DOE/GO/10564-F

DEVELOPMENT OF VISCOMETERS FOR KRAFT BLACK LIQUOR

FINAL REPORT — PHASES I, II, IIA, AND III

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Work Performed for the U.S. Department of Energy, Office of Industrial Technologies
Under Cooperative Agreement DE-FC02-93CH10564

November 1999

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EXECUTIVE SUMMARY

This report documents the results of performance evaluations and final recommendations for several prototype sensors designed for use as on-line viscometers for black liquor application. More specifically, the report addresses all the goals established for Phase I through Phase III of the US Department of Energy Cooperative Agreement entitled *Development of Viscometers for Kraft Black Liquor* (DE-FC02-93CH10564).

The motivation for this study is to fulfill the need for an extensive evaluation of instruments for measuring the viscosity of black liquor. The viscosity of the black liquor has a significant effect on the effectiveness of all the major operations used to process the liquor through a series of evaporators and concentrators, storage tanks, and its ultimate firing in the recovery boiler. In spite of its relevance, the viscosity is not routinely measured on-line in most pulping mills. Instrument manufacturers encounter a major economic barrier when developing viscometers for black liquor applications because of the relatively high risk that the costs of testing and design may not be recoverable if the new market fails to provide a large and exclusive customer base. The development process involves significant technical barriers given that the sensors must work in hostile environment characterized by high temperatures, corrosive liquids and gases, and the presence of suspended solids. Furthermore, the viscosity is a sensitive function of temperature and solids content.

During Phase I of the project six prototype on-line viscometers were identified as candidates for further development and evaluation. These instruments represented five different viscosity measurement principles, namely, (1) pressure flow in a tube, (2) drag flow between coaxial cylinders, (3) damping of vibrations of a vibrating element, (4) combined pressure and drag flow on a moving element, and (5) fluid relaxation after exposure to a pulsed magnetic field (nuclear magnetic resonance). Three North-American mills were identified as partners that would provide black liquor samples and participate in a final instrument evaluation at their industrial sites. The participating paper companies were (1) St. Laurent Paper Products Corporation of West Point, Virginia, (2) Canadian Forest Products Ltd. of Prince George, BC, and (3) Georgia Pacific Corporation of Palatka, Florida.

During Phase II of the project the six prototype on-line viscometers were evaluated at the University of Florida using laboratory facilities and a pilot flow-loop plant designed to simulate industrial process flow conditions. The viscometers were tested for accuracy and repeatability under varying process conditions, such as different black liquor species, solids content, temperature, flow rate, and contaminants, as well as for maintenance and reliability. The accuracy

of the on-line instruments was determined by direct comparison with measurements taken under controlled laboratory conditions with reliable off-line viscometers used as reference. It was concluded that three of the viscometers tested satisfied the required performance and reliability requirements, namely a torsional-vibrational viscometer manufactured by Nametre Company of Metuchen, NJ, a capillary-coriolis viscometer manufactured by Micro Motion Inc. of Boulder, CO, and a cylindrical-rotary viscometer manufactured by Brookfield engineering Laboratories Inc. of Stoughton MA. These three instruments produced viscosity measurements with an accuracy better than ±10% with respect to laboratory references, and performed without excessive maintenance requirements, and therefore were deemed as adequate for further evaluation in a subsequent phase of trials in mill environments.

During Phase III of the project the instruments that successfully passed the testing criteria during the previous phase were installed in black-liquor recovery facilities in three different mills. The Nametre instrument was installed in the St. Laurent Paper Products Corporation site, and operated successfully for a period of approximately six months without experiencing any malfunctions or without extensive maintenance needs. The accuracy of the viscosity measurements taken by the on-line instrument was estimated to be approximately ±16% with respect to laboratory references. Based on the successful testing results it can be concluded that the Nametre instrument can be recommended for use in black liquor applications in industrial paper mills.

The Micro Motion capillary-coriolis instrument was installed in the Canadian Forest Products Ltd. mill. The instrument provided on-line viscosity measurements with an accuracy of approximately ±19.5%, in addition to generating density measurement with an accuracy better than ±0.2%, as well as mass flow rate data. Due to the incorrect installation of the side-stream that fed the viscometer it was not possible to test this instrument for extended periods of time in order to obtain representative reliability and maintenance-requirements data; however, no fault can be attributed to the viscometer itself. It is concluded that this instrument can also be recommended for use in a mill environment .

The Brookfield cylindrical-rotary viscometer was installed in the Georgia Pacific Corporation mill. A number of failures were observed due to improper installation of the instrument and damage that occurred due to overstressing of the torque element. In spite of the excellent performance displayed by this instrument in the pilot plant facilities of the University of Florida, the mill trials did not succeed in making the viscometer operational for a significant period of time. Although the few measurements that were taken during the trials appear to be of acceptable accuracy, the lack of more extensive data does not support the conclusion that the instrument is acceptable at this time for reliable use in a typical mill environment. Nevertheless, it is reasonable to expect that this viscometer would operate satisfactorily in a mill environment if it were installed with appropriate protection against suspended solid particles and line pluggage.

The experience during the mill trials made evident that the success of an on-line viscometer is strongly dependent on the quality of the installation of the side-stream that supports the instrument.

In particular, failures were realized in all cases when the side-stream lines were not insulated and when they were not heated via a steam-tracing line or electrical tape. In addition during Phase III of the project the black liquor of three mills involved in the study experienced an increasing presence of large particles in the liquor as a consequence of on-going efforts to close the mill. Therefore, an important part of the installation should be the inclusion of screens or strainers that can prevent blockage of the side stream by large particles.

The other viscometer prototypes tested were not recommended for mill trials for various reasons. In particular, results obtained using a magnetic-resonance viscometer under the joint development of Southwest Research Institute and Quantum Magnetics showed great promise for viscosity measurement and potentially for on-line solids-contents measurement. We tested only a preliminary prototype, and could therefore not pursue its evaluation in a mill site. Based on the results obtained we strongly encourage the further development of this technology for black liquor applications. Much development work was also done with a prototype sliding-element viscometer manufactured by Cambridge Applied Systems. The instrument showed remarkable accuracy and reproducibility for viscosity measurement, but only a laboratory (off-line) version of this instrument was delivered by the manufacturer, and therefore the instrument was not suitable for on-line mill trials. We also believe that this viscometer holds potential for success in black-liquor applications provided that the manufacturer verifies the reliability of the instrument for loading and discharging black-liquor samples on line. Finally, an viscometer design was pursued jointly with a team from Stevens Institute of Technology based on a new method for measuring pressure drop. Although this instrument holds potential for black liquor applications, the developers were not able to provide a prototype for testing under this program.

In summary, this project has successfully attained its ultimate objectives by identifying and testing in a pilot plant environment and in paper mill facilities two on-line viscometers that can be used for black liquor applications in industrial environments. In addition, a third viscometer that has been qualified through extensive pilot plant testing holds promise for deployment in a paper mill with appropriate installation procedures. The work has also produced a viscometer testing methodology that can be used as a paradigm for further instrument development and evaluation. Specific recommendations for the installation of the instruments are provided to ensure reliable operation with reasonable maintenance requirements.

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1. INTRODUCTION

This report documents the results of the evaluation of viscometers designed for the on-line measurement of the viscosity of kraft black-liquor. The testing was carried out at the laboratories and pilot-plant facilities of the University of Florida in Gainesville, Florida, and at three North American mills. The work reported in this document is sponsored by the US Department of Energy under DOE Cooperative Agreement DE-FC02-93CH10564.

Several on-line viscometers marketed by commercial instrumentation companies were selected for the study with the goal of encompassing a wide spectrum of viscosity measurement principles, such as sliding-element, rotating cup, capillary-coriolis, and vibrational-element principles, among others. The majority of the instruments considered are available commercially, with only few exceptions representing instrumentation principles that were still under development.

A comprehensive testing of the instruments was first done at the University of Florida in a fully automated pilot flow loop with precise control of pressure, flow, and temperature, that permitted testing the viscometers under various conditions that emulate a pulping mill environment. The performance of each on-line instrument was quantified in terms of its precision relative to that of laboratory viscometers, and in terms of its reliability and maintenance requirements. Interactions with the instrument manufacturers lead to design or implementation modifications intended to improve the instrument performance. This pilot-plant phase of the project identified three viscometers that successfully demonstrated potential for good performance as an on-line instrument in an actual paper mill environment. In the final phase of the project the three viscometers selected through the pilot-plant studies were subjected to further testing in a mill environment where the study was primarily conducted through the interaction of mill personnel and representatives of the instrumentation companies.

The following sections describe the economic and technical barriers that motivated the comprehensive study undertaken, document the industrial significance of the viscosity of black liquors, and provides a succinct overview of the program.

1.1 Economic and Technical Barriers

The economics of establishing a new market for viscometers, such as black liquor processing applications, is dominated by the fact that viscometer manufacturers are typically small companies, that produce instruments mostly for laboratory use rather than for on-line deployment. These

manufacturers typically lack the capital necessary to invest in the high cost of development instruments for new markets; furthermore, these costs may not be recoverable if they were incurred by a single manufacturer should the new market fail to provide a large and exclusive customer base. Even though instruments manufacturers and the pulp and paper industry would reap benefits from the availability of reliable on-line viscometers, development of such technology would not be possible without external subsidies such as the funding provided under this Department of Energy cooperative agreement.

The successful development of effective and reliable on-line viscometers for black-liquor applications must successfully overcome a number of technical barriers as listed below:

1. The sensors must work in hostile environment characterized by high temperatures, corrosive liquids and gases, and the presence of suspended solids.

2. Since the viscosity may be a very sensitive function of temperature, the accuracy of the viscosity sensors requires the delivery of very precise temperature control of the fluid and of the instrument itself.

3. The viscosity of a material is a transport property, requiring that the correct flow geometry and measurement conditions be carefully controlled. This is in contrast to the measurement requirement of other quantities which are more intrinsically basic such as concentration, intensive or extensive thermodynamic properties (temperature, pressure, etc.), mass, force, length, etc. In general, the determination of viscosity requires the measurement of the flux produced by a driving force under controlled conditions. More specifically, the torque, displacement, or pressure drop produced by a known shear rate must be measured. Therefore, a number of significant technical challenges must be resolved in order to produce a reliable on-line measurement of viscosity.

1.2 Industrial Importance of Kraft Black Liquor Viscosity

Currently, kraft pulping is used for more than 90% of the chemical pulping in the US and Canada. Except for a very small fraction that is used as a feed stock for the production of lignin, kraft black liquor is used on site as a fuel for energy recovery in a Tomlinson furnace. The scale of kraft recovery operations is very large. More than 73 million metric tons of black liquor solids are processed annually in the US and Canada, requiring that nearly 300 million metric tons of water be evaporated annually. Studies have shown that an increase in solids concentration of black liquors fired in recovery furnaces will lead to a very large increase in energy recovery, and also to

increased capacity in recovery systems [Empie *et al.*, 1993]. For example, if the solids concentration were to be increased from 70% to 75%, there would be an additional energy recovery of 3.1 10^{13} BTU/yr (3.27 10^{10} MJ/yr) for the entire U.S. industry [Empie *et al.*, 1995a]. Furthermore, high-solids firing reduces emissions to the environment, improves the efficiency of smelt reduction, and improves boiler efficiency [Karvinen, 1991].

However, operational and safety considerations impede the shift towards firing at increasingly higher solids content. For example, for black liquor samples taken from a typical Northwestern U.S. mill, it was found that at the normal firing temperature the viscosity increased from 90 cP (0.09 Pa·s) at 73% solids to 190 cP (0.19 Pa·s) at 75.6% solids, and to 600 cP (0.60 Pa·s) at 80% solids. Such high viscosities create enormous difficulties in transport, handling, and firing of the liquor in the furnace, which has led to conservative operations in the interest of safety.

Results to date show that the viscosity is a dominant variable affecting the evaporation, transport, heat transfer, and droplet formation of black liquor [Adams *et al.*, 1989] [Empie *et al.*, 1995]. For example, it is known that the black liquor viscosity plays a major role in the operation of the evaporators and concentrators. In the extreme, an increase of viscosity in the concentrator can result in a flow-regime shift from turbulent to laminar, leading to a rapid build up of the film layer, adversely affecting evaporation efficiency and eventually leading to plugging. In addition, the power consumption for pumping and the heat transfer coefficient vary significantly with viscosity in the forced-circulation heat exchangers commonly used in evaporators. There is also evidence that viscosity is one of the key variables in the droplet formation and droplet size distribution in the recovery furnace [Empie *et al.*, 1995].

The literature shows that the liquor viscosity increases exponentially with solids concentration [Wight, 1985; Fricke, 1987; Zaman, 1993; Zaman and Fricke, 1994]. In fact, the change in viscosity expected for solids concentrations from 18% to 80% over a range of temperatures may be as large as four orders of magnitude [Adams *et al.*, 1989]. The viscosity at high solids is also affected by the pulping conditions [Zaman and Fricke, 1996]. Studies with liquors obtained from laboratory-scale pulping processes indicate that the viscosity may also undergo an irreversible decrease when the liquor at high solids concentration is held at temperatures at or above 100°C (373°K) for a period of hours [Small and Fricke, 1985]. In addition, the viscosity of black liquors at the same temperature and percentage solids can vary widely from mill to mill, or even from day to day in the same mill [Adams *et al.*, 1989].

Black liquor taken from the concentrators is typically heated before it is sprayed into the furnace. The purpose of the heating is to maintain constant viscosity [Wells, 1993] and obtain

improved furnace control. In the absence of reliable on-line viscometers, viscosity control is usually attempted by measuring percentage solids and manipulating the temperature. With the availability of an on-line viscometer, control of viscosity could be achieved by direct measurement while still manipulating the temperature and solids content to control viscosity. Furthermore, by monitoring the viscosity and solids content in the evaporator train, a suitable feedforward-feedback control strategy could be designed and implemented. It is clear that on-line measurement of viscosity is necessary for control if the huge energy savings that are possible by operating at high solids are to be realized. It is essential to develop on-line viscometers for measuring the viscosity of black liquors with solids contents levels from 30% to above 75%.

It may be of interest to note that the advantages of using an on-line viscometer are being realized in the food industry. For example, in the manufacture of skim milk powder it is recognized that using an on-line viscometer to control the degree of evaporation leads to a significant reduction of steam consumption and stack losses, and to a minimization of fouling problems [O'Donnell *et al.*, 1994]. Furthermore, maintaining a constant viscosity also results in a more consistent product as the mean powder particle diameter is directly proportional to the viscosity of the skim milk concentrate [O'Donnell *et al.*, 1995].

1.3 Program Overview

This cooperative-agreement project was organized in eight sequential steps which involved the activities described below.

1. Review of viscosity measurement principles and identification of instrument manufacturing companies that can produce on-line sensors utilizing the measurement principles.

2. Development of supporting relationships with these companies to modify their designs for black-liquor application purposes, if necessary, and to obtain access to their instruments and technical support.

3. Procurement of instruments with necessary modifications for testing with black liquor in a laboratory environment at the University of Florida.

4. Development of relations with pulp mills representing different locations in the U.S. and Canada for participation in the program as providers of black liquor and as sites for final testing of the viscometers.

5. Design and construction of a pilot flow-loop plant at the University of Florida for testing the viscometers as on-line instruments for measuring of black liquor viscosity over wide ranges of closely controlled solids concentration, temperature, and flow rate.

6. Measurement and correlation of properties of liquors from the participating mills.

7. On-line testing of the instruments in the pilot flow loop at the University of Florida and identification of sensors that are deemed accurate, reproducible, and reliable for industrial deployment in a pulp mill. Characterization of the performance of the on-line instruments done by comparison with reliable laboratory (off-line) viscometers.

8. On-line testing of the instruments in the manufacturing facilities of participating pulp mills to evaluate performance of all the instruments that are successfully screened using the pilot flow loop tests described in Step 7.

Phase I of the program encompassed Steps 1-5. Cooperative and intellectual property agreements were established with six American viscometer manufacturers that design instruments using various measurement methods such as vibrational, rotational, and coriolis principles, among others. Five of these manufacturers ultimately made instruments available for the testing program. The participation of three mills was secured, and black liquor was delivered by each participant to the University of Florida. The viscosity of all the liquors was determined in the laboratory as a function of temperature and solids content, and all other relevant physical properties were fully characterized in terms of correlations.

Phase II of the program encompassed the activities of Step 7. All five viscometers available were installed on line at the University of Florida pilot flow-loop where they underwent extensive testing. The viscometers recorded measurements for various conditions of liquor flow rate, temperature, and solids contents. The performance of the on-line instruments was determined by comparison with viscosity values predicted by the laboratory analysis. The instruments were also evaluated in terms of reliability and maintenance requirements. This testing step successfully identified three on-line viscometers that met the performance and reliability requirements for black liquor processing environments, namely (*i*) a torsional-oscillatory viscometer manufactured by the Nametre company, (*ii*) a capillary-coriolis viscometer manufactured by Micro Motion Inc., and (*iii*) a cylindrical rotational viscometer manufactured by Brookfield Engineering Laboratories, Inc.

Phase III of the program addressed the activities of Step 8, namely, the mill trials of the instruments. The deployment of the three viscometers identified in Step 7 was made as follows: (*i*)

the Nametre unit was installed in the facilities of St. Laurent Paper Products Corporation Company (formerly Chesapeake Paper Products) in West Point, Virginia, (*ii*) the Micro Motion unit was installed in the facilities of Canadian Forest Products Ltd., in Prince George, BC, Canada, and (*iii*) the Brookfield unit was installed in the facilities of Georgia Pacific Corporation, in Palatka, Florida. Black liquor samples were taken from the mills and shipped to the University of Florida so that the on-line measurements taken at the mill could be compared with laboratory measurements. The testing experience at the mills showed that the Nametre and the Micro Motion units performed with good accuracy and reliability. The success of these two sensors depended critically on four installation factors, namely (*i*) the use of insulation on the instrument housing and on the piping to and from the instrument, (*ii*) the installation of local heating (steam lines or electrical tape) around the instrument housing, (*iii*) the availability of a steam-flushing mechanism to wash the interior of the instrument as a maintenance measure, and (*vi*) the installation of a screen and a strainer to prevent potential damage and obstruction caused by large solid particles. Both units were shown to require minimal periodic maintenance in the form of steam flushes. The Brookfield unit was installed improperly, and therefore it suffered from frequent flow blockages that put it out of service very frequently; however, in the brief periods when the unit was operational it showed that it was capable of producing accurate measurements.

1.4 Organization of the Report

Section 2 of the report presents the specific models of viscometers considered in the study as representatives of the major principles of viscosity measurement. This section also discusses the evaluation plan, which consists of comparing viscosity measurements taken under carefully controlled laboratory conditions with measurements taken by on-line viscometers installed in the University of Florida pilot flow-loop and with those taken by instruments installed in industrial paper mills.

Section 3 presents the results of the evaluation of a torsional-oscillatory viscometer manufactured by Nametre Company. The results include a brief description of the performance of torsional-oscillatory instruments (*i*) as installed in the University of Florida pilot plant, and (*ii*) as installed in the recovery plant of the St. Laurent Paper Products Corporation Mill at West Point, Virginia.

Section 4 presents the results of the evaluation of a capillary-coriolis viscometer manufactured by Micro Motion Inc. The results include a brief description of the performance of capillary-coriolis instruments (*i*) as installed in the UF pilot flow loop, and (*ii*) as installed in the recovery plant of the Canadian Forest Products Ltd. mill at Prince George, British Columbia.

Section 5 presents the results of the evaluation of a cylindrical-rotary viscometer manufactured by Brookfield Engineering Laboratories Inc. The results include a brief description of the performance of cylindrical-rotary instruments (*i*) as installed in the UF pilot flow loop, and (*ii*) as installed in the recovery plant of the Georgia Pacific Corporation mill at Palatka, Florida.

Section 6 discusses the performance of three other viscometers considered in this study, namely (*i*) a magnetic resonance viscometer developed jointly by Southwest Research Institute and Quantum Magnetics, (*ii*) a sliding-element viscometer manufactured by Cambridge Instrument, and (*iii*) a capillary viscometer proposed by a development team working at Stevens Institute of Technology in Hoboken, New Jersey.

Section 7 presents a summary of conclusions and recommendations for the laboratory (off-line) and for each on-line viscometer studied, and discusses recommended locations for deploying the instruments in a paper mill.

Section 8 is the Appendix to the report. It contains a detailed description of the University of Florida pilot flow-loop plant, a detailed description of the experimental design followed for evaluations in the pilot flow loop, a description of laboratory characterizations, and contact information for the key project participants, including instrument vendors and paper mills.

Section 9 contains a list of all the references cited in the body of the report.

2. INSTRUMENTS CONSIDERED AND EVALUATION PLAN

2.1 General Principles of Measurement and Instruments Considered

The viscosity of a fluid is a transport property of the material that is interpreted as a measure of the flux of momentum caused by shearing a force in the case of a non-elastic materials. This is expressed as

$$\tau = \eta\dot{\gamma} \tag{2.1.1}$$

where τ is the stress tensor, $\dot{\gamma}$ is the shear rate tensor, and η is the viscosity. For the case of a liquid (or a two-phase liquid-solid fluid) the viscosity is a function of the temperature and the composition of the material, and may be also be a function of the shear rate.

To determine the viscosity of a fluid it is preferable to make the measurement in an instrument with two axes of symmetry so that only one set of elements of the tensors are active. In that case, the viscosity can be determined as the ratio of the measured shear rate and shear stress tensor components

$$\eta = \frac{\dot{\gamma}_{ij}}{\tau_{ij}} \tag{2.1.2}$$

where τ_{ij} and $\dot{\gamma}_{ij}$ represent the only active tensor components defined along the i-th direction imposed by the axisymmetric geometry of the measuring instrument.

A significant challenge is that an ideal axisymmetric flow pattern required for equation (2.1.2) to be valid can only be approximated in practice even under the best of conditions, and the flow must be laminar. For example, flow through a tube (or a channel), typically called pressure flow, is one common method used as for designing viscometers because the tube has the required two axes of symmetry, the shear rate can be determined from the volumetric flow rate, and the shear stress can be determined from the pressure drop. However, the tube is of finite length and therefore end corrections are required in order to introduce appropriate compensations. Other idealized symmetries for measurement are possible, but every one requires corrections of one type or another. An important instrument design objective of is to minimize the need for such

corrections.

The following five basic types of symmetrical flows have been used frequently in viscometer design:

1. pressure flow in a tube
2. drag flow between coaxial cylinders
3. damping of vibrations of a vibrating element
4. combined pressure and drag flow on a moving element
5. fluid relaxation after exposure to a pulsed magnetic field (nuclear magnetic resonance)

An objective of this program was to evaluate the use of viscometers belonging of each type of the five measurement principles listed above. All commercial instruments considered were reviewed with the goal of selecting manufacturers who, in our opinion, had developed instruments that could be most readily modified for measurements with black liquor. Contacts with numerous manufacturers were pursued diligently, and resulted in the identification of the following instrument models that were included in the scope of the project:

1. Nametre 2010MX Viscoliner torsional oscillatory viscometer. Manufactured by Nametre Company, Metuchen, NJ.
2. Micro Motion CMF025 capillary-coriolis viscometer. Manufactured by Micro Motion Inc., Boulder CO.
3. Brookfield TT-100 cylindrical rotary viscometer. Manufactured by Brookfield Engineering Laboratories, Inc., Stoughton, MA.
4. NMR Viscometer. Prototype under development under a joint effort of Southwest Research Institute and Quantum Magnetics, San Antonio, TX.
5. Cambridge Applied Systems Sliding-Element Viscometer. Manufactured by Cambridge Applied Systems Inc., Medford, MA.
6. Stevens Institute Capillary Viscometer. Designed by the Design and Manufacture Institute of Stevens Institute of Technology, Hoboken, NJ.

The first four manufacturers indicated appearing in the above list were able to deliver

functional on-line prototypes that underwent extensive testing and evaluation. The NMR (nuclear magnetic resonance) sensor (fourth on the list) is a prototype instrument that was under development at the time of our testing efforts, and was included to carry out an initial investigation of the potential of NMR measurement principle for black liquor applications. The Cambridge Applied Systems instrument (fifth in the list) was delivered only in its laboratory (off-line) form. Although it was not suitable for on-line utilization in an industrial environment, the laboratory Cambridge instrument nevertheless underwent a preliminary test of performance in the pilot flow facility. The Stevens Institute of Technology instrument was not delivered by the manufacturer for evaluation. Contact information for the participating instrument manufacturers is given in the Appendix (see Section 8.4).

2.2 Instrument Evaluation Plan

2.2.1 Evaluation in Laboratory Facilities

The viscosity measurements produced by the on-line viscometers being evaluated need to be compared with reference measurements taken in a laboratory environment. The laboratory instruments utilized included a Haake Rotovisco RV12 rotational viscometer, as well as other standard instruments. In addition, three of the participating viscometer manufacturers provided off-line (laboratory) versions of their on-line instruments, making available the following instrument models:

1. Brookfield TT-PVS rotating cup viscometer for laboratory use. Manufactured by Brookfield Engineering Laboratories, Inc., Stoughton, MA.

2. Nametre 2010MX Viscoliner torsional oscillatory viscometer for laboratory use. Manufactured Nametre Company, Metuchen, NJ.

3. Cambridge Applied Systems Sliding-Element Viscometer for laboratory use. Manufactured by Cambridge Applied Systems Inc., Medford, MA.

More extensive details of the procedures followed for laboratory characterizations are given in the Appendix (see Section 8.3).

2.2.2 Evaluation in Pilot Flow -Loop Facility at the University of Florida

A pilot flow-loop facility was constructed at the University of Florida to evaluate the candidate on-line viscometers. The pilot plant is fully automated and features precise control of pressure, flow, and temperature, that permitted testing the viscometers under various conditions that emulate

a pulping mill environment. Figure 2.1 shows a diagram of the plant, featuring a heated main tank that holds up to 600 gal of liquor that is circulated along a main-stream loop using a progressive cavity pump. The viscometers being evaluated are installed on-line along the sample-stream loop and their readings are captured by a centralized data acquisition and control system. The main loop and the sample loop are connected at a sampling point where a portion of the black liquor is diverted into the sample stream. Once in the sample stream, the black liquor temperature may be increased using a static-mixing heat exchanger before encountering a bank of on-line viscometers connected in series (*cf.* Figure 2.1). The liquor is then returned to the main-stream loop after the temperature is reduced, if necessary, using the cooler. A number of viscometers can be installed in series on the section labeled "viscometer bank" in Figure 2.1.

An extensive process and instrumentation diagram for the flow loop is given in Figure 2.2. The tags VIS 1 to VIS 4 represent the deployment points for the viscometers in the sample stream. Note that the plant has a strainer located between the heater and the bank of viscometers in order to capture very large solid particles that may cause damage to the instruments located downstream. A detailed explanation of the diagram is given in the Appendix (see Section 8.1).

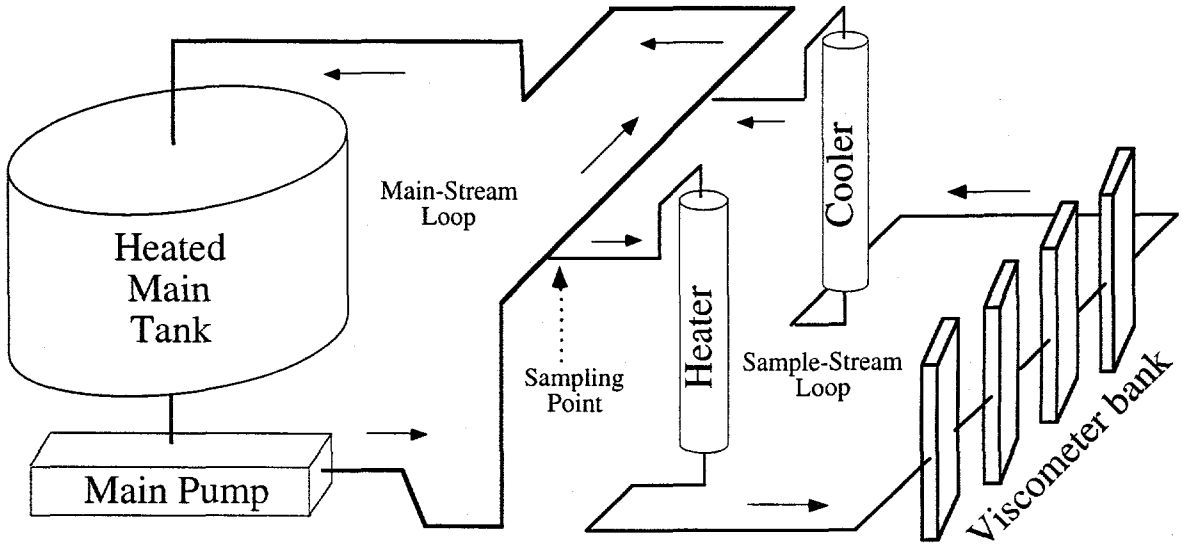


Figure 2.1. Isometric view of the University of Florida plant for instrument evaluation. A viscometer bank located on a sample stream operates on-line to characterize the fluid flowing along the main stream loop.

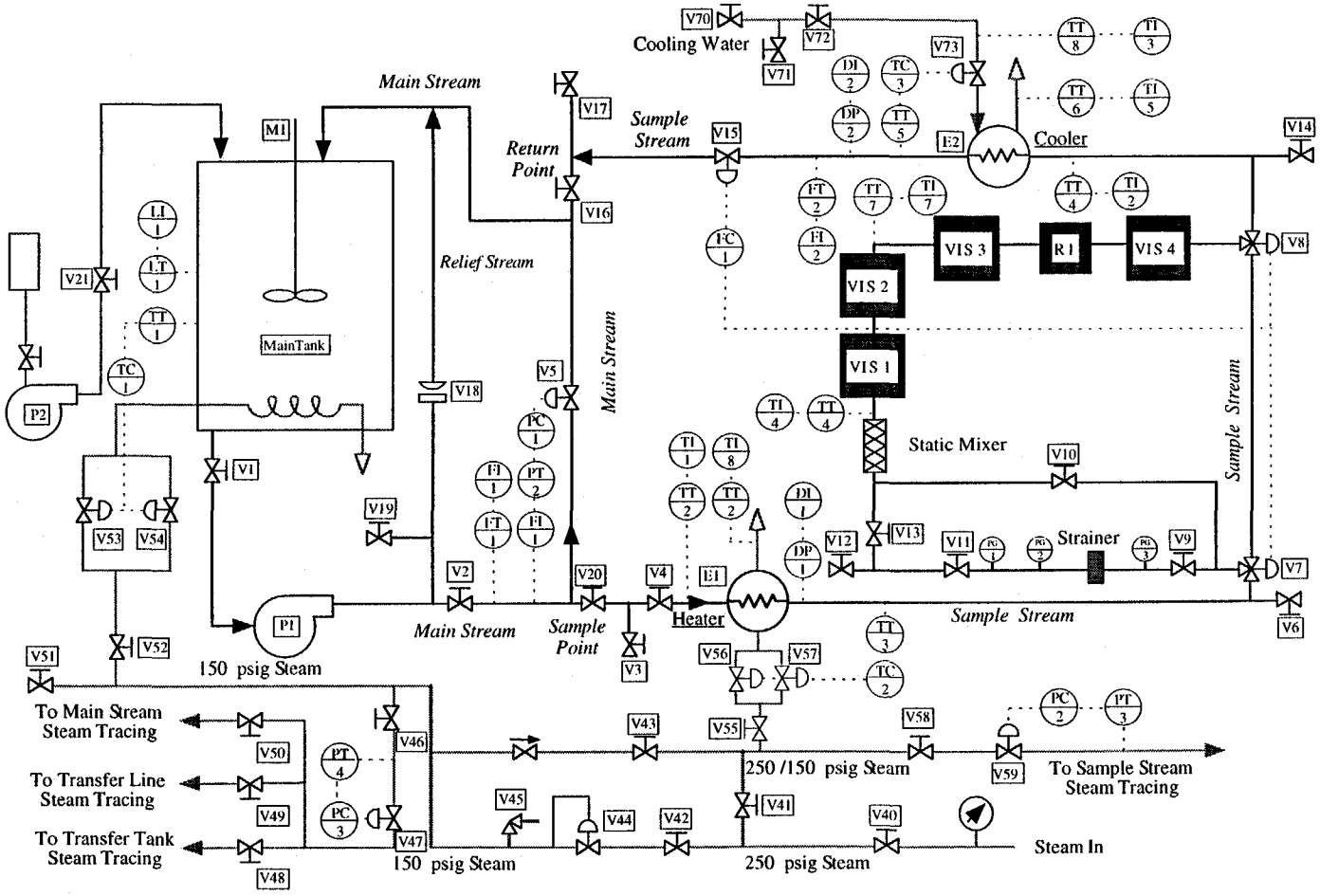


Figure 2.2. Process and instrumentation diagram for the flow loop used in the University of Florida pilot plant to evaluate on-line viscometers. The progressive cavity pump P1 takes black liquor from the main tank and makes circulates it through the closed loop labeled main stream. Valve V20 diverts a fraction of the flow into the by-pass loop labeled sample stream where the on-line viscometers are installed in positions labeled VIS 1 through VIS 4. Relevant symbols are defined in Table 8.1.1 (see Section 8 for further details).

The experimental plan for the pilot-plant work consists of examining the performance of the on-line prototype viscometers under various process conditions to determine the range and suitability of each instrument in typical mill environments. A most relevant issue to consider is that the viscosity of black liquor is dependent not only on temperature but also on the type of liquor (wood species and cooking conditions) and the solids concentration. Four different types of black liquor were loaded in the pilot plant after they were concentrated to various levels of solids content, namely:

1. Black liquor provided by Canadian Forest Products Ltd., Prince George, BC, Canada.
2. Black liquor provided by St. Laurent Paper Products Corporation, West Point VA, USA
3. Black liquor provided by Georgia Pacific Corporation, Palatka FL, USA
4. A mixture of the liquors provided by Canadian Forest Products and Georgia Pacific Corporation

Each prototype on-line viscometer was evaluated in the pilot flow-loop using the four types of black liquors at different levels of solids content and at varying flow rates and temperatures. The accuracy of the viscosity measurements produced by the on-line viscometers was determined by comparing the measurements to those obtained by laboratory measurements taken under well-controlled conditions. A detailed description of the experimental design followed in the pilot-plant evaluations is given in the Appendix (see Section 8.2).

The range, accuracy, and repeatability of the measured response were examined at various temperatures and flow rates under steady state conditions. The time response, sample acquisition, and preparation were additional performance variables that were also under consideration. Finally, the reliability and maintenance of the instruments were long term factors that were considered in the evaluation.

Extensive data were acquired during the evaluation phase using all four black liquor types at 13 different solids concentrations under various temperature and flow rate conditions. The experiments tested the accuracy and the reproducibility of the on-line instruments under steady state conditions, and also tested the speed of response of the instruments through transient-response experiments. The steady-state data were mostly reported in plots of the viscosity as a function of flow rate, and the transient-response tests data were plotted as viscosity as a function of time.

In evaluating the experimental results from the on-line prototype viscometers, the viscosity measurements recorded on-line at the flow loop were compared with the laboratory reference viscosity measurements. The laboratory reference viscosities were calculated using the corresponding Viscometer Line temperature that was recorded on-line. Table 2.1 show the specific temperature sensors that were used to determine the temperature of each on-line viscometer during the on-line experiments. The locations for each temperature sensor is given in Figure 9.1.

Table 2.1. Temperature sensor used for laboratory reference for each instrument.

On-line Viscometer	Temperature Sensor
Brookfield	TT7
Micro Motion	TT7 and Internal sensor
Nametre	TT4 and Internal sensor
SWRI/QM	TT7
Cambridge (Lab version)	Internal sensor

The Micro Motion, Nametre, and the laboratory version of the Cambridge possess internal temperature sensors, as shown in the Table 2.1. The Micro Motion internal temperature sensor is located on the outer casing and not in the flowing fluid. This reading is not accurate for the transient temperature run, but is suitable for steady sate analysis. Near the end of the instrument evaluations program the Nametre internal RTD failed and the external Viscometer Line RTD TT4 was used due to its proximity to the instrument.

It is important to remember that the laboratory reference for a liquor is a function of temperature only since the liquor is Newtonian at a constant solid concentration. In practically all the runs carried out, the viscosity versus flow rate plots show a downward trend of viscosity as the flow rate increases; however, this is not due to increases in shear stress but to increasing temperatures. As the flow rate increases, the net loss of heat in the viscometer line decreases and consequently the temperature in the viscometers increases. The approximate constant temperature attained in any given run can vary by as much as ±5°C. This was especially true for the high temperature runs in which the heater is employed, since maintaining a constant temperature with the fluctuations of black liquor flow rate and steam flow was very difficult.

For the transient temperature response runs, the flow rate is maintained at a low flow rate (~0.75 gpm) by manually adjusting the Sample Line Flow Rate Controller FC1 and the Motor Speed Controller. The Process Heater Controller TC2 is manually opened in small increments to introduce a temperature step change in the Viscometer Line utilizing the Heater E1. The flow rate is kept constant as best as possible though there are some shifts in the flow rate as the temperature

increases. Once the temperature in the Viscometer Line reaches steady state, another step change is introduced by manually adjusting the Process Heater Controller Output. The Viscometer Line RTDs are assumed to instantaneously measure changes in the Viscometer Line during the transient temperature response runs. The time delays of the instruments are calculated from the moment the nearest RTD responds to the Viscometer Line temperature change.

2.2.3 Evaluation in Industrial Paper Mill Sites

All viscometers that satisfactorily passed the evaluation tests in the pilot plant were then installed in various mills for final testing in an industrial environment. The following three mills participated in the testing of instruments:

1. St. Laurent Paper Products Corporation, West Point, Virginia.
2. Canadian Forest Products Ltd., Prince George, British Columbia
3. Georgia Pacific Corporation, Palatka, Florida.

Contact information for the participating mills is given in the Appendix (see Section 8.4).

3. EVALUATION OF NAMETRE'S ON-LINE TORSIONAL-OSCILLATORY VISCOMETER

3.1 Abstract

A torsional oscillatory viscometer manufactured by the Nametre Company was evaluated as an on-line sensor for kraft black liquor in a Pilot Plant environment at the University of Florida and at the site of the St. Laurent Paper Products Corporation mill in West Point, Virginia.

Testing at the University of Florida Pilot Plant was done using a piping loop where black liquor is allowed to circulate under closely controlled solids concentration, temperature, and flow rate. Measurements were made under various flow conditions for liquors at concentrations ranging from 34 to 73% solids and temperatures up to 140°C (413°K). The accuracy and precision of the instrument were determined by comparison with laboratory measurements of viscosity. In general, the viscometer tracked reliably within ±10% of the laboratory reference viscosity for a wide range of operating conditions. The sensor operated satisfactorily under both steady-state and transient conditions. The instrument transient response was rapid, with a lag of approximately two minutes. Varying the flow rate had no effect on the operation of the viscometer. Sensor fouling was noticed with some liquors with a propensity to precipitate solids; however, an on-line steam-cleaning operation effectively restored the performance of the instrument.

Testing at the St. Laurent Paper Products Corporation mill was done by installing the Nametre torsional-oscillatory viscometer in a side stream connected to the ring header line. Reliable operation was sustained for a period of approximately six months, realizing an accuracy of ±16% or better with respect to laboratory reference values. The tests showed that the viscometer can be used on-line in a mill environment to effectively monitor the black liquor viscosity. The instrument requires very little maintenance and is capable of tracking the viscosity with reasonable accuracy. The instrument deployed on-line featured an included angle of 90°, making the viscometer virtually immune to fouling since such highly streamlined sensor profile prevents the accumulation of solids the top surfaces.

3.2 Viscometer Description and Principles of Operation

The Nametre Torsional Oscillatory viscometer evaluated in this study operates in a torsional oscillation mode. Motion of the sensor surface generates freely propagating shear waves within the sample medium. The dimensions of the sensor are such that the waves can be considered to be

planar without introducing significant error. The attenuation of the shear waves depend on the both the viscous and elastic character of the sample. Since the propagation depth of the shear wave is usually small, in most cases the size of the container is not critical [Ferry, 1977].

When the Nametre torsional-oscillatory viscometer is configured in a surface-loading geometry, the mechanical impedance acting on the sensor surface is related to the complex dynamic viscosity coefficient via the expression

$$Z_m^* = R_m + iX_m = \sqrt{i\omega\rho\eta^*}$$

where Z_m^* is the complex mechanical impedance, R_m is the real part of the mechanical impedance, X_m is the imaginary part of the mechanical impedance, ω is the frequency of oscillation, and ρ is the density of the sample, and where the complex dynamic viscosity coefficient [Bird *et al.*, 1977]

$$\eta^* = \eta' - i\eta''$$

consists of a real part η' which represents the energy dissipation or loss characteristics (viscous behavior), and an imaginary-part η'' which reflects the energy storage (elastic behavior) of the material.

It can be shown that the real and imaginary parts of the complex viscosity coefficient are related to the real part and imaginary part of the complex mechanical impedance via the equations

$$\eta' = \frac{2R_m X_m}{\rho\omega}$$

$$\eta'' = \frac{R_m^2 - X_m^2}{\rho\omega}$$

and that the viscosity-density product $\eta_{nom}\rho$ is proportional to the square of the real part of the mechanical impedance via the equation

$$\eta_{nom}\rho = \frac{2}{\omega} R_m^2 \tag{3.1}$$

where η_{nom} is the nominal viscosity defined as follows:

$$\eta_{nom} = \sqrt{\eta'^2 + \eta''^2} + \eta''. \tag{3.2}$$

Thus, the Nametre torsional-oscillatory viscometer measurement of the nominal viscosity-density product $\eta_{nom}\rho$ determined through relationship (3.1) is sensitive to both the viscous and elastic

nature of the sample due to the presence of coefficients η' and η'' in equation (3.2).

Under normal operation, the transducer that causes the sensor motion is driven in such a way that the amplitude of motion of the sensor is held constant at approximately 1 micron, and the average electrical power P required to accomplish this constant amplitude is measured. Since the measured average power is directly proportional to the real part of the mechanical impedance, *i.e.*,

$$P = \phi R_m \tag{3.3}$$

it is possible to infer the value of R_m from knowledge of the empirical factor ϕ which is specified by the manufacturer and is stored in the instrument's on-board electronics. Finally, the value of R_m determined in this fashion is used to obtain the viscosity-density product of the sample using equation (3.1). From this result the nominal viscosity can be calculated if the density is known or is measured using another instrument. Note that the empirical factor ϕ can be found by measuring the average power for a sample of precisely known viscosity and density and from equations (3.1) and (3.3).

For purely viscous materials (*i.e.*, $\eta'' = 0$) the expression for the nominal viscosity given in equation (3.2) reduces to $\eta_{nom} = \eta'$. In such cases the nominal viscosity is referred to as *absolute viscosity* and is simply denoted by the symbol η. Although concentrated black liquors may show viscoelastic behavior, all the liquors used in this study were confirmed to be non-elastic; hence, for these materials $\eta'' = 0$, and the viscosity-density product measured by the instrument is in fact the product of the *absolute viscosity* times the density.

If precise viscosity measurements are needed, then a correction to the viscosity-density product measurement obtained as explained above must be introduced to eliminate the biasing effect of the reference zero of the instrument. The reference zero is simply defined as the viscosity-density product measured when the sensor is surrounded only by air, and is denoted $(\eta\rho)_{air}$. Letting $(\eta\rho)_{meas}$ denote the viscosity density product measured when the fluid sample is black liquor, the corrected reading is obtained through the difference

$$\eta\rho = (\eta\rho)_{meas} - (\eta\rho)_{air} \tag{3.4}$$

Since the reference zero may change with temperature, it is important to carry out a reference zero measurement at the temperature at which the black liquor sample will be measured. Given that the empirical Cox-Mertz rule has been shown to apply to black liquor [Zaman and Fricke, 1995], it follows that the shear rate at which the sample is subjected during measurement is given by $\dot{\gamma} = 2\pi f$, where $\dot{\gamma}$ is the shear-rate (s^{-1}) and $f = \omega/(2\pi)$ is the frequency of mechanical

oscillation of the sensor element [cycles/sec].

One concern with oscillatory viscometers is that vibrations caused by pumps and other machinery can adversely affect the measurement. While the influence of this background noise is essentially negligible at higher viscosities, its influence on lower viscosity measurements is potentially problematic. Nametre torsional-oscillatory viscometers incorporate feedback control circuitry in order to minimize vibration effects [Fitzgerald *et al.*, 1991]. Proper installation and site selection also aid in minimizing background noise effects [Tarrant *et al.*, 1994].

3.3. Viscometer Configuration and Installation Details

The torsional oscillatory viscometer used in the tests is a Nametre 1810MX Viscoliner [Nametre, 1999] with a hollow cylindrical sensor. The main components of the viscometer are shown in Figure 3.1 which features a containment vessel specifically manufactured for this application at the University of Florida. The torsional member comprises a central inner drive-shaft which drives an enlarged hollow cylindrical tip and an outer hollow sheath which serves as a restoring spring. The mass of the sensor must be small enough to attain the rapid thermal stabilization that is necessary for accurate measurements. The viscometer is also equipped with an internal RTD to measure the process temperature at the sensing surface. The black liquor enters through a port at the bottom of the containment vessel and exits through a side port at the top. A deflection plate at the bottom protects the sensor from potential damage caused by debris carried by the flow. Flex hoses of 1" diameter are connected to the input and output ports to isolate the sensor from high-frequency vibrations propagated through the piping network of the pilot flow loop. Careful installation of the sensor is crucial for the stability and reproducibility of the readings. The sensor must be protected from process shocks, vibrations, and electromagnetic interference [Tarrant *et al.*, 1994]. The absence of high frequency interference from day-to-day operation of the pilot flow loop was confirmed by comparing readings taken with and without running process equipment.

