Evaluation of a capillary–coriolis instrument for on-line viscosity and density measurements of kraft black liquor

ANTHONY P. DUTKA, V.R. BASKER, OSCAR D. CRISALLE, ARTHUR L. FRICKE, AND PAUL KALOTAY

ABSTRACT: A capillary–coriolis instrument was evaluated as an on-line viscometer and density meter for kraft black liquor. This instrument features sealed pressure diaphragms connected to a differential pressure cell. It was installed in a pilot plant where liquor could be circulated in a flow loop with the solids concentration, temperature, and flow rate of the liquor closely controlled. Measurements were made under laminar flow conditions for liquors with solids concentrations ranging from 34% to 74% and with temperatures up to 140°C. For determining the accuracy and precision of the instrument, on-line measurements were compared with laboratory measurements of viscosity and density. Under laminar flow conditions, the viscometer tracked reliably within ±10% of the laboratory reference viscosity for highly viscous liquors. The sensor operated satisfactorily under both steady-state and transient conditions and did not experience fouling problems.

Application: The capillary–coriolis viscometer can reliably measure on-line the viscosity and density of kraft black liquors, including liquors with very high solids content.

A by-product of the kraft pulping process, most black liquor is fired in a Tomlison furnace to recover energy. Kraft recovery operations involve more than 73 million metric tons of black liquor processed per year in the United States and Canada. Consequently, there is an economic incentive to increase the solids content of black liquor. If the solids concentration were to be increased from 70% to 75%, for example, there would be an additional energy recovery of 3.1×10^{13} Btu/year for the entire industry [1]. Moreover, such an increase in solids content would reduce emissions, reduce smelt, and improve boiler efficiency [2].

Increasing the solid contents, however, will also increase the viscosity. For example, a black liquor at normal firing temperature would increase in viscosity from 0.09 Pa•s at 73% solids to 0.6 Pa•s at 80% solids. Black liquors are usually heated to maintain constant viscosity before they are sprayed into the recovery furnace [3]. However, since viscosity increases exponentially with solids content [4–8], small variations in solids can cause large variations in viscosity even if the temperature is held constant, making the temperature-control schemes ineffective. Furthermore, black liquor can become non-Newtonian or even viscoelastic at very high solids concentrations [7, 9, 10]. Results show that viscosity is a dominant variable affecting the evaporation, transport, heat transfer, and droplet formation of black liquor [11, 12]. Hence, the availability of an on-line viscometer that can reliably measure the viscosity at high solids content is a necessary tool to obtain adequate control for such liquors.

The work described here pertains to an extensive evaluation of the performance of a capillary–coriolis instrument used to measure the viscosity and density of black liquor on line. The project was undertaken at the University of Florida and was sponsored by the U.S. Dept. of Energy and by participating industries for the purpose of evaluating several on-line viscometers. Evaluation results for other viscometers are reported elsewhere [13, 14].

PILOT FLOW LOOP FOR INSTRUMENT EVALUATION

The experimental system consists of a piping loop, viscometers, and a computerized data acquisition system, designed to emulate the flow of black liquor in a recovery mill. **Figure 1** is a diagram of the system. In the main stream of the piping loop, a progressive cavity pump takes heated black liquor from a 300 gal tank and forces it to circulate along a 2 in. (5.08 cm) stainless steel pipe in a closed circuit that returns the liquid to the tank.

At a sampling point along the main stream, a fraction of the black liquor is diverted into a 1 in. (2.54 cm) piping network, creating a sample stream. This sample stream passes through a heater where the liquor temperature is raised under accurate control. Further upstream, the on-line viscometers take a reading of the liquor viscosity. Extensive details of the setup are reported elsewhere [13–16].

PRINCIPLES OF VISCOSITY MEASUREMENT

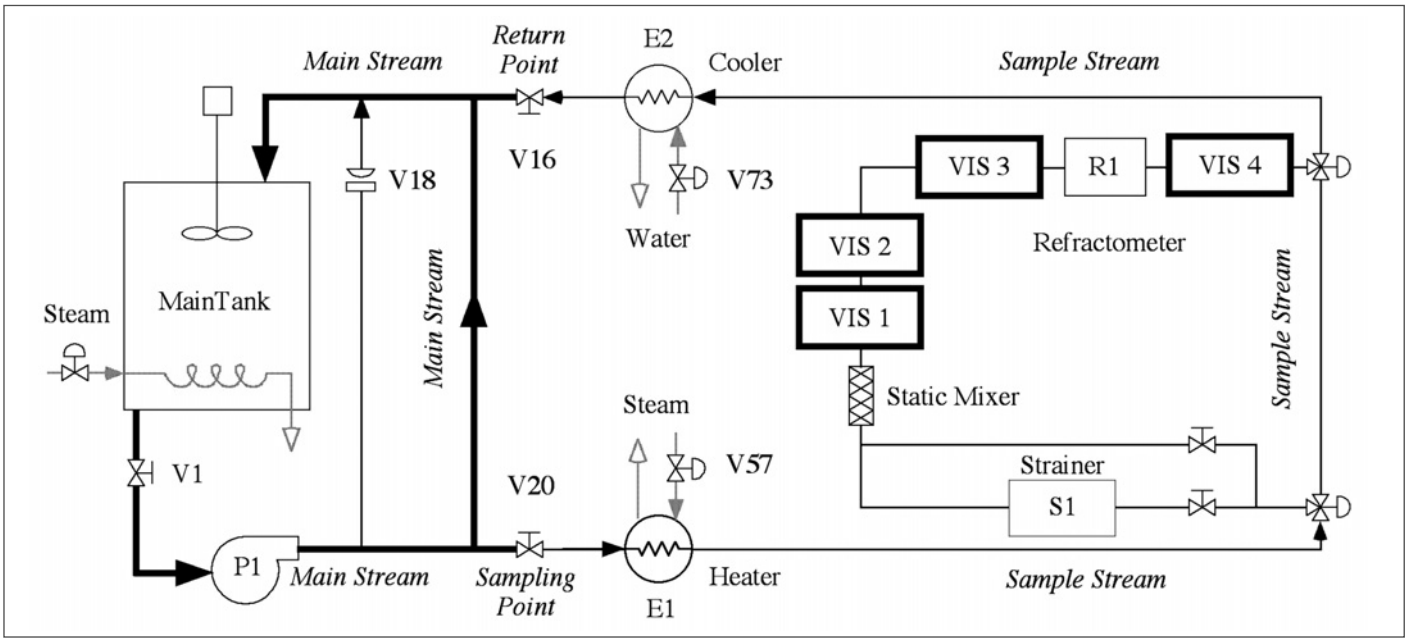
Capillary–coriolis viscometry is based on the Hagen-Poiseuille equation for incompressible Newtonian fluids in fully developed, steady-state laminar flow conditions in a capillary [17]:

