid to a certain depth d.

The measured number (referred by the vendor as "acoustic viscosity") is the product of the density and the dynamic viscosity (Equation 4) and has the unit $g \cdot cm^{-3} \cdot mPa \cdot s$ (Durdag. 2007; 2008).

$$\eta_A = \frac{Z^2}{\omega} = \eta \cdot \varrho \quad [4]$$

The microelectromechanical tuning fork sensor delivers density, viscosity and temperature, thus it measures kinematic and dynamic viscosity simultaneously. The sensor can be seen as a tuning fork with a flat profile at its end, which is electrically stimulated to an elliptical oscillation within a fluid with its resonant frequency. The surrounding medium impacts the resonant frequency of the tuning fork. As the fork profile is different in the two oscillation directions, the detuning in this two directions provides two distinct values, therefore enabling the calculation of both, dynamic viscosity and density.

A rotary viscometer served as a reference instrument, which was placed directly into the ink tank. As the name suggests, the sensor has a cylindrical measuring body which rotates around its axis (Figure 2).

With increasing viscosity of the measured fluid, the torque resistance at the measuring body increases. As the torque of the motor is held constant, the rotational speed decreases. The reduced rotational frequency is captured by the evaluation unit and transformed to dynamic viscosity.

2. Materials and methods

2.1 Inks and solvents

Rotogravure printing inks are organic-solvent- or water-based inks of low viscosity, with a dynamic viscosity η ranging from 10 mPa · s to 200 mPa · s. To achieve the correct viscosity, the inks are mixed with solvent, which affects the pigment concentration and optical density of the colour.

As solvent evaporates out of the ink during printing, the viscosity increases. Additionally, the behaviour of the ink changes in several respects, including colour strength (pigment concentration), fluid behaviour during cell filling and doctoring, and the behaviour within the printing nip and drying section. For this paper, conventional process colour inks with toluene (publication rotogravure printing) and ethanol / ethyl acetate (packaging gravure printing) in different concentration levels were used. Additionally, a white gravure ink was mixed with varying concentrations of ethyl acetate.

2.2 Experiments

Four experiments were conducted to evaluate the different viscosity measuring instruments. The following inks from Siegwerk Druckfarben AG were mixed (Table 1):

Table 1: Inks used in the experiments (for column L% see section 2.4)

Ink	Solvent portion *L%* (%)	Kinematic viscosity (Frikmar 3 mm cup) (s)	Temperature *T* (°C)
Black series NC 133 Solvent: Ethanol Extender: 150 %	51.0	24.5	23.0
	42.9	26.8	22.7
	39.9	27.9	22.5
	36.4	29.0	22.1
	34.1	30.0	22.4
	34.9	30.0	21.9
	30.7	31.9	21.9
	28.9	33.4	21.6
Cyan series NC 133 Solvent: Ethanol Extender: 150 %	60.0	23.1	21.2
	45.8	26.7	21.5
	34.0	31.7	21.6
Black Solvent: Toluol Extender 200 %	44.8	22.8	22.6
	36.5	26.4	22.6
	25.0	32.3	22.7
White – PU-Binder Solvent: Ethyl acetate	33.3	23.1	21.4
	24.8	26.6	21.5
	16.6	32.1	21.6

2.2.1 Experiment 1: Accuracy of the solvent concentration measurement

Reproducibility of the viscosity measurements was tested with different inks representing the whole process range of solvent concentrations. The data obtained from this experiment were used to map the individual measurements to *L%* (solvent content of the ink).

2.2.2 Experiment 2: Equipment capability study

The equipment capability study is a method to evaluate the accuracy and reliability of different measurement systems. This study allows researchers to analyse and determine the best measurement system for respective areas of application. The commonly used method resulted from decades of experience within the automotive industry. Three analytical procedures, as shown in Table 2, are foreseen:

Table 2: Equipment capability study

Procedure	Purpose	Parameter
Procedure 1	Systematic error and repeatability	C_g. C_{gk}. t-Test, confidence intervals
Procedure 2	Repeatability, reproducibility (with operator influence)	%R&R, ndc
Procedure 3	Repeatability, reproducibility (without operator influence)	%R&R, ndc

Procedure 1 is normally used by the manufacturer of the measurement system to prove the suitability and capability. Procedures 2 and 3 are deployed by the user to confirm suitability on-site.