The LED readouts on the front panel of the electronic control unit provided with the viscometer display two output signals, namely, the viscosity-density product ($\eta\rho$) and the temperature of the flowing stream. Both signals are also transmitted to the plant's data acquisition system as 4-20 mA signals. The control unit is capable of handling up to four sensors, and each sensor channel can take a 4-20 mA densitometer input to permit direct on-line calculation of nominal viscosity. The control unit also has user-configurable alarms and an RS-232 serial port to communicate with process computers and other control systems. The instrument has auto-range capability with no need for operator attendance. This is important because in many applications,

such as polymerization processes for example, the viscosity can range over five decades [Fitzgerald *et al.*, 1987]. Since black liquor viscosity can also vary widely, this feature is especially useful.

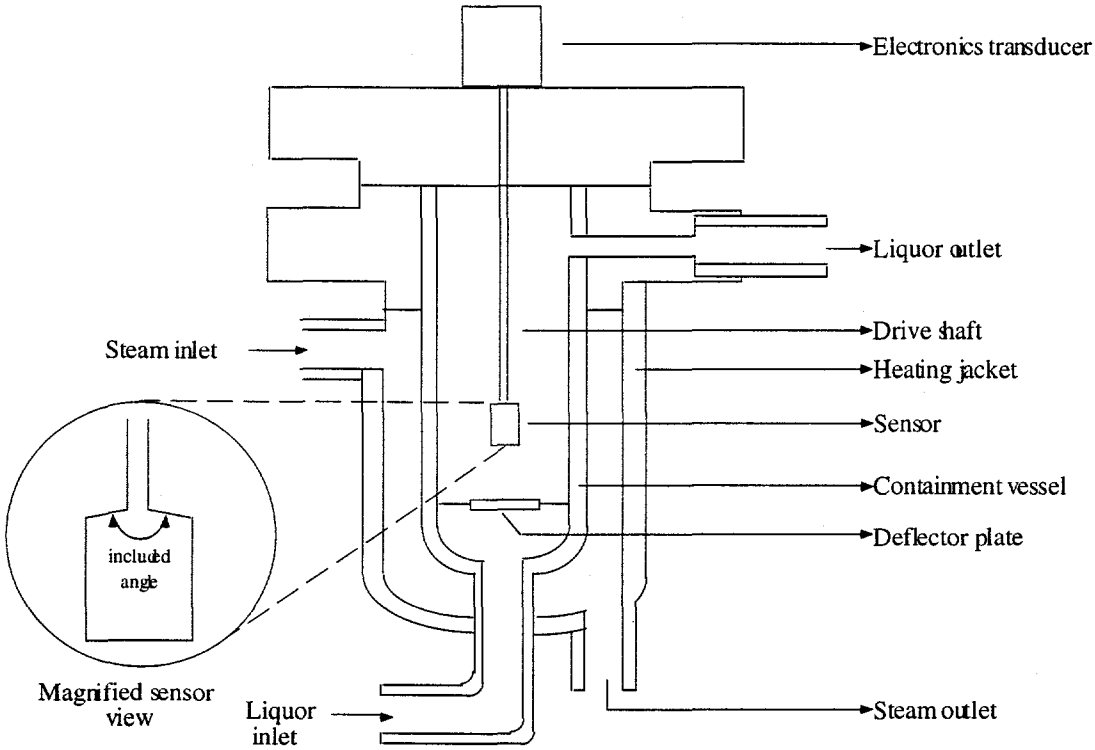


Figure 3.1. Schematic diagram of the Nametre torsional-oscillatory viscometer evaluated in the University of Florida pilot plant.

3.4 Pilot Plant Trials at the University of Florida

The Nametre torsional oscillatory viscometer underwent extensive testing at the University of Florida using the pilot flow loop plant

3.4.1 Experimental Overview

Black liquor was obtained from three kraft mills, namely, Georgia Pacific Corporation in Palatka, Florida; Canadian Forest Products Limited in British Columbia, Canada; and St. Laurent Paper Products Corporation in West Point, Virginia. These mills are respectively representative of southern softwood, western softwood, and mid-Atlantic mixed-species pulping. The liquors were received at concentrations of 40-45% solids, and were further diluted or concentrated as needed in a wiped-film evaporator at the University of Florida. Table 3.1 lists the solids composition of the

black liquors used in the experimental runs. The on-line viscometer was tested at 14 different black liquor concentrations, ranging from a 34.1% Georgia Pacific liquor to a 73.4% liquor obtained by mixing a Georgia Pacific Corporation liquor with a Canadian Forest Products Limited liquor (*cf.* Table 3.1).

The Georgia Pacific liquor was the easiest to handle because of its low level of air entrainment and ease of evaporation; hence, more experiments were performed with this material. The Canadian Forest Products Limited and St. Laurent Paper Products black liquors formed crystalline particles in the main tank at intermediate (53-60%) solids concentrations when stored for several days as was required for the experiments. A 40-mesh strainer installed in the sample stream of the pilot plant protected the on-line instruments from the crystalline particles.

Table 3.1. Black liquor sources and solids concentrations used to test the torsional oscillatory viscometer.

Georgia Pacific Corporation	Canadian Forest Products Limited	St. Laurent Paper Products Corporation	Mixture of Georgia Pacific Corporation & Canadian Forest Products Limited
34.1%	47.9%	49.3%	67.7%
42.2%	57.7%	59.5%	71.4%
48.2%			73.4%
55.8%			
58.3%			
65.5%			
68.4%			

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 the thermal and physical homogeneity of black liquor. To collect samples for laboratory characterization, the flow through the sample loop and through the main loop were stopped briefly and a 1-liter sample was withdrawn. Samples were collected from both the main loop and the sample loop simultaneously, and were compared to verify sample integrity. The flow was stopped to ensure that flashing does not occur during sampling. Flashing destroys the sample integrity by making the liquor appear more concentrated than it actually is. A discrepancy of up to 2% in solids content has been observed due to flashing if this precaution is not taken.

Experiments were run in the pilot flow loop at different temperatures for each of the concentrations listed in Table 3.1. The highest temperature reached in the main tank was limited to 10-20 °C below the boiling point of the black liquor. This was done to minimize evaporation, and

to eliminate or minimize the degradation that can occur in liquors at high solids concentration when held at high temperatures. Experiments at temperatures above the atmospheric boiling point of black liquor were done using the heater in the sample loop to bring the liquor to the desired temperature. The liquor temperature was then reduced back to the main-tank temperature by the cooler and returned to the main stream in the loop. For each concentration-temperature combination, data was collected at different flow rates, including the static condition of zero flow. The upper bound of the flow rate for the experiments is dictated by the output of the main progressive cavity pump at its maximum operational limit of 150 psig ($1.135\ 10^6$ Pa). However, for safety reasons the main line pressure was usually kept at 70 psig ($5.839\ 10^5$ Pa). This pressure upper bound in turn limited the maximum possible flow rates obtainable for highly concentrated liquors.

The black liquor density as a function of temperature was determined in a laboratory environment for each liquor listed in Table 3.1. The resulting density-temperature correlations were used to extract a viscosity value from the viscosity-density product measured on-line by the Nametre torsional-oscillatory viscometer. In addition, an on-line densitometer installed in the loop also provided on-line density measurements that, in the absence of air entrainment, agreed closely with the laboratory values. Hence, the combination of the densitometer with the Nametre torsional-oscillatory viscometer could permit the effective determination of the viscosity on-line. The temperature measured by the internal RTD of the Nametre torsional-oscillatory viscometer was used as the reference temperature for the flowing black liquor stream. This temperature closely matched the temperature indicated by an RTD located immediately before the viscometer inlet port in the sample loop.

Reference-Zero Correction

The reference-zero value of the Nametre torsional-oscillatory viscometer is related to the amount of energy (and hence the average electrical power) necessary to maintain a constant amplitude of oscillation in air. During installation of the viscometer, a zero calibration is performed at the process operating temperature. In the case of the pilot flow loop, where the temperature is one of the adjustable variables, it is necessary to determine the change of the zero reference as a function of temperature in order to quantitatively account for its contribution to the viscosity readings. Figure 3.2 shows the viscosity-density product measured by the instrument in air at various temperatures. Calibration runs conducted at other times for cross-checking purposes produced very similar results. As is evident from the figure, the error over a very wide range of temperatures (from 20°C to 180°C) is less than 0.21 $cP \cdot g/cm^3$ ($2.1\ 10^{-4}$ $Pa \cdot s \cdot g/cm^3$). The data shows that although the air zero correction may be considerable at low viscosity if a large

temperature range is to be considered, it is nevertheless insignificant at higher nominal viscosity-density product values. The nominal viscosity-density product in air for the data in Figure 3.2 is captured by the correlation

$$(\eta\rho)_{air} = 0.209 - 2.197\ 10^{-3}\ T + 6.245\ 10^{-6}\ T^2 \tag{3.5}$$

where $(\eta\rho)_{air}$ is the nominal viscosity-density product measured in air (cP·g/cm^3) and T is the temperature (°C). All measurements taken with the instrument installed in the pilot flow loop were corrected by subtracting the temperature dependent reference zero $(\eta\rho)_{air}$ given by equation (3.5) as prescribed in equation (3.4).

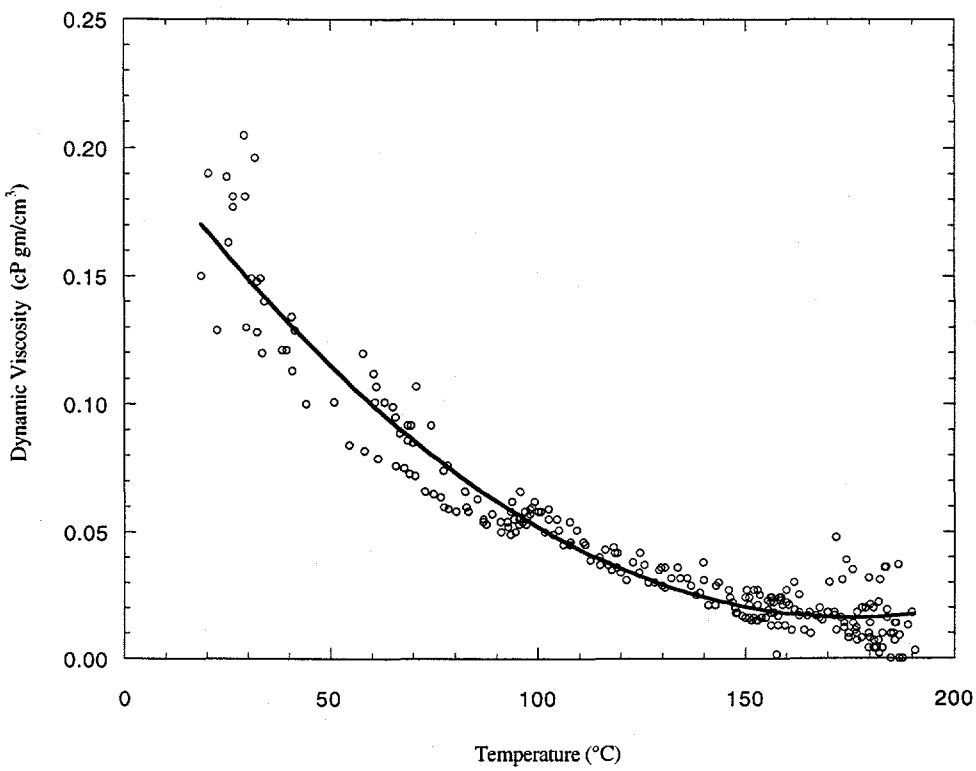


Figure 3.2. Reference zero (defined as the viscosity-density product in air) for the Nametre torsional-oscillatory viscometer as a function of temperature.

3.4.2 Discussion of Results

This section presents typical results obtained with the Nametre torsional-oscillatory viscometer. Extensive results are given in the Phase IIA DOE report on this research [Fricke and Crisalle, 1996]. Since all the black liquors considered in this study were non-elastic, the Nametre

torsional-oscillatory viscometer measurements indicate the product of the absolute viscosity and the density. The results include characterizations of viscosity versus flow rate at different temperatures and solids compositions, and one 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 liquor viscosity control based on widely used strategies such as lambda tuning [Thomasson, 1995].

Figure 3.3 shows the results obtained for a Georgia Pacific 68.3% solids black liquor at approximately 85 °C (358 °K). In this case, the flow rate in the sample stream is restricted to approximately 1.8 gpm (1.1356 10^{-4} m^3/s) in order not to exceed the safe operational limit on the main pump. The viscosity-density product measurement at each flow rate was taken after steady state had been established. The following density correlation as a function of temperature obtained using laboratory studies for this liquor was used to extract the viscosity values from the measured viscosity-density products:

$$\rho = 1.3744 - 5.7594\,10^{-4}T - 1.0900\,10^{-6}T^2 \tag{3.6}$$

where ρ is the density (g/cm^3) and T is the temperature (°C).

The diamond-shaped markers in the upper plot in Figure 3.3 show the viscosity values obtained after the viscosity-density values measured by the instrument were divided by the density predicted from (3.6). It should be noted that the continuous line joining the markers is included as a visual aid to track the data trend, and is obtained through an arbitrary polynomial fit. The upper plot also shows a laboratory reference band of viscosity values defined by two dashed lines. Two to four different laboratory viscometers were used to determine the viscosity as a function of temperature for samples collected from the flow loop. The upper and lower band respectively correspond to the highest and lowest viscosity predictions obtained when considering the measurements produced by several laboratory viscometers, namely, a Haake concentric-cylinder RV12 rotational viscometer (configurable in pressure-cell or open-cup geometries), a Cambridge sliding-element laboratory viscometer, a Brookfield LTV 100 rotating cup viscometer, and a laboratory Nametre 1810MX Viscoliner. Any measurements that fall inside the reference band are considered to be consistent with the laboratory measurements, and hence are deemed acceptable for black liquor viscosity monitoring and for control purposes.

The lower plot in Figure 3.3 is a measure of the percentage relative difference between the viscosity measurement (η) produced by the on-line instrument and the viscosity (η_{ref}) predicted by a correlation generated from reliable laboratory instruments, *i.e.*

$$\% \text{ Relative difference} = 100\,\frac{\eta - \eta_{ref}}{\eta_{ref}}$$

We consider an on-line instrument to be accurate if the relative-difference value ranges between -10% and +10%. For this particular liquor at 68.4% solids concentration, the laboratory reference viscosity was determined from the correlation

$$\eta_{ref} = \exp\left[A + \frac{B}{T + 273.15} + \frac{C}{(T + 273.15)^2}\right] \tag{3.7}$$

where A = 18.2073771, B = -17633.4415, C = 4709530.7768, and where T is the temperature (°C), and η_{ref} is a laboratory-determined reference viscosity (cP). The constants appearing in correlation (3.7) were determined using viscosity data taken with a pressure-cup Haake RV12 viscometer.

A relevant remark concerning Figure 3.3 is that the temperature is not strictly constant during the run; in fact, as the flow rate varies, normally the temperature also varies. The observed temperature fluctuations were typically no greater than 1 °C (1 °K), however, since the viscosity of black liquor is highly sensitive to temperature, such small variations must be taken into account for accurate work. For completeness, Table 3.2 gives the viscosity and temperature data for 10 selected flow rates plotted on Figure 3.3.

Analysis of Figure 3.3 shows that the Nametre torsional-oscillatory viscometer responds with good accuracy, since the measurements lie within the laboratory reference band. The percentage relative difference during the run is better than -5%. Furthermore, the figure shows that the measurements were unaffected by flow rate since the data follows the trend defined by the edges of the laboratory reference band (which in turn are affected only by the temperature). Note that the instrument tracks the changes in viscosity that occur due to the small temperature changes that take place at different flow rates.

Figure 3.4 is another example of a viscosity run for the Canadian Forest Products Limited liquor with 47.9% solids at approximately 30 °C (303 °K). The laboratory data predict a viscosity in the range of 54-62 cP (0.054-0.062 Pa·s) at 30 °C (303 °K). The viscometer measurements are very close to the upper boundary of the laboratory reference band. The lower plot shows that most readings have a relative difference no greater than 5%. Again, the viscosities measured by the instrument were unaffected by flow rate.

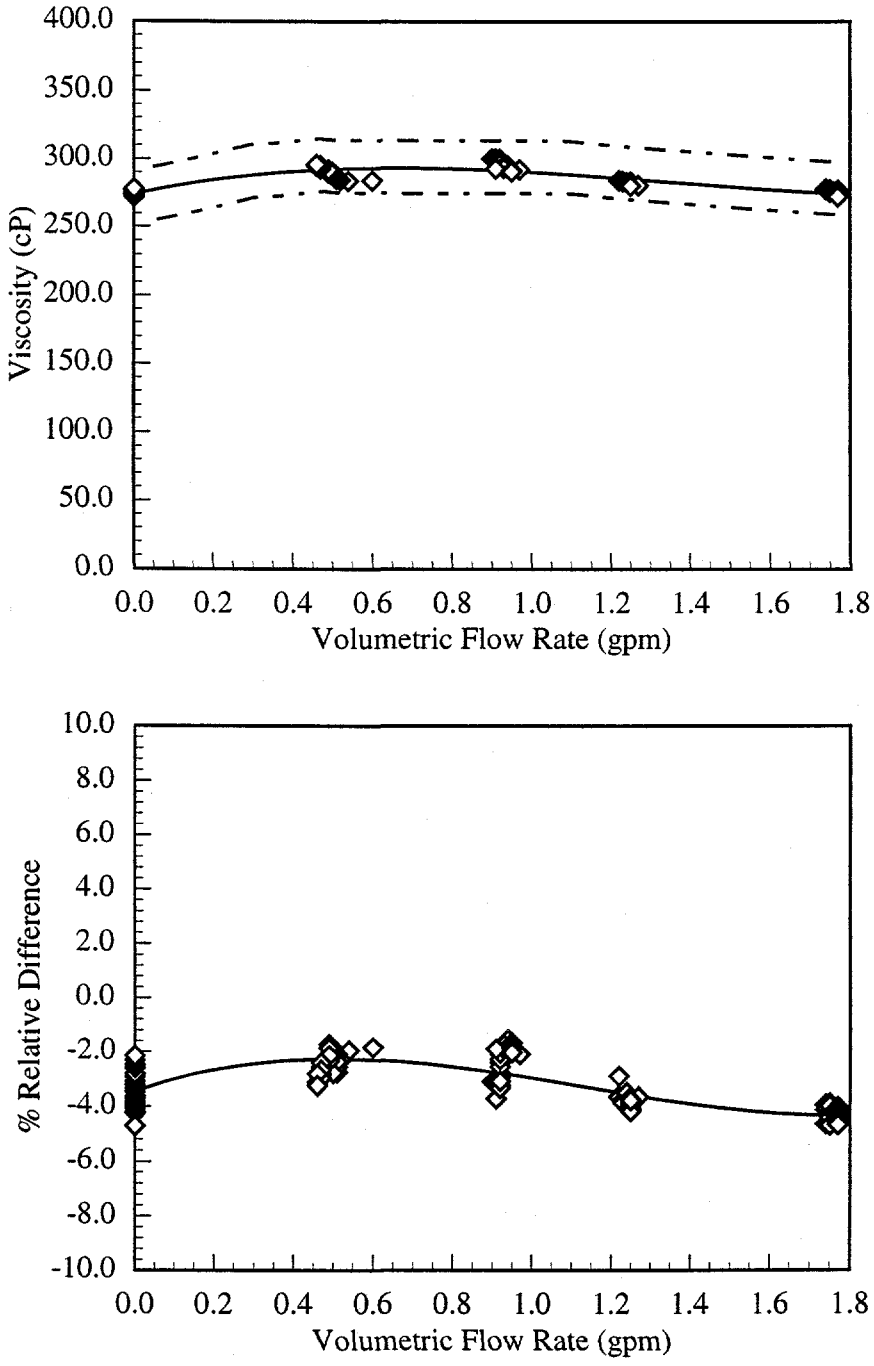


Figure 3.3. Viscosity measured on-line by the Nametre torsional-oscillatory viscometer for a Georgia Pacific black liquor with 68.4% solids at approximately 85 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

Table 3.2. Flow-rate, temperature, and viscosity data for Figure 3.3.

Flow Rate(1)	Temperature(2)	Viscosity(3)	Relative Difference
[gpm]	[°C]	[cP]	[%]
0.60	84.94	283.81	- 4.3
0.49	84.67	290.85	- 3.7
0.51	85.50	285.30	- 0.1
0.51	84.85	287.61	- 3.6
0.91	84.20	300.02	- 3.8
0.92	84.30	299.57	- 3.3
1.25	85.32	280.64	- 2.9
1.75	85.69	276.15	-2.0
0.00	85.49	272.10	- 3.4
0.00	85.58	272.46	- 4.5

(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 multiply by 1000.

Figure 3.5 shows the results obtained for a Canadian Forest Products Limited 47.9% solids black liquor at approximately 50 °C (323 °K). With viscosity readings in the range of 22-25 cP (0.022-0.025 Pa·s), the instrument successfully tracks the changes indicated by the laboratory reference band (upper plot), achieving relative differences that are better than +5% for most readings (lower plot). All measurements were unaffected by the flow rate.

Figure 3.6 shows the viscosity of a Canadian Forest Products Limited liquor with a solids content of 57.7% at a temperature of approximately 80 °C (353 °K). The viscosity measurements from the instrument lie above the laboratory reference band with a relative difference of approximately +60%. In this case, build up of black liquor sediments on the rear (downstream) part of the sensor was suspected to be the cause of the large offset in the measured viscosity. The accumulation of sediments causes an effective increase of the mass of the sensor, and hence affects the vibrational response introducing an error in the reading. Clearly, this fouling effect would be more pronounced for low viscosity liquors, where a small increment in sensor mass can account for a more significant fraction of the total amplitude of the sensor vibration pattern. Note that even though the measurements are affected by a large absolute error, the viscometer nevertheless succeeds in tracking the viscosity variations indicated by the edges of the laboratory reference band. Hence the error in measurement is essentially a constant offset. The sensor used in this work was cylindrical with flow directed upward in a direction parallel to the cylinder. The rear of the cylinder was conical but the included angle was 156° (*cf.* inset in Figure 3.1), *i.e.*, the top the cylinder was virtually flat. When the measuring element was removed and inspected, we found deposits only on the conical section at the rear of the sensor where flow stagnation most likely had occurred. This problem can be greatly reduced or eliminated altogether by streamlining the rear of the sensor via the adoption of a smaller included angle. It should be noted that this particular liquor presented the highest levels of precipitation and deposits formation of all the materials tested.

The problem of sensor fouling can be countered by flushing the instrument with steam. Flushing was done by first cutting off the black-liquor flow to the viscometer, a procedure easily performed in the pilot flow loop. Then 100 psig (7.908 10^5 Pa) steam was injected in the pipes, purging the black liquor and its deposit from the viscometer. Steam flushing was continued until the viscosity-density product measurement returned to a value close to zero. After this was achieved, the liquor flow through the sample loop and the viscometers was resumed, the viscometer line was allowed to thermally equilibrate, and normal operations were restored.

Figure 3.7 shows the behavior of the viscometer after a steam-flushing operation. It is clear that the steam cleaning procedure returned the viscosity measurement to a level consistent with the

laboratory reference band, as shown in the upper plot. Again, the instrument tracks very well the changes in viscosity predicted by the laboratory reference band. The relative difference of the measurements is shown in the lower plot, where the deviation from a reference laboratory correlation is better than -15% for most measurements, and somewhat worse at the static conditions of zero flow.

Figure 3.8 shows the results of a transient response study. The liquor used was a mixture of Georgia Pacific and Canadian Forest Products Limited black liquors with a resulting solids composition of 71.4%. The upper plot in Figure 3.8 shows the viscosity as a function of time. The continuous line represents the measurement produced by the on-line instrument. The lower plot in Figure 3.8 shows the temperature history during the run. In this experiment the temperature of the flowing black liquor steam was changed in a step-fashion by opening the steam control valve (valve labeled as V57 in the process and instrumentation diagram given in Figure 8.1.1 of the Appendix) that feeds the indirect heater (E1). 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) on the figure denote the instants when the valve opening was changed. Initially the temperature in the sample loop was maintained at approximately 102 °C (375 °K) and the system was at steady state. The corresponding steady-state viscosity value was 490 cP (0.49 Pa·s). At approximately 2 minutes into the run, a step change was introduced by opening the heater steam valve to 15% of its maximum lift. This instant is marked by the circle symbol on both the upper and lower plots in Figure 3.8. There was a delay of approximately 1 minute before the effect of this change in steam flow rate was felt in the sample loop temperature, as shown in the lower plot. Furthermore, there was a time lag of approximately 2 minutes (indicated as *lag 1* in the figure) before the Nametre torsional-oscillatory viscometer responded to the temperature change. The temperature in the sample loop kept rising steadily and finally settled at 125 °C (398 °K). The viscosity in turn settled at 206 cP (0.206 Pa·s) approximately 19 minutes into the run. Note that the changes in viscosity shown in Figure 3.8 represent a range that is far in excess of any that would be experienced in a mill due to temperature variations in such a short time. The Nametre torsional-oscillatory viscometer was able to track well these dramatic temperature changes.

The viscometer tracking performance at other times are qualitatively the same. For example, at approximately 23 minutes into the run, the heater steam control valve was opened to 20%. This instant is marked by the square symbol in both the upper and lower plots in Figure 3.8. The time lag (labeled *lag 2*) and the response of the on-line viscometer are similar to the previous change.

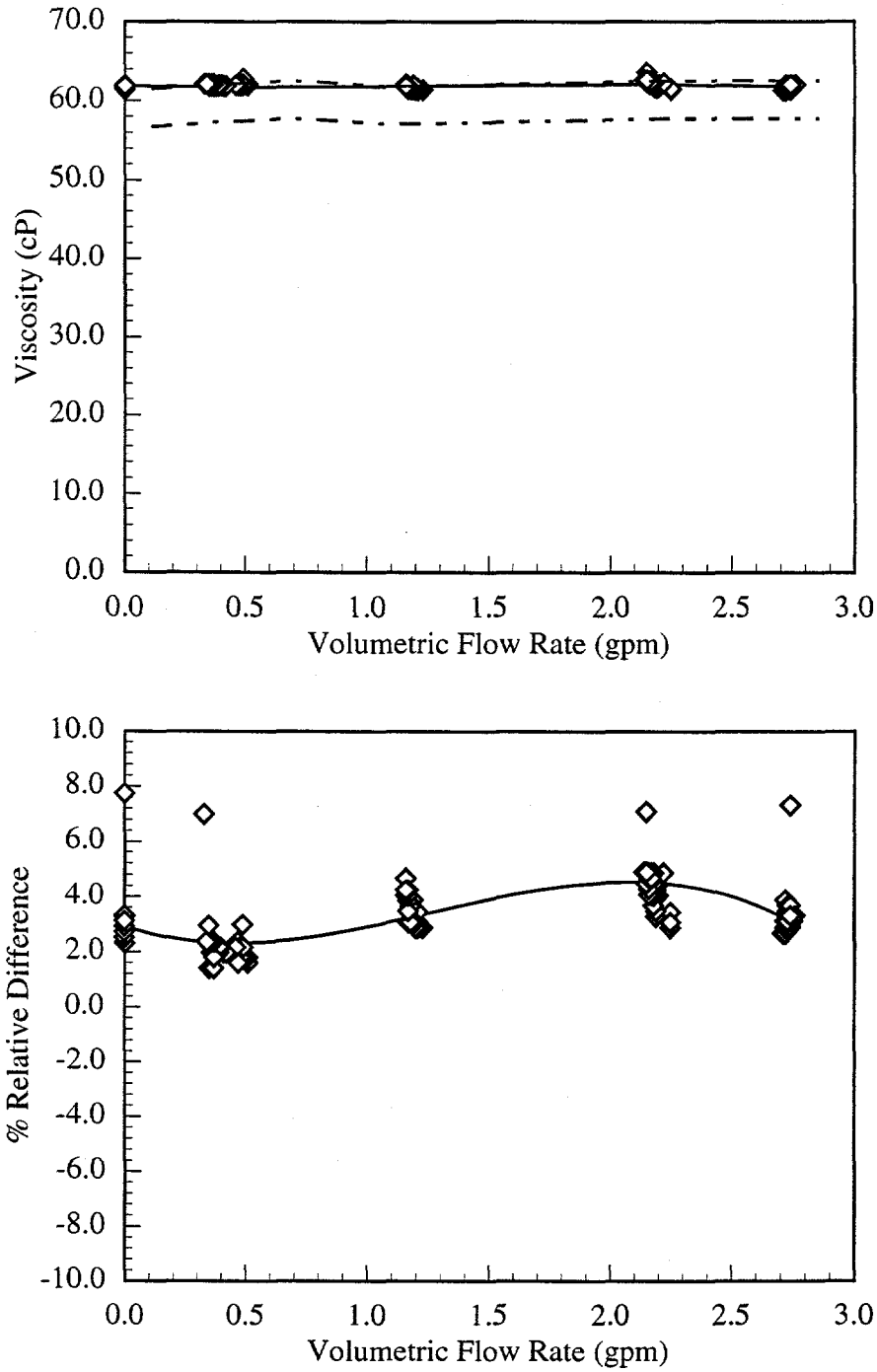


Figure 3.4. Viscosity measured on-line by the Nametre torsional-oscillatory viscometer for a Canadian Forest products Limited black liquor with 47.9% solids at approximately 30 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

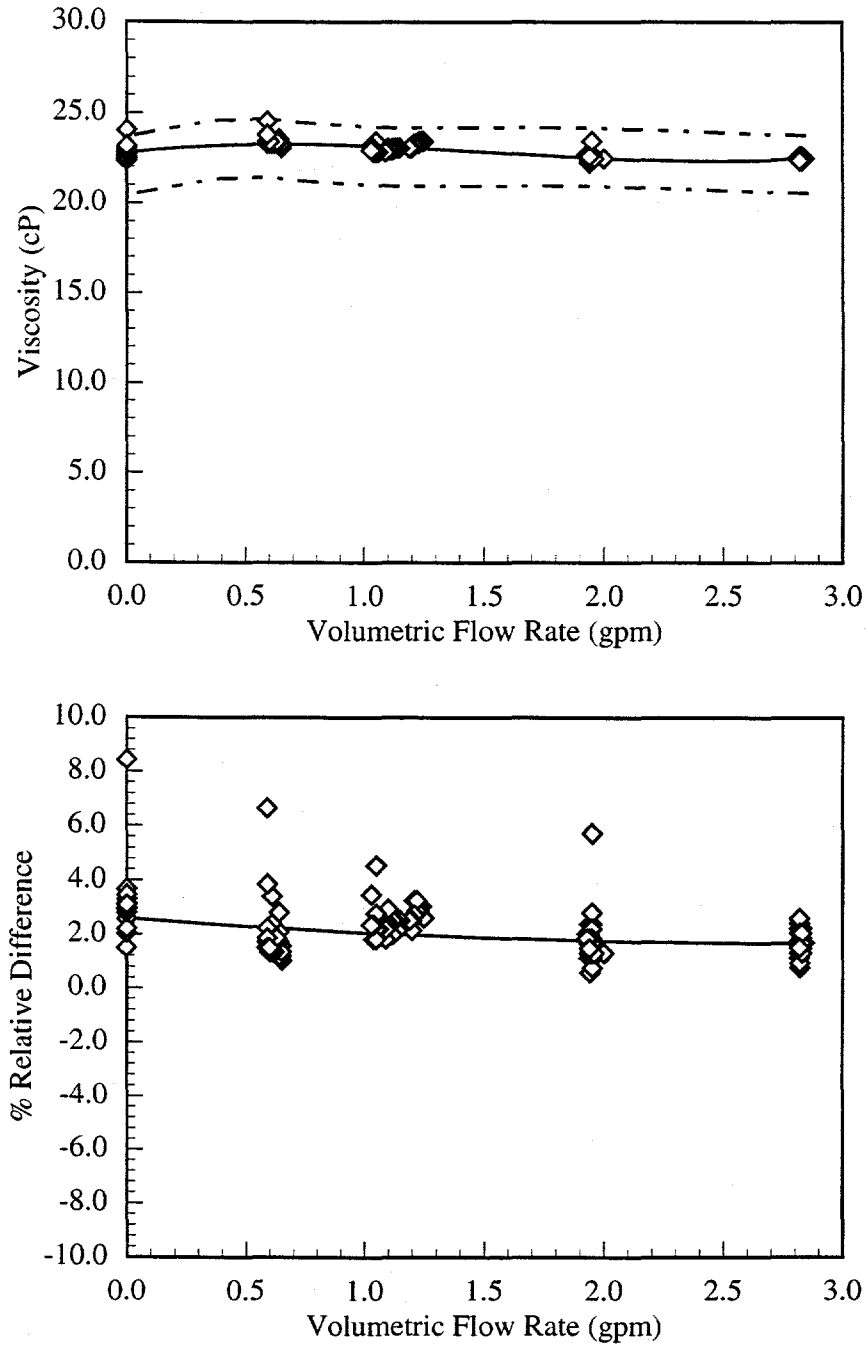


Figure 3.5. Viscosity measured on-line by the Nametre torsional-oscillatory viscometer for a Canadian Forest products Limited black liquor with 47.9% solids at approximately 50 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation

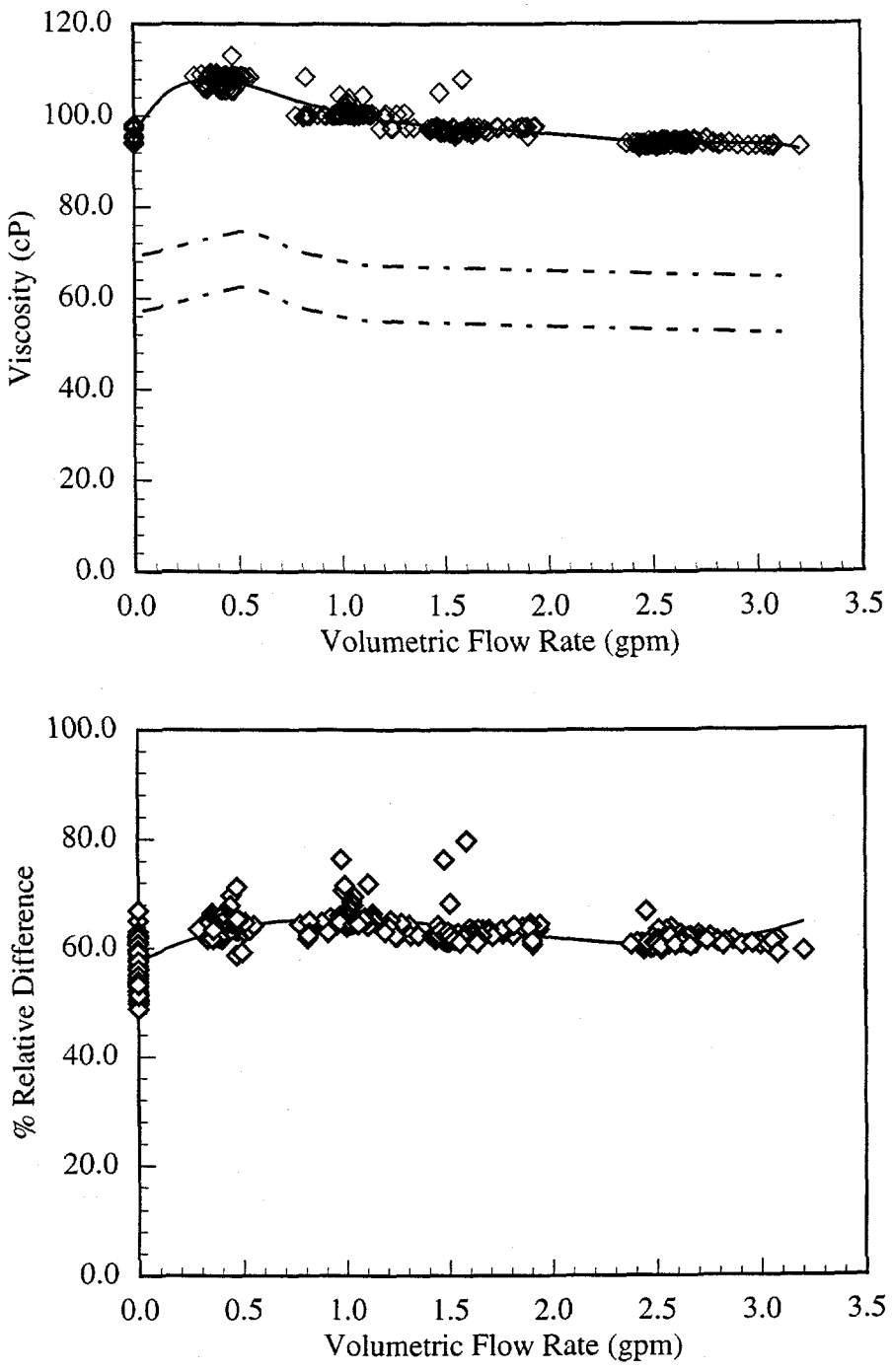


Figure 3.6. Fouling of the sensor caused degradation of the accuracy of the viscosity measured for a Canadian Forest products Limited black liquor with 57.7% solids at approximately 80 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

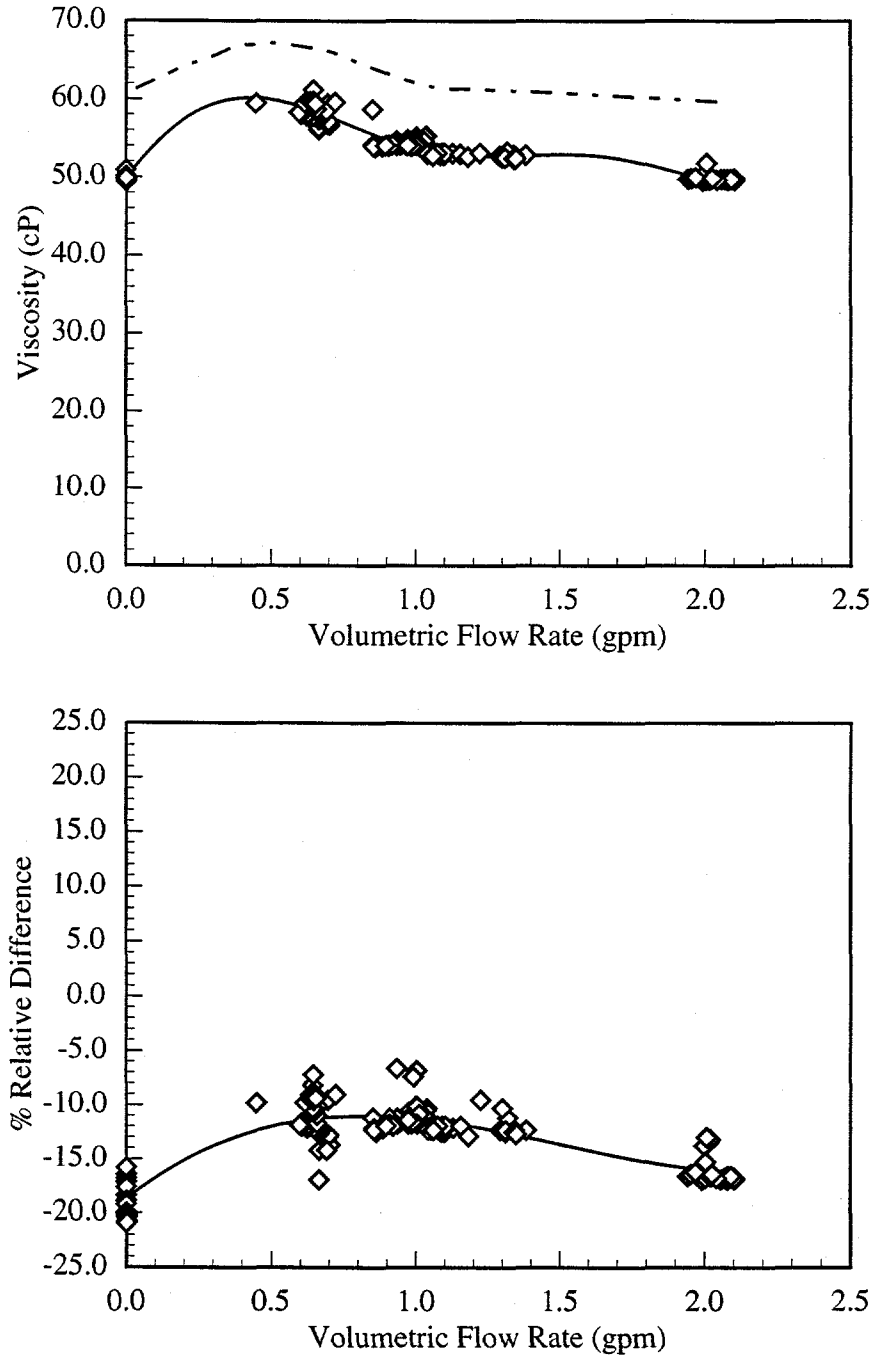


Figure 3.7. After a brief on-line steam-cleaning operation the Nametre torsional-oscillatory viscometer recovers its accuracy. Same liquor type and operating conditions as those reported for Figure 3.6.

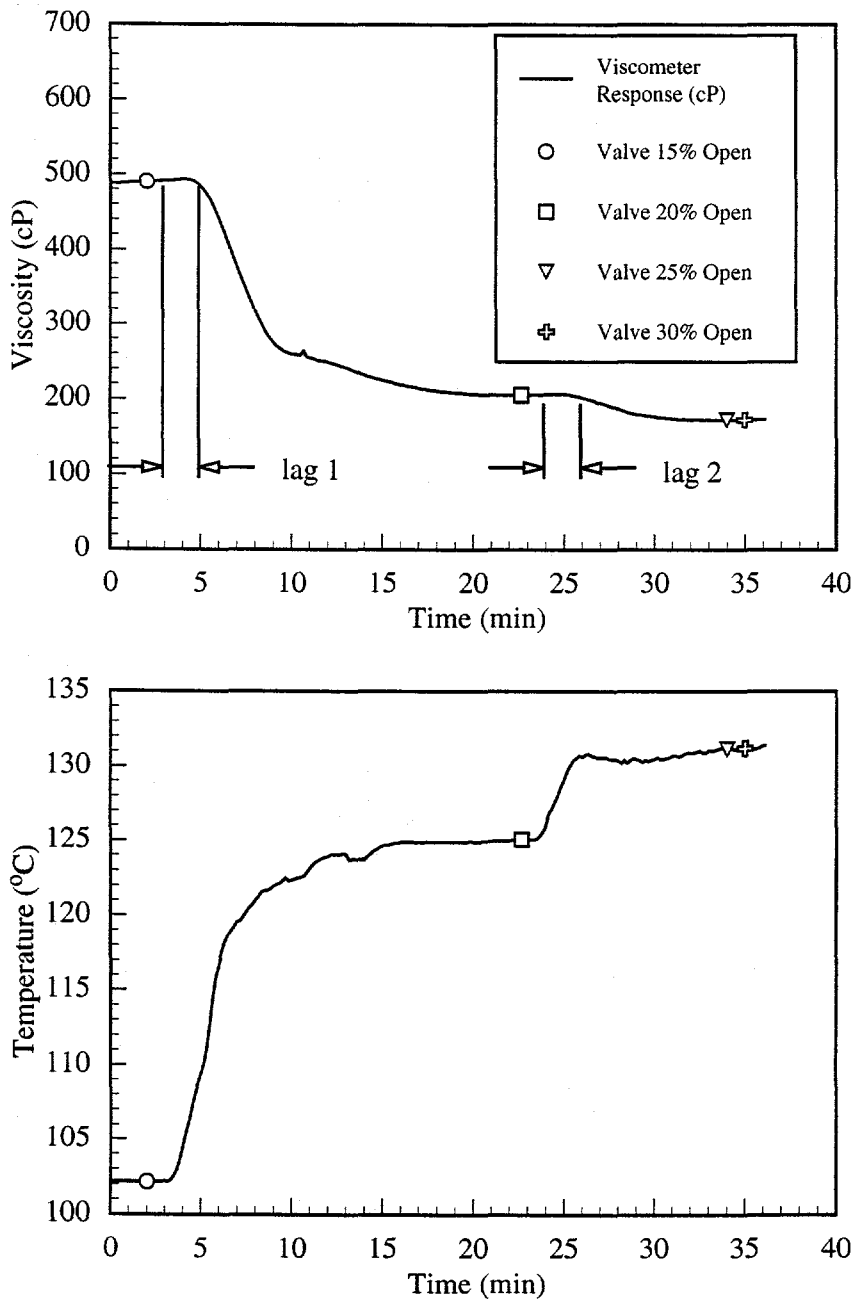


Figure 3.8. Transient response of the Nametre torsional-oscillatory viscometer. The upper plot shows the viscosity measured on-line for a mixture of Canadian Forest products and Georgia Pacific liquors with 71.4% solids as the temperature of the liquor is changed by opening the steam valve of the indirect heater. The lower plot shows the temperature of the black liquor as a function of time. The markers appearing in both plots indicate the instants when the steam valve opening is changed in order to cause a temperature increase.