$$\eta = \phi \frac{\Delta p \rho}{m} \qquad (1)$$

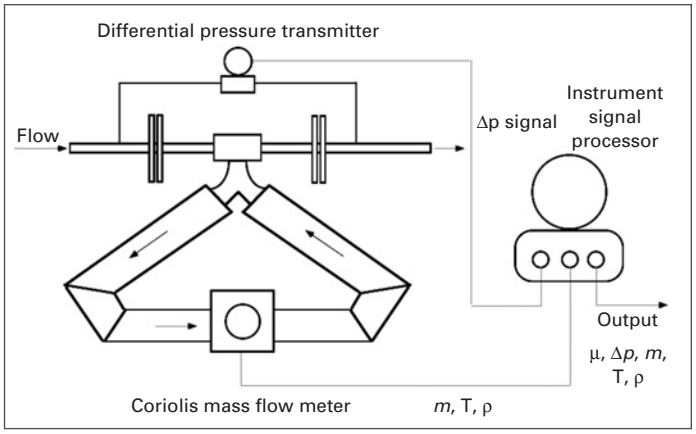
where

η = viscosity
ϕ = proportionality constant
Δp = pressure drop
ρ = density
m = mass flow rate.

The viscometer studied uses a differential pressure cell to measure the pressure drop and a coriolis mass flow sensor to measure both the density and the mass flow rate. Hence, the viscosity can be calculated from Eq. 1. A volumetric flow rate (q) can also be calculated as $q = m/\rho$. Details of the measurement of mass flow rate and density are given in the ensuing subsections.



1. Pilot flow loop process and instrumentation diagram. The labels VIS 1, VIS 2, VIS 3, and VIS 4 denote points where a viscometer can be installed for testing.



2. Diagram of the capillary–coriolis viscometer used in the pilot flow loop.

Georgia Pacific	Canadian Forest Products	St. Laurent Paper Products	Mixture*
34.14	47.96	49.39	67.72
42.24	57.73	59.58	71.38
48.29	—	—	73.40
55.84	—	—	—
58.38	—	—	—
65.51	—	—	—
68.38	—	—	—

*Georgia Pacific and Canadian Forest Products.

I. Solids content of the black liquors, %.

For a Newtonian fluid, the viscosity proportionality constant ϕ is set by calibrating the sensor at a point of known viscosity. A non-Newtonian fluid uses two points, one in the upper part and one in the lower part of the operating viscosity range.

Keep in mind that the Hagen-Poiseuille equation is developed for a straight capillary tube and for fully developed flow, two conditions that do not strictly hold for the coriolis sensor. Operating at low Reynolds number (less than 1150) reduces the possibility of turbulence. At relatively high Reynolds numbers, secondary flow characteristics as well as entry and exit effects can produce pressure drops higher than those assumed in the Hagen-Poiseuille equation, which adversely affects the sensor reading. Therefore, it is important to control the rate of fluid flow through the instrument.

Mass flow rate measurement

The coriolis mass flow-rate sensor measures the effect of coriolis acceleration and deceleration [18] caused by the flow of a fluid in vibrating tubes.

Consider the setup shown in **Fig. 2**. The fluid flows through a tube shaped like a coat hanger, an approximation of an ideal U-shape pipe, rigidly supported at one end and free to vibrate at the other end. The tube is forced to vibrate at the natural frequency f, so that the free end moves up and down along an arc.

In the downward cycle, the fluid entering the sensor at the supported end has only a horizontal component of velocity. However, as the fluid advances, it acquires a vertical velocity component as a result of the downward movement of the tube. This downward component of velocity increases as the fluid travels down the tube and as the fluid negotiates the turn at the free end. This change in velocity results in fluid acceleration, which takes the form of a coriolis acceleration. According to Newton's third law of motion, the downward force attributable to this fluid acceleration is resisted by the upward reactive force applied by the tube wall.

After crossing the tube bend, the fluid climbs towards the supported end, resulting in a coriolis deceleration as the tube applies a reactive force in the downward direction. These opposing upward and downward reactive forces cause the tube to twist. The mass flow rate through the sensor is proportional to the extent of twist:

$$m = \mathrm{k}_m T_w \qquad (2)$$

where

m = mass flow rate
k_m = proportionality constant
T_w = measured twist.

The elastic properties of the sensor tubes are affected by temperature and pressure because they affect the stiffness of the tube [19, 20]. Coriolis mass flowmeters of recent design, like the one used in this study, employ a sensor to minimize or eliminate these effects.

Density measurement

The coriolis mass flowmeters can also be used to determine the density of the fluid because the natural frequency of vibration of the coriolis sensor depends on the mass of the fluid in the sensor. A simple force balance and a mass balance yield the relationship of Eq. 3 [21]:

$$\rho = \left(\frac{K}{4\pi^2 V} \right) \frac{1}{f^2} - \frac{m_{tube}}{V} \qquad (3)$$

where

K = density calibration constant
ρ = density of the fluid
V = volume of the sensor
f = natural frequency
m_{tube} = mass of the fully flooded sensor.