Procedure 1
The two most important quality parameters for the measurement equipment are the C_g and C_{gk} values. Procedure 1 uses these values to determine the parameters within its application. With this procedure, measurement series are made at a calibration standard and out of these series (at least 25 values) the arithmetic average and standard deviation are calculated. After that, in combination with the specified characteristic tolerances, the C_g and C_{gk} values, can be calculated (Polák, Drlička and Žitňanský, 2014). A measurement system is defined capable if the C_g and C_{gk} values are greater than 1.3.

Procedure 2
Procedure 2 is also known as *%R&R* or as the GR&R-Study (Gage Repeatability & Reproducibility). After defining the amount of test objects n and number of testers k, whereby the test objects should cover the process range, the number of repeats (r) are determined, where $n \cdot r \cdot k > 30$ (Dietrich and Schulze, 1998; 2007). An analysis of variation (ANOVA) is made and values obtained are repeatability *EV*, reproducibility *AV* and part deviation *PV*. The *EV* value is a measure of the influence of the measurement equipment, whereas the *AV* value is a measure of the user's influence.

The *R&R* value is calculated by Equation 5:

$$R\,\&\,R = \sqrt{EV^2 + AV^2} \qquad [5]$$

For a particular case, the single values (*EV*, *AV*, *PV*, *R&R*) are set into relation with the benchmark *RF*, which can be seen as the tolerance *T* of the process or the total deviation *TV* out of the process deviation.If the process deviation is unknown, *TV* can be derived from Equation 6:

$$TV = \sqrt{EV^2 + AV^2 + PV^2} \qquad [6]$$

if only the measurement system is under investigation, *PV* is not to be considered and therefore:

$$TV = \sqrt{EV^2 + AV^2} = R\,\&\,R \qquad [7]$$

The relative values *%EV*, *%AV*, *%PV*, *%R&R* result by dividing *EV*, *AV*, *PV*, and *R&R* by *TV*.

The evaluation of the measurement results can be made according to the Ford guideline EU 1880 (1997):

%R&R ≤ 20 % measurement system is capable,
%R&R ≤ 30 % measurement system is partly capable,
%R&R ≥ 30 % measurement system is not capable.

Procedure 3
In this survey, procedure 3 is used. Procedure 3 is a derivation of procedure 2, where the influence of the user is omitted. This is possible if the user has no influence on the results, like in automated measurements or in-line systems. In this case, $n \cdot r > 20$ applies. If there are no different test objects, the amount of repeats has to be increased. As already described, the ANOVA is used for the evaluation of the system, where the reproducibility *AV* equals zero. The *R&R* value corresponds in that case to the repeatability *EV*.

2.2.3 Experiment 3: Temperature behaviour in ink

The change in viscosity was measured as a function of temperature. The aim was to examine the behaviour of the different measuring instruments when handling ink which varies in temperature, e.g. cold ink at start up or ink heated up during running the printing process.

2.2.4 Experiment 4: Influencing factors of the viscosity measurement in the printing process

Typical parameters influencing the measurement process – printing speed, flow rate of ink within the inking system, and micro foaming extent – were varied on press. The aim of the experiment was to determine the viscosity measuring instrument with the best resilience within the process environment.

2.3 Experimental setup

The viscosity measuring instruments were installed in the ink circulation of a Rotomec MW60 gravure printing press, which is driven by a double acting pneumatic pump. Figure 3 illustrates the individual components of the general experimental setup.

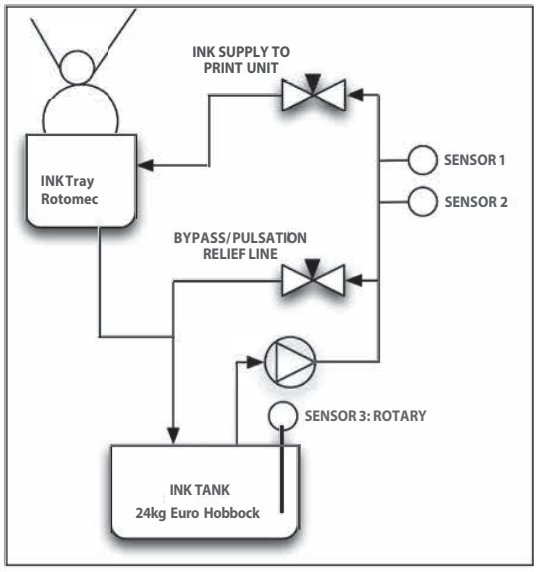

Figure 3: Experimental setup;
Sensor 1: Tuning fork; Sensor 2: Acoustic wave; Sensor 3: Rotary viscometer

To gather the values for each test point, the ink was poured into the tank, the tank was closed and the pump activated. Then the measurement was conducted for 5 min and the values of each sensor collected. After deactivating the pump, the tank was opened, the ink removed and the tank together with the other parts of the ink circulation was cleaned.