3.4.3 Summary of Observations for the Pilot Plant Trials

The torsional-oscillatory viscometer used in this study tracked reliably within ±10% of the laboratory reference viscosity at high viscosities (greater than 175 cP (0.175 Pa·s))and also at low viscosities and low solids (less than 50% solids) with no fouling problems. The following observations were made:

- The instrument performance is unaffected by particles smaller than a 40-mesh size.
- The instrument response is rapid, with a delay of approximately 2 minutes.
- The instrument provides a direct measure of nominal viscosity-density product. Since all the black liquors tested in this study were non-elastic, the nominal viscosity is in fact equal to the absolute viscosity.
- It is possible to measure the absolute viscosity on-line if an on-line densitometer is available.
- The instrument reading is unaffected by the liquor flow rates achieved in the sample stream; therefore, regulating the flow in the slip stream where the viscometer is mounted should not be a concern.
- With the cylindrical sensor used in the pilot flow loop, the instrument was subject to fouling at solids concentrations above about 50%, especially for liquors from which solids could precipitate.

The Nametre torsional-oscillatory viscometer was used in the pilot flow loop to measure viscosities from 4 cP (0.004 Pa·s) up to approximately 1070 cP (1.07 Pa·s). Once the unit was auto-zeroed in the installation phase, the viscometer did not require recalibration throughout the experimental trials. For use in an environment where there is a wide operating temperature range, the instrument requires a reference zero correction, especially when utilized for low viscosity materials. At high viscosities, the zero correction is insignificant.

The fouling effect on the particular instrument tested appears to be a problem for some black liquors with solids content in excess of 50%, and appears to be particularly severe in the range of 53-60% solids. Fouling occurred with black liquors prone to severe precipitation such as the Canadian Forest Products Limited liquor with 57.73% solids, causing a constant offset towards higher viscosity values, as illustrated in Figure 3.6. Steam cleaning quickly restored the performance of the Nametre torsional-oscillatory viscometer; however, given that the liquor

depicted in Figures 3.6-3.7 had a very strong tendency to precipitate solids, it was observed that fouling occurred again within an interval of approximately an hour. In general, restoration of performance was effectively achieved with steam cleaning. Since the offset introduced by sensor fouling is nearly constant, the reproducibility (*i.e.* precision) of the instrument remains unaffected. Consequently, even under severe fouling the instrument remains a perfectly acceptable viscosity sensor for process control applications where reproducibility is typically more important than accuracy. Figure 3.3 is typical of a high-solids and high viscosity liquor where the effect of fouling was not observed in the reading of the instrument. This is most likely due to the fact that at high viscosities the change in sensor mass due to fouling has a smaller effect on the amplitude of vibration. Fouling is most significant at lower viscosities, where the same amount of fouling induces a higher error in viscosity.

Fouling always occurred at the same location on the sensor, namely on the small ledge where the sensor joins the drive shaft, and it appears to be caused by flow separation at the back of the cylinder. The location of the deposits was verified physically by disassembling the containment vessel on several occasions. The sensor showed no fouling on any other surface. This problem could be eliminated through the use of other sensor designs available from the manufacturer. For the sensor used in this study, fouling is likely to be eliminated by streamlining (using a smaller included angle) the rear of the sensor so as to reduce any stagnant flow areas, and by increasing the sample line flow rate. Further studies conducted with another sensor featuring an included angle of 90° showed that negligible levels of fouling can be attained. Aside from its potentially fouling-reducing effects, flow rates greater than those used in this study are likely to have no other effects on the instrument readings.

There was no evidence of erosion of the Teflon coating of the sensor, even though the experiments were run intermittently over a period of more than a year. The efficacy of the coating is undetermined, but it is suspected that Teflon coating is not necessary.

The electronic control unit is capable of handling up to four sensors plus an on-line densitometer input. With the addition of a densitometer, absolute viscosity can be determined on-line. Since the density of black liquors is not as strong a function of temperature as viscosity is, an on-line density measurement is probably not necessary for most applications. In those cases, an acceptable approach would be to estimate the density from temperature-based correlations and use the estimates to produce an absolute viscosity reading.

3.5 Mill Trials at a St. Laurent Paper Products Corporation Site

The Nametre viscometer was selected for trial at the St. Laurent Paper Products Corporation mill at West Point, Virginia, because this instrument has the broadest range for measurement of viscosity and at this mill there was interest initially in measuring viscosity at a number of different points in the recovery system. The vibrating element of the instrument installed at the St. Laurent mill was streamlined to eliminate (or greatly reduce) the buildup of deposits on the downstream side of the element that was observed on the rather blunt cylindrical element used in the flow line tests. This was achieved by reducing the included angle to 90° (see inset in Figure 3.1).

As was the case with all the mill trials attempted in this study, long delays in mill trials were experienced due to the necessity to wait for shut downs for installation and due to unexpectedly long negotiations over agreements between the participating mills and viscometer companies. The mill trial of the Nametre instrument at the St. Laurent site began eight months after the completion of Phase IIA of the program.

3.5.1 Experimental Overview

The viscometer was installed in a side stream line from the furnace ring header so that the liquor viscosity would be measured at the liquor firing conditions, but so that this line could be isolated for cleaning the instrument without interrupting furnace operation. The liquor diverted into the side stream line was returned to the suction side of the pump. The side stream line was steam traced and sufficient flow was maintained to exchange the liquor in the chamber containing the instrument in less than a minute to ensure rapid response of the device to changes in viscosity of the liquor in the ring header. The side stream line was piped and valved to isolate the instrument and chamber for flushing and cleaning and to allow removal of the instrument for maintenance.

A plan for evaluation of the instrument was developed in cooperation with The Nametre Company and St. Laurent mill personnel. Instrument readings were to be continuously recorded on the distributed computer system (DCS) and the pertinent DCS records were to be retransmitted to the University of Florida. Originally, our plan was to provide for on-site laboratory measurements of viscosity and solids concentration during the first several weeks of the trial. A trained technician from the University of Florida would be on-site and an appropriate laboratory viscometer would be transported from the University of Florida to the mill where it would be set up and used by the technician to make measurements on samples to verify the readings of the Nametre viscometer installed on-line in the mill. After that period, the laboratory instrument would be returned to the University of Florida, and samples taken periodically at the mill would be transported to the University of Florida for measurements for verification. These original plans

had to be changed because of long unanticipated delays in starting the mill trials and to the fact that fixed costs associated with the project reduced the available resources.

An alternative mode of operation was established through the availability of increased technical support provided by the mill and a detailed plan for sampling was developed. Samples were to be taken at the mill under technical supervision, and transported by express freight to the University of Florida where verification measurements would be made. Even under this plan, the number of verification measurements had to be severely restricted due to the costly delays that had been experienced.

The mill trial began on July 31, 1997 and samples were taken until September 12, 1997. A total of ten mill samples were evaluated at the University of Florida during the trial period. These were spaced over increasing time periods. Samples were taken one, four, six, eight, twenty, twenty-two, thirty-nine, and forty-three days after the initial sample. The mill trial was continued through November, 1997 and an additional sample was taken and evaluated on November 13, 1997. Several times during the mill trial the instrument and instrument chamber was isolated and steam cleaned. When placed back on line after cleaning, the instrument readings returned to values very near to those produced before cleaning, indicating that there was no buildup of solid deposits on the instrument sensor. The longest period run without cleaning was in excess of 30 days, and there was no evidence of interference in instrument performance by deposits even over this period. The liquor flow through the side stream line was not fitted with a screen to collect suspended particles.

The goal of the program was to demonstrate that the on-line instruments could measure the absolute viscosity of the bulk fluid with a determined absolute accuracy. The Nametre instrument measures the product of the absolute viscosity and the density of the fluid, rather than the absolute viscosity. Also, this measurement operates at a shear rate determined by the mechanical design of the measuring element and the coupling that occurs between the element and the fluid. For verification purposes, laboratory measurements were made using viscometers based on totally different design principles, and density correlations for the samples were experimentally determined. Solids contents were also measured in the laboratory.

3.5.2 Discussion of Results

Mill samples were taken during periods of stable operation. Table 3.3 shows the times at which 10 samples were taken, along with mill readings of liquor temperatures, solids concentration from refractive index readings, and the on-line viscosity measured by the Nametre instrument. Also included are solids concentration as determined at the University of Florida using the

modified McDonald method. As the last column of the table shows, the solids content measurements obtained at the mill and those obtained at the University of Florida are not in very close agreement. This is of particular relevance because the viscosity is a strong function of the solids content of the black liquor. Note that the difference is not necessarily consistent, although most laboratory measurements are an average of about 1.3% higher in solids content than the mill measurements. Such differences are consistent with our usual experience. However, four laboratory measurements are about 1.3% lower than mill measurements. There is roughly an average ±1.3% difference in mill and laboratory solids concentration measurements which (if both measurements are correct) can result in an estimated 7-9% difference between the viscosities measured on-line by and the viscosity measured at the University of Florida laboratories. This, of course, is an uncertainty that we had hoped to eliminate by making laboratory measurements at the mill during the early stages of the trial. For purposes of comparison, the laboratory measurements of solids concentration were taken as being the correct values.

Table 3.3. Analysis of samples collected at St. Laurent Paper mill and readings produced by the on-line Nametre unit. Sampling time, firing temperature, on-line reading (viscosity-density product measured by the on-line Nametre instrument), and solids contents reported as read at the mill site and as determined at the UF lab.

Sample			Firing Temperature		On-Line Reading	Solids Content		
						Mill	UF Lab	Difference
ID	Date	Time	°F	°C	cP-g/ml	%	%	%
SL073197	07/31/97	10:50	258.0	125.6	45	67.7	69.03	1.33
SL080197	08/01/97	10:06	258.4	125.8	58.1	70.1	71.61	1.51
SL080497	08/04/97	09:00	258	125.6	35	66.9	68.69	1.79
SL080697	08/06/97	16:00	259	126.1	56	70.4	70.62	0.22
SL080897	08/08/97	15:00	262	127.8	35	67.8	68.94	1.14
SL082097	08/20/97	15:45	260.7	127.1	40	68.5	67.52	-0.98
SL082297	08/22/97	15:05	261.6	127.6	38	69.6	68.24	-1.36
SL090897	09/08/97	16:35	258.6	125.9	38	65.9	67.89	1.99
SL091297	09/12/97	08:00	259.7	126.5	34	68.7	67.09	-1.61
SL111397	11/13/97	10:28	258.6	125.9	67.4	71.8	70.06	-1.74

The densities of the first seven samples were measured at ambient temperature using an Accupyc Comparison Pycnometer, and the results are summarized in Table 3.4 Multiple measurements were made for each liquor. The densities at ambient temperature were determined to within a standard deviation of ±0.02% or better. Additional analytical work was done on sample SL080197 by determining its reduced density and its thermal expansion coefficient by dilatometry to a precision of better than 0.1%. Results are presented in Table 3.5.

Table 3.4. Measured density at the firing temperature of collected at St. Laurent Paper mill samples, corrected density ad 25^{0}C, and corrected density values at the firing temperature

Sample ID	Measured Density		Corrected Density at 25 °C		Corrected Density at Firing Temperature	
	T	ρ(T)	T	ρ25	T	ρ(T)
	°C	g/ml	°C	g/ml	°C	g/ml
SL073197	23.1	1.4468 ± 0.0002	25.0	1.4294	125.6	1.3594
SL080197	21.8	1.4493 ± 0.0001	25.0	1.4456	125.8	1.3748
SL080497	22.2	1.4468 ± 0.0001	25.0	1.4273	125.6	1.3572
SL080697	22.3	1.4562 ± 0.0002	25.0	1.4394	126.1	1.3683
SL080897	21.7	1.4490 ± 0.0002	25.0	1.4289	127.8	1.3571
SL082097	22.7	1.4441 ± 0.0001	25.0	1.42	127.1	1.3492
SL082297	22.8	1.4374 ± 0.0001	25.0	1.4245	127.6	1.3531
SL090897			25.0	1.4223	125.9	1.3522
SL091297			25.0	1.4173	126.5	1.347
SL111397			25.0	1.4359	125.9	1.3647

Table 3.5. Thermal expansion data and reduced densities for sample SL073197.

Temperature	Density	Reduced Density
°C	g/ml	g/ml
20.5	1.44826	1.0018
35.4	1.43925	0.9956
50.1	1.42946	0.9888
65.4	1.41918	0.9817
80.1	1.40848	0.9743
94.4	1.39880	0.9671
106.0	1.38976	0.9614

These density and thermal expansion results were compared with measurements made in the past on other liquor samples from the same mill recovery system. It was determined that the new data was comparable to earlier results; therefore, we proceeded to use the density and thermal expansion data presented in Tables 3.4 and 3.5 to develop a correlation for determining density as a function of temperature and solids concentration. The correlation in question has been used successfully for more than a hundred black liquors to predict liquor density from a known source to within better than 0.1%; further details are given below.

The thermal expansion data and the ambient temperature density measurements were used to develop a correction formula for experimental density measurements to 25°C. It was found that for a black liquor such as the St. Laurent mill liquor, the relation between ρ_{25} and solids concentration

is linear up to a solids concentration of 75%. The seven experimental liquor density measurements reported on the third column of Table 3.4 were used to determine the following correlation:

$$\rho_{25} = A + B\,S \tag{3.8}$$

with

$$A = 0.99705$$
$$B = 0.626379$$

where ρ_{25} is the liquor density at 25°C [g/ml], and S is the solids mass fraction [dimensionless], and where A and B are the empirical constants obtained experimentally via linear regression techniques. A relationship for the density at other temperatures was developed using the thermal expansion data for sample SL080197 leading to the experimentally determined correlation

$$\rho_R = \rho/\rho_{25} = 1.0 + a\,(T - 25) + b\,(T - 25)^2 \tag{3.9}$$

with

$$a = -4.35682 \times 10^{-4}$$
$$b = -5.20853 \times 10^{-7}$$

where ρ_R is the reduced viscosity [dimensionless], ρ is the liquor density [g/ml] at temperature T [°C], and ρ_{25} is the liquor density at 25°C [g/ml], and where a and b are the empirical constants obtained experimentally for these liquors. Correlation (3.9) was used to calculate densities for all liquors samples collected during the mill trials carried out in St. Laurent Paper Product Corporation's site.

The liquor density at the firing conditions was calculated from the solids concentration and firing temperature. First ρ_{25} (the liquor density at 25°C) was calculated using equation (3.8), then the reduced liquor density at the firing temperature was calculated using (3.9), and finally, these two results were combined to find the liquor density at the firing temperature and solids concentration for the sample using

$$\rho = \rho_R\,\rho_{25} \tag{3.10}$$

These calculated values are given in Table 3.4 and are accurate to within approximately 0.2% or better for a given solids concentration and temperature.

Laboratory viscosity measurements were made using pressurized rotational and sliding element viscometers over a shear rate range of about 200 to 1200 sec^{-1}. Past experience with many black liquors had demonstrated that these data can be extrapolated to at least 3000 sec^{-1} with confidence. All of the mill liquor samples proved to be slightly non-Newtonian (*i.e.*, viscosity

varies with shear rate) at the firing temperatures. The viscosity was determined as a function of shear rate at four temperatures between 108°C and 133°C for mill sample L5073197 using a Haake and a Brookfield LTV pressurized coaxial cylinder viscometers. The Brookfield LTV laboratory instrument can be used for measurements at higher shear rates, since rotating the cup rather than the inner cylinder retards development of secondary flows. Since data taken with the two instruments were in close agreement up to about 800 sec^{-1}, most subsequent data were taken using the Brookfield viscometer.

Viscosity data were taken for each liquor sample at temperatures within less than 1°C of the liquor firing temperature. Using the temperature coefficients for viscosity at selected shear rates that were developed from data on sample SL073197, the experimental data for each liquor were corrected to the firing temperature for each liquor. These corrections were less than 3%, with uncertainties that are probably very nearly an order of magnitude less and are therefore negligible. It is also worth noting that laboratory precision on repeated measurements of viscosity for any one fluid is about ±3%. Measurements with the Brookfield and Haake pressurized laboratory viscometers with calibrating fluids and with liquors fall within this range.

There is negligible error in density determination (less than 0.1%), negligible error in correcting for the difference in firing temperature and temperature at which the viscosity is measured in the laboratory (less than an estimated 0.3%), and the uncertainty in the accuracy of laboratory measurements of about 3%. Therefore, the total uncertainty contribution from these three effects (assuming that the mill temperature measurement is accurate to within better than 0.1°C) is 3.4%. As discussed earlier, differences in solids concentration introduce an uncertainty of an estimated maximum of about 7-9%. If the mill temperature measurements have an uncertainty of about 1°F (0.56°C), this introduces an additional uncertainty of about 3%. Hence, the sum of all uncertainties leads to a maximum estimated difference of about 16%.

As expected from past experience, the dominant factor in the estimated maximum uncertainty is due to difference in solids concentration. Mill solids concentration measurements in the 65-75% range are seldom accurate to better than ±0.5%, and there is the chance in some cases that water is lost due to flashing during sample collection. Based upon the results, we have assumed that there is an average of ±1.3% uncertainty in solids measurement of liquor samples taken during the mill trial. Also, it is seldom that temperature measurement devices used in mills are individually calibrated as is normal practice for laboratory work. Therefore, an assumption of an uncertainty ±0.5°F in temperature measurements is reasonable. Therefore, ±16% was determined to be an acceptable range for differences in viscosity determinations between the on-line Nametre instrument and laboratory instrument measurements on the same liquor.

The relations of viscosity versus shear rate for the laboratory data were examined to estimate the shear rate at which the on-line Nametre instrument operated. This was done at shear rates ranging from 1700 to 2500 sec^{-1}, using as a criterion that the average percentage differences in viscosity measurements should be near zero for all measurements except that known to be during a period of mill upset. A shear rate of 2300 sec^{-1} was selected as most appropriate. It should be noted that the shear rate of operation of the Nametre instrument is affected by the fluid viscosity, but this effect is small. Solids concentrations and viscosities determined in the laboratory at 2300 sec^{-1} were accepted as absolute. The liquor firing temperature at the mill was accepted as absolute. On this basis, liquor density at the firing temperature was determined as discussed and viscosities calculated from the on-line Nametre readings. Viscosities at 2300 sec^{-1} as determined in the laboratory were corrected for the small differences between liquor firing and laboratory viscosity measurement temperatures. These results are presented in Table 3.6. Using the laboratory determinations of viscosity at 2300 sec^{-1} as the reference standards, we calculated the percentage difference as

$$\text{Relative difference} = 100\,\frac{\eta - \eta_{ref}}{\eta_{ref}}\ \%$$

where η is the viscosity measured by the on-line Nametre sensor, and η_{ref} is the viscosity determined using the instruments available at the University of Florida laboratories. These differences are shown in the last column of Table 3.6.

Table 3.6. Summary of physical data for samples collected at St. Laurent Paper mill including the relative difference between the viscosity determined by the on-line Nametre instrument and the viscosity determined at the UF laboratory facilities.

Sample			Firing Temperature		Solids Content	Density	On-Line Reading	Viscosity		
					(mill values)			On-Line	UF Lab*	Relative Difference
ID	Date	Time	°F	°C	wt %	g/ml	cP-g/ml	cP	cP	%
SL073197	07/31/97	10:50	258.0	125.6	67.7	1.3594	45	33.3	30.3	9.90
SL080197	08/01/97	10:06	258.4	125.8	70.1	1.3748	58.1	42.5	48.9	-12.99
SL080497	08/04/97	09:00	258	125.6	66.9	1.3572	35	26.0	25.7	1.13
SL080697	08/06/97	16:00	259	126.1	70.4	1.3683	56	41.0	37.3	9.83
SL080897	08/08/97	15:00	262	127.8	67.8	1.3571	35	25.9	30.3	-14.45
SL082097	08/20/97	15:45	260.7	127.1	68.5	1.3492	40	29.5	30.3	-2.57
SL082297	08/22/97	15:05	261.6	127.6	69.6	1.3531	38	27.9	24.5	13.95
SL090897	09/08/97	16:35	258.6	125.9	65.9	1.3522	38	28.3	21.5	31.86
SL091297	09/12/97	08:00	259.7	126.5	68.7	1.347	34	25.1	27.1	-7.52
SL111397	11/13/97	10:28	258.6	125.9	71.8	1.3647	67.4	49.0	59	-16.95

* Determined at 2300 sec^{-1}

While these results are not as good as we had hoped, they are very reasonable and indicate that the trial was successful. All differences lie within or near the estimated uncertainty of ±16% with scatter approximately about zero (except for sample SL090897 which was taken during a mill upset and is suspect). If the results for sample SL090897 is rejected, only the difference for sample SL111397 exceeds 16% (-16.3%), and this is not far outside the uncertainty band of ±16%. For the first 39 days of the trial, the differences were less than ±16%, and most were within the original target of ±10% in spite of the uncertainties in solids concentration. The difference after 105 days of the trial was still only 16.3%, even though no special attention was given to the instrument nor was it cleaned for the last 66 days of the trial. Therefore, overall, the trial can be considered as successful, indicating that the Nametre instrument can be used with confidence for on-line measurement of viscosity of black liquors at high solids concentrations and temperatures.

3.5.3 Summary of Observations for the Mill Trials

The Nametre instrument operated continuously for a period of approximately six months without experiencing any failures. The instrument required very little maintenance, and did not suffer performance degradation due to solids deposits in the sensor due to the adoption of a streamlined sensor with a 90° included angle. The viscometer produced on-line viscosity measurements that in general differed from reference laboratory measurements by less than ±16%, indicating that the instrument can operate successfully as an on-line viscometer. The trials show that periodic steam cleaning of the instrument is not strictly necessary since the viscometer operated successfully for over three months without steam-cleaning maintenance.

3.6 Specific Conclusions and Recommendations Regarding this Instrument

The torsional oscillatory viscometer can be used to effectively monitor black liquor viscosity on line. The equipment operates satisfactorily under both steady-state and transient conditions, producing fast, accurate, and reproducible measurements. Varying the black liquor flow rate has no effect on the viscosity readings. The equipment is self contained and requires very little attention. This viscometer is capable of handling a very wide range of viscosities, and the electronics package can accommodate multiple sensors. This makes it possible to use one electronics package with multiple sensors deployed at different locations where viscosity could vary widely making it possible to track viscosity at a number of points in the recovery system.

Liquors with a propensity for precipitating solids may foul the sensor and cause a constant offset in the reading. Performance can be restored in most cases with a simple steam cleaning procedure. However, fouling can be avoided altoghether using a streamlined probe with a 90°

included angle. Based on our experience, we recommend that the viscometer be used in a slip stream to provide easy access to the sensor for repairs and maintenance.

3.7 Recommended Installation and Deployment Point

In general, the Nametre instrument should be placed in a side stream as shown in Figure 3.9. We have successfully utilized 1-inch pipes for the side stream, but larger diameters could also be used. The side-stream line containing the instrument should be piped and valved so that it can be drained and steam cleaned as needed to remove black-liquor deposits that constrict flow. A conceptual valving configuration is shown in Figure 3.9. Although the manufacturer correctly claims that the instrument could be installed directly in the main line (avoiding the additional costs associated with a the more extensive installation shown in the figure), we do not recommend such installation because routine instrument maintenance or repair services may cause unacceptable interruption of flow in the main line.

It must be remarked that the flow around Nametre's measuring element need not be laminar, but the measuring element is best mounted in a chamber with at least 2-inches of separation between the element and the walls of the chamber. The instrument itself could be mounted in a chamber in a line that can be periodically isolated and drained for steam cleaning. Preferred deployment locations would be in a side stream line fed with liquor diverted from the ring header line or in a line to a firing gun, as shown in Figure 3.9. Liquor screening is not necessary, since the presence of suspended particles does not affect the instrument; however, a strainer is recommended to provide ultimate protection against large-diameter particles.

In order to prevent the deposit of black-liquor solids on the surface of the sensor head located downstream we recommend an included angle (see inset of Figure 3.1) of 90°. This has the effect of streamlining the profile of the sensor and thus creating flow conditions unfavorable for the deposition of solids.

It is very important to insulate and heat all the side-stream lines as well as the viscometer itself. This precaution is of critical importance for two main reasons. First, the viscosity of black liquor is extremely sensitive to temperature variations; therefore, heating and insulation precautions must be taken to guarantee that the temperature of the black liquor that reaches the instrument is identical to that of the liquor flowing in the main line. The second reason is that lack of heating and insulation will lead to black liquor cooling inside the side stream pipe, eventually causing pluggage of the line. The heating of the side-stream lines can be done using a steam-tracing piping network consisting of copper tubes wrapped around the pipes, or by installing an electrical heating tape

which can be connected to an inexpensive temperature controller.

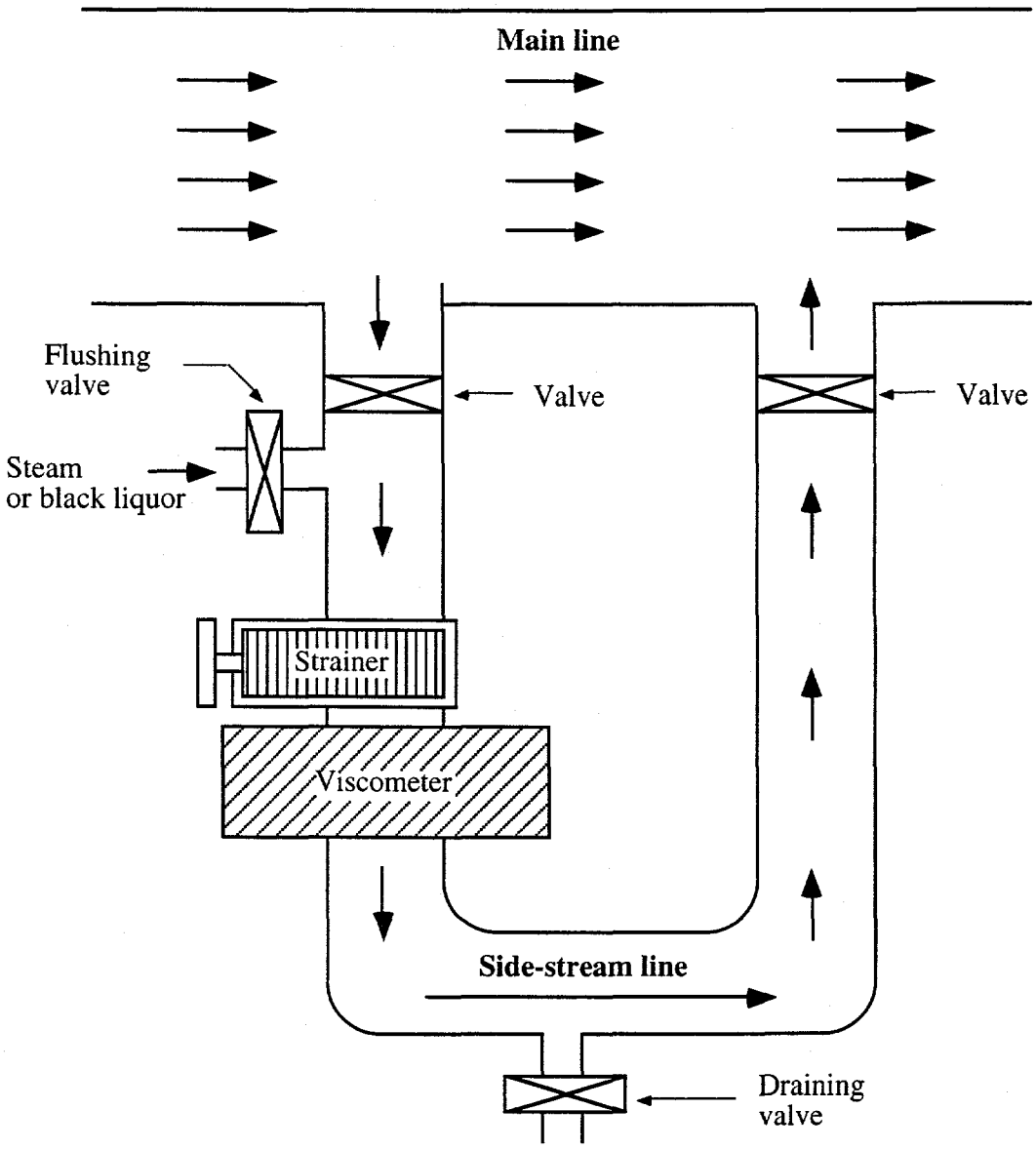


Figure 3.9. Schematic of the recommended installation of the viscometer in a side-stream line. All the side-stream pipes and the viscometer itself should be heated (using steam-tracing tubes or electrical heating tape) and insulated.

The Nametre instrument is relatively expensive in comparison with other devices considered in this study; however, most of the cost is connected with the electronics rather than the measuring element. If multiple measurements are to be made, the cost per measurement is lower, since

multiple measuring elements can be used. The instrument measures the product of viscosity and density at one shear rate. Since the viscosity of liquors varies much more than density with temperature, concentration, and composition, the fact that the instrument does not measure the viscosity directly is not a severe limitation.

For the case of significantly non-Newtonian (pseudoplastic) fluids, the use of a single Nametre sensor would not be able to capture the viscosity of the flowing material if the flow index as well as the viscosity happen to vary. In this case the problem should be solved by installing two Nametre elements, each operating at a different shear rate.

4. EVALUATION OF MICRO MOTION'S ON-LINE CAPILLARY-CORIOLIS VISCOMETER

4.1 Abstract

A capillary-coriolis instrument featuring sealed pressure diaphragms connected to a differential pressure cell was evaluated as an on-line viscometer and density meter for kraft black liquor in a Pilot Plant environment at the University of Florida and at the site of the Canadian Forest Products mill in Prince George, British Columbia

Testing at the University of Florida Pilot Plant was done using a piping loop where black liquor is allowed to circulate under closely controlled solids concentration, temperature, and flow rate. Measurements were made under laminar flow conditions for liquors with solids concentrations ranging from 34% to 74%, and temperatures up to 140°C. The accuracy and precision of the instrument were determined by comparison with laboratory measurements of viscosity and density. In general, under laminar flow conditions, the viscometer tracked reliably within ±10% of laboratory reference viscosity for high viscous liquors. The sensor operated satisfactorily under both steady-state and transient conditions. The instrument transient response was very rapid with a lag not exceeding 30 seconds. The sensor did not experience any fouling problem throughout the long period of testing at the pilot plant. At high flow rates for low viscous liquors, a linear rise in measured viscosity was observed due to the development of secondary flows. A power-law correlation has been developed to compensate for this effect and recover accuracy.

Testing at a Canadian Forest Products mill was done by installing the Micro Motion capillary-coriolis viscometer in a side stream connected to a ring header line. Data collected during a period of over three weeks of continuous operation shows that the instrument can make very accurate measurements of viscosity, and that it can produce viscosity measurements which are in reasonable agreement with reference measurements taken under controlled laboratory conditions. The study of samples available showed a viscosity accuracy better than ±19.5% with respect to laboratory reference values. The long-term reliability and maintenance needs of the instrument could not be tested because pluggage occurred in the side-stream piping network caused by unexpectedly large suspended solid particles. The instrument itself did not fail, and its strong performance in the Pilot Plant trials indicates that it is capable of sustaining long periods of continued operation with virtually no maintenance.

4.2 Viscometer Description and Principles of Operation

The capillary-coriolis viscometer is based on the Hagen-Poiseuille equation for incompressible Newtonian fluids in fully developed steady state laminar flow conditions in a capillary [Bird, *et al.*, 1960; Micro Motion, 1993; Zaman, 1993]

$$\eta = \phi \frac{\Delta p \rho}{\dot{m}} \tag{4.1}$$

where ϕ is a proportionality constant, Δp is a differential pressure, ρ is a density, and $\dot{m}$ is a mass flow rate.

A differential pressure cell provides a direct measurement of the pressure drop (Δp), and the coriolis mass flow sensor provides a direct measure of mass flow rate ($\dot{m}$) and of the density (ρ), hence permitting the calculation of viscosity from Equation (4.1). Since the density of the fluid in the sensor is measured, a volumetric flow rate ($\dot{q}$) can also be calculated as $\dot{q} = \dot{m}/\rho$. The proportionality constant ϕ in Equation (4.1) is determined via calibration using a fluid of known viscosity.

4.2.1 Mass Flow Rate Measurement

The coriolis mass flow-rate sensors, measure the effect of coriolis acceleration and deceleration caused by the flow of a fluid in vibrating tubes. The coriolis force is a force that naturally arises when dynamic problems are analyzed within a rotating frame of reference (for example, the earth is a rotating reference frame). This concept is very familiar in meteorology and oceanography and is often invoked to explain everyday weather phenomenon like wind patterns and ocean currents. For a detailed description about the origin and the nature of the coriolis force, the interested reader is referred to Stommel and Moore [1989].

Consider the schematic setup shown in Figure 4.1. The fluid flows through the U-shaped tube that is rigidly supported at one end and is free to vibrate at the other end. The amplitude of this vibration increases from zero at the supported end to the maximum value at the free end. The tube is vibrated at the natural frequency, so that the curved end of the tube moves in an arc. Consider the downward cycle of the tube vibration. The fluid entering the sensor at the supported end has only the horizontal component of velocity; however as the fluid moves, due to the downward movement of the tube it also acquires a vertical component of velocity. This downward component of velocity increases as the fluid travels down the tube and negotiates the tube turn. This change in velocity results in fluid acceleration. This is denoted as the 'coriolis acceleration'.

According to the Newton's Third law of Motion, the downward force due to this coriolis 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 and this results in 'coriolis deceleration'. In this section, the tube applies a reactive force in the downward direction. This opposing upward and downward reactive forces causes the tube to twist. The amount of twist is proportional to the mass flow rate through the sensor.

The mass flow meter tested in this study, uses a pair of tubes in the shape of a coat hangers. An electromagnetic drive coil located at the center of the tube bend vibrates the flow tubes at their natural frequency. The amount of twist caused by the flow is measured as the time difference in phase between two electromagnetic velocity sensors located on either side of the flow tube. With no flow through the sensor, the two velocity pickups are in phase. With flow, the velocity signals are no longer in phase and a time difference is detected. A flow calibration factor is determined using the mass flow meter with a reference fluid, and along with the time difference, the mass flow rate is ascertained [Micro Motion, 1993].

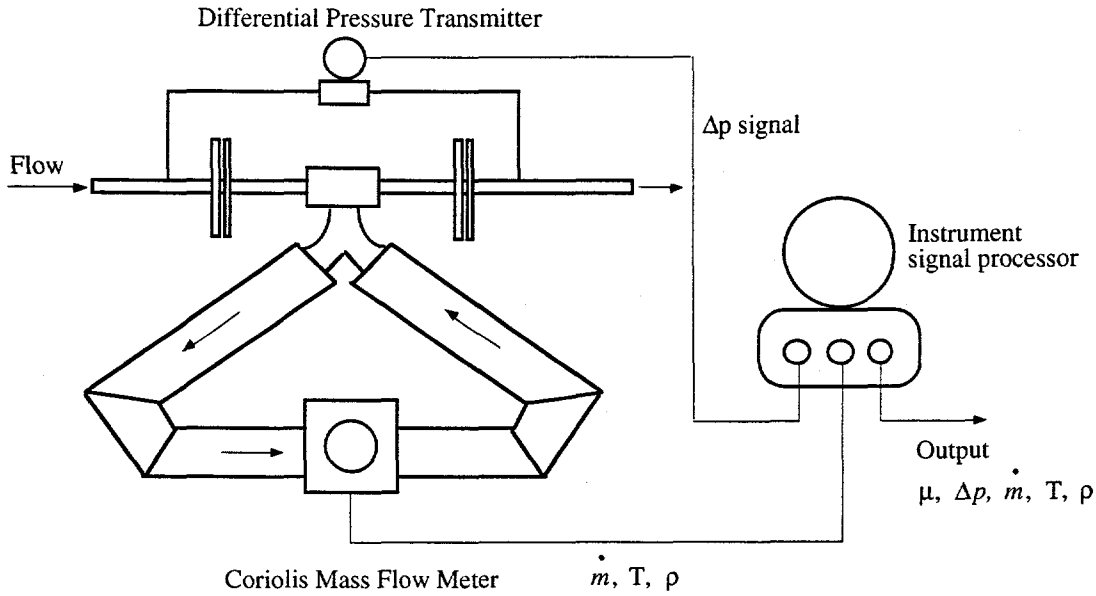


Figure 4.1. Schematic diagram of the Micro Motion capillary-coriolis viscometer evaluated in the University of Florida pilot plant.

An ideal coriolis mass flow meter is not affected by the thermodynamic state of the fluid (i.e. density, pressure, temperature), flow regime, or by the fluid's rheological properties (viscosity). The oscillating behavior of the vibrating system in the sensor depends only on the elastic properties of the vibrating tubes (such as the Young's modulus and the Poisson's modulus), geometric

configuration of the sensor, and the flow behavior of the fluid. However, in actual operation, the elastic properties of the sensor tubes are affected by temperature and pressure and has to be appropriately compensated. For example, the stiffness of the vibrating system decreases with the increase in temperature. Hence, at fixed mass flow rate, at elevated temperatures, an increase in the measured signal is observed [Cascetta *et al.*, 1992]. Commercial mass flow meters employ an electronic thermo-compensation system to offset this effect. The fluid pressure also has an effect on the measurement and has to be compensated. The stiffness of the vibrating tubes increase with increase in the fluid pressure and this results in underestimated readings [Cascetta, 1996]. New generations of coriolis mass flow meter (like the one used in this study) employ a sensor specifically designed to minimize or if possible, eliminate these effects.

4.2.2 Density Measurement

The coriolis mass flow maters can also be used to determine the density of the fluid. The natural frequency of vibration of the coriolis sensor in operation, depends on the mass of the sensor as well as the mass of the fluid in the sensor. The sensor with the mass of the fluid present in the flow tubes can be modeled as a mass-and-spring assembly in which the mass, once set in motion, vibrates at its natural frequency. By a simple force balance, the resonant frequency for the free oscillation of a mass-spring assembly can be shown to be [Micro Motion, 1993]

$$f = \frac{1}{2\pi}\sqrt{\frac{K}{m_{tot}}} \tag{4.2}$$

where f is the resonant frequency, K is a calibration constant, and m_{tot} is the mass of the fully flooded sensor. Using

$$m_{tot} = m_{tube} + m_{fluid} = m_{tube} + \rho V \tag{4.3}$$

where m_{tube} represents the mass of the tubes, m_{fluid} is a calibration constant, and V is the volume of the sensor. From (4.2) and (4.3) the density can then be calculated as

$$\rho = \left(\frac{K}{4\pi^2 V}\right)\frac{1}{f^2} - \frac{m_{tube}}{V} \tag{4.3}$$

The calibration constant K is determined by using two fluids of known density (air and water) as a reference. Note that the mass and volume of the sensor are fixed for any given device, and that the tube frequency is established by the instrument.

From the measurements of the pressure drop, density, and the mass flow rate, the Hagen-

Poiseuille Equation (4.1) can be used to calculate viscosity, provided the flow is laminar and steady-state conditions prevail. The viscosity proportionality constant ϕ is set by calibrating the sensor at a point of known viscosity for a Newtonian fluid. A Non-Newtonian fluid uses two-points: One in the upper part and one in the lower part of the operating viscosity range. It is important to 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 onset of turbulence. At relatively high Reynolds numbers, secondary flow characteristics as well as entry and exit effects can produce pressure drops higher than that predicted by the Hagen-Poiseuille equation, hence adversely affecting the reading of the sensor. Therefore it is important to control the flow rate through the instrument.

4.3 Viscometer Configuration and Installation Details

The specific configuration of the capillary-coriolis viscometer tested is Micro Motion CMF025M Coriolis Mass Flow Sensor (with a pair of coat hanger shaped flow tubes), combined with a Rosemount 3051CD differential pressure cell and a Rosemount RFT9739 transmitter, to give an instrument capable of measuring a pressure drop across a length of tube as well as being capable of measuring mass flow rate and density. These three measurements produced by the coriolis-capillary instrument can be combined to yield a viscosity measurement for the black liquor, as discussed in the previous section. The physical location of this viscometer in the pilot flow loop is indicated by the tag VIS 2 in the process and instrumentation diagram given in Figure 8.1.1 in Appendix 8.1.

A schematic diagram of the viscosity measurement system is shown in Figure 4.1. 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 in one of the flow tubes is used to calculate temperature at the point of measurement. The transmitter also supplies power to the differential pressure cell and the 4-20 mA signal from the DP-cell is fed to the transmitter [Micro Motion, 1992a].

The transmitter has two analog 4-20 mA outputs, a frequency/pulse output and a control output. The analog and frequency outputs can be configured to independently represent any of the following variables: viscosity, pressure drop, mass flow rate, volumetric flow rate, density, temperature, or user defined alarms. The control output represents flow direction, alarms, or transmitter status. In addition, the transmitter also has serial communication capability via Bell 202

or RS 485 serial communication standard.

The communication between the DAQ computer and the transmitter in the pilot flow loop was achieved simultaneously in two independent ways. The two analog signals from the transmitter (configured to be the liquor viscosity and the pressure drop across the instrument) were fed to the DAQ cards and read through the DAQ software. Also digital communication was established with the transmitter via the RS 485 standard under Modbus protocol. The manufacturer supplied software ProLink [Micro Motion, 1992b] was used to effect bi-directional data transfer. Using the software, it is possible to read and calibrate all the variables, set alarms, inquire transmitter status, reset the output types and limits, perform auto-zero and also to directly write to files.

4.4 Pilot Plant Trials at the University of Florida

4.4.1 Experimental Overview

Black liquor was obtained from three kraft mills, namely, Georgia Pacific Corporation at Palatka, Florida; Canadian Forest Products Limited at British Columbia, Canada; and St. Laurent Paper Products Corporation at West Point, Virginia, as being respectively representative of southern softwood, western softwood, and mid-Atlantic mixed-species pulping. These 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 4.1 lists the solids composition of the black liquors used in the experimental runs. The on-line viscometer was tested on 14 different black liquor concentrations, ranging from a 34.14% Georgia Pacific liquor to a 73.40% liquor obtained by mixing a Georgia Pacific Corporation liquor with a Canadian Forest Products Limited liquor (*cf.* Table 4.1).

The Georgia Pacific liquor was the easiest to handle because of its low level of air entrainment and ease of evaporation; hence, more experiments were performed with this black liquor. The Canadian Forest and St. Laurent black liquors formed crystalline particles in the storage tank at intermediate (53-60%) solids concentrations when used for several days as was required for the experiments. 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 the thermal and physical homogeneity of black liquor. To collect samples for laboratory characterization, the flow through the sample loop and main loop was stopped briefly and a 1 liter sample was withdrawn. Samples were collected from both the main loop and

the sample loop simultaneously, and were compared to verify sample integrity. The flow was stopped to ensure that flashing does not occur. Flashing destroys the sample integrity, making the liquor appear more concentrated than it actually is. A discrepancy of up to 2% in solids content has been observed due to flashing if this precaution is not taken.

Extensive laboratory characterizations were done on the samples drawn from the pilot flow loop. The laboratory analysis included the measurement and generation of correlations for viscosity, density, heat capacity, solids content, and boiling point elevation. This data provided complete physical information about the black liquor in the loop, and served as a reference basis for evaluating the performance of the on-line viscometer.

Experiments were run in the pilot flow loop at different temperatures for each of the concentrations listed in Table 4.1. The highest temperature reached in the main tank was limited to 10-20°C below the boiling point of the black liquor. This was done 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. After passing through the sample loop, the liquor temperature was reduced to that of the main-tank temperature by the cooler, and was then returned to the main stream. For each concentration-temperature combination, data was collected at different flow rates, including the static condition of no flow. The upper limit of the flow rate for the experiments is dictated by the output of the main progressive cavity pump at its maximum operational limit of 150 psig. However, the main line pressure was usually kept at no more than 70 psig for safety reasons. This pressure upper bound in turn limited the maximum possible flow rates obtainable for highly concentrated liquors.

Table 4.1. Black liquor types and solids concentrations tested with the capillary coriolis viscometer.

Georgia Pacific Corporation	Canadian Forest Products Ltd.	St. Laurent Paper Products Corp.	Georgia Pacific Corp. & Canadian Forest Products Ltd. 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%			

Prior to the beginning of the runs, the coriolis mass flow meter and the differential pressure cell were zeroed, and a one-point viscosity calibration was performed. In order to achieve this, the viscometer line was fully flooded and then the isolation valves (see valves labeled V7-V8 in the process and instrumentation diagram given in Figure 8.1.1 of Appendix 8.1) are activated to develop a no-flow condition in the viscometer line. The instrument readings were then zeroed via the instrument software. To provide a one-point viscosity calibration, the black liquor was maintained at its most highly viscous level (i.e., at the lowest temperature considered for the series of runs), and 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 is entered into the instrument software. The transmitter calculates the proportionality constant ϕ (see equation 1), and enters it into the instrument microprocessor. For a Newtonian fluid, this constant need not to be changed once it is set. All the black liquor tested in this study were Newtonian.

4.4.2 Discussion of Results

This section presents typical results obtained with the capillary-coriolis viscometer in the pilot flow loop [Dutka *et al.*, 1997]. Extensive results are given in the Phase IIA DOE report on this research [Fricke and Crisalle, 1996]. The results include characterizations of viscosity versus flow rate at different temperatures, solids compositions, and a study of the transient response of the instrument to temperature changes. The transient response study is very important to determine the feasibility of liquor viscosity control based on this instrument.