We can determine the constant K by using two fluids of known density (air and water) as references.

DETAILS OF THE VISCOMETER

The capillary–coriolis viscometer we tested is a Micro Motion CMF025M Coriolis Mass Flow Sensor, combined with a Rosemount 3051CD differential pressure cell and a Rosemount RFT9739 transmitter. The physical location of this viscometer in the pilot flow loop is indicated by the label "VIS 2" in Fig. 1. A diagram of the overall measurement system is shown in Fig. 2.

The RFT 9739 transmitter provides an oscillatory voltage to vibrate the sensor's flow tubes at their natural frequency. The transmitter receives the velocity signals produced by the pickoffs on the flow tubes to calculate mass flow rate and density. The signal from an internal RTD sensor (resistance temperature device) in a flow tube is used to measure temperature. The transmitter also supplies power to the differential pressure cell, and the 4–20 mA signal from the differential pressure cell is fed to the transmitter [22].

EXPERIMENTAL OVERVIEW

Black liquor was obtained from three kraft mills: Georgia Pacific Corp. in Palatka, FL, Canadian Forest Products Ltd. in British Columbia, and St. Laurent Paper Products Corp. in West Point, VA. These mills were taken as being representative of southern softwood pulping, western softwood pulping, and mid-Atlantic mixed-species pulping, respectively. The liquors were received at concentrations of 40–45% solids and were diluted or concentrated as needed in a wiped-film evaporator at the University of Florida.

Table I lists the solids composition of the black liquors used in the experimental runs. The on-line viscometer was tested on these 14 different black liquor concentrations, ranging from a Georgia Pacific liquor of 34.14% solids to a black liquor of 73.40% solids obtained by mixing a Georgia Pacific liquor with a Canadian Forest Products liquor. All of the black liquors tested in this study were Newtonian. A 40-mesh strainer installed in the sample stream of the pilot plant protected the on-line instruments from the crystalline particles.

Before each run, the sample stream was cleaned with low-pressure steam followed by circulating hot water. The main tank and the loop pipes were kept at the nominal temperature for several hours to ensure thermal and physical homogeneity of black liquor. To collect samples for laboratory characterization, we briefly stopped the flow through the sample loop and main loop and withdrew a 1 L sample. Samples were collected from both the main loop and the sample loop simultaneously and were compared to verify sample integrity. The flow was stopped, and a sampling bomb was used to ensure that flashing did not occur.

Experiments were run in the pilot flow loop at different temperatures for each of the 14 concentrations. The highest temperature reached in the main tank was limited to 10–20°C below the boiling point of the black liquor. This limitation was imposed to minimize evaporation and to eliminate or minimize the degradation that can occur in liquors at high solids concentration when held at high temperatures. For experiments at temperatures above the atmospheric boiling point of black liquor, the heater in the sample loop was used to bring the liquor to the desired temperature.

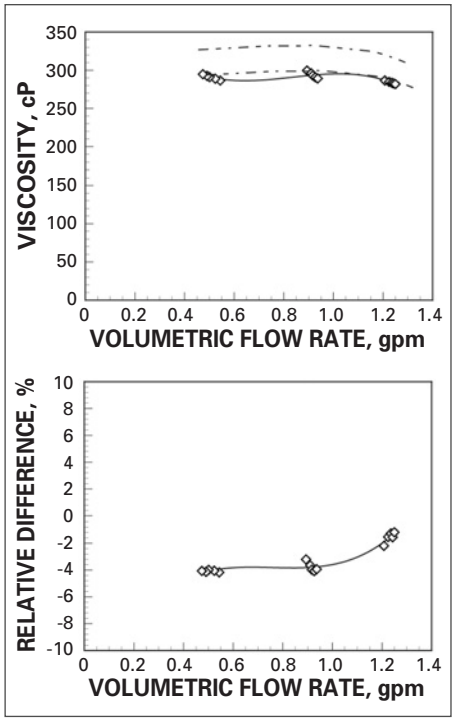
Prior to the beginning of the runs, the coriolis mass flowmeter and the differential pressure cell were zeroed, and a one-point viscosity calibration was performed. The viscometer line was fully flooded, and the two isolation valves shown at the right-most edge of Fig. 1 were activated to develop a no-flow condition in the viscometer line. The instrument readings were zeroed via the instrument software.

To obtain a one-point viscosity calibration, we maintained the black liquor at its most highly viscous level (i.e., at the lowest temperature considered for the series of runs). The flow through the sample loop was maintained at the velocity that creates the highest differential pressure measurable while still in the laminar regime. Under these conditions, the laboratory-measured value of the black liquor viscosity at the temperature of the coriolis sensor was entered into the instrument software. The transmitter calculates the proportionality constant ϕ in Eq. 1 and enters it into the instrument microprocessor.

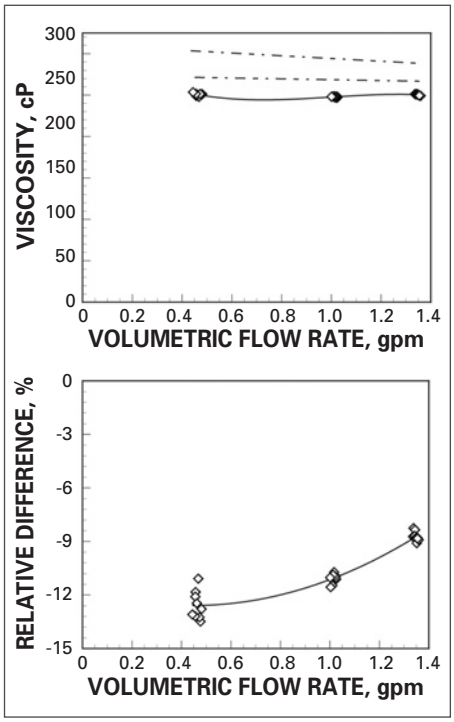
DISCUSSION OF EXPERIMENTAL RESULTS

We characterized the solids compositions of the black liquors and established the relationships between the viscosity and flow rate at different temperatures. We also carried out a study of the transient response of the instrument to temperature changes. Such open-loop studies on the process are necessary to determine the feasibility of controlling liquor viscosity based on widely used strategies such as "lambda tuning" [23].