2.4 Reference system: Solvent concentration

The meaning of the values of the different viscosity measuring instruments differ according to the different measuring principles (see section 1) and not all deliver dynamic viscosity. Therefore, the correlation between the different systems was based on a reference system solvent concentration: *L%* (%), the quotient of the amount of solvent additionally poured into the basic ink, delivered by the ink manufacturer, to the amount of the basic ink itself. As the viscosity is indirectly proportional to this number, the transformation (Equation 8) was used to match the values of the devices to *L%*, with characteristic constants *a* and *b* for each device and ink type.

$$y = \frac{a}{x} + b \qquad [8]$$

where x is solvent concentration *L%* in %. and y is value of viscosity from given measurement device.

3. Results and discussion

3.1 Experiment 1: Accuracy of the solvent concentration measurement

All measurement values were converted through Equation 8 to the solvent content and given as *L%*. Figure 4 shows these *L%* values of the devices compared to the actual solvent concentration *L%*.

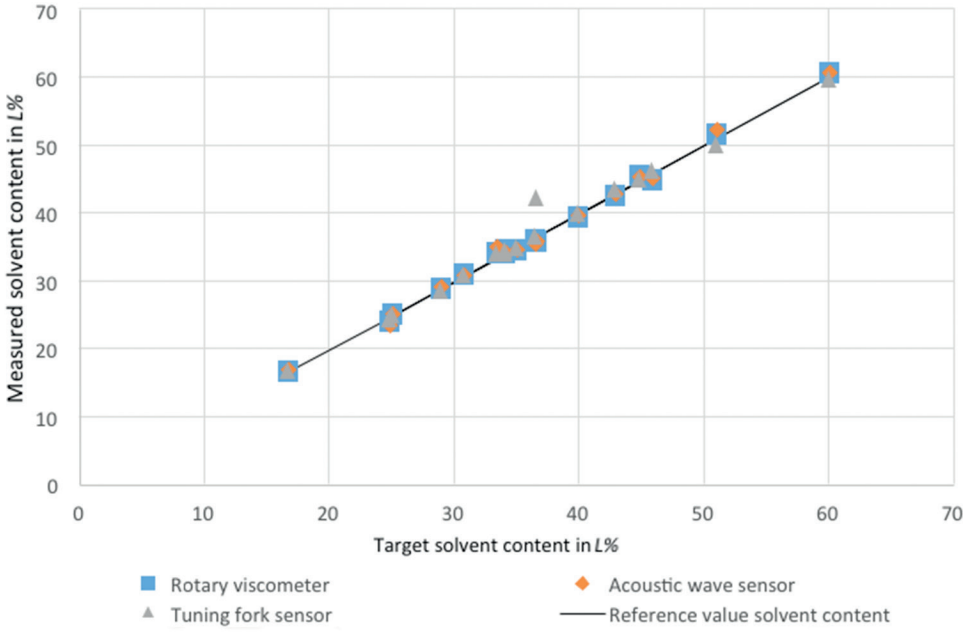

Figure 4: Target-actual comparison of the three sensors for the different dilutions

All instruments can reproduce the different dilutions levels with good accuracy with the exception of one outlier, presumably a faulty measurement. The acoustic wave sensor has the lowest standard deviation of ± 0.07 *L%* of all dilutions, the tuning fork sensor the highest with ± 0.09 *L%*. The rotary viscometer with values of ± 0.08 *L%* lies between the results for other sensors.

3.2 Experiment 2: Equipment capability study

The testing fluid for the evaluation was Black NC 133 from Siegwerk Druckfarben AG with 150 % of extender and ethanol as solvent with a solvent content of 36.44 *L%*. The tolerances were defined to ± 1 s (3 mm DIN cup), which is a common value in order to avoid variations in print. For a solvent content of 36.44 %, flow time of 1 s (3 mm DIN cup) corresponds to a solvent content of 2 %. This leads to a tolerance of 4 *L%*. Table 3 shows the results of the measurements.