Figure 4.2 shows the results obtained for a high viscosity Georgia Pacific black liquor with a 68.39% solids content 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 in Figure 4.2 show the viscosity values measured by the instrument. It should be noted that the continuous line joining the markers is included as a visual aid to track the data trend, and is obtained through an arbitrary polynomial fit. The upper plot also shows a laboratory reference band of viscosity values defined by two dash-dot lines. Two to four different laboratory viscometers were used to determine the viscosity as a function of temperature of 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 viscometer for laboratory measurement. The upper and lower band edges respectively correspond to the highest and the lowest viscosity predictions produced by all the laboratory instruments. Any measurements that fall inside the reference band are considered to be consistent with the laboratory

measurements, and hence are deemed accurate. One should note that in Figure 4.2 the width of the reference band approximately corresponds to a ± 5% deviation from the mean viscosity measured by all the laboratory instruments.

The lower plot in Figure 4.2 is a measure of the percentage relative error between the viscosity measurement (η) produced by the on-line instrument and the viscosity (η_{ref}) predicted by a correlation generated from a reliable laboratory instrument, *i.e.*:

$$\% \text{ Relative Error} = \frac{\eta - \eta_{ref}}{\eta_{ref}} \times 100 \tag{4.4}$$

We consider an on-line instrument to be accurate if the relative error is ±10% or less.

For this particular liquor at 68.39% solids concentration, the laboratory reference viscosity was determined from the experimental correlation

$$\eta_{ref} = \exp\left(A - \frac{B}{(T+273.16)} + \frac{C}{(T+273.16)^2}\right) \tag{4.5}$$

where A = 18.2073771, B = -17633.4415, C = 4709530.7768, and where T is the temperature (°C) and η_{ref} is the laboratory-determined reference viscosity (cP).

A relevant remark concerning Figure 4.2 is that the temperature is not strictly constant during the run; in fact, as the flow rate varies, normally the temperature also varies. The observed temperature fluctuations were typically no greater than 1°C. However, since the viscosity of black liquor is temperature-sensitive, such small variations must be taken into account for accurate work. Table 4.2 gives the viscosity, and temperature data for 10 selected flow rates plotted on Figure 4.2.

Table 4.2. Flow-rate, temperature, and viscosity data for Figure 4.2.

Flow Rate (gpm) [gpm]	Temperature [°C]	Viscosity [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

Analysis of Figure 4.2 shows that the Capillary-Coriolis viscometer responds with good accuracy, since the measurements lie within the laboratory reference band. The percentage relative error during the run is better than -5%. Furthermore, the figure shows that the measurements were unaffected by flow rate since the data follows the trend defined by the edges of the laboratory reference band (which in turn are affected only by the temperature). Note that the instrument tracks the changes in viscosity that occur due to the small temperature changes that take place at different flow rates. It should be noted that the maximum flow rate for this run was 1.25 gpm.

Figure 4.3 shows another high viscosity run with Georgia Pacific 65.51% solids black liquor at approximately 80°C. Again, the viscometer tracks just below the lower laboratory reference boundary with a percentage relative error better than -12%. Higher flow rates were not possible due to limitations of the differential pressure cell selected for this application. Figures 4.2 and 4.3 are typical high-viscosity results for this instrument. In both cases, there is a minor offset with excellent tracking.

Figure 4.4 shows a mid-ranged viscosity run with Georgia Pacific 65.51% solids black liquor at approximately 95°C. Initially, the viscosity of the black liquor is approximately 140 cP, and then it slowly decreases to about 120 cP as the flow rate increases. At 0.5 gpm, the viscometer is within the laboratory reference band; however, as the flow rate increases, so does the viscosity reading. This run shows a transition to a linear rising response in the viscosity measurement, where the instrument loses accuracy though it retains a remarkable level of reproducibility (i.e. precision). This deviation effect is discussed in more detail in the following section.

Figure 4.5 shows a low viscosity run with Georgia Pacific 68.39% solids black liquor 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 gpm is slightly above the laboratory reference boundary with a value of 55 cP, and the viscosity measurement steadily increases as the flow rate increases, even though the laboratory reference viscosity remains relatively constant.

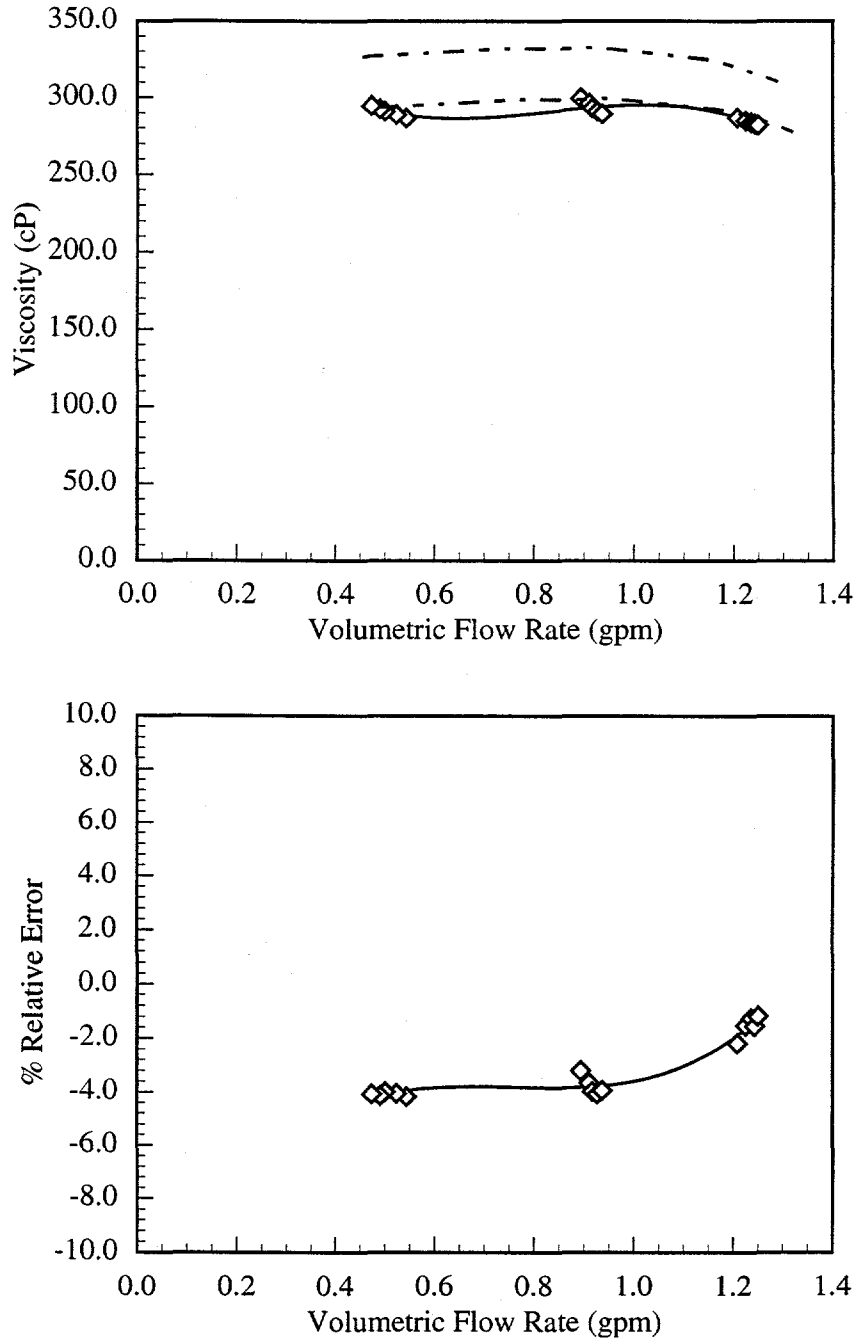


Figure 4.2. Viscosity measured on-line by the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 68.39% solids at approximately 85 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

Figure 4.6 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 resulting solids composition of 67.46%. The upper plot in Figure 4.6 shows the viscosity as a function of time measured by the on-line instrument. The lower plot in Figure 4.6 shows the temperature history during the run. In this experiment the temperature of the flowing black liquor steam was changed in a step-fashion by opening the steam control valve (valve labeled as V57 in the process and instrumentation diagram given in Figure 8.1.1 of the Appendix) 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) on the figure denote the instants when the valve opening is 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 660 cP. At approximately 2 minutes into the run, the first step input is introduced by manually opening the steam valve to 10%. A second temperature step input is applied 3 minutes into the run. There is approximately a 3-minute process time-delay before the RTD registers a temperature change at the point where the liquor exits the heat exchanger. After this process delay, it is apparent that the capillary-coriolis viscometer responds with an instrument delay that does not exceed 30 seconds. During this temperature step change there is a drop of viscosity from 660 cP to 250 cP. For the third step input (where the valve is set to 20% open), the time-delay of the viscometer is actually slightly shorter than the process delay. The viscosity decreases from 250 cP to 100 cP. In order to maintain accurate viscosity measurements from the instrument, a low flow-rate was maintained throughout the run.

Figure 4.7 shows the result of on-line density measurement with Georgia Pacific 68.51% solids black liquor at approximately 80°C. This run demonstrates the excellent accuracy and precision of the density measurement of the coriolis mass flow meter, a part of the capillary-coriolis viscometer. The density measurements were within -1% of the laboratory correlations.

Figure 4.8 shows the result of on-line density measurement with Georgia Pacific 42.20% solids black liquor 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.

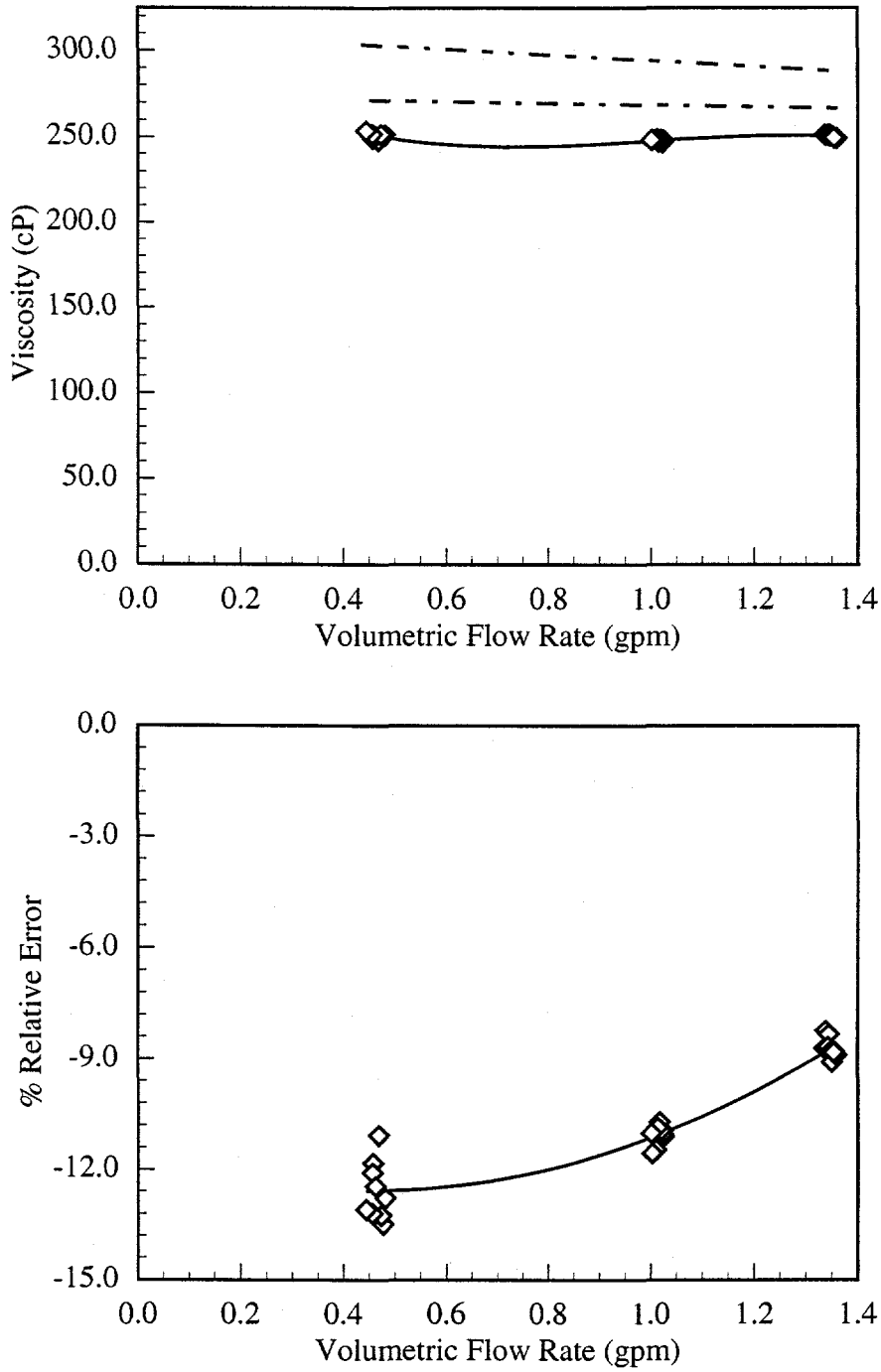


Figure 4.3. Viscosity measured on-line by the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 65.51% solids at approximately 80 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

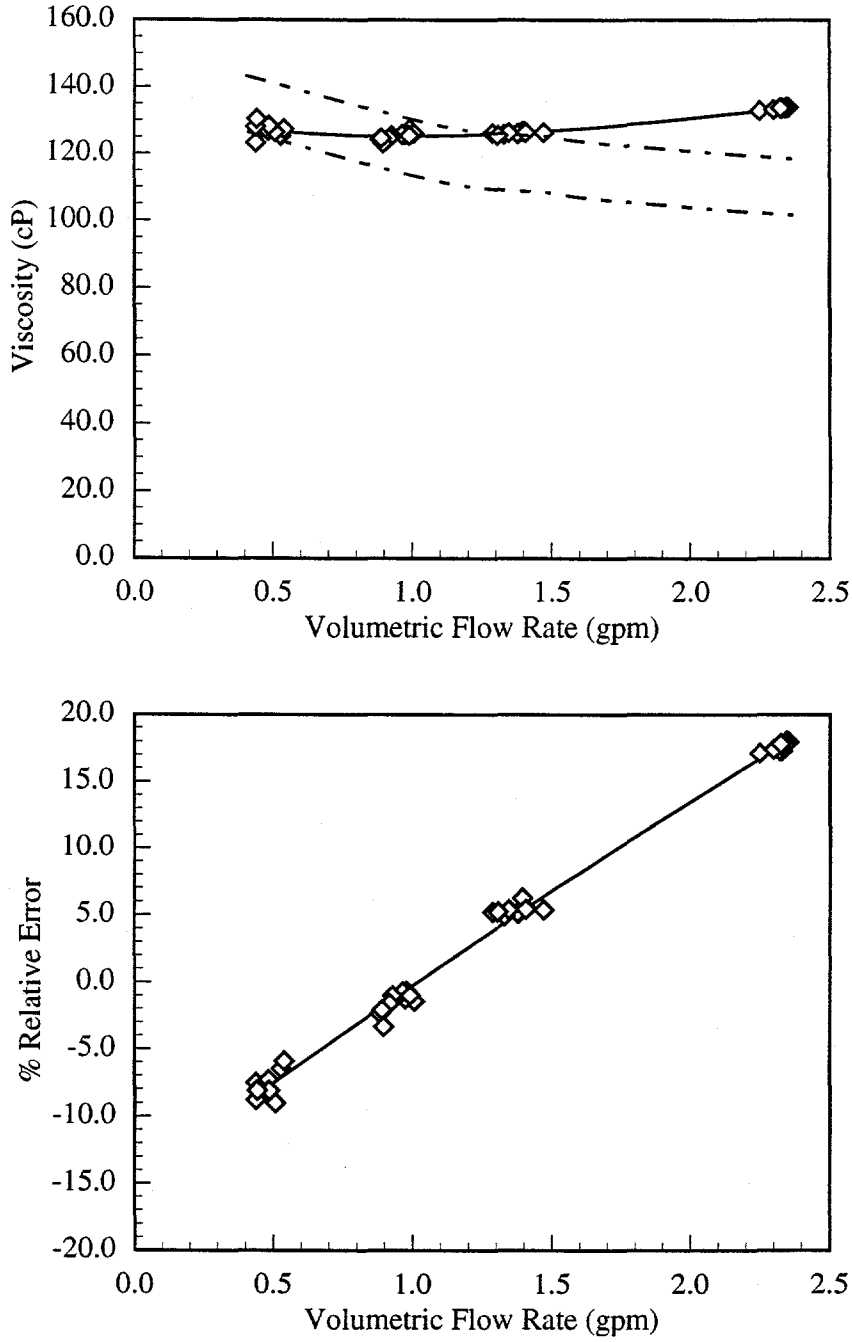


Figure 4.4. Viscosity measured on-line by the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 65.51% solids at approximately 95 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

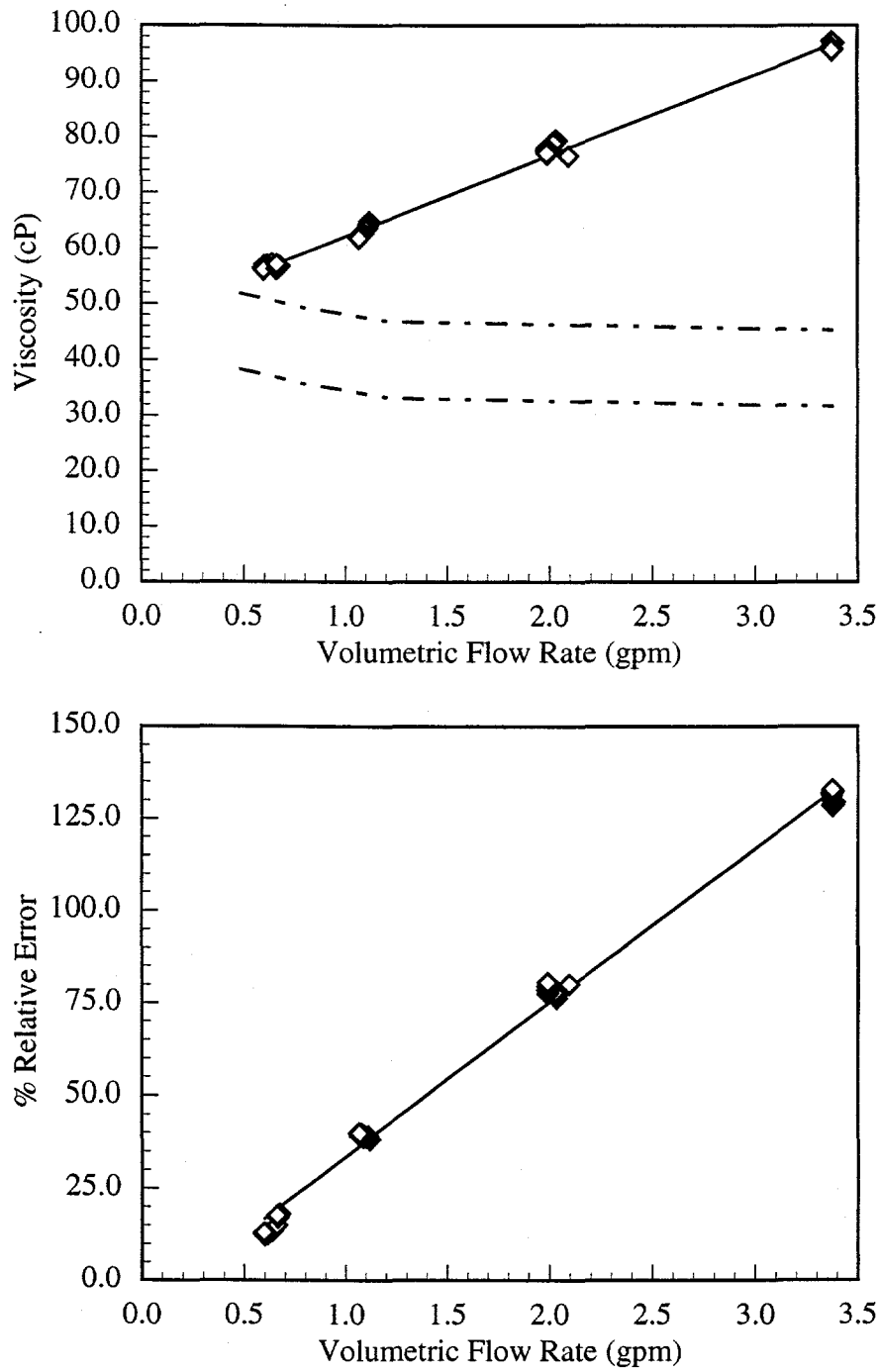


Figure 4.5. Viscosity measured on-line by the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 68.39% solids at approximately 125 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

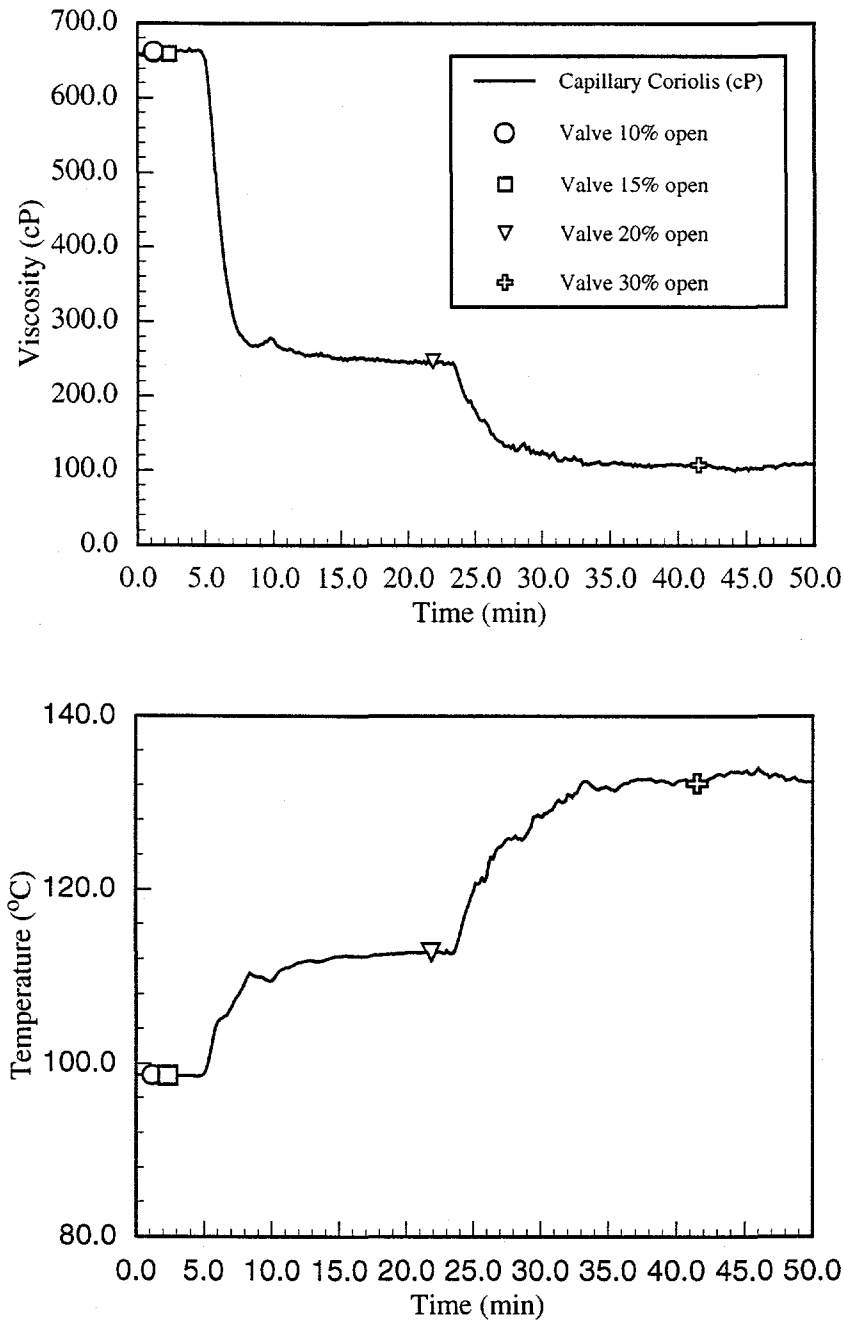
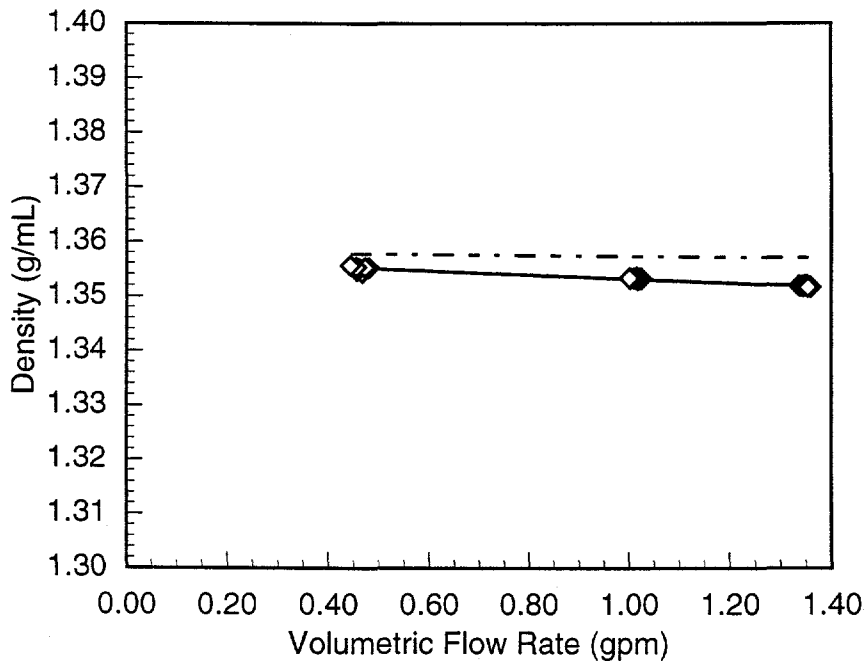


Figure 4.6.. Transient response of the Micro Motion capillary-coriolis viscometer. The upper plot shows the viscosity measured on-line for a mixture of Canadian Forest products and Georgia Pacific liquors with 67.46% solids as the temperature of the liquor is changed by opening the steam valve of the indirect heater. The lower plot shows the temperature of the black liquor as a function of time. The markers appearing in both plots indicate the instants when the steam valve opening is changed in order to cause a temperature increase.



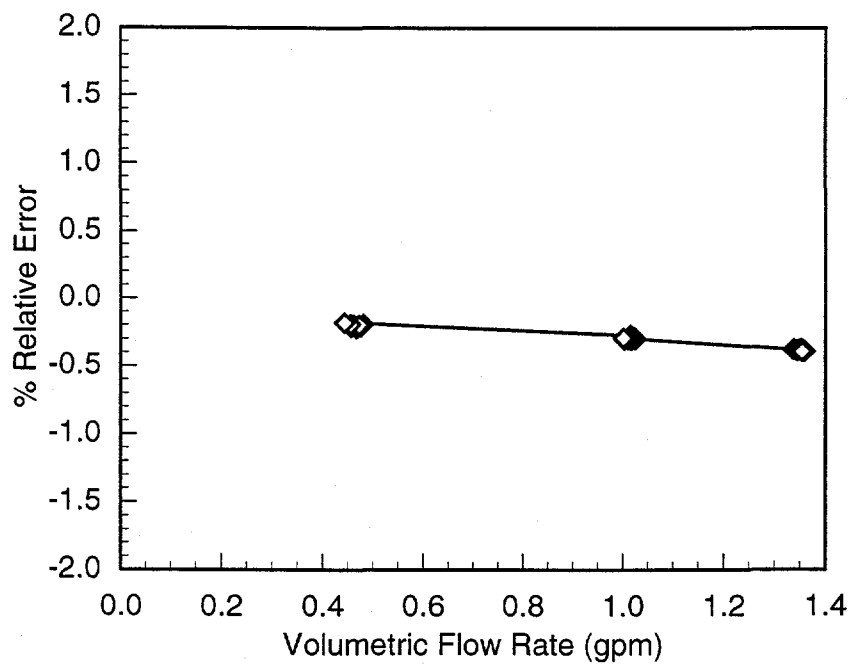


Figure 4.7. Density measured on-line by the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 65.51% solids at approximately 80 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

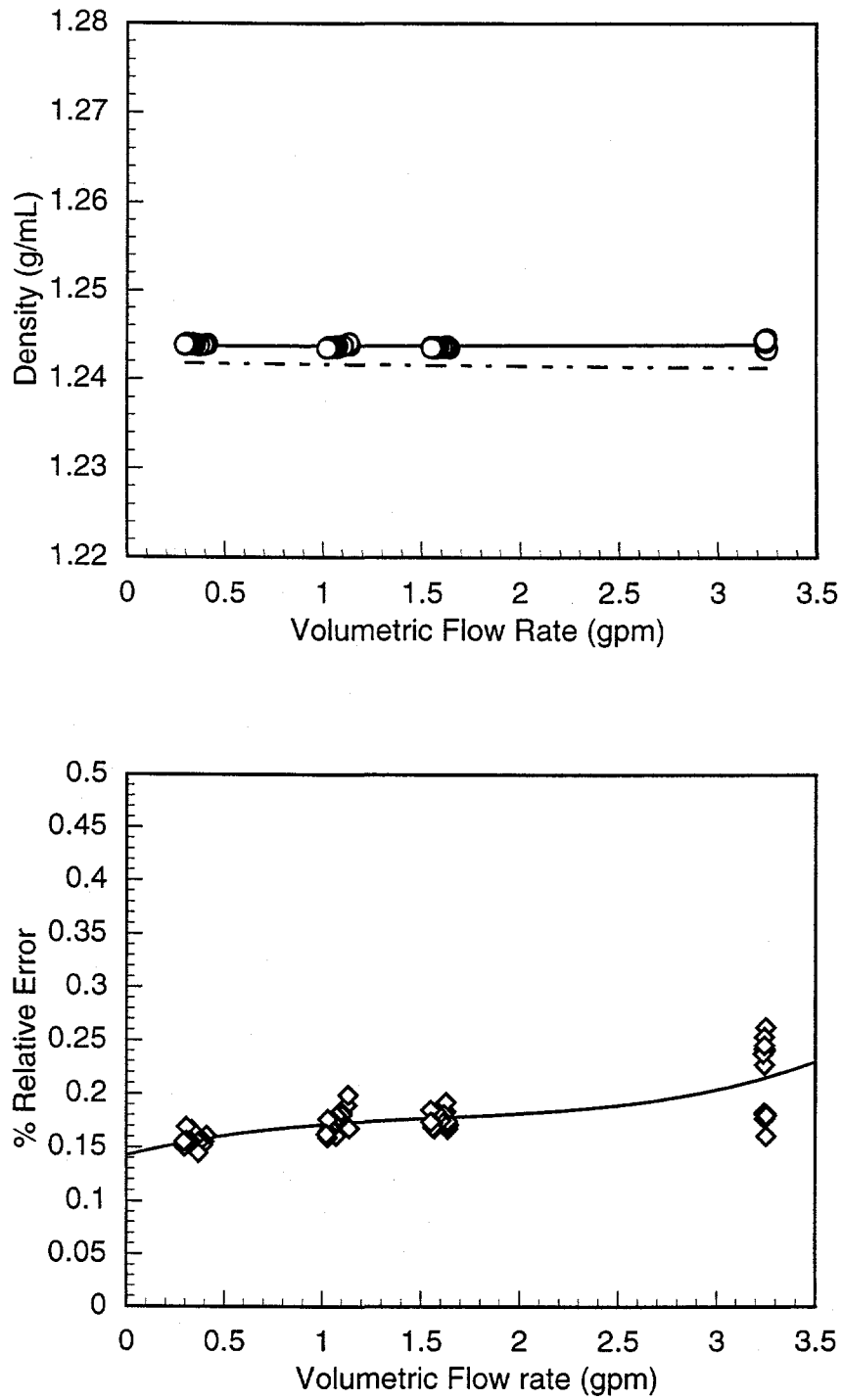


Figure 4.8. Viscosity measured on-line by the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 42.24% solids at approximately 35 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

4.4.3 Summary of Observations for the Pilot Plant Trials

The general trends observed with the on-line capillary-coriolis viscometer tested in the pilot flow loop are summarized as follows:

- The viscometer used in this study tracked reliably within ±10% of the laboratory reference at high viscosities.
- There was no evidence of fouling during the trials
- 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 not exceeding 30 seconds.
- There are no moving parts in the process stream, hence making the instrument more robust.
- In addition to viscosity data, the instrument provides information on mass flow rate, volumetric flow rate, temperature, differential pressure, and density.
- Secondary flows affect the viscosity measurement for low viscosity and high flow-rate systems.
- This instrument requires a one-point calibration with a laboratory reference for Newtonian fluids.

The capillary-coriolis viscometer was used in the pilot flow loop to measure viscosities from 5 cP up to about 1040 cP with acceptable accuracy. The setup in the pilot plant used in this study includes a coriolis mass flow meter with 0.25" flow tubes, and a differential pressure cell with a range of 0-36 psig. The viscosity measurement and the flow-rate range are limited by the pressure drop across the instrument.

In experiments run with low-viscosity liquors, a linear increase of the measured viscosity as a function of flow rate was encountered. We suspect that the secondary flow effects within the coriolis flow tubes causes the erroneous viscosity measurement. At flow rates lower than 0.5 gpm, the instrument measured the viscosity accurately even at low viscosities (80 cP); however, on the test runs with low solids, the instrument may have been inadvertently calibrated under conditions where the secondary flows were significant, instead of the normal conditions where it is normally calibrated in the regime of high viscosity and high pressure drop. We have verified that

the viscosity measurement typically evolves as a linear function of the flow rate whenever the adverse secondary-flow effects are present.

Secondary Flow Effects

The problem of secondary flows arises whenever a fluid passes through a curved section of the pipe. The onset and the effect of secondary flows in curved pipes has received much attention in the fluid mechanics literature due to its importance in engineering and biological systems [Berger *et al..,* 1983]. When a fluid flows through a curved pipe, the fluid at the center moves faster than the fluid at the outer walls. Due to the centrifugal forces, the faster moving fluid at the center is forced outwards, while the slower moving fluid is forced inwards where the pressure is less. This 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

$$\text{Dean number} = \text{Reynolds number} \times \frac{\text{diameter of the pipe}}{\text{radius of curvature}}$$

The Dean number represents the product of the (inertial force)/(viscous force) and (centrifugal force)/(viscous force). Depending on the radius of curvature and the Dean's number, the axial velocity distribution is affected resulting 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 also experimentally verified by flow visualization experiments. The interested reader is referred to Ito [1987], and Cheng *et al..* [1995] and the references therein. 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 attention on the effect of the secondary flow on the viscosity measurement and the possible ways to overcome the adverse effect by empirical fitting of 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 St. Laurent 59.28% solids black liquor at approximately 65°C demonstrated that the viscosity measurement remained constant at 200 cP for flow rates up to 1.8 gpm before the differential pressure cell reached its operational limit. . The upper flow rate limits with black liquor viscosities between 200-300 cP were not reached in the pilot flow loop due to limitations in the differential pressure cell.

Correction by Empirical Correlations

Under conditions of high flow rate and low viscosities, an additional pressure loss is created due to the effect of secondary flows causing 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. Hence, for operation at a particular volumetric flow rate, a correction factor may be applied by comparing with laboratory viscosity. However, it was found that the response to the secondary flows appears 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 in an attempt to provide a correction to the secondary-flow effects:

$$\eta = k\frac{\Delta p}{\left(\dot{m}/\rho\right)^{n}} \qquad (4.6)$$

where n is a correction factor to compensate for secondary-flow effects. The viscosity compensation parameters n and ϕ in (4.6) must be determined experimentally. The original formulation of Equation (4.1) uses $n = 1$ and $k = \phi$, a calibration constant.

An optimization study was carried out, leaving both n and k as the free unknown variables. The optimal compensation parameters obtained are reported in Table 4.3 for two liquors: (i) a Georgia-Pacific liquor with a 65.51% solids content (Figure 4.9), and (ii) a St. Laurent Paper Products liquor with a solids content of 59.28% (Figure 4.10). Note that in Table 4.3 the units of constant k is cP-psig/(gpm)^n.

For the data represented in Figure 4.8, it is obvious that the secondary flow affects the measurement at experiments conducted at flow rates greater than 1 gpm. 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 gpm. Figure 4.9 shows that the compensation parameters of Table 4.3 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 by Figure 4.10, the compensation parameters produce corrections that are within ±5% of the laboratory reference. Based on our experience, the better way to operate is to limit the viscosity measurements to 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 where the effect of secondary flow is appreciable, then the above empirical correction scheme could be employed. We hope that further

analysis of the data gathered at the pilot plant may yield a universal model for the coriolis mass flow sensor used in the study.

Table 4.3. Capillary-coriolis compensation parameters for two black liquors: a Georgia-Pacific liquor with a 65.51% solids content (Figure 4.9), and a St. Laurent Paper Products liquor with a solids content of 59.28% (Figure 4.10)

Parameter	Figure 4.9	Figure 4.10
k	10.05	6.982
n	1.322	1.316

Alternate 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/unit length. The actual design of the coriolis mass flow meter used in the pilot flow-loop has a pair of tubes in the shape of coat hangers. The pressure drop is measured across the coriolis mass flow meter. The fluid passes through as many as eight curved sections between the pressure taps. This causes the secondary flow effects which leads to increased pressure drop, and hence, increased value of the measured viscosity. The easiest way to get rid of this problem is to measure the pressure drop for the fully developed flow through a piece of straight tube either before or after the coriolis mass flow meter. In order ensure the development of a good laminar flow profile, a straight pipe run of at least 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. While this has not been tested in our pilot flow loop, we believe that this is a very promising approach that needs to be studied in detail.

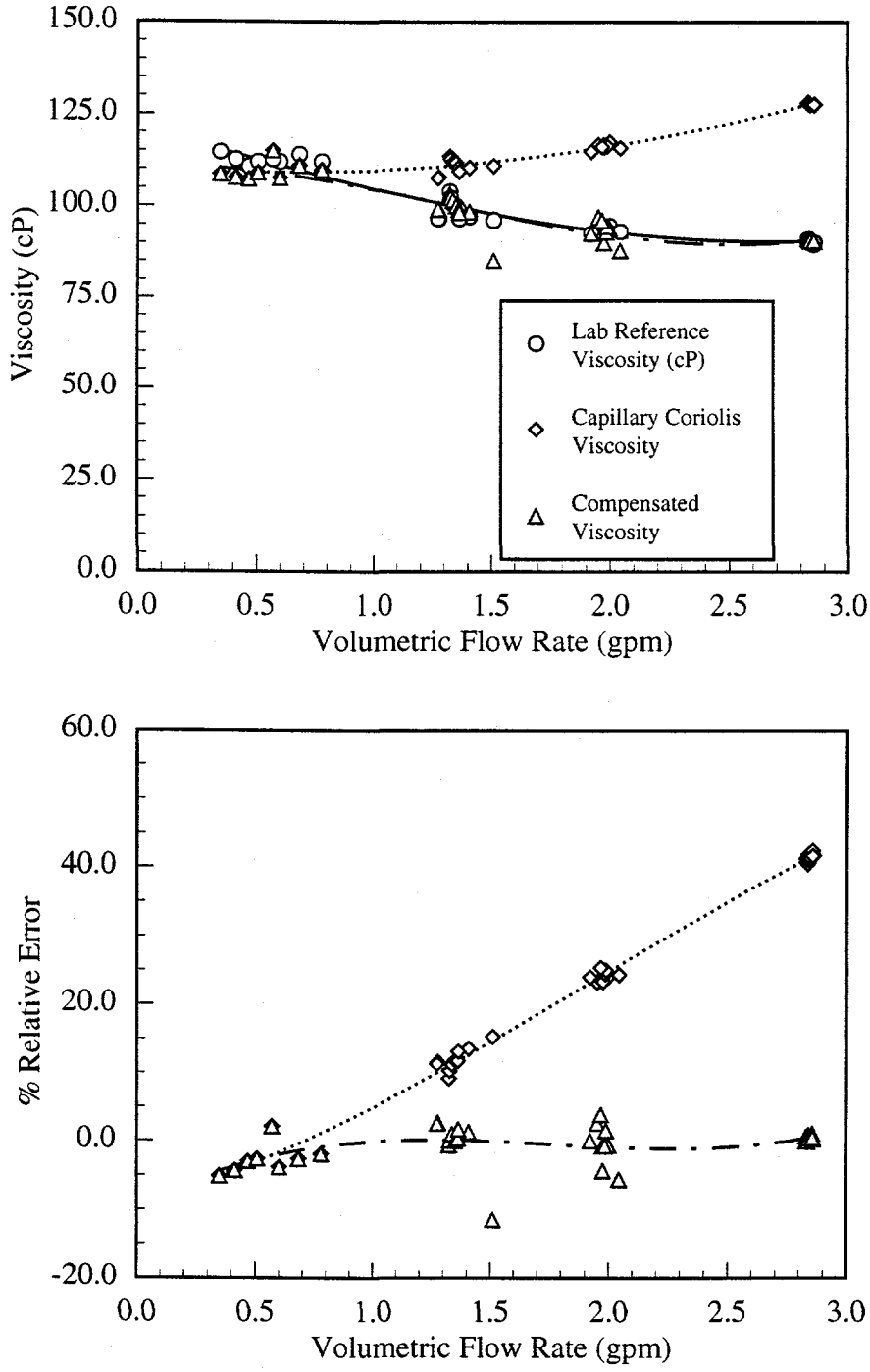


Figure 4.9. On-line viscosity as-measured and after compensation obtained with the Micro Motion capillary-coriolis viscometer for a Georgia Pacific black liquor with 65.51% solids at approximately 128 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

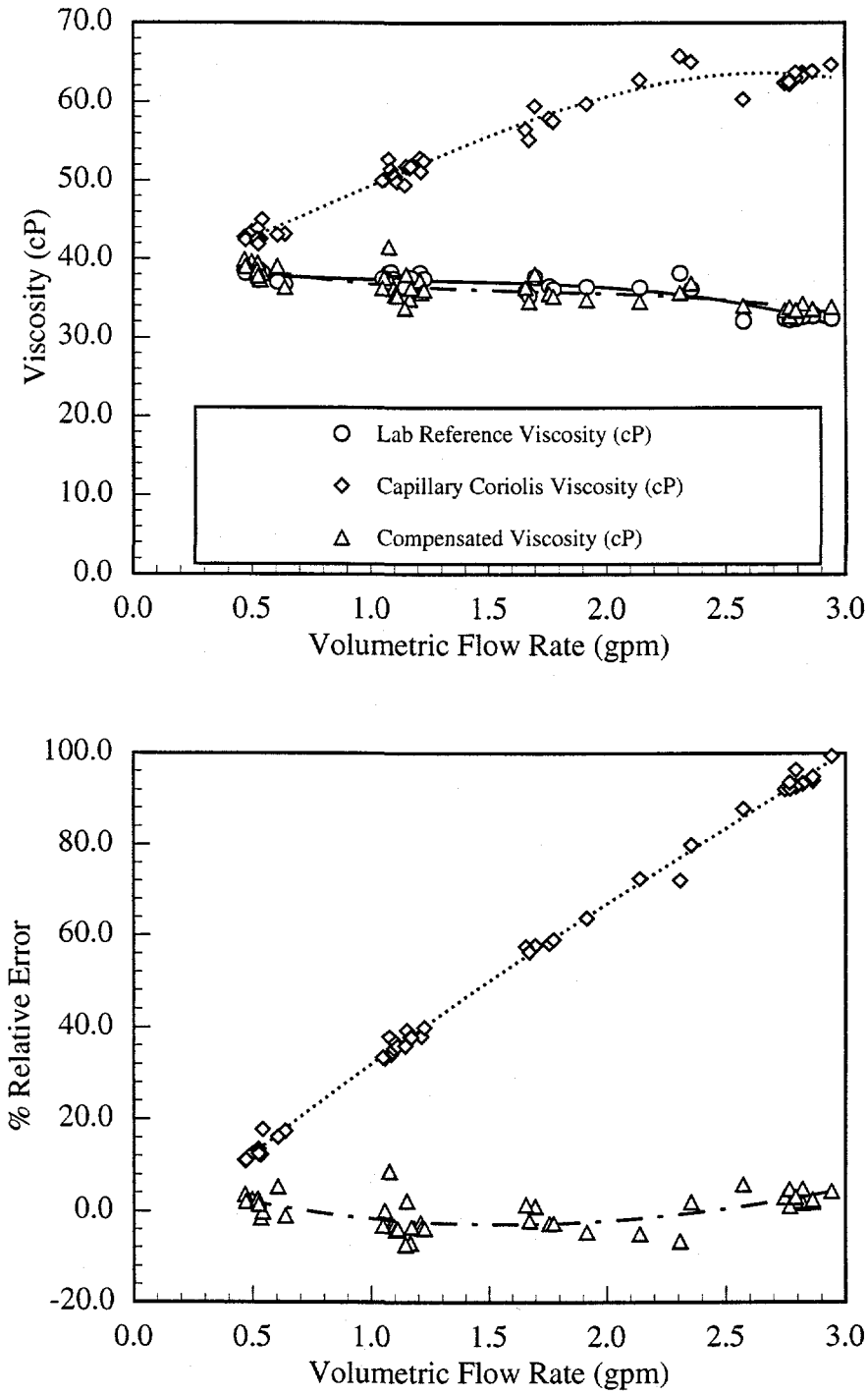


Figure 4.10. On-line viscosity as-measured and after compensation obtained with the Micro Motion capillary-coriolis viscometer for a St. Laurent Paper Products black liquor with 59.28% solids at approximately 75°C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

4.5 Mill Trials at a Canadian Forest Products Site

The Micro Motion capillary-coriolis viscometer was selected for trial at the Canadian Forest Products mill located in Prince George, British Columbia, Canada, a site representative of a northwestern mill. This viscometer was selected for trial at this location because the black liquor at firing conditions in this mill has the highest viscosity of the three mill sites available for the study. Furthermore, the mill wished to eventually operate at even higher viscosities—up to 400 cP. This made the mill an ideal site for the Micro Motion instrument given that the laminar flow conditions required for operating the instrument are most easily met at higher viscosities. Also, the mill personnel were interested in determining on-line the density and the mass flow rate of the black liquor fed into the recovery furnace, making the Micro Motion instrument ideally suited for this application since the device also provides the additional measurements desired. The liaison established between Micro Motion, Canadian Forest Products, and University of Florida personnel was very good. Micro Motion designed and supplied the instrument free of charge and also provided technical and technical support at their own expense, whereas the absorbed all the costs of installing and testing instrument. An initial planning meeting was held at Prince George in the Fall of 1996 where guidelines were set for conducting trials starting on the first quarter of 1997.