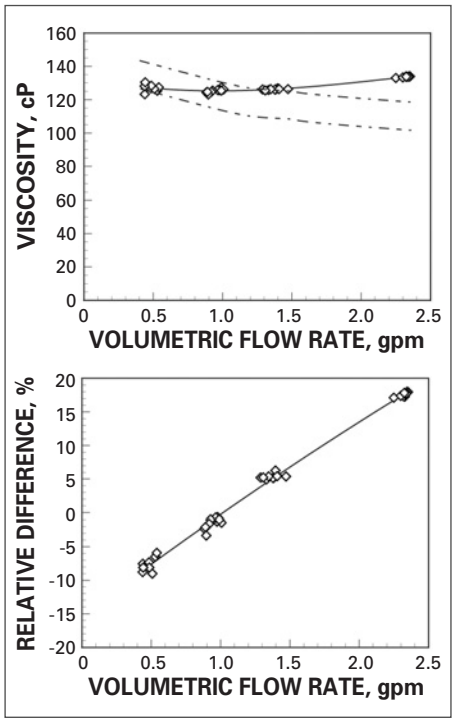
Figure 3 shows the results obtained for a high-viscosity Georgia Pacific black liquor (68.39% solids) at approximately 85°C. The viscosity measurement at each flow rate was taken after steady state had been established. The diamond-shaped markers in the upper plot show the viscosity values measured by the instrument. The continuous line joining the markers is included as a visual aid to track the data trend and was obtained by an arbitrary polynomial fit. The upper plot also shows a laboratory reference band of viscosity values defined by two dash–dot lines.



3. Capillary–coriolis viscometer with black liquor at about 85°C (Georgia Pacific, 68.39% solids). (To convert viscosity to Pa•s, divide by 1000. To convert volumetric flow rate to m^3/s, multiply by 6.309×10^{-5}).



4. Capillary–coriolis viscometer with solids black liquor at about 80°C.



5. Capillary–coriolis viscometer with black liquor at about 95°C (Georgia Pacific, 65.51% solids).

Flow rate,[1] gal/min	Temp.,[2] °C	Viscosity,[3] cP
0.544	84.8	286.62
0.524	84.7	288.94
0.474	84.4	294.85
0.911	84.4	296.12
0.937	84.7	289.28
1.208	85.1	286.67
1.225	85.3	284.75
1.250	85.5	281.95
1.243	85.4	282.78
1.250	85.5	281.95

[1] To convert to m^3/s, multiply by 6.309×10^{-5}.
[2] To convert to K, add 273.15.
[3] To convert to Pa•s, divide by 1000.

II. Flow rate, temperature, and viscosity data for Fig. 3.

Two to four different laboratory viscometers were used to determine the viscosity as a function of temperature for the samples collected from the flow loop. We used a Haake Rotovisco RV12 Rotational viscometer, a Cambridge Sliding-Element laboratory viscometer, a Brookfield Rotating Cup viscometer, and a Nametre 1810 MX viscometer for laboratory measurements. The upper and lower band edges correspond to the highest and the lowest viscosity predictions, respectively, produced by all the laboratory instruments. Any measurements that fall inside the reference band are considered to be consistent with the laboratory measurements and are deemed accurate. The width of the reference band corresponds to a deviation of approximately ±5% from the mean viscosity measured by all the laboratory instruments.

The lower plot in Fig. 3 is a measure of the percentage relative difference between the viscosity measurement produced by the on-line instrument (η) and the viscosity predicted by a correlation generated from a reliable laboratory instrument (η_{ref}):

$$\%\ \text{rel. diff.} = \frac{\eta - \eta_{ref}}{\eta_{ref}} \times 100 \qquad (4)$$

We considered an on-line instrument to be accurate if the relative difference was no more than ±10%. For this particular liquor at 68.39% solids concentration, the laboratory reference viscosity was determined from the experimental relationship defined by Eq. 5:

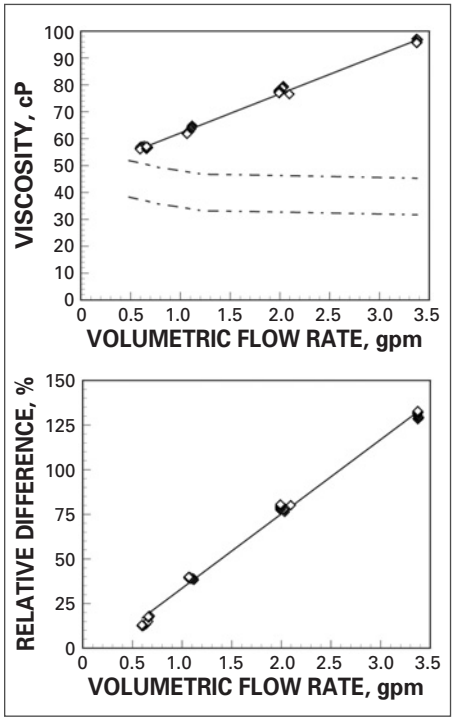
$$\eta_{ref} = \exp\left(A - \frac{B}{T_K} + \frac{C}{T_K^2} \right) \qquad (5)$$