Table 3: Results for the equipment capability study procedure 1 (C_g, $T_{min/Cg}$) and procedure 3 (%EV and %R&R)

Ink	Rotary viscometer in *L%*	Acoustic wave sensor in *L%*	Tuning fork sensor in *L%*
Basic size in *L%*	36.44	36.44	36.44
Tolerance in *L%*	4.00	4.00	4.00
Mean in *L%*	36.11	35.54	36.01
Standard deviation in *L%*	0.13	0.05	0.70
C_g	1.61	4.00	0.29
$T_{min/Cg}$ in *L%*	3.309	1.32	18.59
%EV	17.5	5.76	71.61
%R&R	17.5	5.76	71.61

The equipment capability study showed that the rotary viscometer and the acoustic wave sensor are capable with a C_g of 1.61 and 4.00, respectively. The tuning fork sensor is with C_g of 0.29 far below the critical value of 1.33 and therefore it is not capable. For the tuning fork sensor, a tolerance of 18.59 *L%* can be achieved at the best. That means that variations of ± 4.5 s flow time (3 mm DIN cup) are possible. A reason for the poor results of the tuning fork sensor could be the experimental setup, where the cross section of the tube the sensor was integrated in was too low and the measurements were affected by the pulsation of the pump.

The results of procedure 3 confirm the results of procedure 1 and show that the acoustic wave sensor has a very good repeatability (*%EV* = 5.76). The rotary viscometer with *%EV* of 17.5 is also below the limit of 20 % and therefore is capable.

3.3 Experiment 3: Temperature behaviour in ink

The black ink was stored in a closed tank for 24 h at 7 °C. During the measuring operation, the ink container was heated from 12 °C to 36 °C. The measured values of the three sensors are shown in Figure 5.

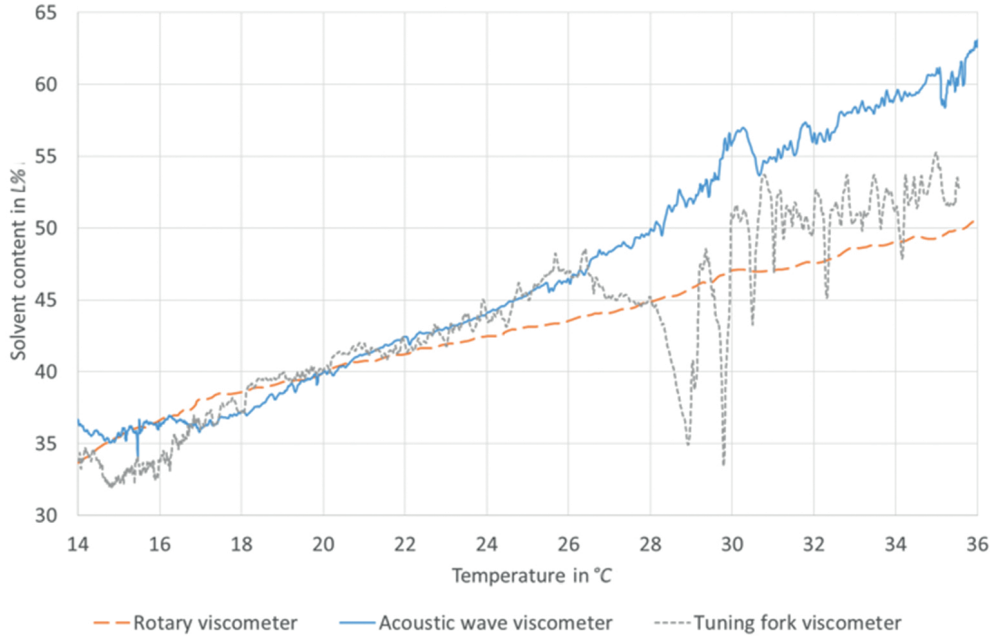

Figure 5: Temperature behaviour in ink (higher L% corresponds to lower viscosity); normalised at 20 °C

The viscosity change due to increasing the ink temperature is accurately reproduced by the sensors. Again, the tuning fork sensor shows big variations. Additionally, the slope of the increase is significantly higher for the acoustic wave sensor. This can be explained by density changes, which occur in parallel to the lowering of the viscosity. As this sensor measures "acoustic impedance" acc. to Equation 2, the density decrease affects the measurement with an additional linear factor. As a result, each sensor has to be calibrated separately to compensate for temperature changes.

3.4 Experiment 4: Influencing factors of the viscosity measurement in the printing process

Firstly, the effect of printing speed was investigated. The diving of the cylinder with the empty cells into the ink brings many small air bubbles into the ink and creates micro foaming. The higher the speed, the bigger this effect is. The following tables (Table 4, Table 5 and Table 6) show the sequence of the test procedures. Viscosity measurements during test procedures, by using different viscometers, are presented in Figure 6.