4.5.1 Experimental Overview

The viscometer was installed in a 1-inch side stream line from the furnace ring header at the exit of the indirect liquor heater so that viscosity could be measured at the liquor firing conditions. The installation allowed the isolation of the side-stream line for cleaning the pipes without interrupting the furnace operation. The side stream was also piped and valved to isolate the instrument itself for flushing and cleaning or for removing the instrument for maintenance. It was recommended that the side stream line be heat traced and that it include a control valve adjustable from the Micro Motion instrumentation package so that the flow rate through the instrument could be controlled. No screens or strainers were recommended at this the time of the initial installation because the liquor was not expected to contain large suspended particles and the diameter of the instrument tubes was greater than 1/2-inch. The differential pressure cell was installed across the points of flow entrance and exit of the Micro Motion instrument.

A sample of the Canadian Forest Products black liquor was delivered to the University of Florida for analysis prior to the initiation of the trial period. The liquor viscosity was determined as a function of temperature and shear rate and the thermal expansion coefficient was characterized. These results were compared with those obtained for black liquor from the same mill that had been used at the University of Florida pilot plant several months earlier, and it was concluded that the

black liquor characteristics had not changed appreciably. Estimates of the viscosity range expected at the mill were provided to Micro Motion Inc. for the purposes of sizing the instrument.

A plan for evaluation of the instrument was developed with Micro Motion and Canadian Forest Products mill personnel. Arrangements were made for a laboratory viscometer to be transported from the University of Florida to the mill in Prince George, British Columbia, where it would be set up in the mill's laboratory for on-site measurement the black liquor samples. A conventional sampling method that consists of drawing a sample from the line into a container open to the atmosphere was used regularly at the mill. A major drawback of this so-called "open-sampling" method is that volatile elements are lost during sampling due to flashing into the atmosphere. To counter this problem sampling vessels known as "bombs" that avoid the flashing problem during sampling were designed and built by the University of Florida researchers and supplied to the mill. In mid January of 1977 a trained technician from the University of Florida was sent to the mill for a period of two weeks to supervise the sampling operations and to make solids-content and viscosity measurements in the laboratory. Portions of each sample would be packaged and transported to the University of Florida to determine if shipping time affected the results of viscosity measurements.

4.5.2 Discussion of Results

A first instrument trial of the Micro Motion viscometer was conducted at the Canadian Forest Products mill site on January of 1997 and a second trial on November of 1997.

First instrument trial - January 1997

The first mill trial began on January 13, 1997 and total of 35 samples were collected over a period of 10 days. The analysis results are summarized in Tables 4.4a and 4.4b which show the solids contents and the viscosity of each sample. The tables also differentiate between the two sample collection methods used, namely samples labeled as "bomb" which were taken using specially designed closed vessels that prevent flashing during sampling, and samples labeled as "open" which were collected into containers that were open to the atmosphere.

For selected samples the viscosity was determined in the laboratory facilities of the mill and later in the laboratories at the University of Florida. It was found that these values were consistent with each other, and that any differences were primarily attributable to differences in the temperature of measurement. These observations confirmed that the mill has good in-house capability for determining the viscosity of black liquor in a laboratory environment, a necessary capability needed to regularly verify the accuracy of an on-line viscometer.

The solids concentration reported in Tables 4.4a were determined on-line at the mill using two different refractometers and also at the University of Florida laboratories using standard procedures. The values reported in the table suggest that in general, the solids concentration of the bomb samples are in agreement with the refractometer readings while the solids concentrations of the open samples are typically about 2% higher. Since the viscosity of black liquor is such a strong function of the solids contents, this table illustrates the necessity of using a bomb to prevent flashing during the acquisition of samples for laboratory uses.

The viscosity data summarized on Table 4.4a cannot be used for assessing the accuracy of the on-line instrument because throughout the period reported the Pressure drop readings were extremely erratic. Furthermore, after 13 days of operation flow through the line stopped due to pluggage caused by the cooling of the black liquor. The instrument was removed and an inspection revealed that the membranes separating the black liquor from the differential pressure cell fluid had been ruptured due either to corrosion or improper installation, causing the erratic pressure drop readings. The line pluggage in the side-stream was due to the fact that the mill did not follow the recommendation to heat treat the line. The pluggage was due to liquor cooling rather than caused by suspended particles.

Several months elapsed while this problem was discussed. Even though there should not have been a problem with corrosion (especially, in so short a time) and the problem was probably due to improper installation, it was decided that the defective differential pressure cell would be replaced with one with separation membranes made of Hastelloy or an equivalent material. Since these were special-order, there was a long delay before the instrument could be reinstalled for another trial.

Second instrument trial - November 1977

A second trial of the Micro Motion instrument at Canadian Forest Products began in November, 1997. Eleven samples were taken during a twenty-five day trial, and the samples were shipped to the University of Florida for analysis. Table 4.5 summarizes the data recorded by the mill's distributed computer system for all the samples. Note that the solids concentration, as determined from refractometer readings, does not vary much, and the temperature variation range was less than 3°C throughout the testing period. The on-line density measurements made by the Micro Motion sensor appear to be accurate to better than ±0.2%, assuming that both the solids concentration and temperature measurements are absolutely accurate.

Table 4.4a. Samples collected at Canadian Forest Products Limited mill during the first instrument trial (January 1997). The table shows sample-collection information, the firing temperature of liquor at the time of sampling, the method of collection (open atmosphere or closed-bomb container), and the solids contents measured by two on-line refractometers along with that determined at the University of Florida laboratories.

Sample			Firing Temp	Collection Method	Solids Content		
					Refract A	Refract B	UF Lab
ID	Date	Time	°C		%	%	%
CF1131G	01/13/97	15:00	127	open	-	-	77.46
CF1141G	01/14/97	14:15	128	open	-	-	77.08
CF1151B	01/15/97	9:30	129.6	bomb	73.6	74.6	74
CF1151G	01/15/97	9:30	129.6	open	73.6	74.6	75.86
CF1152B	01/15/97	13:30	129	bomb	73.78	74.71	74.11
CF1152G	01/15/97	1330	129	open	73.78	74.71	76.11
CF1153B	01/15/97	15:30	129	bomb	73.72	74.71	73.6
CF1153G	01/15/97	15:30	129	open	73.72	74.71	76.39
CF1161B	01/16/97	09:00	127.8	bomb	74.16	74.8	73.95
CF1161G	01/16/97	09:00	127.8	open	74.16	74.8	76.38
CF1162B	01/16/97	11:00	128	bomb	74.15	75.2	73.79
CF1162G	01/16/97	11:00	128	open	74.15	75.2	76.95
CF1163B	01/16/97	13:00	128.1	bomb	74.16	75.5	73.82
CF1163G	01/16/97	13:00	128.1	open	74.16	75.5	76.9
CF1164B	01/16/97	15:00	127.9	bomb	74.09	75.3	74.01
CF1164G	01/16/97	15:00	127.9	open	74.09	75.3	76.52
CF1201B	01/20/97	10:45	127	bomb	75.1	75.13	-
CF1202B	01/20/97	11:15	127.1	bomb	75.14	75.16	-
CF1203B	01/20/97	12:50	127	bomb	75.03	75.1	-
CF1204B	01/20/97	14:00	126.9	bomb	74.93	75.04	-
CF1205B	01/20/97	14:40	127.5	bomb	74.78	74.91	-
CF1206B	01/20/97	15:20	127.1	bomb	74.92	74.99	-
CF1211B	01/21/97	08:45	126.9	bomb	75.15	75.28	-
CF1212B	01/21/97	09:50	127.5	bomb	75.12	75.24	74.34
CF1213B	01/21/97	10:25	127.7	bomb	75.03	75.22	-
CF1214B	01/21/97	11:15	127.8	bomb	75.12	75.22	-
CF1215B	01/21/97	12:40	127.6	bomb	75.12	75.24	75
CF1216B	01/21/97	13:35	127.6	bomb	75.03	75.2	-
CF1217B	01/21/97	15:00	127.4	bomb	75.07	75.2	-
CF1221B	01/22/97	09:35	127.1	bomb	75.06	75.35	71.82
CF1222B	01/22/97	10:40	127.1	bomb	75.09	75.31	-
CF1223B	01/22/97	11:20	126.9	bomb	75.11	75.35	-
CF1224B	01/22/97	12:50	127.1	bomb	75.09	75.37	-
CF1225B	01/22/97	13:50	127	bomb	75.06	75.36	-
CF1226B	01/22/97	14:50	127	bomb	75.1	75.39	74.59

Table 4.4b. Samples collected at Canadian Forest Products Limited mill during the first instrument trial (January 1997). The table shows sample-collection information, the firing temperature of liquor at the time of sampling, the method of collection (open atmosphere or closed-bomb container), and the viscosity determined on-line with the Micro Motion instrument and that determined at the University of Florida laboratories.

Sample			Firing Temp	Collection Method	Viscosity			
					On-Line	Temp	UF Lab	Temp
ID	Date	Time	°C		cP	°C	cP	°C
CF1131G	01/13/97	15:00	127	open	126.3	132.6	237.8	126.7
CF1141G	01/14/97	14:15	128	open	183.9	129.4	-	-
CF1151B	01/15/97	09:30	129.6	bomb	-	-	-	-
CF1151G	01/15/97	09:30	129.6	open	191.6	129.6	-	-
CF1152B	01/15/97	13:30	129	bomb	95.4	129.1	-	-
CF1152G	01/15/97	1330	129	open	207.4	129	-	-
CF1153B	01/15/97	15:30	129	bomb	109.4	128.6	96.54	128.8
CF1153G	01/15/97	15:30	129	open	150.1	126.3	180.3	129
CF1161B	01/16/97	09:00	127.8	bomb	257.3	127.7	-	-
CF1161G	01/16/97	09:00	127.8	open	107.6	128.4	-	-
CF1162B	01/16/97	11:00	128	bomb	-	-	132.7	128
CF1162G	01/16/97	11:00	128	open	192.6	128.6	220.5	127.9
CF1163B	01/16/97	13:00	128.1	bomb	-	-	-	-
CF1163G	01/16/97	13:00	128.1	open	192.9	128	-	-
CF1164B	01/16/97	15:00	127.9	bomb	112.4	128.5	-	-
CF1164G	01/16/97	15:00	127.9	open	202.4	128.6	-	-
CF1201B	01/20/97	10:45	127	bomb	67.4	128.8	-	-
CF1202B	01/20/97	11:15	127.1	bomb	-	-	122.3	122.6
CF1203B	01/20/97	12:50	127	bomb	-	-	78.6	127.2
CF1204B	01/20/97	14:00	126.9	bomb	-	-	-	-
CF1205B	01/20/97	14:40	127.5	bomb	109.2	129.6	-	-
CF1206B	01/20/97	15:20	127.1	bomb	126.2	127.6	-	-
CF1211B	01/21/97	08:45	126.9	bomb	-	-	-	-
CF1212B	01/21/97	09:50	127.5	bomb	-	-	82.3	127
CF1213B	01/21/97	10:25	127.7	bomb	-	-	132.7	127.5
CF1214B	01/21/97	11:15	127.8	bomb	-	-	118	127.6
CF1215B	01/21/97	12:40	127.6	bomb	-	-	108.9	127.6
CF1216B	01/21/97	13:35	127.6	bomb	-	-	129.7	127.8
CF1217B	01/21/97	15:00	127.4	bomb	-	-	79.4	127.2
CF1221B	01/22/97	09:35	127.1	bomb	-	-	59.5	126.9
CF1222B	01/22/97	10:40	127.1	bomb	-	-	80.1	127.4
CF1223B	01/22/97	11:20	126.9	bomb	-	-	-	-
CF1224B	01/22/97	12:50	127.1	bomb	-	-	70.7	127
CF1225B	01/22/97	13:50	127	bomb	-	-	67.9	126.7
CF1226B	01/22/97	14:50	127	bomb	-	-	112.2	127.2

Table 4.5. Samples collected at Canadian Forest Products Limited mill during the second instrument trial (November 1997). The table shows sample-collection information, the firing temperature of liquor at the time of sampling, the solids-content of the samples ad determined by an on-line refractometer. The three measurements of density, flow rate, and pressure drop (ΔP) produced by the Micro Motion sensor are shown along with the corresponding viscosity determined by the sensor.

Sample			Firing Temp	Solids Content	Measurements			On-Line Viscosity
					Density	Flow rate	ΔP	
ID	Date	Time	°C	mass %	g/ml	lb/min	psi	cP
CF1120	11/20/97	12:43	130.3	75.2	1.3953	230.9	11.56	108.2
CF1121	11/21/97	08:14	130.0	75.1	1.3941	228.3	11.66	109.8
CF1124	11/24/97	08:15	130.9	75.2	1.3935	233.9	11.72	106.8
CF1125	11/25/97	08:32	129.9	75	1.3937	233.4	11.22	102.7
CF1126	11/26/97	08:16	130.3	74.7	1.3929	232.2	11.01	101.9
CF1127	11/27/97	08:10	130.0	75.1	1.3978	229.8	11.19	106.1
CF1128	11/28/97	08:13	128.0	74.9	1.3967	230.2	11.2	104.7
CF1203	12/03/97	08:05	129.4	74.4	1.3967	238.2	10.28	92.2
CF1205	12/05/97	-	-	-	1.3943	-	-	-
CF1210	12/10/97	08:13	129.3	75.5	1.3951	238	11.48	102.7
CF1215	12/15/97	08:23	129.8	74.4	1.3972	234.7	10.74	98.3

At the University of Florida laboratory an analysis was carried to determine the solids concentration was by the modified McDonald's method and to determine the viscosity as a function of shear rate. Table 4.6 shows comparisons of the solid-contents data obtained at the mill, and that obtained at the University of Florida laboratories. The table also shows a comparison of the Micro Motion viscosity data (acquired on-line) and the University of Florida viscosity measurements, along with the percentage difference of mill and lab viscosity data at a shear rate of 500 sec^{-1}. The relative-difference values shown in the last column of the table are calculated using the relationship

$$\text{Relative Difference} = 100\,\frac{\eta - \eta_{ref}}{\eta_{ref}}\ \%$$

where η is the viscosity measured by the on-line Micro Motion sensor, and η_{ref} is the viscosity determined using the instruments available at the University of Florida laboratories.

Table 4.6 shows that there are differences in mill and laboratory solids concentration measurements that in some cases that are rather large. This discrepancy has an impact on the viscosities measured because of the strong dependence of the viscosity on the solids content. The differences in solids is particularly large for the samples labeled CF1120, and CF1210, whose on-line solids contents values are significantly larger than the values obtained at the University of

Florida laboratories. However, the viscosities measured in the laboratory for these samples were also below 100 cP. We know from our pilot flow loop trials that measurements at viscosities below 100 cP can be affected by secondary flow effects; viscosity measurements will have positive errors (*i.e.*, larger values) as we see here. It is obvious that the large positive viscosity difference for the CF1128 sample is due to secondary flow effects present at such low viscosity; therefore, we should ignore this datum. Except for the CF1128 sample and taking into consideration the solids-content differences for samples CF1120 and CF1210, it can be concluded that the relative differences in viscosity lie within ±19.5% with respect to the values determined at the University of Florida laboratories. The four largest positive differences are all for samples whose viscosities lie below 90 cP. The viscosity error can be significantly reduced for viscosities below 90 cP by eliminating secondary flow effects which can be accomplished by installing the differential pressure cell across a straight run of tube rather than across the Micro Motion meter. Furthermore, it is recommended that the differential pressure be measured over a tube with a length-over-ratio value of approximately 50 or higher.

Table 4.6. Samples collected at Canadian Forest Products Limited mill during the second instrument trial (November 1997). The table shows sample-collection information, the firing temperature of liquor at the time of sampling, the solids-content of the samples determined on line via a refractometer and that measured at the University of Florida laboratories. The trailing columns show the viscosity measured on-line by the Micro Motion viscometer along with the viscosity determined at the University of Florida laboratories at four different shear rates. The last column shows the percentage difference between the on-line measurement and the laboratory measurement at 500 sec^{-1}.

Sample ID	Firing Temp	Solids Content		Viscosity					
				On-Line	UF Lab				Difference
		On-Line	UF Lab		400 sec^{-1}	500 sec^{-1}	600 sec^{-1}	680 sec^{-1}	@500sec^{-1}
	°C	mass %	mass %	cP	cP	cP	cP	cP	%
CF1120	130.3	75.2	73.9	108.2	90.5	88.6	86.3	84.1	22.1
CF1121	130	75.1	74.5	109.8	99.3	97.1	94.9	93.1	13.1
CF1124	130.9	75.2	74.7	106.8	121	114.6	108	102.5	-6.8
CF1125	129.9	75	74.2	102.7	129.6	124.1	116.5	109.1	-17.2
CF1126	130.3	74.7	74.9	101.9	113.1	109.5	104.9	100.4	-6.9
CF1127	130	75.1	75.8	106.1	130.8	126.2	120.3	114.6	-15.9
CF1128	128	74.9	72.4	104.7	64	61.7	59.1	56.8	69.7
CF1203	129.4	74.4	74.5	92.2	80	77.0	74.7	73.3	19.7
CF1205	-	-	74.5	-	-	-	-	-	-
CF1210	129.3	75.5	74.0	102.7	85.6	82.6	80.6	79.3	24.3
CF1215	129.8	74.4	74.2	98.3	95.6	92.9	90.5	88.9	5.8

Efforts to coordinate further instrument trials - January 1998 to March 1999

Through continued negotiations additional trials were attempted during 1998. For various reasons, these could not be coordinated to develop comparative mill and laboratory data. In late 1998, the mill began to experience severe problems due to the intermittent plugging of the side stream line and instrument. There was flow stoppage at erratic times. This was traced to plugging of the entire side stream line by large particles of scale originating from the concentrator system. Apparently, scale from the concentrator heating surfaces was breaking loose and was being carried into the liquor lines feeding the furnace. These particles were large enough to rapidly plug the 1-inch side stream line. This was a new and unexpected problem. Such unprecedented large particles had not been observed in any of the earlier samples taken from the mill, which included strong liquors that were further concentrated to solids contents levels above 75% at the University of Florida laboratories and in a pilot in a one square foot wiped-film evaporator. Clearly there had been changes in mill operations in the meantime that increased scaling tendencies of the liquor. It is worth noting that many mills report increases in scaling in evaporators and concentrators over the past several years because of changes in liquor chemistry caused by efforts to decrease certain emissions into the environment.

It was then suggested that a screen or a strainer be installed. The screen suggested was of 6- or 8-mesh cylinder to be installed the point where the side-stream inlet connects with the main liquor line. The screen would be installed with the cylinder axis parallel to the flow in the main liquor feed line so that large particles would be swept by the screen by the flow in the main liquor feed line. Alternatively, it was recommended that a strainer vessel with a 6- or 8-mesh screen be installed in the side stream line. The mill agreed to consider these suggestions, but the tasks could not be done until May, 1999. Therefore, the experiments were "on hold" until that date in order to make another attempt. There was every reason to believe that this trial to start in May, 1999 would be successful. However, the mill did not install the strainers and abandoned the project.

4.5.3 Summary of Observations for the Mill Trials

The mill trials carried out with the Micro Motion viscometer at the Canadian Forest Products succeeded in showing that the instrument can measure the density of the black liquor with good accuracy (±0.2% or better in all cases). The on-line viscosity measurements realized during the trial have a relative accuracy better that ±19.5% under conditions where the differential pressure cell is installed across the meter and the viscosity is greater than 90 cP. At lower viscosities very good accuracy will be attained if the differential pressure cell is installed across a straight segment of pipe upstream or downstream from the instrument.

The reliability of the instrument was not tested beyond a three-week period of operation because the side-stream installation was inappropriate due to a lack of a screen or a strainer to protect the side stream from pluggage. Although the U tubes of the Micro Motion sensor considered were sufficiently large to be immune to plugging by the kinds of particles typically seen in black liquor, at the time of the trial the mill experienced the development and release of an enormous amount of particles from the evaporator and concentrator surfaces that would clog any line of 1-inch diameter. In summary, the pluggage of the side-stream was due to the lack of screening and to the development of an unprecedented amount of solid debris in the liquor, but to no fault of the viscosity sensor itself.

Even though considerable effort was devoted to this mill trial by all parties, only one meaningful set of comparative data taken over a three week period was obtained in nearly two years time. This was very frustrating because the Micro Motion device performed extremely well in the pilot flow loop trials with liquors at high viscosity, and performed well with liquors of lower viscosity (20-100 cP) when the pressure drop measurements were corrected for entry and exit effects and for Coriolis flow effects.

4.6 Specific Conclusions and Recommendations Regarding this Instrument

The testing of the Micro Motion capillary-coriolis viscometer at the University of Florida Pilot Plant proved that under the Micro-Motion capillary-coriolis viscometer can be used to effectively monitor black liquor viscosity on line. It was shown that the equipment operates satisfactorily in laminar flow, under both steady-state and transient conditions, producing very fast, accurate, and reproducible measurements. The viscometer has negligible time delay (less than 30 seconds) and is free from fouling problem. A linear increase of measured viscosity as a function of flow rate was observed with low viscosity liquors due to the adverse effect of secondary flows. An empirical correlation was developed to effectively compensate for these effects and retain accuracy in the viscosity reading. Alternately, to overcome the adverse effects it is suggested that the pressure drop be measured across a straight piece of pipe where the flow is fully developed. Overall, the Micro Motion viscometer performed extremely well in the pilot plant environment.

The mill testing of this instrument was done at the Canadian Forest Products site in Prince George, where data was successfully collected for a period of over three weeks. The instrument produced very accurate density measurements on line. In general, the on-line viscosity measurements taken by the viscometer differed from laboratory measurements by less than ±19.5%, indicating that the instrument is capable of producing good measurements. Efforts to test the reliability and ease of maintenance of the instrument for a period greater than three weeks were

not successful due to mechanical problems with the side-stream installation that were totally independent of the instrument itself. During the testing period the black liquor developed large amounts of suspended solids that would clog any one-inch line without the protection of a screen or strainer. Previous experience with this instrument in pilot plant studies provides strong evidence to expect that the Micro Motion viscometer would operate reliably and without significant maintenance requirements for very long periods of time provided that the side stream is protected from clogging by suspended solid particles.

4.7 Recommended Installation and Deployment Point

We recommend that the Micro Motion instrument be placed in a side stream as shown in Figure 4.11. In general, the instrument should not be exposed to liquors with suspended particles of sizes that would pass a 12-mesh screen. Note that the side-stream features a 12-mesh tubular screen at the entrance, installed in such a way that the cylinder axis is parallel to the flow in the main liquor feed line. This configuration precludes large-diameter particles from entering the side stream while at the same time minimizes the potential for the screen to experience pluggage. The valving configuration should permit back-flushing the screen with back liquor as necessary should pluggage develop. The screen can be mounted in a flanged tee arrangement for ease of removal. Installing the screen downstream from the side-stream entrance point is strongly discouraged because large particles accumulating on the screen may eventually cause a plug that would stop the flow through the line. For cases where the black liquor contains large amounts of suspended solids, such as the liquors encountered at the Canadian Forest Products mill, a screen of mesh 8 or 10 should be used. On the other hand, for black-liquor applications where few particles are present, the viscometer could be used without a screen protection provided that the instrument U tubes are of half-inch size or larger.

In addition to a 12-mesh screen, we also recommend the installation of a strainer at a point upstream from the viscometer, as shown in the figure, to provide ultimate protection for the instrument and ensure long periods of continued operation. Provisions must be made so that the strainer can be flushed or be isolated for cleaning. We do not recommend ignoring the need for the tubular screen and installing only the strainer. While the strainer would protect the instrument from particle-induced damage it would not necessarily prevent line pluggage should the maintenance of the strainer be relaxed at a critical period of operation.

The side-stream line containing the instrument should be piped and valved so that it can be drained and steam-cleaned as needed to remove black-liquor deposits that constrict flow. A conceptual valving configuration is shown in Figure 4.11. We have successfully utilized 1-inch

pipes for the side stream, but larger diameters could also be used.

The side-steam line where the Micro Motion instrument is mounted must have laminar flow. This requires that the entrance valve to the side-stream line be regulated to adjust the flow rate to laminar conditions. It must be noted that even under laminarity conditions the readings are affected by secondary flow effects that take place at the entrance of the viscometer's internal U-shaped tubes; however, if the viscosity is greater than about 90 cP, the secondary flow effects are minor and the differential pressure cell can be mounted across the points where the instrument entrance and exit tubes are connected. If the viscosity is less than about 90 cP, it is preferred to place the differential pressure cell across a straight length of tube located on the side stream at a point either upstream or downstream of the Micro Motion flow instrument. If flow is laminar, the instrument could alternately be mounted in a firing gun line.

The separation membranes of the differential pressure cell can be made of stainless-steel 316 if the black liquor has a low concentration of chlorides. If the chloride concentration is higher (say, in excess of 1.5% by weight), we recommed the use of Hastelloy membranes because under such conditions stainless steel may degrade through stress corrotion or granular cracking.

It is very important to insulate and heat all the side-stream lines as well as the viscometer itself. This precaution is of critical importance for two main reasons. First, the viscosity of black liquor is extremely sensitive to temperature variations; therefore, heating and insulation precautions must be taken to guarantee that the temperature of the black liquor that reaches the instrument is identical to that of the liquor flowing in the main line. The second reason is that under lack of heating and insulation the black liquor will cool down inside the side stream pipe, eventually causing pluggage of the line, and possibly also causing damage to the delicate internal mechanics of the instrument. The heating of the side-stream lines can be done using a steam-tracing piping network consisting of copper tubes wrapped around the pipes, or by installing an electrical heating tape which can be connected to an inexpensive temperature controller.

The Micro Motion instrument with a differential pressure cell is considerably less expensive than the Nametre instrument discussed in Section 3 of this report. It has the advantages of measuring absolute viscosity and liquor density. The viscosity of non-Newtonian fluids can be determined with this instrument by changing the flow rate into the side stream with a control valve so that different shear rates are established while keeping the laminar nature of the flow.

A potential concern with the Micro Motion instrument is that it requires a significant pressure drop across the differential pressure cell while at the same time keeping a laminar flow profile. These conditions are more easily met with liquids of high viscosities. For accurate measurement of

viscosity we recommend that the differential pressure cell be installed in the side stream across a straight segment of pipe with a length-over-ratio value of approximately 50 or higher.

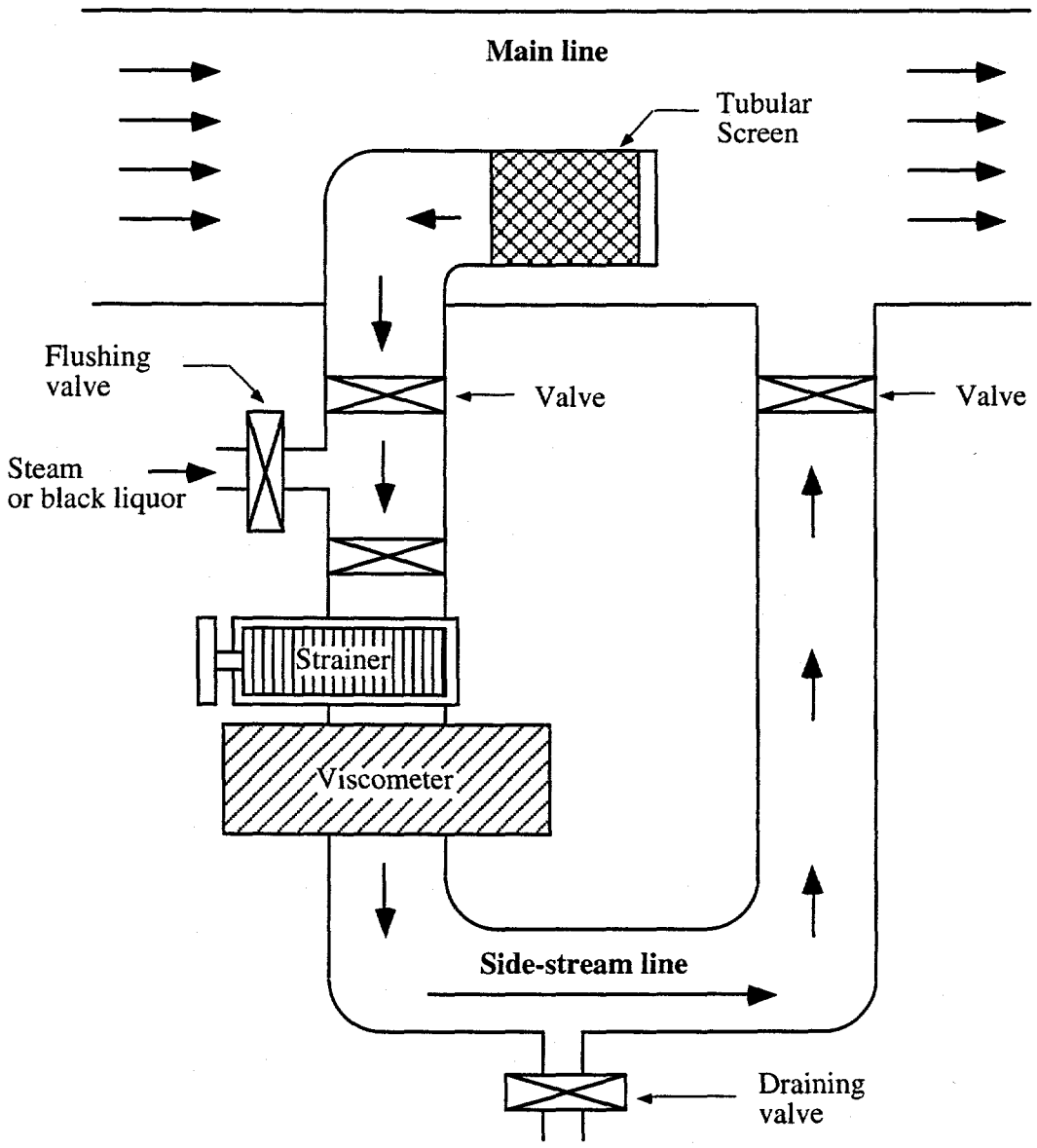


Figure 4.11. Schematic of the recommended installation of the viscometer in a side-stream line. The differential pressure cell can be installed on the side-stream line across the instrument, or elsewhere on a straight-line segment of the side-stream pipe. All the side-stream pipes and the viscometer itself should be heated (using steam-tracing tubes or electrical heating tape) and insulated. The inlet flow should be adjusted with a control valve in order to ensure laminar flow through the instrument.

5. EVALUATION OF BROOKFIELD'S ON-LINE CYLINDRICAL ROTARY VISCOMETER

5.1 Abstract

A cylindrical rotary viscometer manufactured by Brookfield Laboratories was evaluated as an on-line sensor for kraft black liquor in a Pilot Plant environment at the University of Florida and at the site of the Georgia Pacific mill in Palatka, Florida.

Testing at the University of Florida Pilot Plant was done using a piping loop where black liquor is allowed to circulate under closely controlled solids concentration, temperature, and flow rate. Measurements were made under various flow conditions for liquors at concentrations ranging from 42 to 73% solids and temperatures up to 140°C. The accuracy and precision for the instrument were determined by comparison with laboratory measurements of viscosity. In general, the viscometer tracked reliably within ±10% of laboratory reference viscosity for a wide range of operating conditions. The sensor operated satisfactorily under both steady-state and transient conditions. The instrument transient response for step changes in temperature was rapid, with a lag of approximately 45 seconds. The sensor did not experience fouling when tested with liquors over a wide range of solids contents. The instrument can be used to measure the viscosity of non-Newtonian liquors. For high viscosity liquors, a downward trend in viscosity measurement was observed at high flow rates. Hence, it is necessary to regulate the liquor flow rate for improved accuracy.

Testing at a Georgia Pacific mill was done by installing the Brookfield cylindrical rotary viscometer in a side stream that returned the measured liquor to a firing-liquor tank. Three trials were conducted over period of 18 months, but the data collected failed to prove conclusively that the instrument was as reliable and accurate as it was found to be in the pilot-plant test. The identified causes for the instrument failures included incorrect instrument calibration and improper installation procedures (incorrect selection of size for the on-line screen and lack of heating and insulation of the side-stream pipes). In spite of the lack of conclusive evidence to support the accuracy and reliability of the instrument in a mill environment, it is believed this on-line viscometer can operate reliably and with acceptable accuracy in applications where the black liquor is relatively free from large suspended particles provided that a screen of mesh size 8 or 12 is installed and that the instrument is sized so that its measurements are taken at torque values that are 10% or greater than the maximum torque defined in the calibration range.

5.2 Viscometer Description and Principles of Operation

In cylindrical rotary viscometers, the fluid being measured is sheared in the annulus between two concentric cylinders. In the viscometer used in our study, the outer cylinder is rotated at a fixed angular velocity and the inner cylinder is held stationary. In general, it is possible to have other configurations such as the inner cylinder rotating, while holding the outer cylinder stationary, and so forth. The mathematical analysis of these alternative configurations is similar. However, there is one crucial difference. The case where the outer cylinder rotates and inner cylinder is held stationary is immune to hydrodynamic instability because the laminar flow in this system is strongly stabilized by the centrifugal forces [Bird *et al.,* 1960].

In the on-line configuration tested in the pilot flow loop (see Figure 5.1), some of the fluid entering the viscometer is drawn into the measuring annulus between the stationary cylindrical stator and the cylindrical rotor. The resulting fluid motion has an axial component in addition to the dominating rotational component caused by the rotating outer cylinder. This type of fluid motion is generally referred to as 'spiral flow' and the hydrodynamic stability of such a flow has received much attention in the literature from both the theoretical and the experimental points of view [see Donnelly and Fultz, 1960; Chandrasekhar, 1961; Chung and Astill, 1977]. Fortunately, just as in the case of pure rotational flow, the case of outer cylinder rotating while the cylinder is held stationary, is free from flow instability. At low inlet flow rates the axial component of motion in the measuring annulus can be ignored, and it can be assumed that the measured torque is essentially due to the rotational motion. In the following theoretical analysis such an assumption is made. Of course, at high flow rates the assumption will be flawed, as secondary flow can develop and affect the torque measurement. The development of secondary flows is dependent on the design of the instrument, the liquor flow rate and the viscosity of the liquor.

Consider the case where the outer cylinder of radius R_o is rotated at a measured angular velocity ω, and the stationary inner cylinder of radius R_i is suspended by a torsion wire. The viscous drag on the stator induces a deflection of the torsion wire. The viscosity is calculated from the measured torque and the angular velocity. Given shear stress at the wall and the shear rate, the viscosity can be calculated by

$$\eta(\dot{\gamma}) = \frac{\tau}{\dot{\gamma}} \tag{5.1}$$

where η is the viscosity of the fluid τ is the shear stress at the stator wall $\dot{\gamma}$ is the shear rate.

The following assumptions are used in the mathematical analysis of the concentric cylinder

system: (5.1) steady-state laminar flow, (5.2) incompressible fluid in the gap, (5.3) isothermal system, (5.4) negligible inertial effects, and (5.5) no-slip condition at the cylinder walls. With these assumptions, the equation of continuity and motion can be solved to yield the fundamental equations of the system [Bird *et al.,* 1977]. From the measured torque, the shear stress at the surface of the stator is given by

$$\tau = \frac{T}{2\pi R_o^2 L_e} \tag{5.2}$$

where T is the torque and L_e is the equivalent length of the stator.

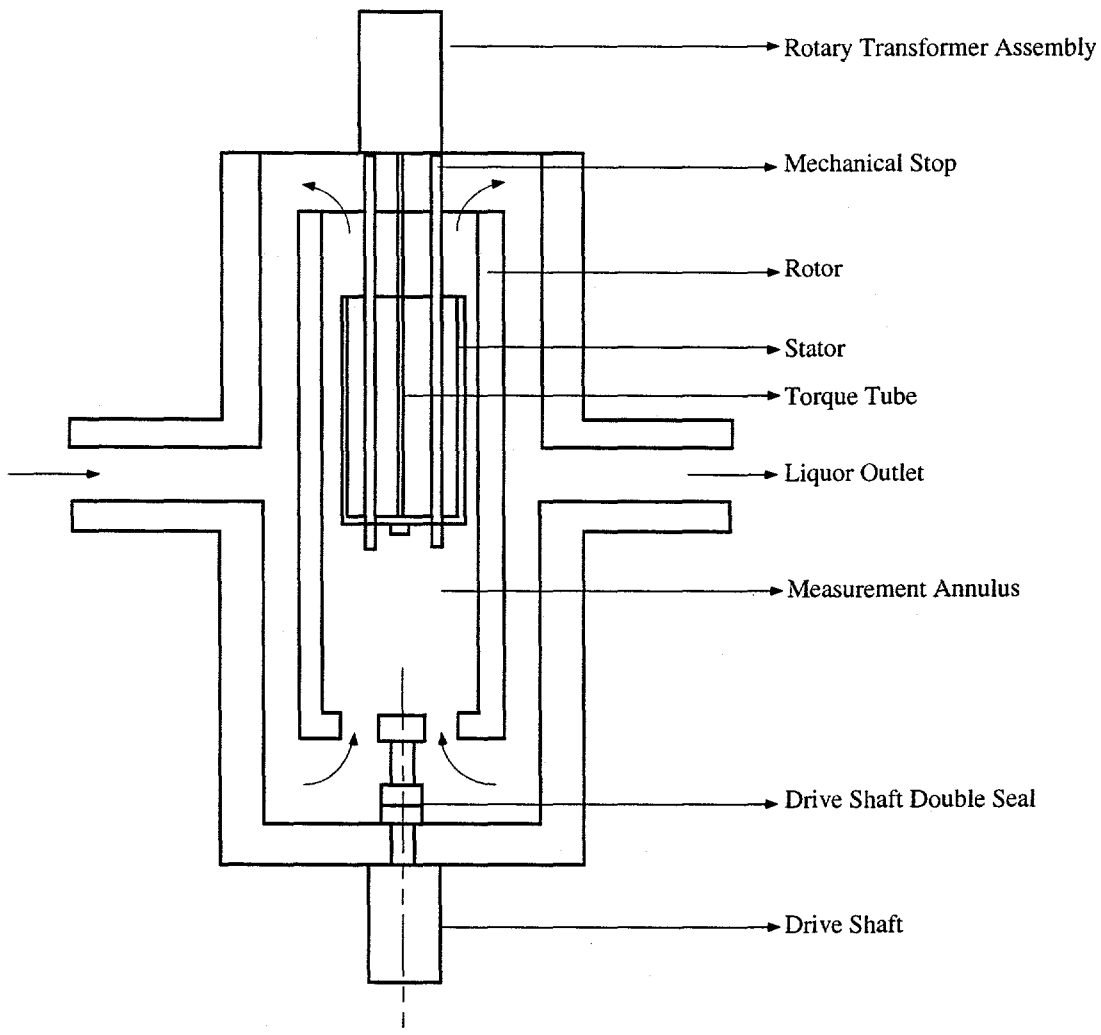


Figure 5.1. Schematic diagram of the Brookfield cylindrical-rotary viscometer evaluated in the University of Florida pilot plant.

The factor L_e in equation (5.2) accounts for end effects in the flow field and can be determined experimentally using standard fluids of known viscosity. The calculation of shear rate is given by [see Skelland, 1967, for a detailed analysis]

$$\dot{\gamma} = \frac{4\pi N}{1 - S^{-2}} C_F \tag{5.3}$$

where

$$C_F = 1 + \frac{S^2 - 1}{2S^2}\left(1 + \frac{2}{3}\ln S\right)\left(\frac{1}{n''} - 1\right) + \frac{S^2 - 1}{6S^2}\ln S\left[\left(\frac{1}{n''} - 1\right)^2 + \frac{d\left(\frac{1}{n''} - 1\right)}{d(\log T)}\right] \tag{5.4}$$

where C_F is the correction factor, N is the rotational speed in rpm, $S = R_O/R_i$ is the ratio of outer-cylinder radius to inner-cylinder radius, and n'' is the slope of the logarithmic plot of torque versus rotational speed.

Once the shear stress and the shear rate are determined, the viscosity can be found from (5.1)-(5.3). Note that for a Newtonian fluid, since the torque is proportional to the rotational speed, $n'' = 1$, and hence $C_F = 1$.

5.3 Viscometer Configuration and Installation Details

The on-line coaxial cylindrical rotary viscometer tested is a TT100SD Rotational Viscometer along with a companion control unit (64-C0795 Rev A) manufactured by Brookfield Engineering Laboratories, Inc. The physical location of this viscometer in the pilot flow loop is indicated by the tag VIS 3 in the process and instrumentation diagram given in Figure 8.1.1 of Appendix 8.1. The basic idea of the design is the use of a rotating-cup configuration [Brookfield, 1997]. In operation, the process stream flows into a sample chamber where it impinges on the rotor (*cf.* Figure 5.1). Due to the force created by the rotor, the fluid sample is drawn into the measuring annulus. The viscous drag of the process fluid on the stator is resisted by the torque tube, which transmits an angular deflection to the rotary transformer. A mechanical stop limits the maximum deflection of the torque tube. The deflection signal is then processed into a 4-20 mA current that is proportional to the torque.

The control unit for the Brookfield cylindrical-rotary viscometer provides two output signals, a linear 4-20 mA torque signal and a non-linear 0-30 Vac signal that quantifies the rotational speed of the rotor. The 4-20 mA torque signal is continuously measured by the DAQ system, whereas the 0-30 Vac signal is noted and the value is manually input to the software program that calculates viscosity. When the rotational speed is changed, this introduces a one time operational time delay

of approximately 30 seconds. The instrument has a dual-range torque capability for improved response in the low viscosity range.

The instrument torque can be calibrated in the field by applying a known torque to the stator by installation of a calibration bar. The factory calibration was done at 300 rpm. The following equation relates the calibration bar rating to the full scale viscosity range at 300 RPM [Brookfield, 1997]:

$$\underset{(@\ 300\ \text{rpm})}{\text{Full - Scale Range}} = \frac{100\ \text{rpm}}{300\ \text{rpm}} \times G_F \times \frac{\text{Calibration Bar Rating} \times 100}{(\text{Full - Scale Reading} - \text{Zero Reference})} \tag{5.4}$$

For the cylindrical-rotary viscometer used in the flow loop, the G_F is 1.1 and the torque signal range is 4 to 20 mA. The full scale viscosity range at any other RPM is given by

$$\underset{(@\ \text{operating rpm})}{\text{Full - Scale Range}} = \underset{(@\ 300\ \text{rpm})}{\text{Full - Scale Range}} \times \frac{300}{N} \tag{5.5}$$

A series of calibration bars are available from the manufacturer, and the instrument can be field calibrated to cover a wide range of viscosities.

Before the start of the experiments in the pilot flow loop, a field calibration was performed to relate the rotational speed measured with a stroboscope to the rotational-speed voltage output from the instrument yielding the correlation

$$N = 2.1591V + 0.0401V^2 \tag{5.6}$$

where N is the rotational speed measured [rpm] and V is the measured voltage [V]. The viscosity at any operating RPM is given by

$$\eta = \frac{(\text{Torque reading} - 4)}{(20 - 4)} \times \text{Full Scale Range}(@\ \text{operating rpm}) \tag{5.7}$$

Substituting (5.4) and (5.5) in (5.7) yields

$$\eta = \frac{300}{(2.1591V + 0.0401V^2)} \times \frac{(\text{Torque reading} - 4)}{(20 - 4)} \times \text{Full Scale Range}(@300\text{rpm}) \tag{5.8}$$

It should be noted that the above calculations for viscosity are valid only for the Newtonian fluids. Since the black liquors tested in the pilot flow loop were Newtonian, the above formula proved to be sufficient for our study. This instrument can also be used to study non-Newtonian

viscosity behavior. However, the calculation procedure is rather elaborate and must proceed as given in the previous section. By measuring the torque signal, the shear stress at the wall can be estimated from equation (5.2). After corrections to the apparent shear rate [Skelland, 1967], equation (5.1) can be used to get non-Newtonian viscosity at a particular shear rate. By varying the rotational speed, and hence the shear rate, the non-Newtonian viscosity as a function of shear rate can be measured. With on-line data acquisition, this can also be used to estimate on-line, the parameters of an appropriate constitutive equation (for example, the power law model, the Carreau-Yasuda model, *etc.*, that can be used for control purposes [see Bird *et al.*, 1987].