where

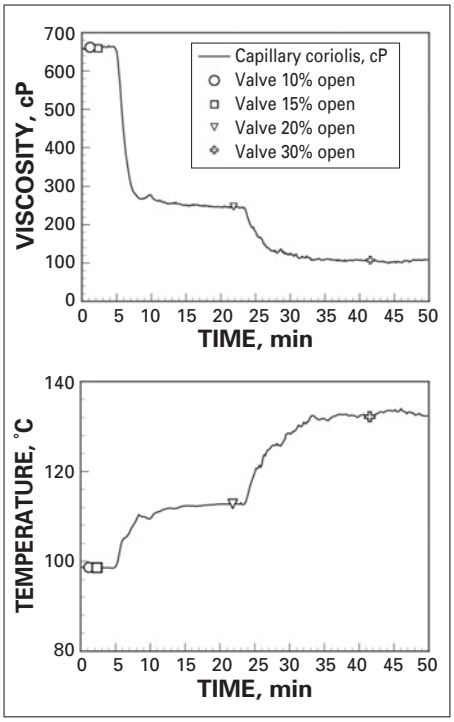
η_{ref} = laboratory-determined reference viscosity, cP
T_K = temperature, K
A = 18.2073771, dimensionless
B = –17633.4415, dimensionless
C = 4709530.7768, dimensionless.

In regard to Fig. 3, the temperature was not strictly constant during the run; in fact, as the flow rate varied, normally the temperature also varied. The temperature fluctuations observed were usually no greater than 1°C. However, since the viscosity of black liquor is highly sensitive to temperature, such small variations must be taken into account for accurate work. **Table II** gives the viscosity and temperature data for 10 selected flow rates plotted in Fig. 3.

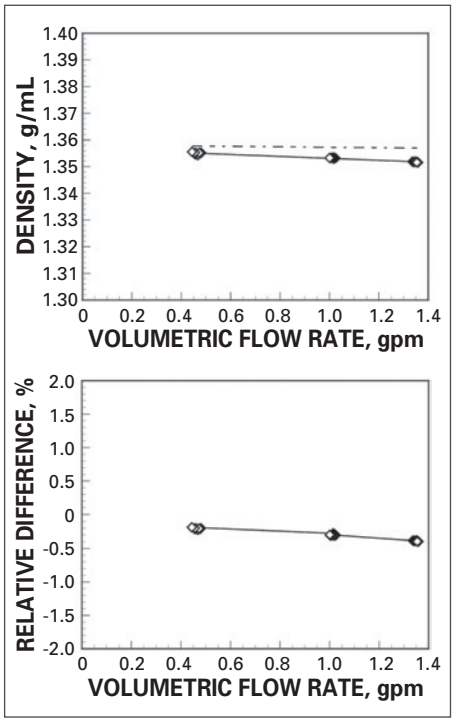
Figure 3 shows that the capillary–coriolis viscometer responds with good accuracy, since the measurements lie close to the lower edge of the laboratory reference band. The percentage relative difference during the run was less than –5% (as shown in the lower plot in Fig. 3), which satisfies the ±10% accuracy specification. Furthermore, the figure shows that the measurements were unaffected by flow rate, since the data follow the trend defined by the edges of the laboratory reference band (which in turn are affected only by the temperature).



6. Capillary–coriolis viscometer with solids black liquor at about 125°C (Georgia Pacific, 68.39%).



7. Capillary–coriolis viscometer transient temperature run (mix of Georgia Pacific and Canadian Forest Products black liquors, 67.46% solids).



8. Density measurement from the capillary–coriolis viscometer system at about 80°C (Georgia Pacific, 65.51%).

Note that the instrument tracked the changes in viscosity that occurred as a result of small temperature changes that took place at different flow rates. The maximum flow rate for this run was 1.25 gal/min. [1 gal/min = 6.3090 10^{-5} m^3/s]

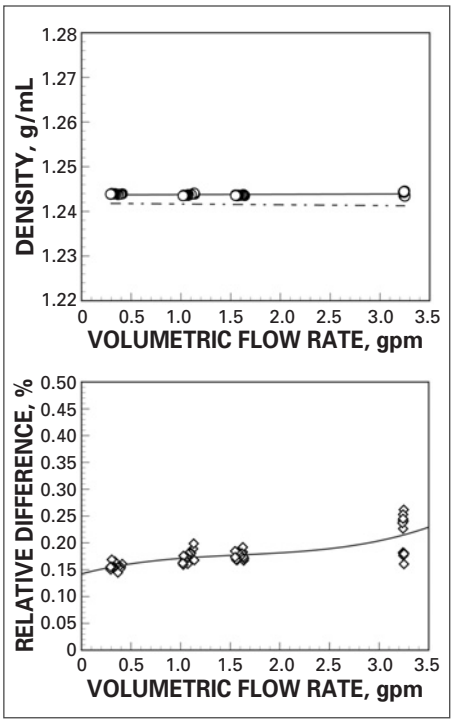
Figure 4 shows another high-viscosity run, this time with a Georgia Pacific black liquor of 65.51% solids at approximately 80°C. Again, the viscometer tracks just below the lower laboratory reference boundary with a percentage relative difference of less than –12%. Higher flow rates were not possible because of the limitations of the differential pressure cell selected for this application. Figures 3 and 4 show typical high-viscosity results for this instrument. In both cases, there is a minor offset with excellent tracking.

Figure 5 shows a mid-range viscosity run with the same black liquor (Georgia Pacific, 65.51% solids) at approximately 95°C. At 0.5 gal/min, the viscometer reading is within the laboratory reference band; however, as the flow rate increased, so did the viscosity reading. This run shows a transition to a linear rising response in the viscosity measurement, where the instrument loses accuracy although it retains a remarkable level of reproducibility, or good precision. (This deviation effect is discussed in more detail later.)