Additionally, the pumping creates foaming too. This effect was investigated in a second measurement series by varying the pressure to the pneumatic pump, therefore modifying the ink flow and additionally the temporal pressure profile within the tubes. The higher the flow rate, the more foam is created. Moreover, as the tuning fork sensor and the acoustic wave sensor are located within the pumping tube line, the flow through or along the sensor was expected to affect the measurement.

Table 4: Process description: Printing speed

Influence factor	Time	Process description
1	11:32	Rotary viscometer not surrounded with ink – values excluded
2	11:44	Added ink
3	12:10	Printing speed: 180 m · min^{-1}
4	12:15	Rotary viscometer not surrounded with ink – values excluded
5	12:30	Added ink
6	12:35	Reduced printing speed: 60 m · min^{-1}
7	12:54	Increased printing speed: 240 m · min^{-1}
8	12:57	Reduced printing speed: 60 m · min^{-1}

Table 5: Process description: Pump pressure

Influence factor	Time	Process description
9	13:05	Bypass: off
10	13:08	Air throttling: open
11	13:11	Pump pressure: 3.5 bar / Bypass: closed / Air throttling: open
12	13:14	Pump pressure: 3.5 bar / Bypass: closed / Air throttling: open / Ink throttling: slightly open
13	13:18	Pump pressure: 3.5 bar / Bypass: closed / Air throttling: open / Ink throttling: half open
14	13:21	Pump pressure: 3.5 bar / Bypass: closed / Air throttling: open / Ink throttling: fully open
15	13:23	Pump pressure: 2.2 bar / Bypass: closed / Air throttling: open / Ink throttling: fully open
16	13:26	Pump pressure: 2.2 bar / Bypass: open / Air throttling: open / Ink throttling: fully open
17	13:29	Standard-setting as 13:05 clock

Often a stirring rod is used to ensure a stable dispersion of the ink, especially with special effect pigments (metallic, iriodine) or with white pigments. This again results in increased foaming. In additio, the rotary sensor could be affected by the ink flow generated in the tank. Therefore, a third measuring series was set up with a stirring rod.

Table 6: Process description: Micro foam

Influence factor	Time	Process description
18	14:18	Printing speed: 300 m · min^{-1} (without substrate)
19	14:24	Activation of stirrer
20	14:39	Aspiration of foam from the ink surface
21	14:42	Without ink tray
22	14:46	Aspiration of foam from the ink surface

Results: rotary viscometer

The addition of solvent at 11:53 is correctly reproduced by the rotary viscometer. The graph shows the ongoing test procedure in a relative smooth curve.

Between 12:35 and 13:00 the printing speed was changed and between 13:05 and 13:30 parameters of the pump stroke varied. To the end of the experiment, the influence of micro foam was checked. The curve slightly descends, as the solvent evaporates slowly. The rotary viscometer can be described as highly resistant to the tested influencing factors.

Results: Acoustic wave sensor

The addition of solvent at 11:53 is correctly reproduced by the acoustic wave sensor. Nevertheless, the values decrease after reaching a local maximum more than expected, exaggerating the effect of the solvent evaporation.

Results: Tuning fork sensor

The tuning fork sensor is susceptible to pump strokes and micro foam and produces a lot of incorrect measurement values. During the variation of the pump settings, measurement errors resulted when the ink throttle was opened. The reason may be that the diameter of the tube, where the sensor unit sits in, has a diameter which is too small, therefore causing high flow speeds and high pressure peaks, which disturb the measurement. Consequently, a change in the ink flow has a negative effect on the value acquisition by the tuning fork system.