5.4 Pilot Plant Trials at the University of Florida

5.4.1 Experimental Overview

Black liquor was obtained from three kraft mills, namely, Georgia Pacific Corporation at Palatka, Florida; Canadian Forest Products Limited at British Columbia, Canada; and St. Laurent Paper Products Corporation at West Point, Virginia, as being respectively representative of southern softwood, western softwood, and mid-Atlantic mixed-species pulping. These 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 5.1 lists the solids composition of the black liquors used in the experimental runs. The on-line viscometer was tested on 13 different black liquor concentrations, ranging from a 42.24% Georgia Pacific liquor to a 73.40% liquor obtained by mixing a Georgia Pacific Corporation liquor with a Canadian Forest Products Limited liquor (*cf.* Table 5.1).

The Georgia Pacific liquor was the easiest to handle because of its low level of air entrainment and ease of evaporation; hence, more experiments were performed with this black liquor. The Canadian Forest and St. Laurent black liquors formed crystalline particles in the storage tank at intermediate (53-60%) solids concentrations when used for several days as was required for the experiments. 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 the thermal and physical homogeneity of black liquor. To collect samples for laboratory characterization, the flow through the sample loop and main loop was stopped briefly and a 1 liter sample was withdrawn. Samples were collected from both the main loop and the sample loop simultaneously, and were compared to verify sample integrity. The flow was

stopped to ensure that flashing does not occur. Flashing destroys the sample integrity, making the liquor appear more concentrated than it actually is. A discrepancy of up to 2% in solids content has been observed due to flashing if this precaution is not taken.

Extensive laboratory characterizations were done on the samples drawn from the pilot flow loop. The laboratory analysis included the measurement and generation of correlations for viscosity, density, heat capacity, solids content, and boiling point elevation. This data provided complete physical information about the black liquor in the loop, and served as a reference basis for evaluating the performance of the on-line viscometer. The specific configuration of the Brookfield cylindrical-rotary viscometer used in our study did not have an internal RTD. Hence, measurements from an upstream RTD labeled TT 7 in Figure 8.1.1 (see Appendix 8.1) was used as the reference temperature.

Experiments were run in the pilot flow loop at different temperatures for each of the concentrations listed in Table 5.1. The highest temperature reached in the main tank was limited to 10-20°C below the boiling point of the black liquor. This was done 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. After passing through the sample loop, the liquor temperature was reduced to that of the main-tank temperature by the cooler, and was then returned to the main stream. For each concentration-temperature combination, data were collected at different flow rates, including the static condition of no flow. The upper limit of the flow rate for the experiments is dictated by the output of the main progressive cavity pump at its maximum operational limit of 150 psig. However, the main line pressure was usually kept at no more than 70 psig for safety reasons. This pressure upper bound in turn limited the maximum possible flow rates obtainable for highly concentrated liquors.

Table 5.1. Black liquor types and solids concentrations tested with the cylindrical rotary viscometer.

Georgia Pacific Corporation	Canadian Forest Products Limited	St. Laurent Paper Products Corporation	Georgia Pacific Corporation & Canadian Forest Products Limited Mixture
42.24%	47.96%	49.39%	67.72%
48.29%	57.73%	59.58%	71.38%
55.84%			73.40%
58.38%			
65.51%			
68.38%			

In addition to temperature and flow rate, the rotational speed of the Brookfield cylindrical-rotary viscometer was also investigated as an experimental variable. Normally, for each flow rate, three different rotational speeds were run. In some cases, the maximum attainable rotational speed was limited by the viscosity range of the instrument, especially at high viscosities. Due to the drift in the rotational-speed voltage, very low rotational speeds introduced large offsets in voltage and adversely affected accuracy; therefore, for a voltage range of 0-30 Vac, the minimum rotational speed examined was 50 rpm (or 5.4 Vac). The viscometer was factory-calibrated at 300 rpm and the high and low ranges were 0-15.57 cP and 0-70.23 cP respectively. Since the measured viscosity is linearly proportional to the angular defection of the torque element, the high range could be expanded by operating at lower rpm. For example, at 50 rpm the high range becomes 0-421 cP (*i.e.*, 0 - 70.23 x 300/50). For experimental runs with high-solids black liquor, the instrument was modified by changing the gear ratio from 9:1 to 18:1, which allowed considerably lower rotational speeds (for example, 18 rpm) without affecting accuracy. With this modification, liquor viscosities as high as 1040 cP were measured in the pilot flow loop.

5.4.2 Discussion of Results

This section presents typical results obtained with the cylindrical rotary viscometer. Extensive results are given in the Phase IIA DOE report on this research [Fricke and Crisalle, 1996]. The results include characterizations of viscosity versus flow rate at different temperatures, solids compositions, rotational speeds, and a study of the transient response of the instrument to temperature changes. The transient response study is very important to determine the feasibility of liquor viscosity control based on this instrument.

Figure 5.2 shows a low viscosity run at 300 rpm with Georgia Pacific black liquor with a 58.38% solids content at approximately 118°C. The viscosity measurement at each flow rate was taken after steady state had been established. The diamond-shaped markers in the upper plot in Figure 5.2 show the viscosity values measured by the instrument. It should be noted that the continuous line joining the markers is included as a visual aid to track the data trend, and is obtained through an arbitrary polynomial fit. The upper plot also shows a laboratory reference band of viscosity values defined by two dash-dot lines. Two to four different laboratory viscometers were used to determine the viscosity as a function of temperature of 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 viscometer for laboratory measurement. The upper and lower band edges respectively correspond to the highest and the lowest viscosity predictions produced by all the laboratory instruments. Any measurements that fall inside the reference band are considered to be consistent with the laboratory

measurements, and hence are deemed accurate. One should note that in Figure 5.2 the width of the reference band approximately corresponds to a ± 10% deviation from the mean viscosity measured by all the laboratory instruments.

The lower plot in Figure 5.2 is a measure of the percentage relative error between the viscosity measurement (η) produced by the on-line instrument and the viscosity (η_{ref}) predicted by a correlation generated from a reliable laboratory instrument, *i.e.*:

$$\% \text{ Relative Error} = \frac{\eta - \eta_{ref}}{\eta_{ref}} \times 100 \tag{5.9}$$

We consider an on-line instrument to be accurate if the relative error is ±10% or less.

For this particular liquor at 58.38% solids concentration, the laboratory reference viscosity was determined from a correlation

$$\eta_{ref} = \exp\left(A - \frac{B}{(T+273.16)} + \frac{C}{(T+273.16)^2}\right) \tag{5.11}$$

where A = 24.6362, B = -20322.9, C = 4594277, and where T is the temperature (°C) and η_{ref} is the laboratory-determined reference viscosity (cP).

A relevant remark concerning Figure 5.2 is that the temperature is not strictly constant during the run; in fact, as the flow rate varies, normally the temperature also varies. The observed temperature fluctuations were typically no greater than 1°C (with the exception of data taken at zero flow rate where the temperature was approximately 10°C lower, as shown in Table 5.2). However, since the viscosity of black liquor is temperature-sensitive, such small variations must be taken into account for accurate work. Table 5.2 gives the viscosity, and temperature data for 10 selected flow rates plotted on Figure 5.2.

Analysis of Figure 5.2 shows that the Brookfield cylindrical-rotary viscometer responds with good accuracy, since the measurements lie within the laboratory reference band. The percentage relative error during the run is better than -10%. Furthermore, the figure shows that the measurements were unaffected by flow rates up to 1.2 gpm, since the data follow the trend defined by the edges of the laboratory reference band (which in turn is affected only by the temperature). Note that the instrument tracks the changes in viscosity that occur due to the temperature changes that take place at different flow rates. It should be noted that the maximum flow rate for this run was 1.15 gpm.

Table 5.2. Flow-rate, temperature, and viscosity data for Figure 5.2.

Flow Rate (gpm)	Temperature (°C)	Viscosity (cP)
0.00	108.46	20.2
0.00	108.36	20.4
0.74	119.56	14.75
0.79	118.08	14.25
0.80	118.17	14.18
0.81	118.08	14.26
0.88	118.45	13.99
0.98	117.62	15.02
1.05	117.89	14.76
1.06	118.82	14.78

Figure 5.3 is a mid-range viscosity run at 50 rpm with Georgia Pacific 65.51% solids black liquor at approximately 95°C. The laboratory data predict a viscosity of 100-120 cP for the temperature range encountered in this run. The instrument again tracked within the laboratory reference bounds with a ±10% relative difference from the laboratory results, except at the highest flow rate (2.4 gpm) where the relative error was close to -14%. The instrument demonstrated a slight downward trend in the viscosity measurement at the highest flow rate considered.

Figure 5.4 is a high-viscosity run at 50 rpm with Georgia Pacific 65.51% solids black liquor at approximately 88°C. With viscosity readings in the range of 168-200 cP, the instrument tracked the changes indicated by the laboratory reference band (upper plot), achieving relative errors of less than +10% or better (lower plot). A slight downward trend in the viscosity measurement with increasing flow rate is detectable.

Figure 5.5 shows the results obtained for a Georgia Pacific 68.38% solids black liquor at approximately 95°C and at a rotational speed of 100 rpm. For this run, the flow rates were varied from a static-flow condition to 2.25 gpm. At the static flow condition and at low flow rates, the Brookfield cylindrical-rotary viscometer tracked the changes indicated by the laboratory reference band, achieving relative errors that are better than ± 10% for most readings. However, as the flow rate was increased, a clear downward trend in the measured viscosity was visible. The development of a cross flow across the stator was suspected to be the cause for a reduced torque measurement, and hence, lower viscosity readings. As the black liquor enters the measuring cell, the flow is diverted from the stator and torque tube by the spinning rotor (see Figure 5.1). Under low flow conditions, the annulus has essentially static flow and the measurement is made at drag flow conditions. However, as the flow rate increases, some axial flow develops inside the annulus which reduces the torque generated by the viscous drag of the spinning rotor. Such an effect is expected to be more severe in the case of higher viscosity black liquors.

Figure 5.6 shows the results obtained for the same liquor as depicted in the previous figure at an elevated temperature of approximately 110°C (and hence at a lower viscosity range) and at a rotational speed of 300 rpm. For this run, the flow rates were varied from 0.5 to 3 gpm. The instrument readings were relatively unaffected by the increasing flow rates. The instrument provided viscosity readings in the range of 55-68 cP, and tracked the changes indicated by the laboratory reference band (upper plot), achieving relative errors that are less than ± 8% for most readings (lower plot).

Figure 5.7 shows the transient response study of the cylindrical rotary viscometer for step changes in temperature at a rotational speed of 50 rpm. The liquor used was a mixture of Georgia Pacific and Canadian Forest Products black liquors with a resulting solids composition of 67.46%. The upper plot in Figure 5.7 shows the viscosity as a function of time. The continuous line represents the measurement produced by the on-line instrument. The lower plot in Figure 5.7 shows the temperature history during the run. In this experiment the temperature of the flowing black liquor steam was changed in a step fashion by opening the steam control valve (valve labeled as V57 in the process and instrumentation diagram given in Figure 8.1.1 of the Appendix) 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) on the figure denote the instants when the changes were made.

At time zero the temperature in the sample loop was maintained at approximately 96°C and the system was at steady state. The corresponding steady-state viscosity was above the measurement range of this instrument since, at the rotational speed used in this run (50 rpm), the highest viscosity that can be measured is 421 cP. Hence, until the viscosity falls to this level due to temperature raise (at approximately 20 minutes into the run), the Brookfield cylindrical-rotary viscometer response remains saturated at the maximum value of 421 cP.

At approximately 36 minutes into the run, a step change was introduced by opening the heater steam valve to 20% of its maximum value. This instant is marked by the square symbol in both the upper and the lower plot in Figure 5.7. Prior to this change, the temperature in the sample loop was at 115°C and the viscosity value had settled around 250 cP. There was a lag of approximately 1 minute before the effect of this step change in valve opening was felt in the sample loop temperature. The Brookfield cylindrical-rotary viscometer starts responding to this temperature change almost immediately. There was a negligible time lag of approximately 45 seconds before the measured viscosity value starts decreasing in response to the temperature raise. The temperature in the sample loop keeps raising until 58 minutes into the run, when a further change

in the valve opening is effected. The Brookfield cylindrical-rotary viscometer was able to track the corresponding viscosity very well over this period of time.

The viscometer tracking performance at other times are qualitatively the same. For example, at approximately 66 minutes into the run, the heater steam control valve was suddenly opened to 30%. This instant is marked by the cross symbol in both the upper and lower plots in Figure 5.7. The time delay and the response of the on-line viscometer was similar to the previous change. At approximately 69 minutes the viscometer reads lower values for a brief time (of approximately 1-minute duration), but the performance is quickly restored and proper operation is observed.

Figure 5.8 plots the viscosity as a function of time to examine the transient response to changes in rotational speed of the Brookfield viscometer. The liquor used was a mixture of Georgia Pacific and Canadian Forest Products black liquors with a resulting solids composition of 67.46%. Obtaining rapid and accurate response while changing the rotational speed is an important feature if this instrument is to be used for measuring the on-line viscosity of non-Newtonian fluids. By varying the rotational speed, the effect of different shear rates on the apparent viscosity can be examined in rapid succession. The viscosity reported in the figure is initially measured running the instrument at a shear rate corresponding to 50 rpm. Then at time t = 1.8 min the instrument's rotational speed is suddenly changed to 100 rpm. This instant is identified in Figure 5.8 using a circle as a marker. Following this change, the viscosity is recorded again as a function of time after a period of approximately 30 seconds denoted as "Lag 1" in the figure. This lag period represents the time that it took the operator to manually enter the value of the new rotational speed into the data acquisition program in order to update the equation that converts the 4-20 mA torque signal into a viscosity value. The torque response from the Brookfield cylindrical-rotary viscometer is almost instantaneous, and all the lag shown in the figure can be attributed almost entirely to the delay associated with manually entering the new rotational speed into the software. Further consecutive changes of rotational speeds to 200 rpm (at t = 3.0 min), 50 rpm (at t = 4.38 min), and a final change back to 200 rpm (at t = 5.38 min) show that the value of viscosity remains approximately constant at all rotational speeds, and that similar operator-induced lags are observed after each change. Such insensitivity of the viscosity to the instrument's rotational speed represents the correct physical behavior since the black-liquor being tested was proven to be a Newtonian fluid (*i.e.*, its viscosity is independent of the shear rate). In summary, the Figure 5.8 demonstrates that the Brookfield cylindrical-rotational viscometer is capable of quickly responding to changes in rotational speed; therefore it could be used to characterize the viscosity of non-Newtonian fluids on-line by taking measurements at different shear rates. This capability would require that the instrument be equipped with software that can automatically and very rapidly input the rotational speed changes to avoid undesirable time lags.

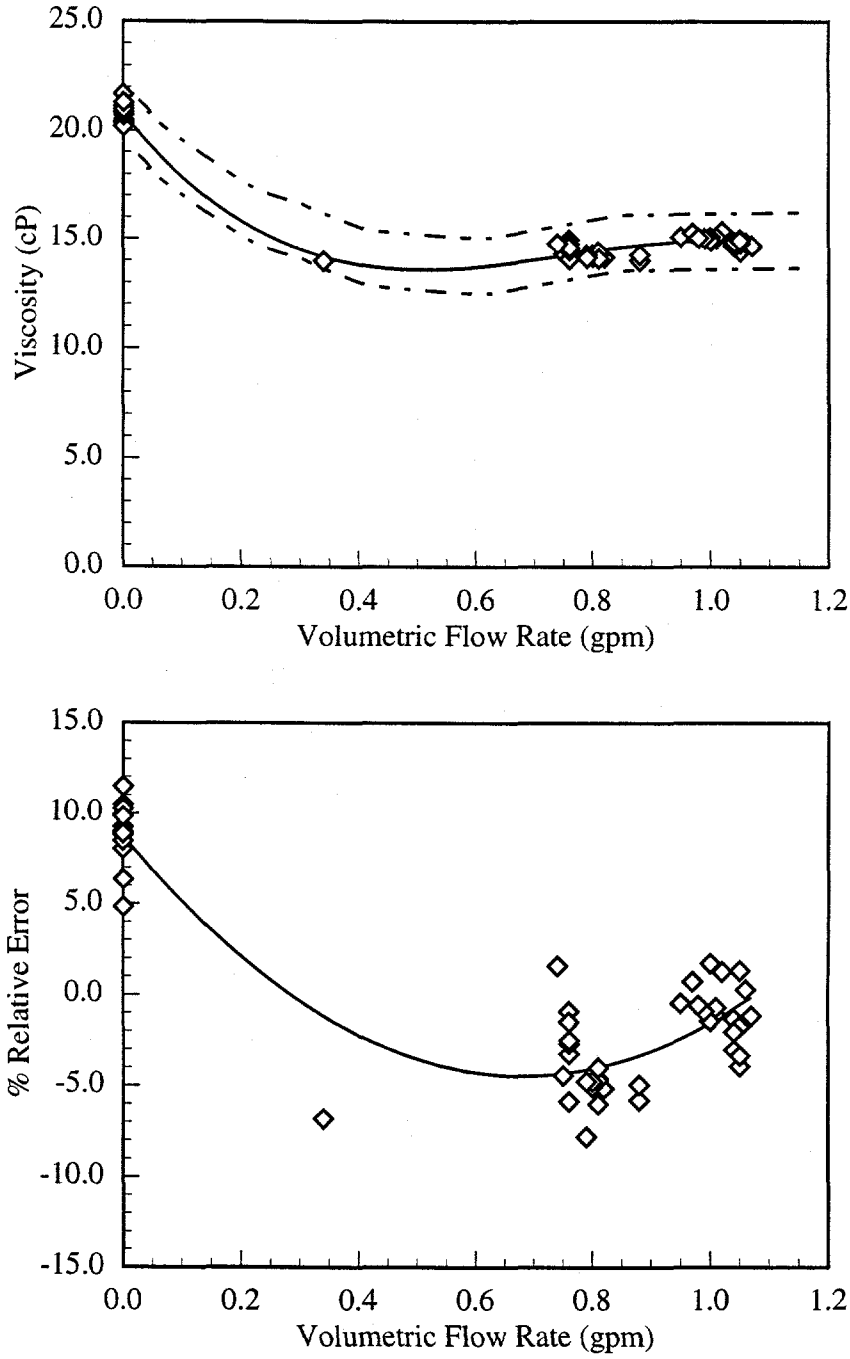


Figure 5.2. Viscosity measured on-line by the Brookfield cylindrical-rotary viscometer at 300 rpm for a Georgia Pacific black liquor with 58.38% solids at approximately 118 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

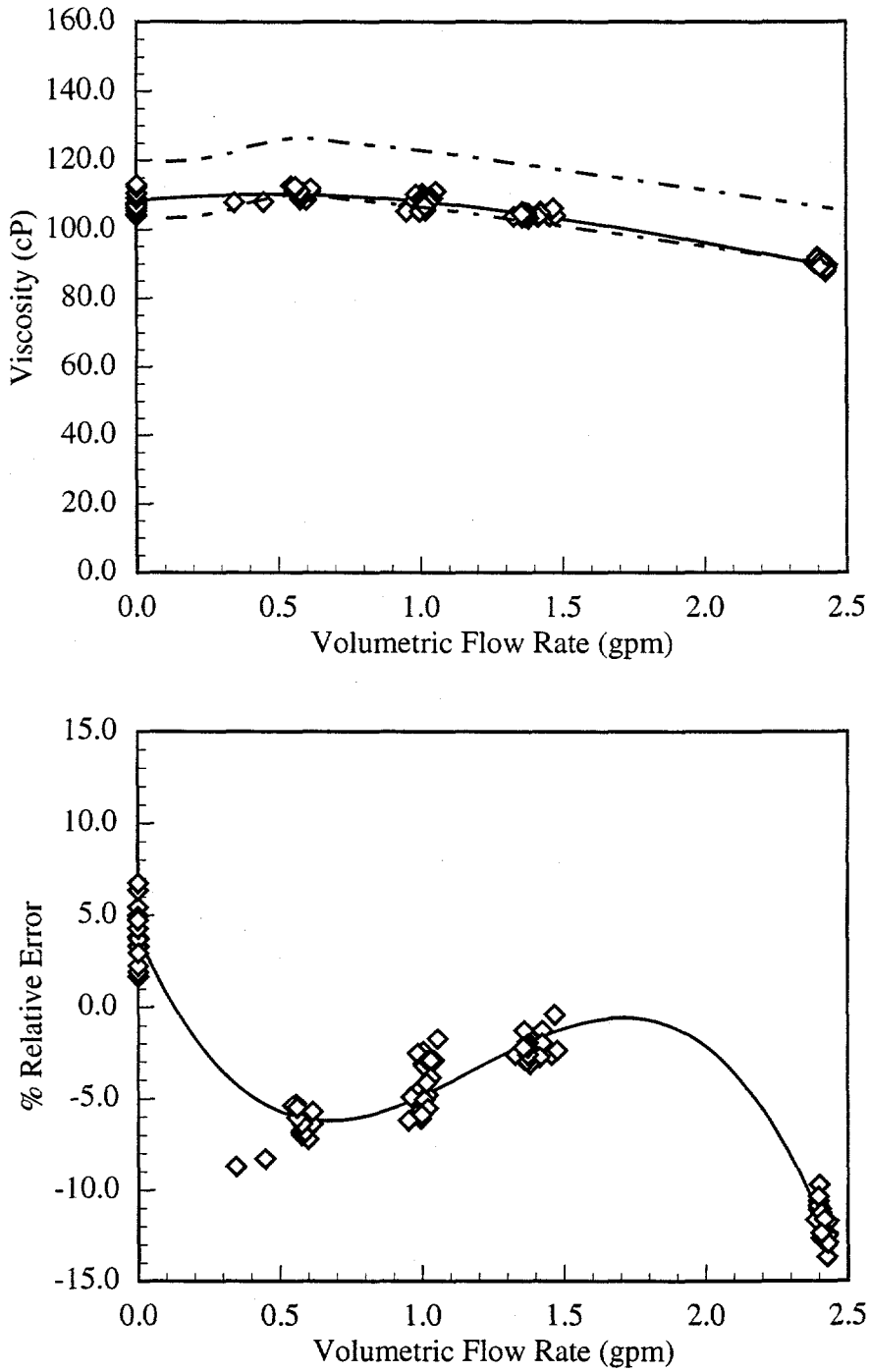


Figure 5.3. Viscosity measured on-line by the Brookfield cylindrical-rotary viscometer at 50 rpm for a Georgia Pacific black liquor with 65.51% solids at approximately 95 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

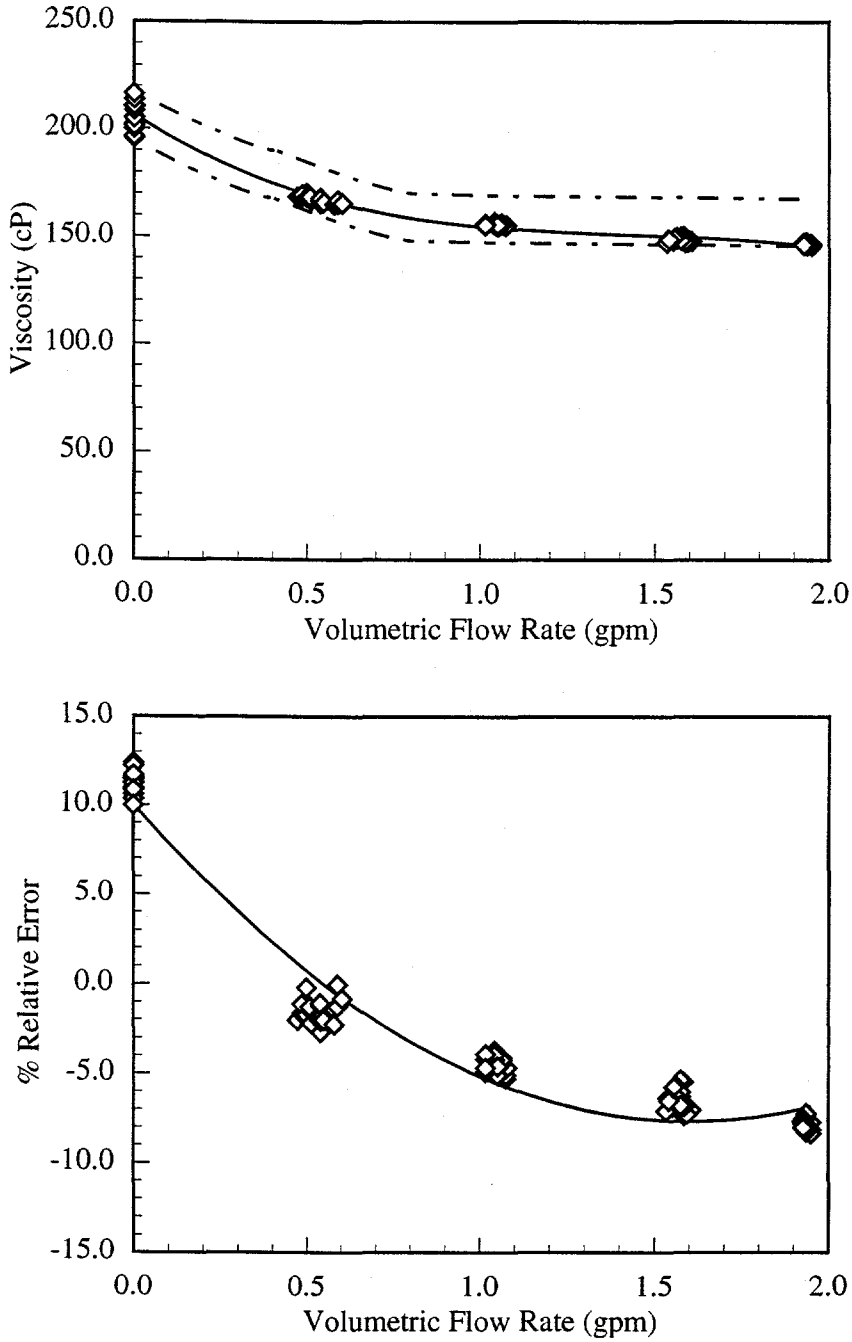


Figure 5.4. Viscosity measured on-line by the Brookfield cylindrical-rotary viscometer at 50 rpm for a Georgia Pacific black liquor with 65.51% solids at approximately 88 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

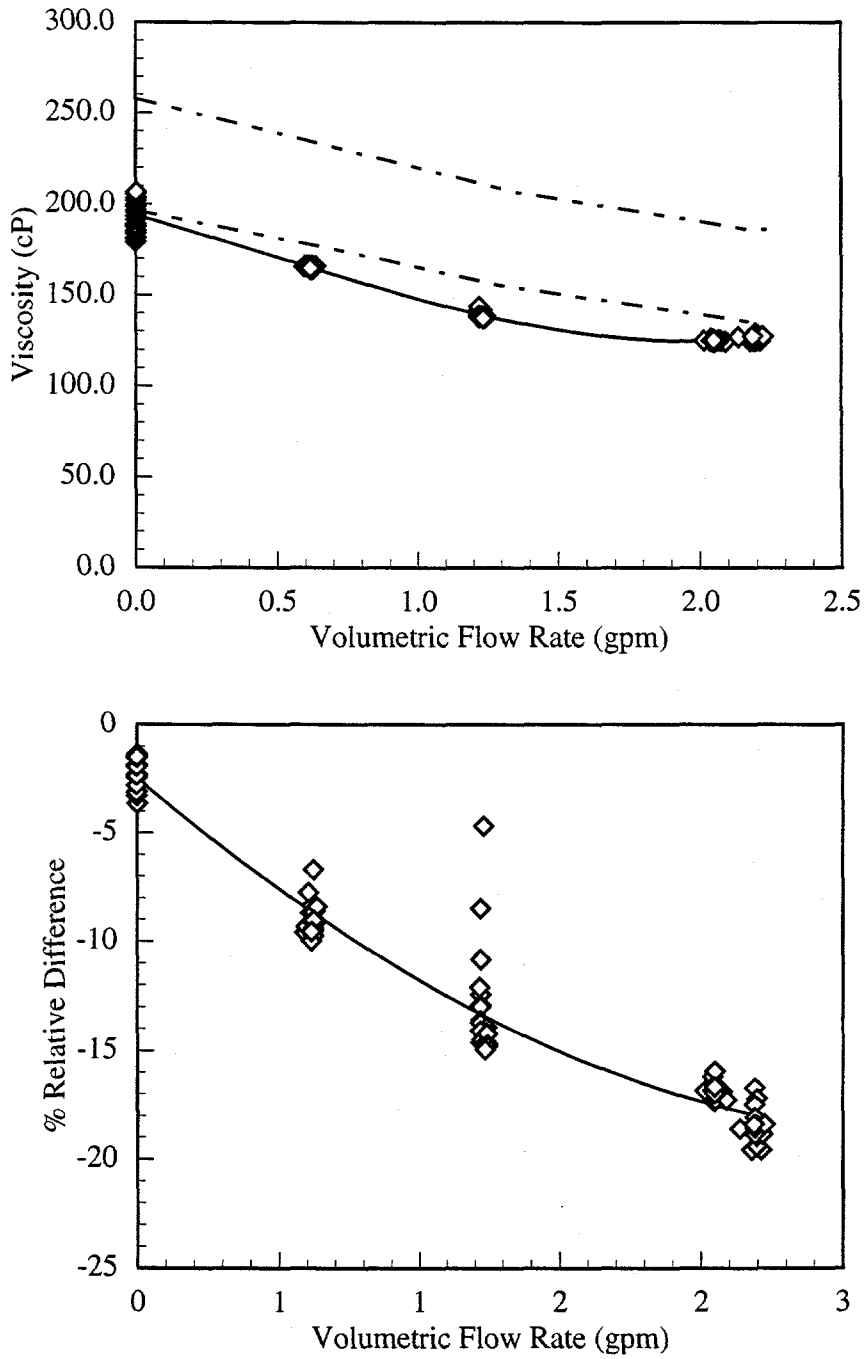


Figure 5.5. Viscosity measured on-line by the Brookfield cylindrical-rotary viscometer at 100 rpm for a Georgia Pacific black liquor with 68.38% solids at approximately 95 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

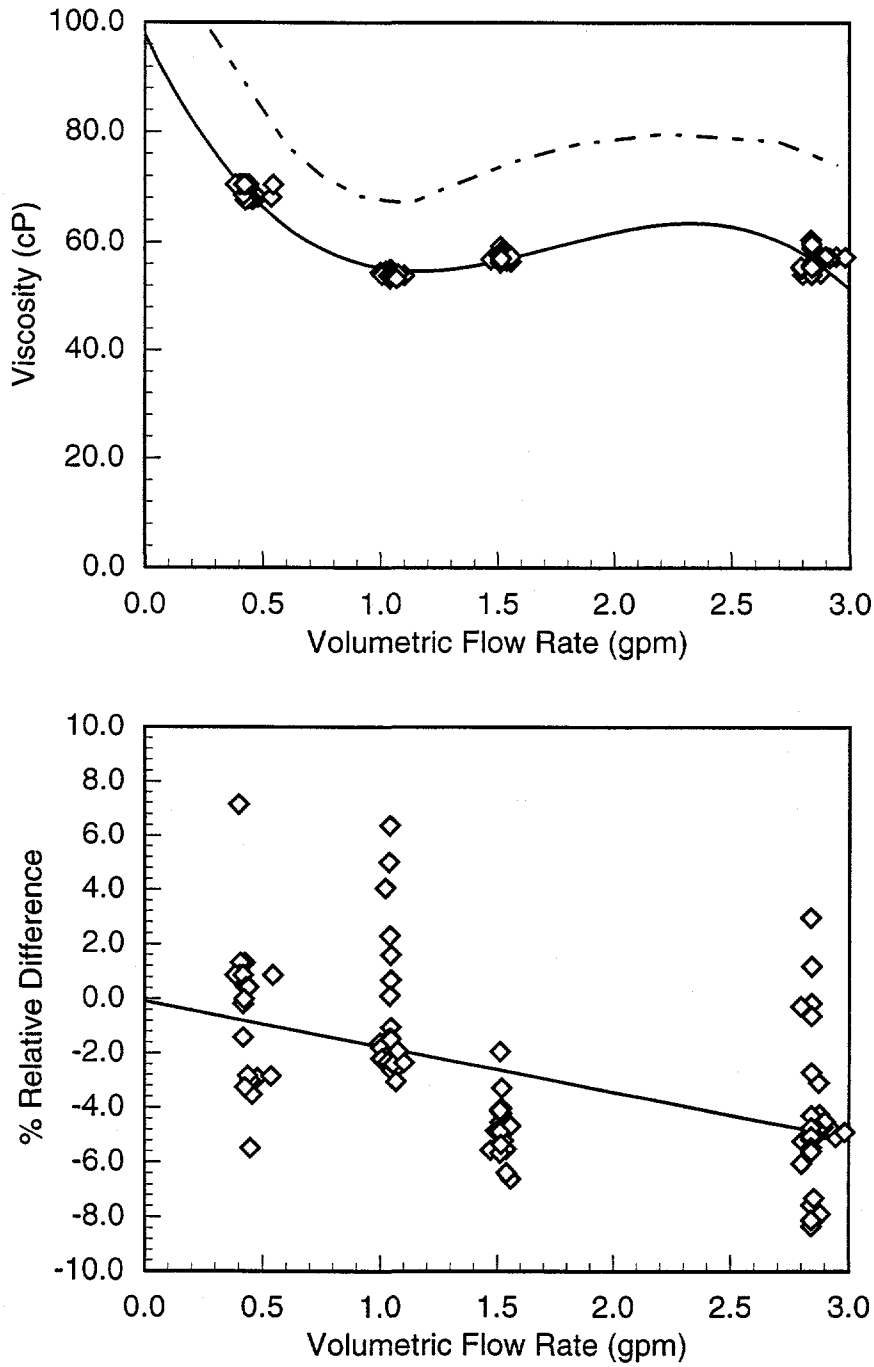


Figure 5.6. Viscosity measured on-line by the Brookfield cylindrical-rotary viscometer at 100 rpm for a Georgia Pacific black liquor with 68.38% solids at approximately 110 °C. The lower plot represents the difference with the viscosity predicted by a laboratory correlation.

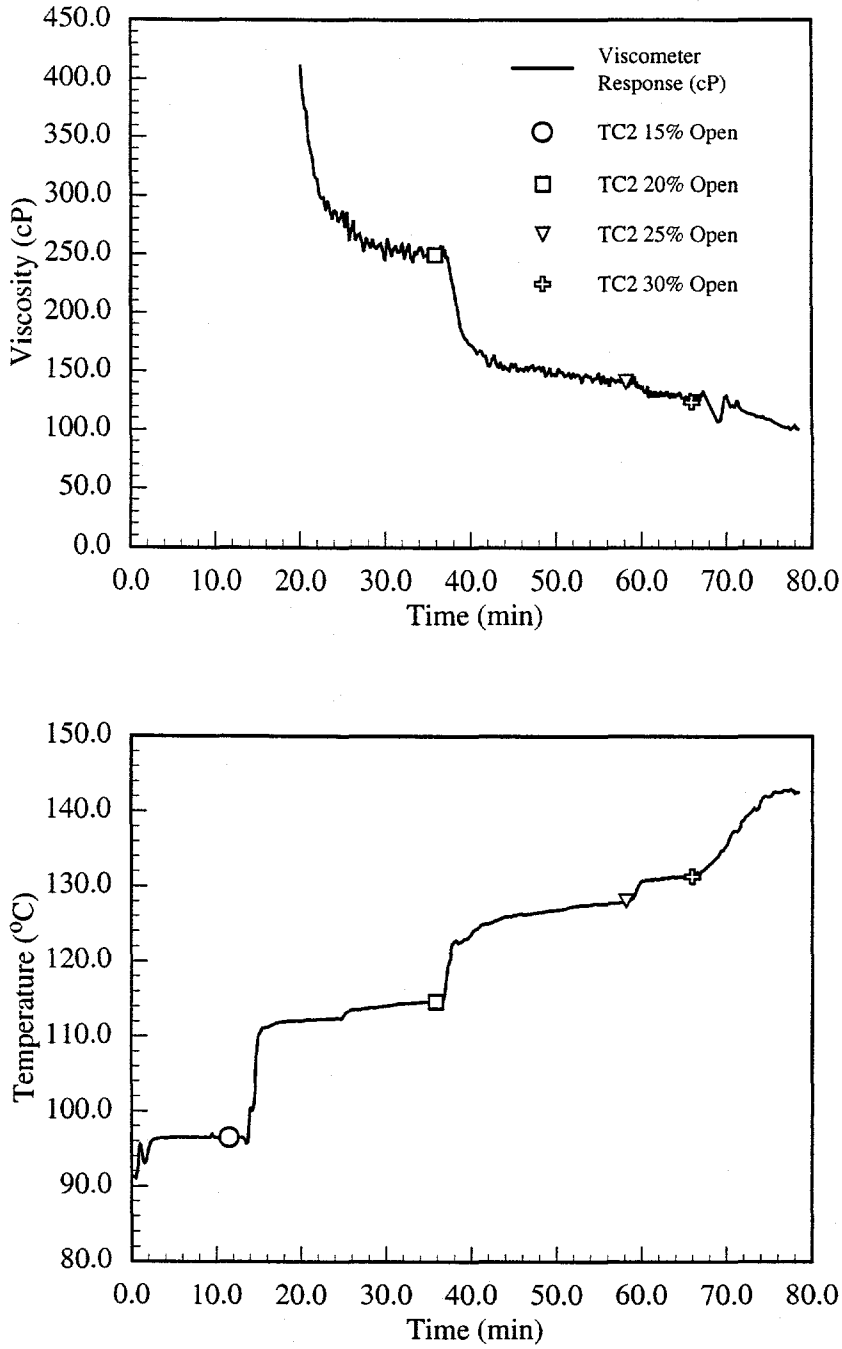


Figure 5.7. Transient response of the Brookfield cylindrical rotary viscometer. The upper plot shows the viscosity measured on-line for a mixture of Canadian Forest products and Georgia Pacific liquors with 67.46% solids as the temperature of the liquor is changed by opening the steam valve of the indirect heater. The lower plot shows the temperature of the black liquor as a function of time. The markers appearing in both plots indicate the instants when the steam valve opening is changed in order to cause a temperature increase.

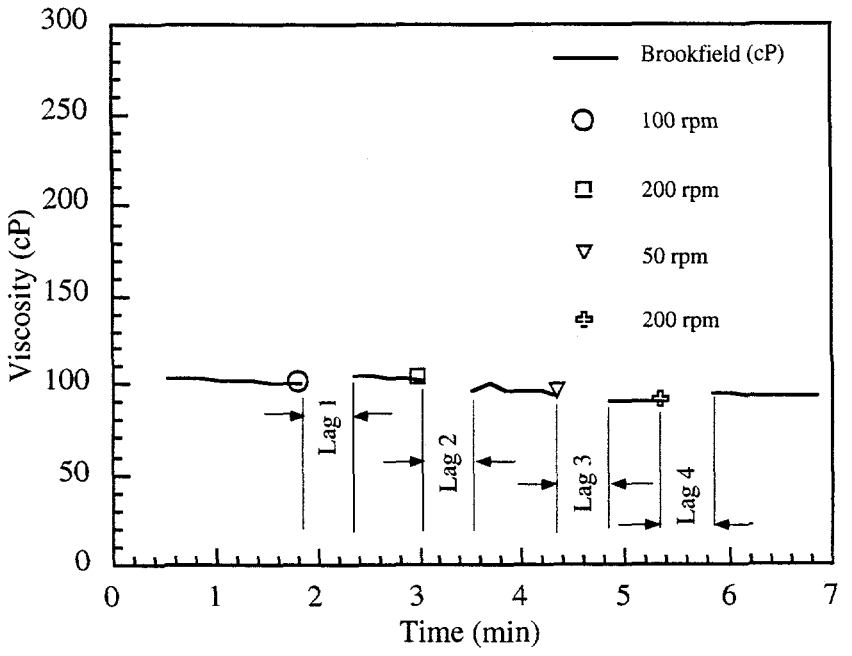


Figure 5.8. Effect of changing the rotational speed of the Brookfield cylindrical-rotary viscometer. The liquor used is mixture of Georgia Pacific and Canadian Forest Products black liquors mix with a resulting solids composition of 67.46%.

5.4.3 Summary of Observations for the Pilot Plant Trials

The general trends observed with the on-line cylindrical rotary viscometer tested in the pilot flow loop are summarized as follows:

- The viscometer used in this study tracked reliably within ±10% or less of the laboratory reference viscosity.
- The viscometer suffered no fouling problems.
- The instrument reading was unaffected by particles passing a 40-mesh size.
- The instrument response to temperature changes is very rapid with a lag not exceeding one minute.
- The rotational-speed transition has negligible time delay (of the order of few seconds); hence the instrument is capable of measuring the viscosity of non-Newtonian fluids.
- Low flow rate conditions do not affect the accuracy of this instrument.

- At higher flow rates, the instrument reading tended to be lower than the laboratory reference for high viscosity black liquors due to axial flow effects.
- The current control unit suffers an offset in both the torque and rotational speed output signals. This had to be manually compensated for by the operator.
- The instrument does not require laboratory calibration for operation. However, laboratory verification is desirable to evaluate accuracy.

The cylindrical rotary viscometer was used in the pilot flow loop to measure viscosities from 12 cP up to about 1040 cP with acceptable accuracy. Increasing the stator length by nearly a factor of two will yield an instrument that can measure viscosities of 3.5 cP with an accuracy of ±10%.

During the trials there was no evidence of fouling or solids build up in the instrument. This conclusion tends to be supported by the fact that, on several occasions, the viscometer line was steam purged with 100 psig saturated steam with practically no change in the instruments performance. The measuring chamber in the viscometer was physically opened and inspected after long exposure to black liquor, and no significant build up on any surface was noticed.

For high viscosity black liquors, a downward trend in viscosity measurement is observed as the flow rate is increased. This phenomenon is depicted in Figure 5.5. However, for liquors with mid-range viscosities this problem does not exist (see Figure 5.6). A development of cross flow across the stator is suspected to be the cause of this problem. Due to the limitations of the viscometer line (in order not to exceed the safely limit on the main pump pressure outlet), it was not possible to evaluate the upper flow rate limits of the instrument. However, in our experience, with flow rates less than 3 gpm the effects are relatively minor. Although the black liquors tested in the flow loop were Newtonian, this instrument can be used to measure the non-Newtonian liquors without any hardware modification. This is a major advantage. Only the calculation procedures need to be changed, to obtain the apparent viscosity from the torque and the rotational speed signals.

The unit installed at the University of Florida suffered a offset drift in both the torque and rotational speed signals and had to be manually corrected for each run. This necessitated extra record keeping to adjust the effect of the offset for each run. An auto zeroing function for the viscosity and rotational speed signals would significantly reduce errors caused by drifts and offsets. Various approaches, such as regularly checking the instrument and electronics ground connections, and addition of chassis grounds did not succeed in eliminating the drift problem. According to the manufacturer, this problem has not been experienced in any other installation.

The only possible non-instrument source of this offset drift may be from the line current, which is not isolated.

The original unit included a prototype single-flush seal to reduce maintenance on the viscometer; however, the seal failed and the standard double-flush seal was installed. The double-flush seal requires a slight positive pressure on the side of the flush fluid. Water under air pressure was used as the flush fluid. In mill installations, oil may be required as a seal fluid. The double-flush type of seal requires more maintenance than the single seal design.

With the current unit as installed in the pilot flow loop, the viscosity calculation is done by the UF DAQ System at a fixed rotational speed. Manual input of the 0-30 Vac signal is necessary whenever the rotational speed is changed. This introduces an unnecessary time delay in computation of apparent viscosity for non-Newtonian studies.

5.5 Mill Trials at a Georgia Pacific Corporation Site

The Brookfield Cylindrical Rotary Viscometer was selected for trial at the Georgia-Pacific Corporation mill at Palatka, Florida, a southern mill.. There were several reasons for selecting the Georgia-Pacific Corporation mill at Palatka, Florida for testing this instrument. It was desired to measure the liquor viscosity at firing conditions. From measurements of viscosity of liquors from this recovery system that had been made over a number of years, the liquor viscosity was known to vary from about 25 to 60 cP and was known to be slightly pseudo-plastic. The Brookfield viscometer, due to the small spacing between the coaxial cylinders used to measure the viscosity, is most susceptible to fouling or damage caused by large suspended particles, and the liquor at the Georgia-Pacific mill proved to exhibit the least solids precipitation of the liquors from the three sites available for mill trials. The mill at Palatka, Florida is less than 50 miles from the University of Florida; therefore, close liaison could be provided.

It was recommended that a viscometer with a maximum range of 200 cP (preferable 100 cP) be installed in a side stream line that was steam traced with a screen no finer than 40-mesh (12-mesh was indicated as preferred). The exact recommendation is discussed in more detail later. Three trial attempts extending over an 18-month period were made. There was a prolonged delay in beginning the first trial due to prolonged negotiations between the mill and the instrument manufacturer over conditions followed by delays awaiting a shutdown to install the instrument.