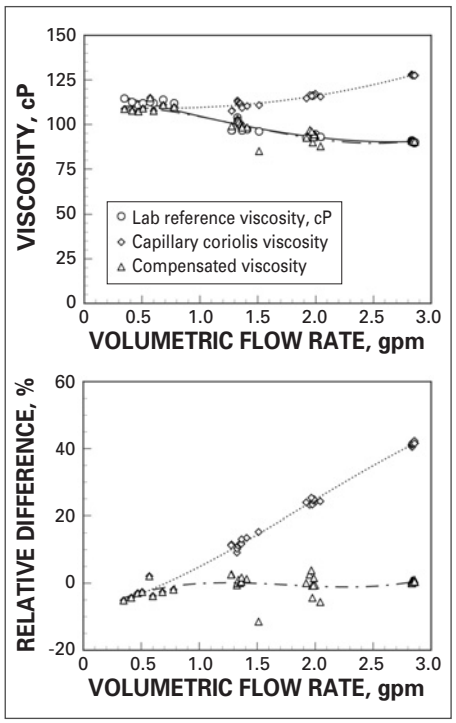
Figure 6 shows a low-viscosity run with a Georgia Pacific black liquor (68.39% solids) at approximately 125°C. This run demonstrates a more dramatic increase in the viscosity measurement as the flow rate increases. Here, the viscosity measurement at 0.75 gal/min was slightly above the laboratory reference boundary with a value of 0.055 Pa•s. The viscosity measurement steadily increased as the flow rate increased, even though the laboratory reference viscosity remained relatively constant. Effective techniques for eliminating the observed errors are discussed in the next section.

Figure 7 shows the transient response study of the capillary–coriolis viscometer for step changes in temperatures. The liquor used was a mixture of Georgia Pacific and Canadian Forest Products black liquors with a solids content of 67.46%. The upper plot shows the viscosity as a function of time measured by the on-line instrument. The lower plot shows the temperature history during the run. In this experiment, we changed the temperature of the flowing black liquor steam in a stepwise fashion by opening the steam control valve (V57 in Fig. 1) that feeds an indirect heater. The step changes in the valve opening were made in discrete increments, as shown in the legend of the figure, until the temperature reached a saturation value. The four symbols (circle, square, triangle, and cross) denote the instants when the valve opening was changed.

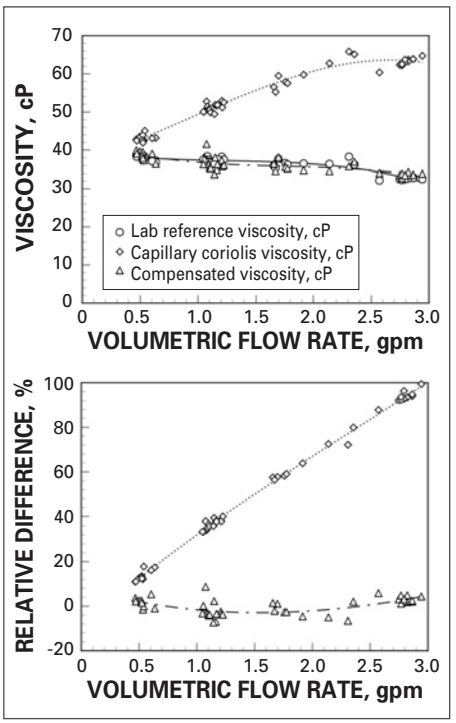
At time zero, the temperature in the sample loop was maintained at approximately 98°C, and the system was at steady state. The corresponding steady-state viscosity was 0.66 Pa•s. At approximately 2 min into the run, we introduced the first step input by manually opening the steam valve to 10%. A second temperature step input was applied 3 min into the run. There is approximately a 3-min process delay before the RTD registers a temperature change at the point where the liquor exits the heat exchanger. After this process delay, the capillary–coriolis viscometer responds with an instrument delay that does not exceed 30 s. During this temperature step change, there is a drop in viscosity from 0.66 Pa•s to 0.25 Pa•s. For the third step input (where the valve is set to 20% open), the delay of the viscometer is actually slightly shorter than the process delay. The viscosity decreases from 0.25 Pa•s to 0.10 Pa•s. To maintain accurate viscosity measure-



9. Density measurement from the capillary-coriolis viscometer system at about 35°C (Georgia Pacific, 42.20%).



10. Capillary-coriolis viscometer viscosity compensation at about 128°C (Georgia Pacific, 65.51%) .



11. Capillary-coriolis viscometer viscosity compensation at about 75°C (St. Laurent, 59.28%).

ments from the instrument, a low flow rate was maintained throughout the run.

Figure 8 shows the result of on-line density measurements with the Georgia Pacific black liquor of 65.51% solids at approximately 80°C. This run demonstrates the excellent accuracy and precision of the density measurement of the coriolis mass flowmeter, a part of the capillary–coriolis viscometer.The density measurements were within –1% of the laboratory correlations.

Figure 9 shows the results of on-line density measurements with the Georgia Pacific black liquor of 42.20% solids at approximately 35°C. The density measurements were within 0.3% of the laboratory correlations. In general, it was found that the density measurements were within ±2% of the laboratory correlations for a wide range of operating conditions.

EFFECTS OF SECONDARY FLOW

The problem of secondary flows arises whenever a fluid passes through a curved section of pipe.The onset and the effect of secondary flows in curved pipes has received considerable attention in fluid mechanics literature because secondary flow is important in engineering and biological systems [24]. When a fluid flows through a curved pipe, the fluid at the center moves faster than the fluid at the outer walls. As a result of centrifugal forces, the faster-moving fluid at the center is forced outwards, while the slower-moving fluid is forced inwards where the pressure is lower. This phenomenon creates a secondary flow at right angles to the main axial flow. A dimensionless number, called the Dean number, is typically used to characterize the flow in a curved pipe and is defined as being equal to the Reynolds number times the quotient of the diameter of the pipe divided by the radius of curvature.