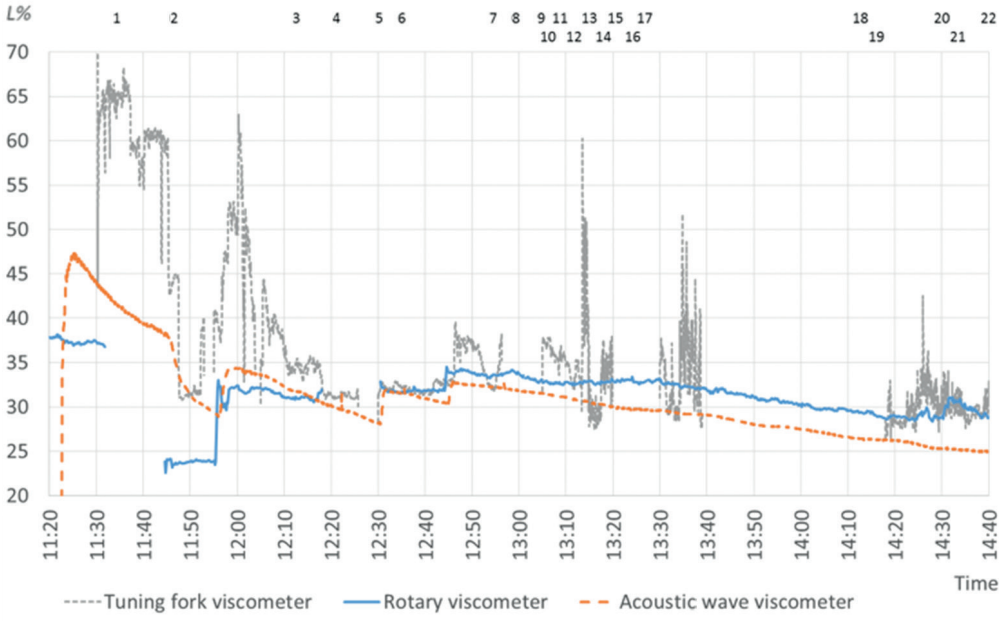

Figure 6: Measurements of viscometers through measurement series; normalised at 12:30

4. Discussion

The evaluation of the three investigated measuring sensors showed that the tuning fork sensor is least tolerant to the pump strokes and to the microfoaming. Pneumatic pumps create a pulsing ink flow, therefore strongly varying fluid velocities and pressures. The measurement is performed through the elliptical oscillations of a rod positioned within the tube for the ink transport from bucket to ink pan, perpendicular to the vector of ink flow. Varying pressures might impact on the rod during pulse cycles, therefore generating erroneous values. A special shunt with homogenized and slow ink flow might solve this problem, however adding to the effort on installation and cleaning. Additionally, it can be assumed that micro bubbles, which come in proximity of the oscillating rod, change the behaviour of the fluid significantly, as they change the mean density and, as relatively big "particles" within the fluid, the mean mobility of the fluid. As this is the number to be measured, these micro bubbles influence every measurement system. Nevertheless, the tuning fork principle seems to be most sensitive to this and seems to be more applicable for unperturbed fluids.

The acoustic wave sensor amplifies the decrease of viscosity due to solvent evaporation as well as by temperature increase. As this sensor measures the "acoustic viscosity", which is the product of dynamic viscosity and density, and both parameters increase with lower solvent content or higher temperature, it could be assumed that the overall signal is more sensitive compared to measuring dynamic viscosity alone. In any case, the density and its change have to be taken into account when calibrating and transforming the measured values from this measuring principle to one which measures the dynamic viscosity. Unfortunately, we did not measure the density with a separate measuring

device, so we cannot confirm this assumption yet. (The tuning fork sensor delivers values for the density too, but the quality of these values was uncertain to the same extent as the values for the dynamic viscosity itself.)

The rotary viscometer performed as expected over the entire experimental period. If it is ensured that the sensor is always completely surrounded with ink, print speed and pump strokes do not influence the measurement and micro foam does only to a small extent.

5. Conclusion

The tests showed that basically all considered sensors are capable of gathering viscosity. The different levels of dilution are captured accurately by all measurement systems. However, the acoustic wave sensor delivers the smallest measurement variations over all levels of dilution. This result is verified in the equipment capability study, where the acoustic wave sensor proved to be the most capable of the three sensors. The tuning fork sensor could not prove its capability in the test set up used.

As the sensors use different measuring principles, they respond differently to temperature variations of the ink, i.e. the coupled viscosity and density changes, and have to be calibrated to correctly response on the process variability.

The tuning fork sensor reacts very sensibly to pump strokes of the pneumatic pump and to micro foam and therefore it is not suitable for an in-line viscosity measurement system without substantial change of the setup used. The acoustic wave sensor is much more insensitive to these effects. Nevertheless, before applying this sensor it has to be investigated if the measuring principle (product of dynamic viscosity and density) sufficiently explains the measured stronger decline compared to the rotary viscometer when solvent evaporates out of the ink.

The rotary viscometer is capable and represents the current state of the art in the printing industry. However, it was the aim to find a system, which is accurate. minimize cleaning effort and can be installed on the press with minimal handling issues. In this context, the acoustic wave sensor is to be preferred.

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