5.5.1 Experimental Overview

The viscometer was installed in a side stream line from the furnace ring header feed line so that

the liquor viscosity would be measured at the liquor firing conditions, but so that this side stream line could be isolated for cleaning the instrument without interrupting furnace operation. The liquor diverted to flow through the side stream line was to be returned to the firing liquor feed tank. Flow through the side stream line could be throttled, but flow control was not necessary. Since most of the flow through the instrument chamber bypasses the measurement gap between the cylinders, measurement is not sensitive to flow rate over moderate ranges of flow rate. However, there should be sufficient flow to provide reasonable response to process changes. A screen or strainer should be located upstream of the instrument, preferably 12-mesh was recommended, and the preferred location for a screen would be over an enlarged entrance to the side stream with the screen parallel to the flow in the ring header feed line to minimize plugging tendencies and to permit back flushing with hot liquor, if necessary. The side stream line was piped and valved to permit the instrument to be isolated for flushing and cleaning or for removing the instrument for maintenance, if necessary. The instrument and side stream line were to be steam traced to prevent freeze up.

A plan for evaluation of the instrument was developed with Georgia-Pacific mill personnel and the Brookfield regional representative. Instrument readings were to be recorded on the DCS system continuously and the pertinent DCS records to be transmitted to us. Samples would be taken using sample bombs designed to prevent flashing during sampling, and the samples split between the mill lab and our lab at the University of Florida. In addition to DCS data, the mill lab would determine solids concentration by three methods, the liquor heating value and liquor density. The liquor solids concentration and the liquor viscosity would be determined at the University of Florida. Liquor viscosity would be determined in the laboratory at the same temperature recorded for the on-line viscometer in the mill. The instrument was mechanically zeroed and calibrated by mill personnel who followed instructions provided by the instrument manufacturer.

5.5.2 Discussion of Results

Three instrument trials were carried out with the Brookfield viscometer at the Georgia Pacific mill in Palatka. These trials were respectively initiated in July 1977, August 1998, and March of 1999.

First instrument trial - July 1977

The first mill trial began on July 8, 1997 and continued through July 30, 1997 for a total of 22 days. During this time a total of twelve samples were collected at the mill site and shipped for analysis at the University of Florida. A summary of the properties of the samples is given in

Tables 5.3a and 5.3b. The variations in solids concentration measurements obtained by various methods (Table 5.3a) illustrate the difficulty in accurately ascertaining this quantity. A graph comparing the solids concentrations determined by all the methods considered given in Figure 5.9a. The solids concentration determined with diatomaceous earth is in closest agreement with the University of Florida measurements, but both are generally below the values obtained via the pH cookout method and from the refractometers. However, the trends are generally the same for all the methods. The temperature measured by the sensor installed inside the on-line Brookfield viscometer and the temperature used for the measurement of viscosity at the University of Florida are compared on a greatly expanded temperature scale in Figure 5.10. With the exception of the first two samples, these two temperatures differ by less than 0.5 °C.

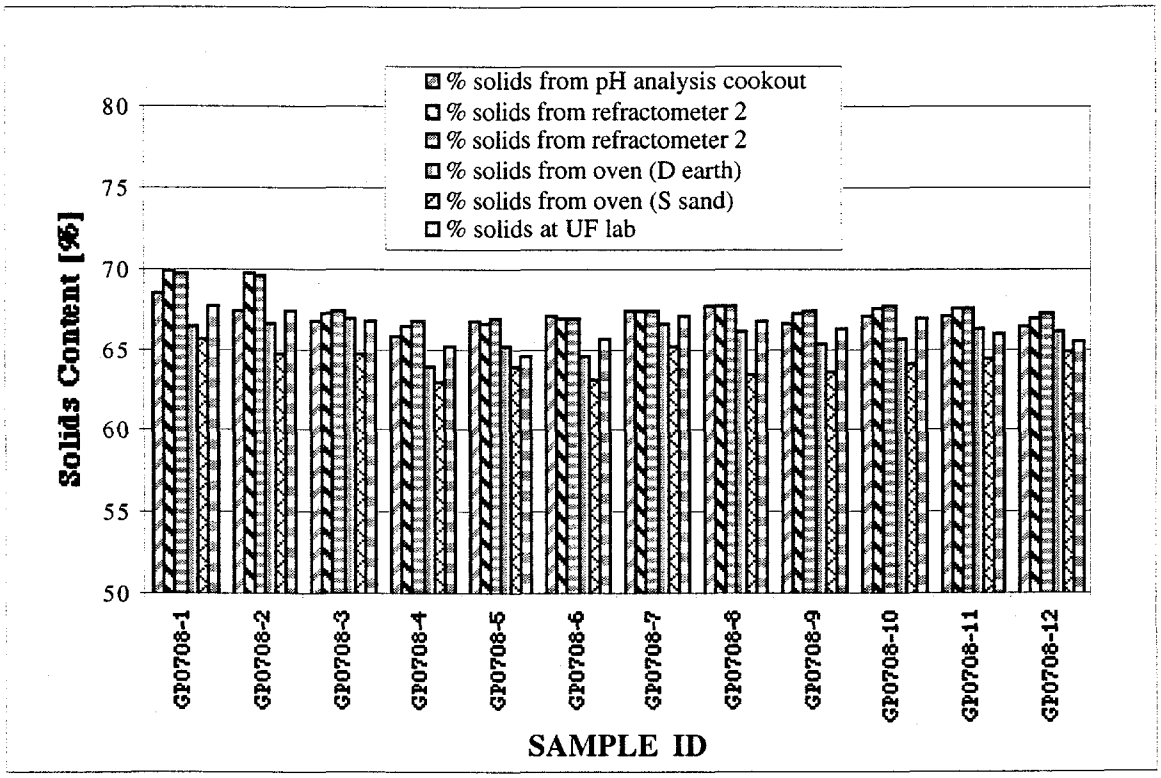


Figure 5.9. Solids contents for 12 samples collected at Georgia Pacific paper mill during the first trial (July 1997).

Table 5.3a. Analysis of 12 samples collected at Georgia Pacific paper mill during the first instrument trial (July 1997). The firing temperature at the recorded at the ring-header lines by the distributed computer system (DCS) is shown along with the temperature recorded by the on-line viscometer (Brookfield unit). The solid-contents values obtained at the Georgia Pacific laboratory along with those obtained at the University of Florida laboratories are shown in the last columns. The specific methods used for solids contents are shown at the bottom of the table.

Sample			Firing Temperature		Solids Content					
					GP Lab					UF Lab
			DCS	On-Line	Method 1	Method 2	Method 3	Method 4	Method 5	
ID	DATE	TIME	°C	°C	%	%	%	%	%	%
GP0708-1	07/08/97	14:58	130.6	128.3	68.50	70.00	69.80	66.50	65.70	67.69
GP0708-2	07/10/97	12:45	130.0	128.3	67.50	69.80	69.70	66.60	64.80	67.38
GP0708-3	07/15/97	12:45	130.0	128.3	66.80	67.30	67.40	66.90	64.80	66.82
GP0708-4	07/16/97	15:13	128.9	127.8	65.80	66.50	66.80	64.00	63.00	65.20
GP0708-5	07/17/97	07:32	129.4	127.8	66.80	66.70	67.00	65.21	64.02	64.54
GP0708-6	07/18/97	07:42	128.3	127.2	67.10	66.90	67.00	64.58	63.26	65.69
GP0708-7	07/22/97	13:20	128.3	126.7	67.40	67.50	67.50	66.70	65.26	67.10
GP0708-8	07/23/87	09:41	128.3	126.7	67.70	67.70	67.80	66.10	63.50	66.79
GP0708-9	07/24/97	-	129.4	127.2	66.70	67.30	67.40	65.33	63.67	66.35
GP0708-10	07/25/97	-	130.0	127.2	67.10	67.60	67.80	65.70	64.10	67.02
GP0708-11	07/29/97	-	128.3	126.1	67.10	67.60	67.60	66.30	64.50	66.00
GP0708-12	07/30/97	-	128.3	127.2	66.50	67.00	67.30	66.20	64.90	65.55

Method 1: Percentage solids obtained from pH analysis cookout

Method 2: Percentage solids obtained from refractometer 1

Method 3: Percentage solids obtained from refractometer 2

Method 4: Percentage solids obtained from oven (D earth)

Method 5: Percentage solids obtained from oven (S sand)

Table 5.3b. Analysis of 12 samples collected at Georgia Pacific paper mill during the first trial (July 1997). The temperature recorded by the Brookfield viscometer (On-Line) is followed by columns that summarize the properties of the black liquor flow along the ring-header lines as recorded by the mill's distributed computer system.

Sample			Instrument	Liquor information are the ring header lines				
			Temperature (On-Line)	Temperature DCS	Flow Rate	Pressure	Heating Value	Density
ID	DATE	TIME	°C	°C	gal/min	psi	BTU/lb	lb/gal
GP0708-1	07/08/97	14:58	128.3	130.6	309	18.6	6415.07	10.26484
GP0708-2	07/10/97	12:45	128.3	130.0	405	19.0	6315.18	11.33197
GP0708-3	07/15/97	12:45	128.3	130.0	412	18.8	6119.17	11.39867
GP0708-4	07/16/97	15:13	127.8	128.9	421	17.8	6601.41	11.38914
GP0708-5	07/17/97	07:32	127.8	129.4	418	18.7	6168.97	11.38279
GP0708-6	07/18/97	07:42	127.2	128.3	419	18.2	6241.19	11.48442
GP0708-7	07/22/97	13:20	126.7	128.3	410	18.0	6146.30	11.36056
GP0708-8	07/23/87	09:41	126.7	128.3	414	18.2	6237.89	11.35421
GP0708-9	07/24/97	-	127.2	129.4	410	19.0	6287.65	11.29704
GP0708-10	07/25/97	-	127.2	130.0	419	18.9	6067.51	11.37644
GP0708-11	07/29/97	-	126.1	128.3	419	17.5	5962.03	11.48760
GP0708-12	07/30/97	-	127.2	128.3	419	17.6	6062.94	11.40185

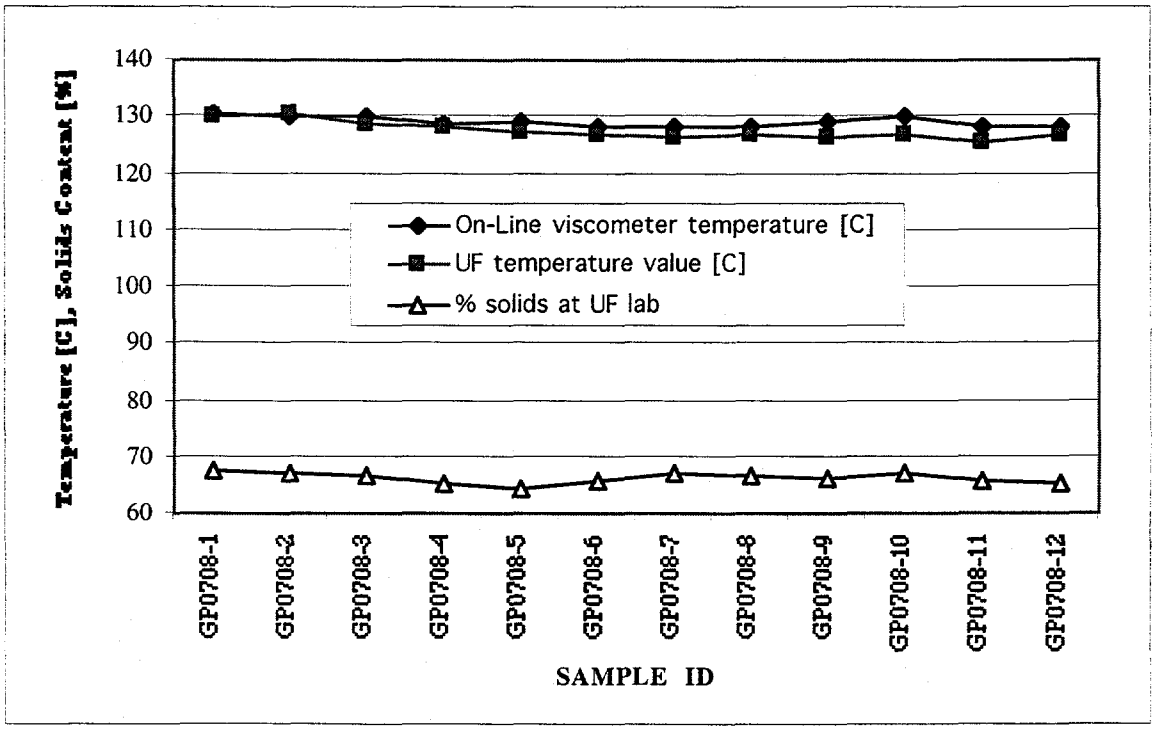


Figure 5.10. Relevant temperatures of 12 samples collected at Georgia Pacific paper mill during the first trial (July 1997). The bottom plot shows the solids content for the samples as determined at the University of Florida laboratories.

The viscosity measurement results are summarized in Table 5.4 and on Figure 5.11. The viscosity reported by the University of Florida laboratories was measured using a Brookfield LTV pressurized rotating cup viscometer, which is based on the same rotating-cup principle of measurement as the on-line Brookfield instrument (model TT100SD) installed at the Georgia Pacific mill. The liquors proved to be slightly pseudoplastic as will be discussed later. The viscosity values determined at the University of Florida laboratories are reported in Table 5.4 as the average values obtained for various shear rates in the range of 200-800 sec^{-1}. The last column of the table shows the relative difference between the on-line measurements and the UF laboratory measurements calculated using the relationship

$$\text{Relative Difference} = 100\,\frac{\eta - \eta_{ref}}{\eta_{ref}}\ \%$$

where η is the viscosity measured by the on-line Brookfield sensor, and η_{ref} is the viscosity measured use the instruments available in the UF laboratories.

Table 5.4. Analysis of 12 samples collected at Georgia Pacific paper mill during the first trial (July 1997). The temperatures shown correspond to that of the ring-header lines (DCS), that of measured by the Brookfield instrument (On-Line), and the temperature used at the UF laboratories The last three columns show the viscosity measured by the Brookfield instrument (On-Line), the viscosity determined at the University of Florida laboratories, and the relative difference between these two values.

Sample			Temperature			Viscosity		
			DCS	On-Line	UF Lab	On-Line	UF Lab (1)	Relative Difference
ID	DATE	TIME	°C	°C	°C	cP	cP	%
GP0708-1	07/08/97	14:58	130.6	128.3	130.22	128.0	51.39	149.1
GP0708-2	07/10/97	12:45	130.0	128.3	130.64	122.5	40.30	204.0
GP0708-3	07/15/97	12:45	130.0	128.3	128.77	127.5	28.47	347.8
GP0708-4	07/16/97	15:13	128.9	127.8	128.38	90.5	22.90	295.2
GP0708-5	07/17/97	07:32	129.4	127.8	127.56	149.0	31.92	366.8
GP0708-6	07/18/97	07:42	128.3	127.2	127.08	181.0	30.96	484.6
GP0708-7	07/22/97	13:20	128.3	126.7	126.66	165.0	45.96	259.0
GP0708-8	07/23/87	09:41	128.3	126.7	127.13	170.0	39.64	328.9
GP0708-9	07/24/97	-	129.4	127.2	126.70	32.5	36.16	-10.1
GP0708-10	07/25/97	-	130.0	127.2	127.05	40.5	35.27	14.8
GP0708-11	07/29/97	-	128.3	126.1	125.81	95.5	27.65	245.4
GP0708-12	07/30/97	-	128.3	127.2	126.92	124.5	28.64	334.7

(1) Average viscosity value for shear rates in the range 200-800 sec^{-1}

A most salient point regarding the values reported in Table 5.4 is that the values measured at the University of Florida laboratories (which lie in the range of 22.9 to 51.4 cP) are consistent with the expected viscosity of the liquor, whereas the values reported by the on-line Brookfield instrument (which range from 90.5 to 181 cP, except for two measurements) are affected by a large error (see last column of Table 5.4). Figure 5.11 shows that the on-line measurements are erratic in comparison with the reference laboratory values, and in general are significantly different from the reference values.

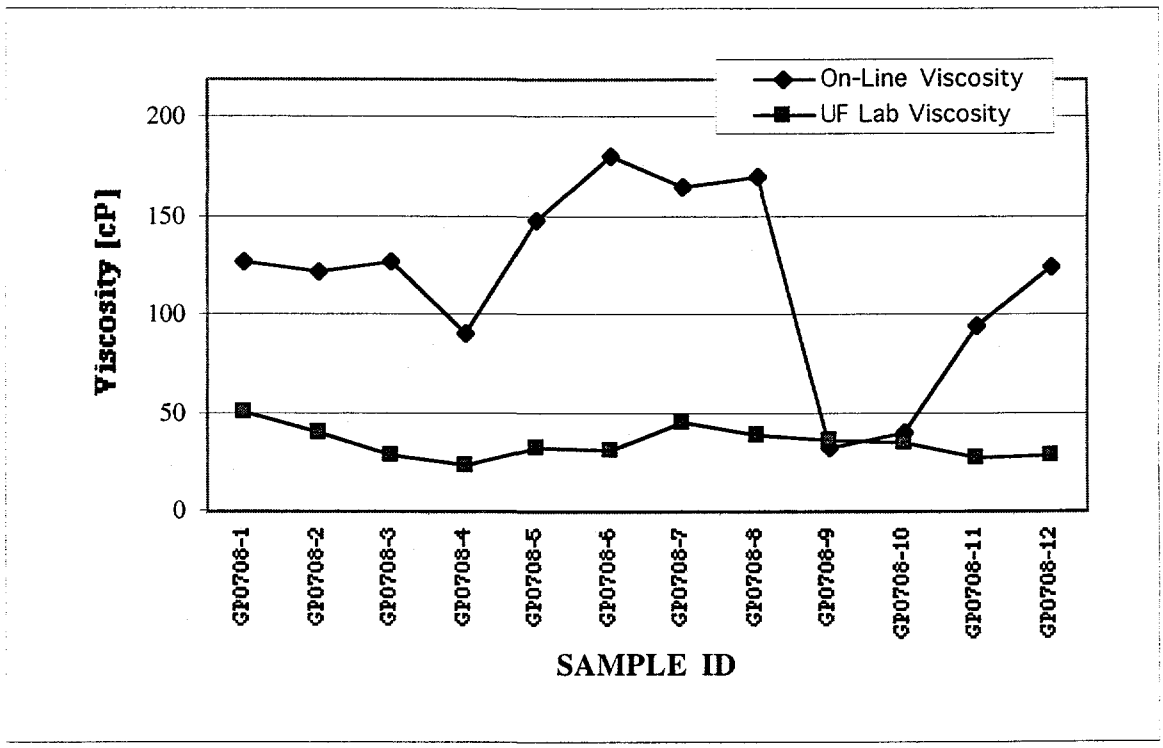


Figure 5.11. Viscosity values for 12 samples collected at Georgia Pacific paper mill during the first trial (July 1997).

The reason for the significant viscosity errors evident in Figure 5.11 is that Brookfield Instruments delivered to Georgia Pacific an instrument that was erroneously designed to measure viscosities in the range of 0-500 cP. This instrument differed significantly from the University of Florida recommendation of an instrument designed for a viscosity range of 0-200 cP, and preferably in the range 0-100 cP. Given the low viscosity values of the liquor, the incorrectly sized instrument operated at torque values that were a very small percentage of its maximum torque, therefore making accurate measurements unfeasible. For example, measurement of viscosity with an error less than 10% for a liquor with a 40 cP viscosity would require a

measurement at ±0.8% of maximum torque. This explains the lack of accuracy of the on-line Brookfield viscometer installed given that this instrument is not recommended for measurements below 10% of maximum torque, even if the instrument has been calibrated with a standard fluid.

In general, except for the fact that the instrument operated for 22 days on-line without plugging, the trial was inconclusive and unsuccessful. The instrument supplied and installed was incapable of making precise and accurate measurements of viscosity in the range to be expected and experienced. More specifically, for the viscosities considered the instrument supplied would exhibit torques ranging from 4.6% to 10.2% of the maximum torque at full speed.

At the end of this trial period the on-line Brookfield instrument suffered physical damage. The reason for this problem was that the Georgia Pacific team had not heat-traced the side-stream where the instrument was deployed. When liquor flow was interrupted the side-stream line was not closed-off, drained and flushed, as recommended. Unfortunately, the instrument cup was allowed to continue rotating as the liquor in the line cooled. As a result, the torque element was overstressed and damaged.

At this point, prolonged negotiations ensued regarding the responsibility for the cost of repairing the damaged instrument and the arrangements for replacement with an instrument having the proper range. Mill personnel agreed to heat-trace the instrument as well as the side stream line to prevent recurrence of the incident that damaged the instrument. It was then necessary to wait for a shut down for installation. In the meantime there had been changes in the mill management and the utility of the project was questioned which led to further delays.

Second instrument trial - August 1978

A second mill trial with the new instrument was begun on August 28, 1998. The side-stream line and the Brookfield on-line instrument were heat traced with electrical heating tape, and the instrument torque system was mechanically calibrated for operation in the range 0-200 cP and at a shear rate of 500 sec^{-1}. A screen was installed upstream from the instrument to protect it from particle damage. The installed instrument was not calibrated against a standard fluid.

The University of Florida proposed a sampling plan that called for the collection samples at 8 hour intervals for the first day, then at 24 hour intervals for the following three days, and then at one-week intervals for three weeks. After the last set of samples were collected the instrument was to run the viscometer in a continuous mode for a total period of 3 months from the first sampling date. Unfortunately the mill was able to take only the first two samples in this plan, spanning less than 10 hours of continuous operation, because the screen in the side stream line plugged and the

liquor stopped flowing.

The two samples were shipped to the University of Florida where the viscosity was measured at the firing temperature at three different shear rates, namely 340 sec^{-1}, 680 sec^{-1}, and 850 sec^{-1}. The viscosity at 500 sec^{-1} was determined by interpolation. The results are summarized in Tables 5.5a and 5.5b, and in Figure 5.12. The viscosity of each sample was also characterized as a function of temperature for three shear rates, and the results are reported in Figures 5.13 and 5.14. It is clear that the Georgia Pacific liquors collected on this date are slightly pseudoplastic and that the variation of viscosity with temperature is significant.

A comparison between the on-line viscosity measurement produced by the on-line Brookfield instrument at a shear rate of 500 sec^{-1} and the viscosity value determined at the UF laboratories is shown on the last column of Table 5.5b. The Brookfield instrument shows a difference of approximately 24% with respect to the reference laboratory values. In spite of this large discrepancy, the Brookfield instrument is nevertheless successful in tracking the drop in viscosity that occurs between the times of the first and second sample collections.

It must be remarked that the viscosities at the firing conditions are very much lower than normally seen for liquors taken from the Georgia Pacific recovery system in the past (compared, for example, with the viscosity measured at the University of Florida laboratories for the twelve samples collected during the fist trial and reported in Table 5.4 and Figure 5.11). Furthermore, the pluggage of the side stream that occurred within 10 hours of operation was totally unexpected since, in the previous test carried out in July of 1998, the system had been operated for 22 days without plugging. Apparently, significant changes in liquor characteristics had taken place in the time between trials, leading to lower viscosities and the presence of larger solid particles. At this point the second mill trial was halted and a review meeting was held.

Table 5.5a. Analysis of samples collected at Georgia Pacific's mill during the second instrument trial (August 1998). The sampling time, firing temperature, solids contents, and the viscosity determined at the University of Florida Laboratories at several shear rates are reported.

Sample			Firing Temp.	Solids Content (1)	UF Lab Viscosity (2, 3)			
					340 sec^{-1}	680 sec^{-1}	850 sec^{-1}	500 sec^{-1}
ID	Date	Time	°C	%	cP	cP	cP	cP
GP0808-1	08/08/98	08:44	128.9	68.4 - 69.1	18.0	17.0	16.3	25.0
GP0808-2	08/08/98	14:20	129.4	68.4 - 68.7	26.0	24.0	21.8	17.4

(1) Solids contents determined at the mill's laboratory facilities
(2) Viscosity measured at the University of Florida laboratory facilities
(3) Viscosity at 500 sec^{-1} was obtained by interpolation

Table 5.5b. Analysis of samples collected at Georgia Pacific's mill during the second instrument trial (August 1998). The sampling time, firing temperature, as well as the viscosity readings obtained on-line (Brookfield unit), and at the University of Florida Laboratory are reported.

Sample			Firing Temperature		Solids Contents(2)	Viscosity (2)		
						On-Line	UF Lab	Difference
ID	Date	Time	°F	°C	%	cP	cP	%
GP0808-1	08/08/98	08:44	264	128.9	68.4 - 69.1	19.0	25.0	-24.0
GP0808-2	08/08/98	14:20	265	129.4	68.4 - 68.7	13.4	17.4	-23.0

(1) Solids contents determined at the mill's laboratory facilities
(2) Viscosity evaluated at 500 sec^{-1}

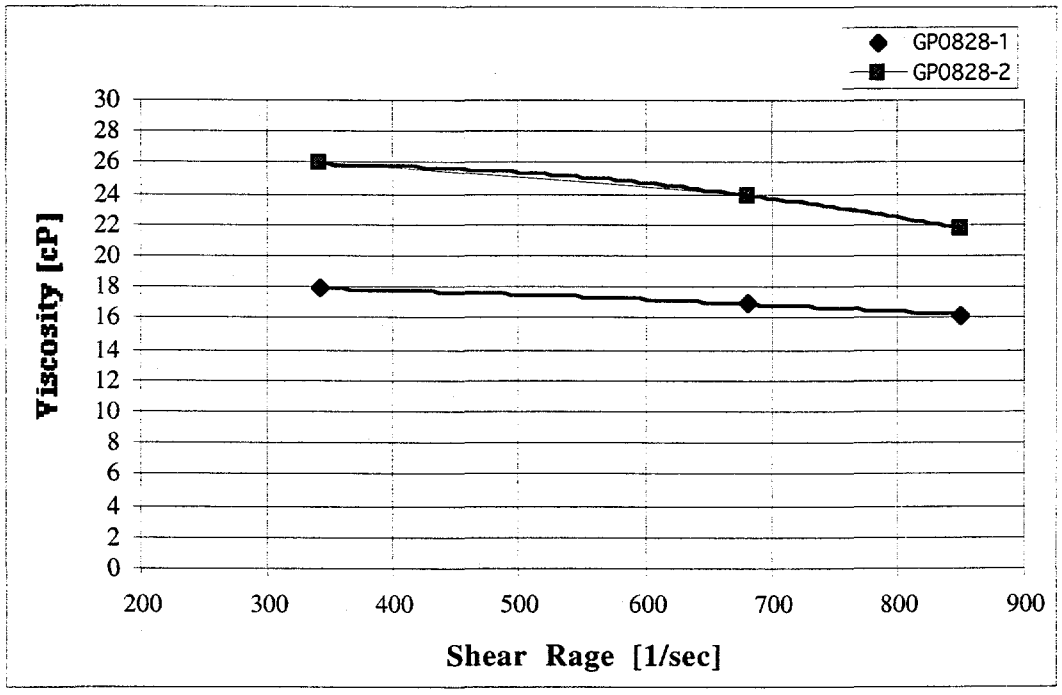


Figure 5.12. Viscosity determined at the University of Florida laboratories as a function of shear rate for the two samples collected at the Georgia Pacific mill on August 28, 1998.

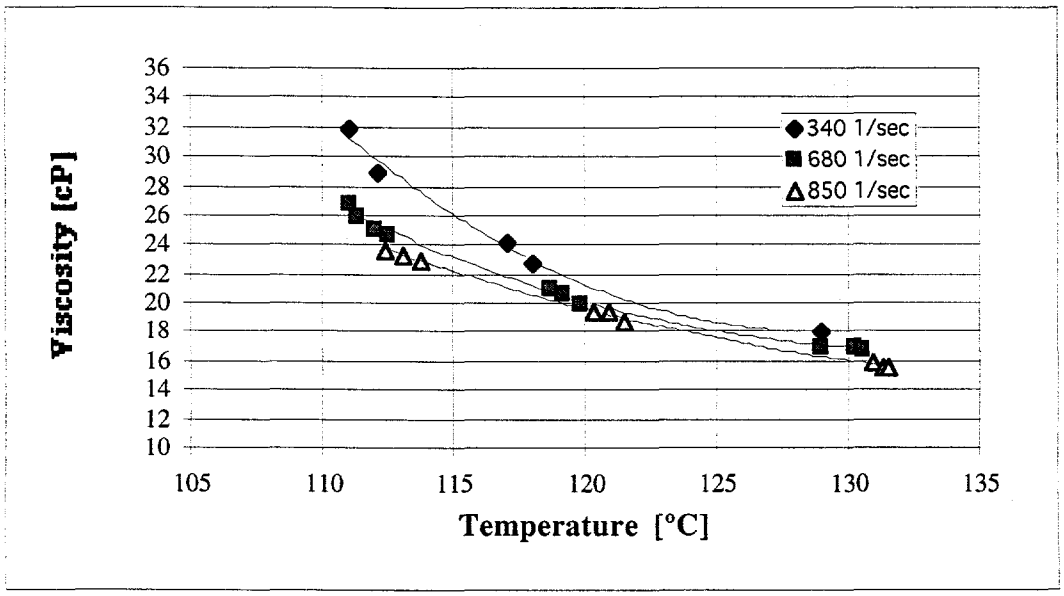


Figure 5.13. Viscosity determined at the University of Florida laboratories as a function of temperature and shear rate for sample CF0828-1 collected at the Georgia Pacific mill on August 28, 1998.

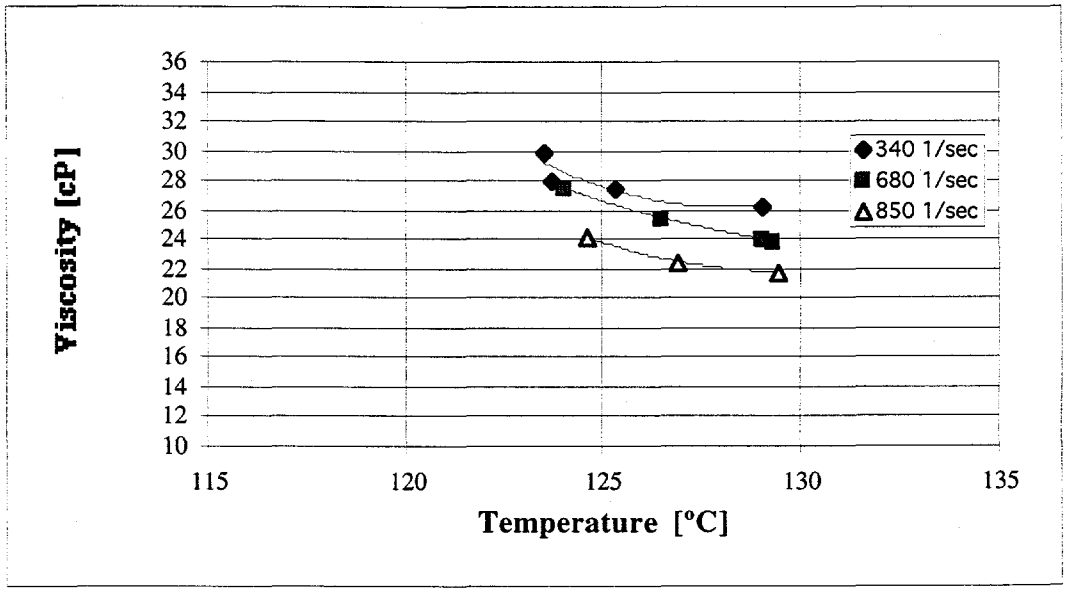


Figure 5.14. Viscosity determined at the University of Florida laboratories as a function of temperature and shear rate for sample CF0828-2 collected at the Georgia Pacific mill on August 28, 1998.

Review of events showed that the Brookfield instrument had been properly installed and mechanically calibrated, and that the side stream line had been insulated and fitted with an electrical heating tape to prevent pluggage problems due to liquor cooling. However, an 80-mesh screen had been installed within the side stream line rather than the recommended 12-mesh screen. This arrangement apparently had also been used in the first trial that had run for 22 days without plugging; therefore, mill personnel saw no reason to change it by following the University of Florida recommendations. Apparently, however, liquor characteristics changed since the first instrument trial, so that this screen arrangement was no longer satisfactory. The lower than expected liquor viscosity also led to low torque readings which in turn resulted in an offset in viscosity of approximately 24% with respect to the UF laboratory values. After prolonged discussion, it was agreed that a third attempt would be made after installation of a recommended 8- or 12-mesh installed at the entrance of the side stream so that it is flush with the main line flow. This would require waiting for the next shutdown scheduled for late-1998 or early-1999 to make the appropriate mechanical change.

Third instrument trial - March 1999

The mechanical changes were allegedly made by the mill personnel and a third mill trial was begun on March 5, 1999 with a sample plan similar to the one planned for the second mill trial. One sample was taken and then the side stream line plugged in less than 10 hours. The on-line viscosity of this single sample was 28.1 cP at 258°F (125.6°C). The solids concentration and the viscosity at 125.6°F and 680 sec^{-1} determined at the University of Florida were 63.3% and 30 cP; therefore, the Brookfield on-line viscosity measurement was found to have a difference of less than 6.3% with respect to the University of Florida laboratory measurement. This single measurement suggests that with the correct sizing specifications the Brookfield viscometer is in principle capable of producing on-line readings with good accuracy, as has been verified by our earlier experiments in the UF Pilot Plant.

Inquiry revealed that a 100-mesh screen had been installed in the side stream line rather than the specified 8 to 12-mesh. Furthermore, the screen was installed upstream from the instrument, but downstream from the point where the side-stream entrance pipe meets with the main line. The incorrect mesh size and the improper screen location point caused the almost immediate pluggage of the line. At this point, mill personnel refused to continue with the project.

5.5.3 Summary of Observations for the Mill Trials

The three instrument trials carried out with the Brookfield viscometer at the Georgia Pacific mill in Palatka, Florida, were inconclusive since its results cannot be used to prove that the

instrument can be used reliably and accurately on line. The first trial demonstrated the necessity of locating the instrument in a side stream line with steam or electrical tracing to protect the instrument from damage due to liquor cooling. The trial also demonstrated that the instrument could be run for extended periods of time without scaling deposits being formed if protection is provided to prevent large suspended particles from entering the instrument measuring zone. Unfortunately, the instrument used for this initial trial was incorrectly sized, and therefore its on-line readings are not meaningful or useful. The second and third trials show that viscosity measurements that track viscosity changes can be made with the Brookfield instrument, and that the on-line viscosity measurement can be accurate if made at torque levels above about 10% of maximum torque and if the instrument is suitably calibrated. It must be noted, however, that these observations regarding accuracy are made using only three measurements for lack of more extensive data.

Although the few viscosity measurements taken with the Brookfield on-line instrument during the second and third trials are encouraging with respect to the accuracy of the instrument, the trials failed to demonstrate that the viscometer could be operated reliably for a sustained period of time. The quick failure of the instrument due to line pluggage in the second and third trials was due to the fact that the installation of the protective screens in the side stream was flawed. The installation of the Brookfield viscometer at Georgia Pacific was flawed because the screens used for the second and third trials led to the rapid plugging of the line. The screens were simply too small, and were not installed flush with the flow of liquor in the main line to minimize the accumulation of black liquor solids. The amount of solid particles in the Georgia Pacific black liquor apparently increased significantly from that observed in the first trial, leading to rapid plugging of the side stream line. It is hypothesized that the successful continued operation of this viscometer would require the installation of an 8-mesh or 12-mesh screen at the entrance of the side stream pipe. Alternatively, a strainer of comparable screening capability could be installed upstream from the instrument, provided that the strainer can serviced via regular maintenance by backflushing with steam or other method of cleaning. Although it is reasonable to expect that the on-line Brookfield instrument would work for a prolonged period of time under the protection of an appropriate screening arrangement, the mill trials conducted at Georgia Pacific do not prove conclusively that such sustained levels of operation are achievable. It must also be remarked that the conditions of increased solid-particle contents observed at the Georgia Pacific mill in Palatka between the first and second trials can be expected to occur with increasing frequency elsewhere as mill cycles are progressively closed to greater extent to meet environmental requirements.

5.6 Specific Conclusions and Recommendations Regarding this Instrument

The testing of the Brookfield cylindrical rotary viscometer at the University of Florida Pilot

Plant proved that the instrument can be used to effectively monitor black liquor viscosity on line. Under pilot-plant conditions it was shown to operate satisfactorily under both steady-state and transient conditions, producing fast, accurate, and reproducible measurements. The response of the viscometer has negligible time delay, and the instrument can be used for Newtonian as well as non-Newtonian measurements. The viscometer suffered no fouling problem. With proper sizing of the rotating cylinder and the torque range, the instrument is capable of satisfactory operating over the expected viscosity ranges encountered in a mill environment. In our opinion, it is highly desirable to enhance the instrument electronics to have an auto-zero option, a feature that is not currently available from the manufacturer. For black liquor application, we strongly suggest choosing the temperature sensor option along with the instrument, which will facilitate verification of the on-line measurements with laboratory measurements taken at the same temperature.

Even though the instrument proved to be successful in an extensive trial period at the UF Pilot Plant, the mill trial results at the Georgia Pacific facilities did not show in a conclusive fashion that the instrument can sustain reasonably long periods of continuous operation. Limited data collected during the second and third mill trials show that accurate measurements can potentially be obtained on-line; however, the small amount of data available does not provide the basis for a conclusive verification of the accuracy of the instrument.

In spite of the lack of evidence of acceptable performance of the Brookfield instrument in the three mill trials conducted, we believe that this viscometer could be used for the measurements of black liquor viscosity if (1) the liquor stream is relatively clear of large suspended particles, (2) an 8-mesh or 12-mesh straining or screening of the liquor is provided (along with the capability of backflushing or cleaning the strainer or screen), (3) the instrument is positioned in a side stream line with provisions for heating the line to prevent freeze-ups, and (4) the instrument is sized for measurements above at least 10% of maximum torque and is properly calibrated. The Brookfield viscometer is definitely <u>not recommended</u> for measurements with liquors containing significant quantities of large-particles as suspended solids.

5.7 Recommended Installation and Deployment Point

The Brookfield instrument must be installed so that it is exposed only to liquors with suspended particles of size no greater than those that would pass a 12-mesh screen. In general, the instrument should be installed in a side stream as shown in Figure 5.14. Note that the side-stream features a 12-mesh tubular screen at the entrance, installed in such a way that the cylinder axis is parallel to the flow in the main liquor feed line. This configuration precludes large-diameter particles from entering the side stream while at the same time minimizes the potential for the screen

to experience pluggage. The valving configuration should permit back-flushing the screen with back liquor as necessary should pluggage develop. The screen can be mounted in a flanged tee arrangement for ease of removal. Installing the screen downstream from the side-stream entrance point is strongly discouraged because large particles accumulating on the screen may eventually cause a plug that would stop the flow through the line.

In addition to a 12-mesh screen, we also recommend the installation of a strainer at a point upstream from the viscometer, as shown in the figure, to provide ultimate protection for the instrument and ensure long periods of continued operation. Provisions must be made so that the strainer can be flushed or be isolated for cleaning. We do not recommend ignoring the need for the tubular screen and installing only the strainer. While the strainer would protect the instrument from particle-induced damage it would not necessarily prevent line pluggage should the maintenance of the strainer be relaxed at a critical period of operation.

The side-stream line containing the instrument should be piped and valved so that it can be drained and steam-cleaned as needed to remove black-liquor deposits that constrict flow. A conceptual valving configuration is shown in Figure 5.14. We have successfully utilized 1-inch pipes for the side stream, but larger diameters could also be used.

It is very important to insulate and heat all the side-stream lines as well as the viscometer itself. This precaution is of critical importance for two main reasons. First, the viscosity of black liquor is extremely sensitive to temperature variations; therefore, heating and insulation precautions must be taken to guarantee that the temperature of the black liquor that reaches the instrument is identical to that of the liquor flowing in the main line. The second reason is that under lack of heating and insulation the black liquor will cool down inside the side stream pipe, eventually causing pluggage of the line, and possibly also causing damage to the delicate internal mechanics of the instrument. The heating of the side-stream lines can be done using a steam-tracing piping network consisting of copper tubes wrapped around the pipes, or by installing an electrical heating tape which can be connected to an inexpensive temperature controller.

Concerning maintenance issues, it should be noted that due to the presence of a rotating part this instrument requires regular attention to prevent damage to the instrument due to pluggage or interactions with large particles.

The instrument should be calibrated so that the minimum viscosity to be measured is at least 10% of the maximum value of the instrument range. Flow though the instrument chamber need not be laminar nor should the flow rate be controlled since only a small portion of the flow through the chamber enters the measuring gap between the cylinders; therefore, only a small pressure drop

over the side stream line is necessary.

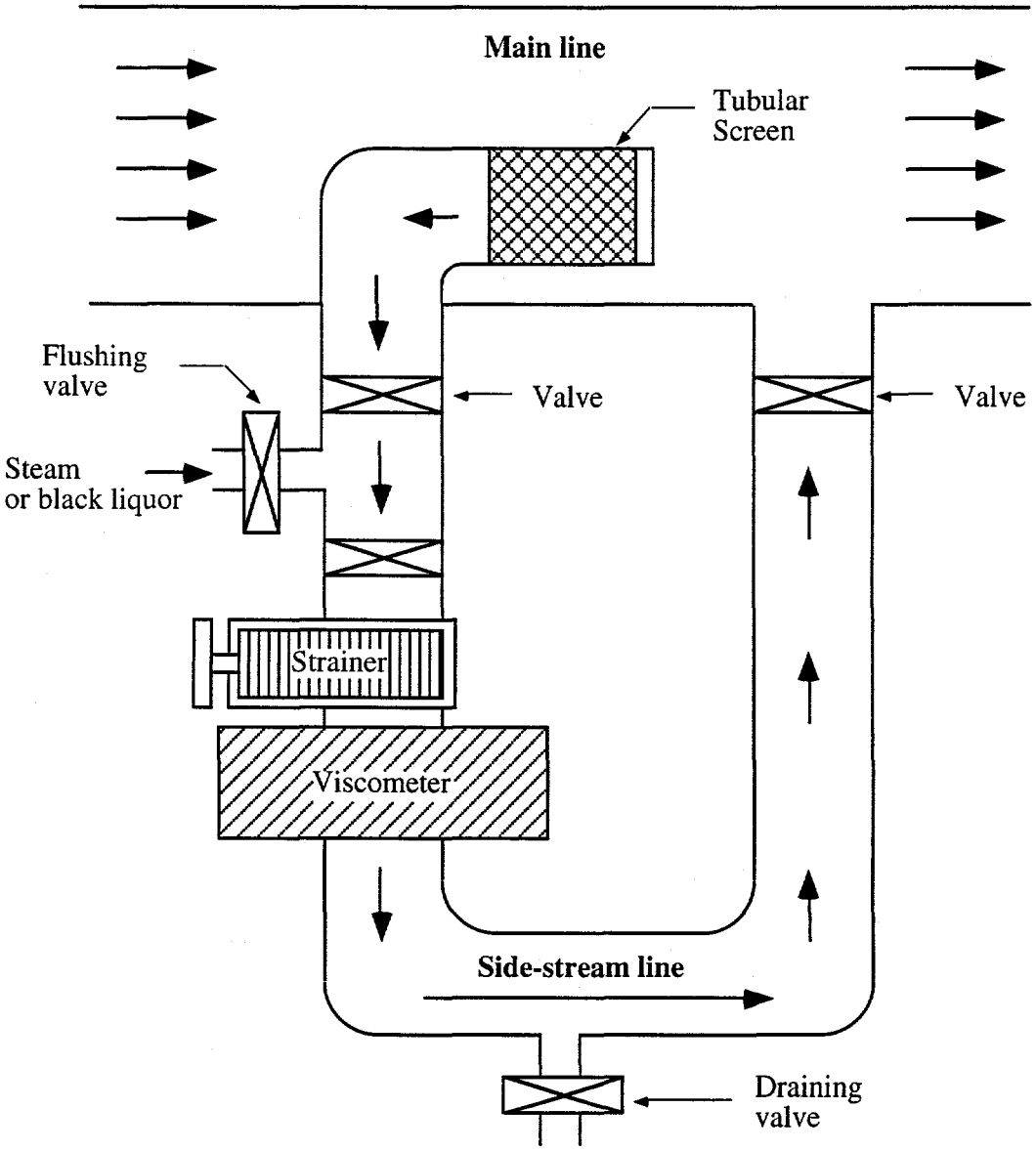


Figure 5.14. Schematic of the recommended installation of the viscometer in a side-stream line. All the side-stream pipes and the viscometer itself should be heated (using steam-tracing tubes or electrical heating tape) and insulated.

The on-line Brookfield instrument is less expensive than the Nametre and the Micro Motion instruments considered in Sections 3 and 4 of this report. The Brookfield instrument measures absolute viscosity and the cup speed can be varied to determine viscosity as a function of shear rate

for non-Newtonian fluids. The instrument is limited to measurements of viscosity above at least 10 cP. The electronics appear to be not as reliable as those for the other two instruments, and the readings sometimes experience drift due to the electronics package. The instrument has a moving part and requires a double mechanical seal with a pressurized seal fluid for use with black liquor.