The Dean number represents the product of the ratio of (inertial force)/(viscous force) and the ratio of (centrifugal force)/(viscous force). Depending on the radius of curvature and the Dean number, the axial velocity distribution is affected, which results in increased pressure drop.The actual flow patterns and the stability and the symmetry of the secondary flows depend on the magnitude and interaction of the viscous, inertial, and the centrifugal forces in the system. For various geometries, the secondary flow effects have been theoretically predicted via numerical and analytical studies and have also been verified by flow visualization experiments [25, 26].

While it may be possible to study and quantify the secondary flow effects for the specific geometry used in this instrument, such an analysis is beyond the scope of this work. Rather, we will focus on the effect of the secondary flow on the viscosity measurement and the possible ways to overcome the adverse effect by empirically fitting the measured data or by modifying measurement strategies.

The secondary-flow effects can be minimized by examining the viscosity ranges of the application and limiting the flow through the mass flow sensor. For example, experimental trials with the St. Laurent solids black liquor of 59.28% at approximately 65°C demonstrated that the viscosity measurement remained constant at 0.2 Pa•s for flow rates up to 1.8 gal/min before the differential pressure cell reached its operational limit. The upper flow rate limits for black liquors with viscosity in the range of 0.2–0.3 Pa•s were not reached in the pilot flow loop, however, because of limitations in the differential pressure cell.

Correction by empirical correlations

Under conditions of high flow rate and low viscosity, an additional pressure loss

is created because the secondary flows cause a linearly rising viscosity measurement with increasing flow rate. Even though the instrument loses accuracy under such conditions, we found from our experience that it still maintains excellent precision and reproducibility. Hence, for operation at a particular volumetric flow rate, a correction factor may be applied by comparing the result with laboratory viscosity. However, we found the response to the secondary flows to be systematic; hence, a compensation correlation may be developed that covers a wider range of operating conditions.

The instrument manufacturer has proposed using the following empirical power-law equation for the measured viscosity to provide a correction to the effects of secondary flow:

$$\eta = k \frac{\Delta p}{(m/\rho)^n} \qquad (6)$$

where

η = measured viscosity, cP
n = correction exponent, dimensionless
k = proportionality constant, cP•gal^m/min^n/psi.

Here, n and k are compensation parameters introduced to account for secondary-flow effects that must be determined experimentally. The original formulation of Eq. 1 is recovered from Eq. 6 for the special case where n = 1 and k = ϕ.

We carried out an optimization study, leaving both n and k as the free parameters. The results are plotted in the graphs of **Figs. 10 and 11**. In Fig. 10, for the Georgia Pacific liquor with 65.51% solids, the optimal compensation parameters were 1.322 for n and 10.05 cP•gal$^{1.322}$/min$^{1.322}$/psi for k. In Fig. 11, for the St. Laurent Paper Products liquor with a solids content of 59.28%, the optimal parameters were n = 1.316 and k = 6.98 cP•gal$^{1.316}$/min$^{1.316}$/psi.

For the data represented in Figs. 10 and 11, the secondary flow has an obvious effect on the measurements of experiments conducted at flow rates greater than approximately 1 gal/min. At lower flow rates, the measurements agreed with the laboratory measurements. Hence, the compensation scheme was applied to data collected for flow rates greater than 1 gal/min.

Figure 10 shows that the compensation parameters for the Georgia Pacific liquor are within ±10% of the laboratory reference; however, the optimal n value is not necessarily a constant for different black liquor types. For the data represented in Fig. 11, the compensation parameters produce corrections that are within ±5% of the laboratory reference.

Based on our experience, it is best to limit the viscosity measurements to operation either above or below the critical flow rate at which the onset of secondary flow occurs. If the operation is below the critical flow rate, no correction is required. On the other hand, if the operation is in the range in which the effect of secondary flow is appreciable, then the empirical correction scheme can be employed.

Alternative measurement strategy for pressure drop

For calculating the viscosity of an incompressible Newtonian fluid via the Hagen-Poiseuille equation, one needs the volumetric flow rate and the pressure drop per unit of length. The actual design of the coriolis mass flowmeter used in the pilot flow loop has a pair of tubes in the shape of coat hangers, rather than a U shape. The pressure drop is measured across the coriolis mass flowmeter itself.

The fluid passes through as many as eight curved sections between the pressure taps. These turns cause the secondary flow effects that lead to an increased pressure drop and hence to an exaggerated value for the measured viscosity.

The simplest way to eliminate this problem is to measure the pressure drop for the fully developed flow across a piece of straight tube placed either before or after the coriolis mass flowmeter. To ensure the development of a fully developed laminar flow profile, a straight pipe run of about 20 tube diameters must be provided on either side of the pressure taps. In this case, the standard Hagen-Poiseuille equation can be used without any modification.

CONCLUSIONS AND RECOMMENDATIONS

The capillary–coriolis viscometer can be used to monitor black liquor viscosity effectively on line. The equipment operates satisfactorily in laminar flow, under both steady state and transient conditions, producing very fast, accurate, and reproducible measurements. Furthermore, there was no evidence of fouling during the trials. Because there are no moving parts in the process stream, the instrument is very robust. In addition to viscosity data, the instrument provides information on mass flow rate, volumetric flow rate, temperature, differential pressure, and density.

Some specific points need to be made:

- The instrument reading was unaffected by particles smaller than a 40-mesh size.
- The transient response time to temperature changes is very rapid, with a lag time of 30 s or less.
- This instrument requires a one-point calibration with a laboratory reference for Newtonian fluids.
- The viscometer used in this study tracked reliably, close to the ±10% of the laboratory reference at high viscosities.

The viscometer was used in the pilot flow loop to measure viscosities from 0.005 × Pa•s up to about 1.04 Pa•s with acceptable accuracy. In experiments run with low-viscosity liquors, a linear increase of the measured viscosity as a function of flow rate was encountered, causing erroneous measurements, most likely as a consequence of secondary flow effects. Such errors on low-viscosity runs were compounded by the fact that the instrument may have been inadvertently calibrated under conditions under which the secondary flows were significant, instead of being calibrated under the normal conditions of a regime of high viscosity and high pressure drop.

At flow rates lower than 0.5 gal/min, the instrument measured the viscosity accurately, even at low viscosities. For low-viscosity flows, an empirical correlation was developed to compensate for these effects, effectively eliminating the error. Alternatively, to overcome the adverse effects, the pressure drop could be measured across a straight pipe for fully developed flow.

With proper sizing of the mass flow rate sensor and of the differential pressure cell, the instrument can operate reliably over the ranges expected to be

encountered in a mill. Based on our experience, we recommend that the viscometer be used only in a slip stream for flow rate control through the instrument. **TJ**

ACKNOWLEDGMENT

The authors are grateful for the financial support of the U.S. Dept. of Energy provided through grant number DE-FC-02-93CH10564 and for the financial and technical support from many industrial partners. Support for the project was also received from the National Science Foundation under grant number CTS 9502936. The authors are grateful to the following companies for providing kraft black liquor: Canadian Forest Products Ltd., St. Laurent Paper Products Corp., and Georgia Pacific Corp. The authors are grateful to Micro Motion Inc. for donating equipment. The authors are grateful for the laboratory assistance of Barbara Speck and the technical assistance of A. A. Zaman and Santiago Tavares.

LITERATURE CITED

1. Empie, H. J., Lien, S. J., Yang, W., and Samuels, D. B., *J. Pulp Paper Sci.* 21(2): J63(1995).
2. Karvinen. R., Hyoty, P., and Sisskonen, P., *TAPPI J.* 74(2): 171(1991).
3. Wells, M. C., "Chemical Recovery Area," in *Mill-Wide Process Control & Information Systems* (Donald B. Brewster, Ed.), TAPPI PRESS, Atlanta, 1993, Vol. 10, Ch. VIII.
4. Wight, M. O., "An investigation of black liquor rheology versus pulping conditions," Ph.D. thesis, University of Maine at Orono, ME, 1985.
5. Fricke, A. L., "Physical properties of kraft black liquors: final report—Phase I and II", U.S. Dept. of Energy Report No. DOE/CE 40606-T5(DE88002991), 1987.
6. Zaman, A. A. and Fricke, A. L, *1991 Forest Products Symposium Proceedings*, American institute of Chemical Engineers, Los Angeles, CA, p. 59.
7. Zaman, A. A., "An investigation of the rheological properties of high solids kraft black liquors," Ph.D. thesis, University of Florida, Gainesville, FL, 1993.
8. Zaman, A. A., and Fricke. A. L., *I & EC Research* 33: 428(1994).
9. Zaman, A. A., and Fricke, A. L., *I & EC Research* 34: 382(1995).
10. Zaman, A. A., and Fricke, A. L., *Chem. Eng. Commun.* 139: 201(1995).
11. Empie, A. J., "Kraft black liquors delivery systems, final report," U.S. Dept. of Energy Contract No. DE-FG02-88CE40839, DOE/CE/40839-TE (DE95015552), February 1995.
12. Adams, T. N., and Frederick, J., *Kraft Recovery Operations Seminar Notes*, TAPPI PRESS, Atlanta, 1989, p. 123.
13. Basker, V. R., Dutka, A. P., Crisalle, O. D., and Fricke, A. L., *Paperi Puu - Paper and Timber* 28(7): 464(2000).
14. Fricke, A. L., and Crisalle, O. D., "Development of viscometers for kraft black liquor, final report – Phase I, II, IIA, and III," U.S. Dept. of Energy Contract No. DEFCO2-93CH10564, November 1999.
15. Gibbs, R. A., "The design of a flow loop for the evaluation of on-line viscometers for black liquor streams in kraft pulping mills," M.S. thesis, University of Florida, 1994.
16. Dutka, A. P., "The development and evaluation of on-line viscometers for kraft black liquor," M.S. thesis, University of Florida, Gainesville, FL 1996.
17. Bird, R. B., Stewart, W. E., and Lightfoot, E. N., *Transport Phenomena*, John Wiley & Sons, New York, 1960.
18. Stommel, H. M., and Moore, D, W., *An Introduction to the Coriolis Force*, Columbia University Press, New York, 1989.
19. Cascetta, F., Cignolo, R. G., Martini, A. R., and Vigo, P., *Trans. Institute of Measurement and Control* 14(2): 99(1992).
20. Cascetta, F., *ISA Transactions* 35(4): 365(1996).
21. *DMS Density Monitoring Instruction Manual*, Micro Motion Inc., Boulder, CO, 1993.
22. *ELITE Model RFT 9739 Field-Mount Transmitter Instruction Manual*, Micro Motion Inc., Boulder, CO, 1992.
23. Thomasson, F. Y., "Controller Tuning Methods," in *Process Control Fundamentals for the Pulp & Paper Industry* (Nancy J. Sell, Ed.), TAPPI PRESS, Atlanta, 1995, Ch. VI.
24. Berger, S. A., Talbot, L., and Yao, L. S., *Ann. Rev. Fluid. Mech.* 15: 461(1983).
25. Ito, H., *JSME International J.* 30(262): 543(1983).
26. Cheng, K. C., Takuma, M., and Kamiya, Y., *J. of Flow Visualization and Image Processing* 2: 1(1995).

Received: November 26, 1997
Revised: July 22, 2003
Accepted: October 20, 2003

This paper is also published on TAPPI's web site <www.tappi.org> and summarized in the March *Solutions! for People, Processes and Paper* magazine (Vol. 87 No. 3)

Dutka, Basker, Crisalle, and Fricke are with the Chemical Engineering Dept., University of Florida, Gainesville, FL 32611-6005. Kalotay, now retired, was with Micro Motion Inc. at the time of this work, Boulder, CO 80301. Email Crisalle at crisalle@che.ufl.edu.


Dutka


Basker


Crisalle


Fricke


Kalotay