6. EVALUATION OF OTHER VISCOMETERS

6.1 Magnetic Resonance Viscometer by Southwest Research Institute and Quantum Magnetics

Magnetic resonance (MR) is a non-radioactive, non-invasive, non-destructive measuring technique based on the interactions of matter with a static and radio-frequency (RF) pulsed magnetic fields. The fluid is passed through the sensor probe that contains a static magnetic field, and a pulsed RF field is applied, after which the RF signals re-emitted from the fluid are analyzed to determine viscosity from laboratory correlations [Barrall, 1996a; Barrall, 1996b; De Los Santos, 1996]. The specific configuration tested is as follows:

- Sensor: Magnetic Resonance Probe
- Control Cabinet: Controller and RF Power Amplifier

This instrument is being developed jointly by Southwest Research Institute of San Antonio, TX, and Quantum Magnetics of San Diego, CA. The instrument is currently an experimental unit, and is not yet fully developed for black-liquor viscosity applications. The objective of the experiments described in this section is to provide initial black liquor viscosity data to support the development of the MR Viscometer.

A prototype instrument was delivered to the University of Florida and was installed in the pilot flow loop by personnel from Southwest Research Institute. Data were taken on black liquors during experimental testing of instruments on-line with black liquors in the pilot flow loop at the University of Florida.

6.1.1 Principles of Operation

The nuclei in the sample fluid are arranged in random orientation until the sample comes into contact with the static magnetic field of the MR probe, where the nuclear spins align with the field direction and develop a magnetic moment. Since the alignment with the magnetic field is not instantaneous, the alignment time is characterized by the spin-lattice relaxation time (T_1), which is one parameter that may be used in correlating viscosity. The parameter T_1 is also useful for determining the time the sample needs to be exposed to the magnetic field before a measurement is acquired.

Once the sample has been magnetically aligned by the permanent magnetic field, the MR probe fires a pulse of RF energy that causes the magnetic moment of the aligned sample nuclei to tip away from the direction of the constant magnetic field (B_0). The frequency of the RF pulse depends upon the strength of the constant magnetic field and the nuclei of the sample [Barrall, 1996a; Barrall, 1996b; De Los Santos, 1996]

$$\omega_0 = \gamma B_0 \tag{6.1}$$

where ω_0 is the frequency of RF pulse (Larmor Frequency), and γ is the magnetogyric ratio

The constant γ has a different value for each nuclide, and in effect, determines the MR frequency for a given applied magnetic field. The MR Viscometer employs ^{1}H MR, with the magnetogyric ratio of $\gamma = 2.675 \times 10^8 \, {}^{rad}/_{s \cdot T}$. At the proper Larmor Frequency, the magnetic moment is shifted 90° from its equilibrium state. After the RF pulse, the nuclei realign with B_0 at its equilibrium state, and, as this occurs, the nuclei re-emit the RF energy that was absorbed. This energy is detected by the sensor coil in the MR probe. The magnitude of the MR signal depends upon the magnitude of the magnetization at the time of the pulse. The equilibrium magnetization, M_0, in an applied field, B_0, is given by [Barrall, 1996a]

$$M_0 = \frac{N\gamma^2 hI(I+1)B_0}{12\pi^2 kT} \tag{6.2}$$

where I is the 1/2 for protons, N is the total number of nuclei, h is the Planck's constant, k is the Boltzmann's constant, and T is the absolute temperature.

To provide for maximum detection sensitivity in a single-pulse MR experiment, the RF pulse must by of sufficient energy to cause the nuclei to be tipped 90° from the applied static magnetic field.

Of primary importance in the evaluation of any MR application is the signal-to-noise ratio (SNR). The SNR of an MR system is a function of a large number of variables and is highly configuration-dependent. For the purposes of this study, Southwest Research Institute and Quantum Magnetics took on the responsibility for adjusting the key operational variables. The reader may contact them for further information. For a given configuration, the SNR of an MR system can be represented by [Barrall, 1996a]

$$SNR \propto \frac{\rho V_s^{1/2} \omega_0^{7/4}}{\delta_f^{1/2}} \tag{6.3}$$

where ρ is the density of resonant nuclei in the sensitive region of the system; V_s is the volume of the sensitive region of the instrument, which is assumed to be filled with the material under study; and δ_f is the bandwidth of he MR receiver. Due to the strong dependence of the SNR on ω_0, it is desirable to operate at as high a field as is practical since higher field entails increased cost, while observing a nuclide with a relatively large magnetogyric ratio. Since γ for hydrogen nuclei is among the largest of the magnetogyric ratios and black liquor contains a high density of hydrogen nuclei, observation of the hydrogen nucleus is the natural choice for MR measurements for this fluid.

The magnetization created by the initial pulse of the MR experiment described above decays with respect to two different time constants, T_1 and T_2. Constant T_1 (spin-lattice relaxation time) is the characteristic time for the nuclear spin system to return to equilibrium with its surroundings after a disturbance. The characteristic time for the nuclear spin system to return to internal equilibrium after a disturbance is characterized by the spin-spin relaxation time (T_2). The magnitude of T_2 is dependent on the magnetic field generated by the presence of the neighboring nuclei. In a solid with large internal magnetic fields, the T_2 value is very small (10-100 μsec). In a very low viscosity fluid, such as a gas, the T_2 value is large (Sec) where there is practically no internal magnetic field. The T_2 parameter can be useful in determining information about the molecular state of the sample in question [Barrall, 1996a; De Los Santos, 1996].

Southwest Research Institute and Quantum Magnetics first considered the relationship between the two relaxation times and the viscosity of the fluid. Both of these relaxation times are affected by intermolecular and by intramolecular motion. This is due to the nature of the spin-spin and spin-lattice couplings which drive the relaxation of the magnetization. Given certain assumptions, an analytical relationship between T_1, T_2, and the molecular reorientation time, τ_c, may be determined. Since the molecular reorientation time is proportional to the ratio of the zero shear viscosity to the absolute temperature, there should be a dependence of the MR relaxation parameters to viscosity [Barrall, 1996a; De Los Santos, 1996].

The analysis sequence utilized in the pilot flow loop was designed to gather as much information as possible, since a particular correlation has not yet been developed. In particular, this plan required long residence time (2-4 minutes) between measurements. On the other hand, for a practical application of the fully developed MR Viscometer, the measurement time should be in the milliseconds range. Since this viscometer is an experimental prototype, the objectives for our testing of this device are different from those of the on-line viscometers discussed in previous sections. Data acquired by the MR Viscometer was sent to Southwest Research Institute and to Quantum Magnetics to support the development of a correlation for black liquor viscosity utilizing

MR principles, and to determine the effects of flow rate, liquor type and contaminants.

6.1.2 Experimental Overview

Table 6-1 lists the experimental runs that were performed at the University of Florida pilot flow-loop using the Magnetic Resonance Viscometer.

Table 6.1. Black liquor types and solids content tested with the SWRI-Quantum Magnetics MR Viscometer.

Georgia Pacific	Canadian Forest	Chesapeake	GP & Canadian Forest Mix
55.84% 65.51%	57.73%		67.72% 71.38% 73.40%

The instrument was installed late in the evaluation phase and only a few experimental trials were performed. The main goals with this instrument were:

- To determine the MR parameter that best correlates with black liquor viscosity and solids content.
- To examine the effects of flow rate on the MR measurement.
- To determine the effects of various black liquor types on the viscosity correlations.

The control unit of the MR viscometer includes its own DAQ (Data Acquisition) system where all the MR parameters are collected and stored on disk. In turn, the sample temperature and the constant T_1 are retransmitted to the UF DAQ System. Time synchronizing of the two sets of data was very difficult, since the only flow-loop variable measured by the MR Viscometer was the sample temperature. Since the instrument has a 2-4 minute lag time between measurements, the flow rate was averaged from the number of flow rate samples collected by the UF DAQ System in the Pilot Plant divided by the number of samples taken by the MR instrument. It was found that the temperature measurement taken by the MR Viscometer is not very accurate; hence the Viscometer Line RTD TT7 (*cf.* PI&D diagram Figure 9.1) was used instead. A temperature correlation between the MR sample temperature and TT7 was developed and this correction was then used to correct the sample temperature data from the MR instrument.

For measurements with no flow, the UF DAQ System was turned off and the MR Viscometer was set to automatic control. The MR control unit is capable of sending signals to control the relay

isolation valves V7 and V8 (*cf.* PI&D diagram Figure 9.1), and of gathering information automatically. Before the MR instrument was allowed to take control of the viscometer line, the operating conditions of the viscometer line were fixed and the isolation control valves were set for remote-control operation. The delay time in the MR Viscometer DAQ program, which permitted flow through the viscometer line to provide a fresh sample for analysis, was set and then the program was activated. Flow entered the viscometer for the set delay time. Once this time had expired, the isolation valves closed for a zero-flow run. Once the analysis was completed, the delay time was reset, and flow through the viscometer line began again.

Selected Experimental Results

The data that was gathered during the experimental trials were first analyzed at UF where the files from the MR Viscometer were synchronized with the UF DAQ System. The data files were then sent to Southwest Research Institute and Quantum Mechanics for further analysis and development of MR-viscosity correlations. Southwest Research Institute and Quantum Magnetics investigated correlations between the viscosity of the black liquor sample and all of the measured MR parameters (relaxation time and amplitudes). They found that the constant T_1 and the long component of the constant T_2 are well correlated with the black liquor viscosity, but that the relative amplitudes of the relaxation times and the short component of the constant T_2 had poor correlations with viscosity. Viscosity as a function of relaxation time was fitted by using nonlinear regression to the model [Barrall, 1996a]

$$\eta_H = c_0 + \frac{c_1}{T_m} + \frac{c_2}{T_m^2} + \frac{c_3}{T_m^3} \tag{6.4}$$

where T_m refers to either T_1^{sr} (saturation recover), T_1^{osir} (one-shot inversion recovery), or to T_2; with c_i represents the correlation coefficients, and η_H is the viscosity of the black liquor determined in the laboratory using a Haake Rotational Viscometer. Equation 6.4 does not represent a true physical relationship between viscosity and MR relaxation times; the model was simply chosen to provide a empirical relationship between these variables.

The coefficients of the model (6.4) were found by linear regression for the three possible choices of T_m. The resulting correlations are plotted in Figure 6.1, for three black liquor types, namely, a Canadian Forest liquor with 57.73% solids (shown as CF 57.73% in the figure), a Georgia Pacific liquor with 55.84% solids (GP 55.84%), and a mixture of Canadian Forest and Georgia Pacific liquors with a 73.40% solids content (GP & CF 73.40%). The T_1^{osir} constant appears to correlate best with viscosity, followed by T_1^{sr}, and then by T_2. The relative difference with respect to the Haake-measured viscosities for the models plotted on Figures 6.1 A, B, and C,

are respectively shown in Figure 7.2 A, B, and C. As a measure of overall error in the predicted viscosities, the average root mean square deviation from the laboratory reference viscosities of the model were calculated. The results are reported in Table 6.2 [Barrall, 1996a].

Due to the design of the sensor, the flow of black-liquor may have a considerable effects on the relaxation times and viscosity. Once the sample has become magnetically aligned, the MR probe fires a pulse of RF energy which causes the magnetic moment to tip away from the direction of the constant magnetic field B_0. As the sample realigns itself with B_0, the RF energy that was absorbed is re-emitted and detected. However, in a flowing sample stream, the sample may have had time to fully realign with B_0 before the sample leaves the measuring chamber. This would cause an erroneous shorter relaxation time as long as the relaxation decay is longer than the necessary residence time. The constant T_1^{osir} is less affected by increasing flow rate, primarily because T_1^{osir} is consistently shorter than the other relaxation times.

Table 6.2. Average root mean square deviation of the predicted viscosity from the actual viscosity.

Black Liquor	Constant T_m in Model (6.4)		
	T_1^{sr}	T_1^{osir}	T_2
CF 57.73%	2.1%	1.1%	4.6%
GP 55.84%	5.3%	1.7%	12.8%
GP 65.51%	4.7%	2.2%	8.8%
GP&CF 67.73%	4.9%	2.3%	13.0%
GP&CF 71.38%	9.5%	2.5%	18.3%
GP&CF 73.40%	10.0%	1.8%	11.4%
Combined	59.4%	40.0%	52.4%

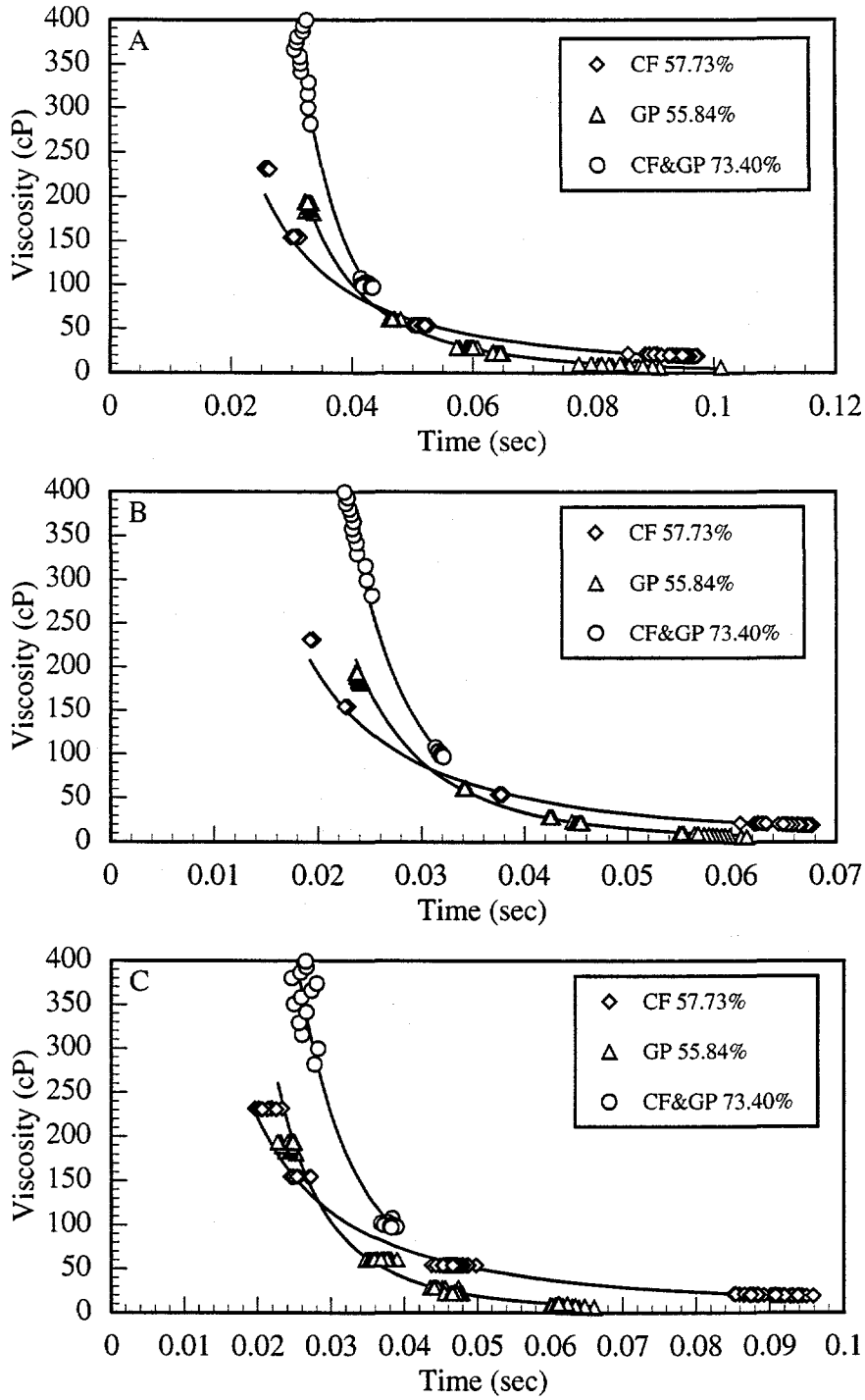


Figure 6.1. Relaxation times for the magnetic-resonance viscometer with selected black liquors for three experiments: (A) T_1^{sr}, (B) T_1^{osir}, and (C) T_2.

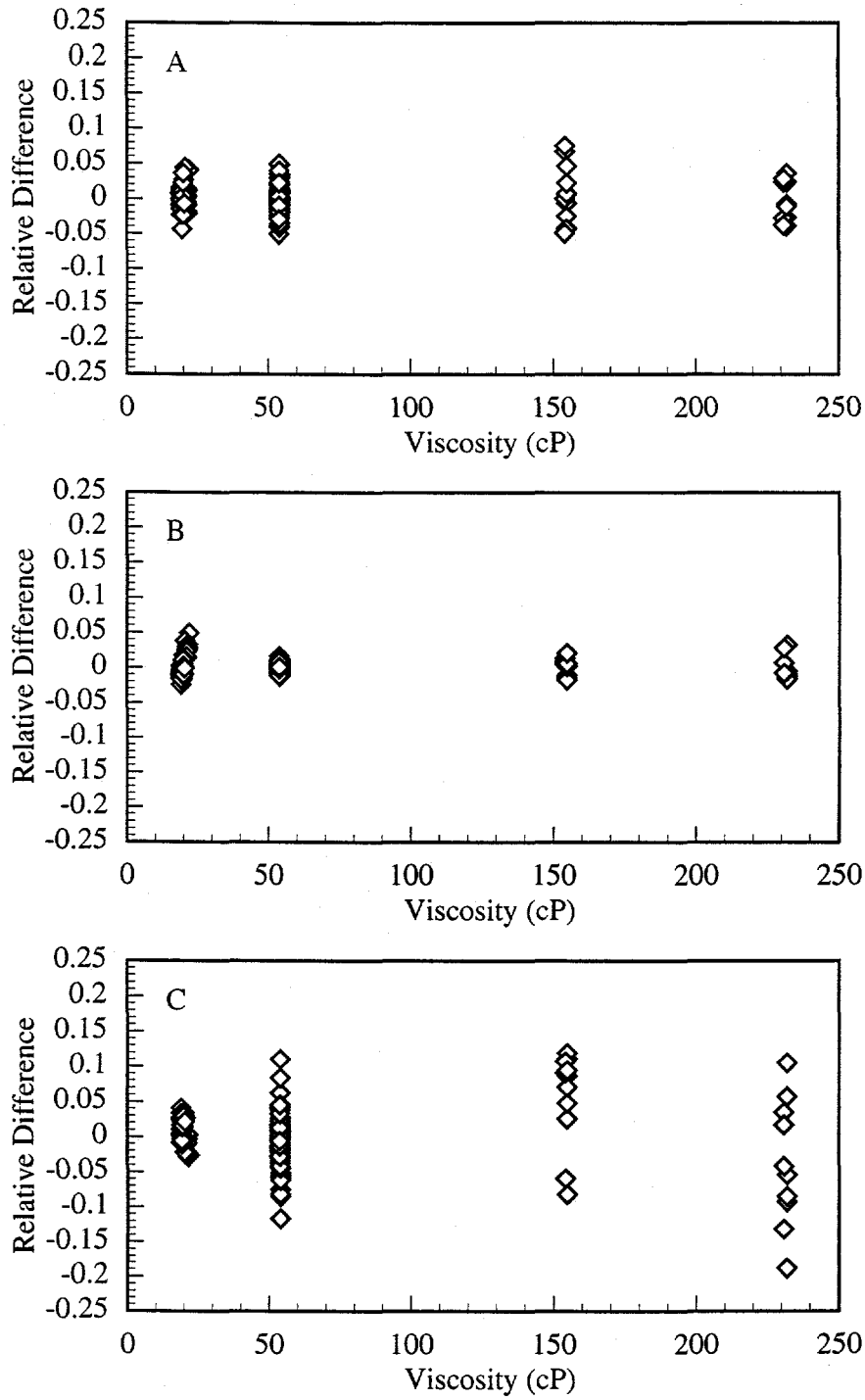


Figure 6.2. Relative difference between the viscosity determined by the magnetic-resonance viscometer and the viscosity determined by a laboratory reference for a Canadian Forest black liquor with a solids content of 57.73%: (A) T_1^{sr}, (B) T_1^{osir}, and (C) T_2.

6.1.3 Specific Conclusions and Recommendations Regarding this Instrument

The MR Viscometer is a new and unique method of indirectly measuring viscosity and possibly solids content. The potential advantages of this technique are very significant, namely, the MR viscometer is non-invasive with no moving parts in the process stream, non-destructive to the sample, and non-radioactive. The MR instrument is an experimental prototype and therefore its performance for practical black-liquor applications was not evaluated. The intention of the study was to provide initial black liquor viscosity data to support further development.

Preliminary work has demonstrated that useful correlations exist between molecular properties and the viscosity. Further work needs to be done to determine if it is possible to develop a model that is independent of black liquor type, and if different MR properties may yield improved correlations for viscosity and solids content. Results demonstrated that a correlation relating relaxation times to fluid viscosity as measured on-line could indeed be developed for a liquor of constant composition; however, black liquor composition was shown to affect the correlation. Results were very reproducible for any one liquor and the response signal exhibited sufficient range for good precision in determining viscosity. The instrument could possibly be used for on-line viscosity measurement of a fluid of constant composition, but not for black liquor, since composition is known to vary considerably and experiments have shown that the relation between relaxation time and viscosity for varies with these composition changes. A mill trial was not recommended, even though this instrument showed promise, due to cost and the demonstrated variation in response due to changes in black liquor composition.

The tests conducted at the University of Florida demonstrate that the instrument can be used to determine viscosity of a fluid under harsh conditions with no restrictions on flow through the instrument. The instrument very possibly could be used as a combined viscometer and analytical instrument on-line. This could be very valuable in a number of applications, such as applications in the food industry. However, considerable development would be required for a specific application, and such development was beyond the scope of our work.

The long residence time between measurements can be reduced to milliseconds if only a single molecular parameter is to be measured and the effects of flow rate can be reduced or eliminated through redesigning the sensor to account for the measured relaxation decay.

6.2 Cambridge Applied Systems Sliding-Element Viscometer

The Cambridge Applied Systems sliding-element viscometer was chosen as the best candidate viscometer of this type. An element is driven back and forth in a tube by an alternating electrical

field, and the element position is determined as a function of time as it passes through the fluid by an LVDT. The viscosity is determined from the element velocity with the assumption that the fluid behaves as a Newtonian fluid. This viscometer cannot be used to determine viscosity of a non-Newtonian fluid.

The available instrument had inadequate temperature control for our purposes, and was not arranged for liquid flow through the instrument; fluid was exchanged in the instrument by the pumping action developed by the sliding element as it passed through the fluid contained in the instrument tube. Based upon our recommendations, Cambridge Applied Systems redesigned the temperature control system. Two separate heater coils were included with a balancing variac. These were connected to a temperature control. With this system, the temperature of the viscometer could be maintained uniform and controlled to within ±0.1°C from 40 to 150°C. We provided Cambridge Applied Systems with details of mechanical modifications to permit flow through the instrument with provisions for isolation of the fluid within the instrument for measurement. Cambridge Applied Systems manufactured a prototype incorporating these changes which we purchased for our laboratory.

The modified instrument that we purchased for the laboratory was used extensively in the laboratory to determine viscosities of black liquors that behaved as Newtonian fluids and that had been screened through a 40-mesh screen to remove large particles. The instrument proved to be quite accurate. The measurement range could be changed by changing the diameter and mass of the sliding element. Measurements were made in the laboratory at temperatures up to 135°C. This instrument, as modified for our program, is an inexpensive and accurate device for measuring viscosity of Newtonian fluids at up to 130°C and viscosities ranging from 0.8 to about 300 cP, if the fluids contain no suspended particles that will not pass a 40-mesh screen.

Based upon our laboratory experience, we requested that Cambridge Applied Systems supply us with a prototype instrument for on-line measurements that would include mechanical and control provisions for automated loading and purging of the instrument measuring chamber. Cambridge Applied Systems was not able to supply the requested prototype. The laboratory instrument purchased for this study was modified to permit manual manipulation of liquor flow through the instrument and was then installed in the pilot flow loop for testing. The instrument was then tested with black liquors that were Newtonian and that exhibited viscosities typical of low to medium solids concentrations black liquors (up to about 55% solids) that contained no suspended solids that would not pass a 40-mesh screen. The questions to be answered were interference from line vibrations, minimum residence time of fluid in the chamber necessary for measurement, and the effects of fouling. Tests indicated that system vibrations did not affect readings and that at least 3

minutes residence time of fluid in the measuring chamber was required for accurate measurement. Fouling could not really be evaluated with the tests that were conducted; the liquor had been screened and the experimental time for on-line measurements was limited, since most of our work was concentrated on studies of other instruments. It is worth noting the response of this device is very sensitive to changes in the clearance between the instrument chamber tube and the sliding element; therefore, this device has to be very susceptible to fouling.

6.2.1 Specific Conclusions and Recommendations Regarding this Instrument

The Cambridge Applied Systems sliding element viscometer, as modified for laboratory use in this program, is recommended as an inexpensive instrument for laboratory use to determine viscosities of screened (screened through 40-mesh or less) black liquors that behave as Newtonian fluids. Measurements can be made under pressure at temperatures up to 150°C. Fouling is not a problem, because the instrument can be disassembled, inspected, and cleaned, if necessary.

The Cambridge Applied Systems sliding-element viscometer is unproven as an on-line viscometer for use for measurements of black liquor. At best, its use is restricted to determine viscosity of liquors at concentrations below about 50% solids. It is undoubtedly susceptible to problems due to fouling, and estimated liquor residence time in the measuring chamber of at least 3 minutes for accurate measurement is considered as excessive.

6.3 Stevens Institute of Technology Capillary Viscometer

A development team working at the Stevens Institute of Technology produced the design of a Capillary Viscometer conceived to utilize a new principle of pressure-drop (Δp) measurement. According to the design, the device was to be mounted on the process line and the sample was to be drawn into the measuring chamber by the upward stroke of a piston. On the downward stroke of the piston, the sample was to be forced back into the main stream, and the viscosity could be determined through the measurement of the Δp across a section of the tube and velocity of the piston [Bird, *et al.*, 1960].

Excellent initial contact was established between the University of Florida and Dr. Starita, the proponent of the new design at Stevens Institute of Technology. In spite of the extensive interactions between the two groups, the Stevens Institute of Technology team was not able to deliver to the University of Florida a prototype laboratory or on-line model. Dr. Starita had very diligently resolved numerous challenges posed by the new design, but the project was abandoned when he later left Stevens Institute of Technology and the administration of the Institute abandoned all developments. For this reason, no evaluation work for this instrument was done at the

University of Florida.

Relevant details of the design are given in the following section to provide the reader with information that might be useful for the understanding of new instruments using this technology that may become available in the market in a near future.

6.3.1 Principles of Operation

In the early stages of this program, very few examples of the use of capillary viscometers for on-line measurements were available; only Seisor and Rheometrics offered these and the instruments offered were useful only for highly viscous polymer melts. Dr. Joseph Starita was aware of some new technical developments that could possibly be used to measure very small pressure differences with high accuracy. While president of Rheometrics, Dr. Starita agreed to develop a new type of capillary viscometer adapting this technology. Shortly afterwards, Dr. Starita sold his controlling interest in Rheometrics and joined Stevens Institute of Technology. Dr. Starita gained approval to continue development at Stevens Institute.

This device would provide a direct measure of Δp, piston velocity, and temperature. The viscosity would be determined from measured values of Δp and shear rate as follows [Bird, *et al.*, 1960]

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{R^2 \Delta p}{8Lv} \tag{6.5}$$

where L is the length of measuring chamber, R is the radius of measuring chamber, and v is the velocity of the piston

This viscometer, like the Cambridge Applied Systems device, takes discrete (as opposed to continuous) measurements of viscosity. The residence time for this device should be very small and the problem of achieving thermal equilibrium with the process line should be easier to handle than with the Cambridge Applied Systems instrument. In addition, this viscometer should be able to measure the viscosity of non-Newtonian fluids by varying the velocity of the piston which in effect varies the shear rate.

The heart of the instrument would be a tube with two very thin wall sections separated by a short distance. Optical fibers would be wound about each of the thin sections and light passed through each fiber. The difference in stresses on the walls of the very thin sections due to the pressure difference would result in a difference in fiber length and a difference in time for light to pass through the optical fibers. This could be related to the pressure drop over the length of tube

between the very thin tube wall sections. This technology had been developed by the Navy for measuring very small differences in pressure at very high static pressure. Additional development was necessary to adapt this for use on a tube made of corrosion resistant material and maintained at a temperature up to at least 150°C.

Dr. Starita's concept was to use a reasonably long tube (24-30") with a short measuring section (about 6") located at the end of the tube to be connected to the pipe carrying the process fluid. The capillary would contain a reciprocating rod with seals that would wipe the walls of the capillary. The fluid to be measured would be drawn into the tube by withdrawing the rod. The direction of the rod would then be reversed and the rod driven through the capillary at a constant controlled rate. During discharge of the fluid, the pressure drop across the short measuring section would be measured. Knowing the geometry, the pressure drop, and the flow rate, one could calculate the viscosity.

Conceptually, this viscometer would be ideal for our intended application. The instrument could be connected directly to a main flow line, such as the ring header line, but could easily be isolated for cleaning and repair without interruption of the main flow. The reciprocating rod with its tight seals would make the device self cleaning. Since the instrument would be isolated from the main flow, the main flow need not be laminar. Also, since the rod speed could be varied, measurements could be made at different shear rates so that viscosities of non-Newtonian fluids could be determined. However, its use would be restricted to applications for black liquors with higher viscosities (approximately 150 cP at 3000 sec^{-1} and 300 cP at 800 sec^{-1}).

Significant development advances were made by the Stevens Institute of Technology group. The problem of adhesion of the optical fibers to the very thin sections of the tube walls remained to be solved, but other mechanical features had been designed and some components were purchased with program funds to support this development. Unfortunately no further work was done at Stevens Institute of Technology to complete the development and deliver a prototype for testing at the University of Florida

6.3.2 Specific Conclusions and Recommendations Regarding this Instrument

The Stevens Institute Viscometer was not delivered for evaluation trials in the University of Florida pilot flow loop. By utilizing a unique method in determining the Δp, theoretically the instrument should have been capable of measuring very small pressure drops for fully developed flow; however, this technology remains unproven for black liquor applications. We anticipate that the Stevens Institute Viscometer would be particularly useful in high viscosity applications so that a sufficient pressure drop would be generated across the measuring chamber. The unanswered

questions for this instrument include: What is the residence time for a measurement, what is the sampling rate or time between measurements, what are the possible effects of fouling in the instrument, and what is the reliability of the Δp measurement. This instrument design has the potential of being a valuable rheological device for industrial applications and it is hoped that further development would be done in the future by a different sponsor coupled with a comprehensive evaluation program to assess the suitability of the instrument for practical applications.

7 OVERALL CONCLUSIONS AND RECOMMENDATIONS

7.1 Laboratory Viscometers for Black Liquor Applications

Laboratory instruments for measuring viscosity of black liquors were thoroughly explored during this and earlier work. Kraft and sulfite liquors always behave as Newtonian fluids at solids concentrations below 50%. At these low concentrations, a correlation based upon principles of corresponding states relates the reduced viscosity of the liquor (the liquor viscosity divided by the viscosity of water at the same temperature) to the ratio of solids concentration and absolute temperature for any liquor of constant solids composition.

Viscosity measurements made with glass capillary viscometers below the liquor boiling point at several concentrations can be used to establish a correlation that can be used to predict the liquor viscosity at any temperature and solids concentration below 50%. At <u>least</u> three measurements at different temperatures and solids concentration are required.

Laboratory instruments for other, more direct measurements have been tested and proven. The Cambridge Applied Systems sliding-element viscometer, as modified for use in this project, can be used for direct measurement of viscosity for liquors with low solids concentration (liquors that behave as Newtonian fluids) at temperatures up to 150°C, *i.e.*, well above the normal boiling point of the liquor. The sample size required is small (less than 50 cc), and measurement is rapid (less than 10 minutes). Liquor samples must be screened, however, to remove suspended particles that would interfere with the movement of the sliding element. The instrument is rugged and easy to operate. With two elements to give a range of about 0.8 to 100 cP, the instrument cost is less than $6,000.

The Nametre laboratory viscometer can also be used for direct measurement of viscosities for liquors at low solids concentrations when the instrument zero is accurately calibrated for the effect of temperature. When housed in a sealed, jacketed vessel, the instrument can be used to measure viscosity accurately at pressures up to 300 psia. Alternatively, it can be used to measure viscosities of liquors at temperatures below the normal boiling point of the liquor by placing the element in the liquor in an open container. The sample size required is large (about 1.5 liters) and the instrument cost is about $35,000. However, the viscometer is rugged, has many uses, can be used to

measure an extremely wide range of viscosity, and is unaffected by the presence of suspended particles.

Measurements of viscosities of liquors at solids concentrations above about 50% solids usually require different instruments. If the liquor is known to behave as a Newtonian fluid and if large suspended particles (above about 80-mesh) are not present, glass capillary viscometers and the Cambridge Applied Systems sliding element viscometer described earlier can be used, subject to the same restrictions. The Nametre laboratory viscometer can also be utilized, and restrictions on its use are not as severe as those for use of glass-capillary or sliding-element viscometers.

The Brookfield laboratory rotating cup viscometer with Fann geometry has proven to be an effective instrument for measurement of liquor viscosity at high solids concentrations and at temperatures up to 150°C. With cup rather than bob rotation, secondary flows are retarded and measurements can be made up to about 1200 sec^{-1}. The cup rotational speed can be adjusted to vary the shear rate. With proper calibration for end effects, the instrument can be used to measure apparent viscosities as low as 10 cP with an accuracy better than ±10%. The sample size required for a measurement is less than 30 cc. With proper training, one can operate the instrument easily without fear of damaging the moving parts. Non-Newtonian relations for viscosity can be developed easily, and reliable measurements can be made on fluids containing suspended particles that pass a 12-mesh or finer screen. The instrument cost with a temperature bath accessory is approximately $30,000.

A Haake pressurized rotating cup viscometer with jacketed cup or with the cup immersed in an oil bath can be used to measure viscosities of liquors at high solids concentrations and at temperatures up to at least 150°C. The instrument can be used to measure viscosities at up to about 800 sec^{-1} without development of secondary flows, and it can be used to measure viscosities as low as 10 cP with an accuracy of about ±2% with proper calibration for end effects. It can also be used for measurements of fluids containing suspended particles subject to restrictions similar to those that apply to the Brookfield laboratory rotating cup viscometer. The Haake instruments costs approximately $75,000 with temperature bath controls.

Both the Brookfield and Haake instruments can be used to determine non-Newtonian viscosity behavior of liquors over ranges of approximately 200-1200 sec^{-1} or 80-800 sec^{-1}, respectively. The Nametre instrument can be used to determine if viscoelastic effects are significant; however, measurement can be made at only one shear rate that can only be approximately determined.

7.2 Recommended Viscometer Deployment Points in a Mill

On-line viscometers can be installed at different locations in the recovery area of a paper mill. In general, monitoring the viscosity over time should prove helpful in identifying trends in black liquor viscosity, and eventually lead to a correlation between such trends with difficulties experienced in the firing in the recovery furnaces. The deployment point of the viscometer depends on the specific process-control objectives sought; however, two locations appear to be particularly promising as discussed below:

1. *Installation in the ring header lines*

   This installation is best suited for eventual closed-loop control of the black liquor firing in the recovery furnaces since the sensors make a direct measurement of viscosity, which is the variable of interest. Note that the common practice is to control the temperature (rather than the viscosity) at this point; however, such control strategy is not optimal because the viscosity can vary widely at constant temperature (due to variations of solids contents, for example). Clearly, it is more desirable to control the viscosity by adjusting the temperature.

2. *Installation at the exit of the concentrator or the multi-effect evaporator chain*

   This type of installation would permit adjusting the solids content of the liquor exiting the concentrator or evaporator units until a target desired viscosity is attained. The beneficial consequence would be the production of a black liquor of more predictable viscosity which in turn should assist in attaining better controlled firing of the recovery furnace.

7.3 On-Line Viscometers for Black Liquor Applications

At least one example of an instrument operating on one of five principles—rotating element, vibrating element, sliding element, pressure flow through a tube, and fluid relaxation after exposure to a pulsed magnetic field—were tested on-line in the pilot flow loop with black liquor over wide ranges of solids concentration, temperature, and flow rates. The following subsections provide a summary of conclusions and recommendations regarding each of the instruments tested.

7.3.1 Nametre Torsional-Oscillatory Viscometer

Conclusions

In tests conducted at the University of Florida pilot plant the Nametre Torsional-Oscillatory Viscoliner viscometer produced measurements that tracked within ±10% the laboratory reference at high viscosity (greater than 175 cP) and also at low viscosities with low solids (less than 50% solids). The instrument was unaffected by particles smaller than those that would pass a 40-mesh screen, by varying flow rates, or by second phases (such as air) in the process fluid. The Nametre viscometer provides a direct measure of force and does not require laboratory calibration. The instrument's output signal is the product of the density times the viscosity of the fluid, therefore, an independent on-line measurement of density is required to obtain an absolute viscosity measurement. The instrument tested at the University of Florida pilot plant experienced fouling (deposit of solids on the sensor probe) when measuring high-solids liquors; this eroded the instrument's ability to produce reproducible measurements. In most cases, steam purging the viscometer line was sufficient to clean the sensor probe and restore the performance of the instrument. The fouling problem was completely eliminated in the mill trials after the manufacturer introduced a design to streamline the sensor probe.

The pilot-plant studies of the Nametre instrument revealed a number of appealing advantages. First, this instrument has demonstrated good accuracy, precision, and tracking when the sensor probe is free of fouling. Second, there were no effects of particles smaller that 40-mesh. Third, the viscosity measurement is independent of flow rate. Fourth, the instrument is unaffected by second phases (such as air entrainment) in the process fluid. Fifth, this instrument has the widest operational range of the viscometers evaluated. Finally, this viscometer has an excellent electronics package ready for on-line deployment; in addition, the control unit is capable of handling multiple sensors.

The tests conducted at the mill of St. Laurent Paper Products Corporation in West Point, Virginia, were done with a Nametre Viscoliner instrument with a sensor probe streamlined with an inclusion angle of 90°. The instrument was in operation for approximately six months without experiencing any malfunctions and it remained free of fouling with virtually no maintenance. The viscosity measured on-line differed from laboratory references by less than ±16%.

A concern that must be kept in mind with using this instrument includes the fact that an on-line density measurement is necessary if there is a need for measuring absolute viscosity (rather than the product of viscosity and density). Since in comparison with the viscosity, the density of black

liquor is a much weaker function of temperature and of solids-content, the viscosity-product measurement should be a useful indicator of viscosity variation in most on-line applications. A second concern is that for accurate viscosity measurement the unit requires a zero correction to compensate for temperature effects; however, such corrections are insignificant when measuring liquors of high viscosity. Finally, background vibrations must be taken into account for optimal installation to guarantee that the sensor probe dynamics is unaffected by the surrounding environment.

Recommendations

The torsional oscillatory viscometer can be recommended as an instrument effective for the on-line monitoring black liquor viscosity. The instrument is expected to operate reliably with minimal maintenance. It is recommended that the viscometer be installed in a side stream in order to avoid interruption of the process flow in case of needed repairs or maintenance. This viscometer can handle a very wide range of viscosity; in fact, with one electronics package the Nametre viscometer is able to monitor several sensor locations even though the liquor viscosity may be very different at each of these locations. For specific installation recommendations see Section 3.7.

7.3.2 Micro Motion Capillary-Coriolis Viscometer

Conclusions

In tests conducted at the University of Florida pilot plant the Micro Motion capillary-coriolis viscometer was able to track the viscosity values within ±10% of the laboratory reference at high viscosities (> 150 cP) and at lower viscosities with low flow rates (< 0.5 gpm) through the instrument. There was no evidence of any fouling or affect of particles smaller than 40 mesh on measurements during the experimental runs. The Micro Motion viscometer provides a direct measure of mass flow rate, density, and pressure drop, along with measurements of volumetric flow rate and sensor temperature.

The pilot-plant studies of the Micro Motion instrument identified a number of excellent features. First, this instrument has demonstrated good accuracy at high viscosities (> 150 cP) during testing at the University of Florida pilot plant. Second, the instrument demonstrated excellent reproducibility at all viscosity ranges tested in the pilot plant. Third, there were no significant fouling effects observed. Fourth, there were no effects of particles smaller that 40-

mesh. Fifth, it may be possible to use this instrument for non-Newtonian fluid measurements at high viscosities by varying the flow rate through the instrument. Sixth, the viscosity measurement for a Newtonian fluid is nearly independent of flow at high viscosities. Seventh, this instrument provides excellent measurements of density and mass flow rate which are independent of flow rate. Finally, this viscometer has an excellent response time (fastest of all the instruments evaluated).

At flow rates lower than 0.5 gpm the instrument measured the viscosity accurately even at lower viscosities (80 cP). At flow rates greater than 0.5 gpm the viscometer developed a linear increase in the measured signal, perhaps due to the onset of secondary flows; consequently, if the instrument must be installed under higher-flow conditions the linear trend must be compensated for through the use of appropriate empirical correlations. Note however that the Micro Motion viscometer is capable of accurate and repeatable viscosity measurements even at low viscosities if the mass flow meter is properly sized and if the side stream flow rate is maintained at a level below the critical flow rate specified by the manufacturer.

Mill tests conducted at the facilities of Canadian Forest Products in Prince George, British Columbia, Canada, were attempted through an extended period of time. Improper installation of the instrument and of the side stream prevented the extensive acquisition of data. Three weeks of continuos operation were eventually recorded yielding viscosity measurements that agreed within ±19.5% of laboratory reference values. The density measurements produced by the instrument were excellent, with an accuracy better than ±0.2%. The instrument eventually stopped operating due to pluggage caused by an inordinate amount of large-diameter solid particles that appeared in the black liquor, leading to the obstruction of tubes of 1-inch diameter. This failure was caused by an improper design of the side stream which did not feature screening to protect the instrument from such large particles.

A concern of relevance when using the Micro Motion capillary-coriolis instrument is that laminar flow must be maintained through the side stream for accurate viscosity measurement. The instrument manufacturer designs the diameter of the coriolis tubes and specifies the maximum flow rate in order to preserve flow laminarity for the viscosity range of interest. Consequently, the flow rate through the instrument must be controlled for accurate viscosity measurements. Another issue that must be kept in mind is that this instrument requires at least a one-point calibration from a laboratory reference (typically a standard calibration oil); however, this service is provided by the manufacturer as part of the instrument design package.

Recommendations

In addition to its capabilities for measuring the density and mass flow rate of flowing black

liquors, the Micro Motion capillary-coriolis viscometer can be recommended for use as an on-line monitor of viscosity. The instrument must be installed in a side stream where the flow rate is controlled to guarantee laminar flow through the instrument. For specific installation recommendations see Section 4.7.

7.3.3 Brookfield Cylindrical-Rotary Viscometer

Conclusions

In tests conducted at the University of Florida pilot plant the Brookfield Cylindrical-Rotary viscometer tracked within ±10% of the laboratory references throughout most of the experimental runs. Even though there was some concern of fouling with this design, there was no evidence of fouling or interference from particles smaller than 40 mesh through out these trials. Since this instrument makes a direct measure of force, laboratory calibrations are not necessary for a viscosity measurement for Newtonian fluids, but laboratory verification would ensure accuracy. The instrument displayed a tendency to output a lower viscosity reading than the laboratory reference at higher flow rates (> 2.5 gpm) with the configuration used. Maintaining a relatively low flow rate should not be difficult when operating in a side stream. The control unit demonstrated an offset in both the Viscosity-Zero and Rotational Speed Voltage which was manually compensated for by the operator. The electronics package needs to be upgraded with an auto-zero for the offsets and on-line viscosity calculation.

The pilot-plant studies of the Brookfield cylindrical-rotary viscometer document a number of advantages of this design. First, this instrument has demonstrated excellent accuracy, precision, and tracking throughout the viscosity ranges encountered during the evaluation phase in the University of Florida pilot plant. Second, low flow rate conditions do not affect the accuracy of the instrument. Third, there were no significant fouling effects observed. Fourth, there were no effects of particles smaller that 40-mesh. Fifth, this instrument can be useful for non-Newtonian fluid measurements without changes in flow rate because of the short rotational speed transient. Finally, this viscometer does not require a laboratory reference for calibration.

Some concerns regarding the use of the Brookfield viscometer are the following. First, there is no temperature sensor inside this instrument; therefore, comparison with laboratory references are more difficult. Second, this instrument is limited to low flow rates for accurate measurements. Third, the unit uses a double-flush seal which can require additional maintenance. Fourth, the instrument is limited to viscosities above 10 cP with the configuration considered. Finally, the

current electronics package is considered to be unsatisfactory for installation in an industrial process.

Even though the instrument proved to be successful in an extensive trial period at the UF Pilot Plant, the mill trial results at the Georgia Pacific facilities did not show in a conclusive fashion that the instrument can sustain reasonably long periods of continuous operation. Limited data collected during the second and third mill trials suggests that accurate measurements can potentially be obtained on-line; however, the small amount of data available does not provide the basis for a conclusive verification of the accuracy of the instrument.

Recommendations

This instrument did not perform successfully in a mill trial, and therefore there is a lack of strong evidence to support the claim that the viscometer would perform adequately in an industrial environment. However, given the good performance of the instrument in the University of Florida pilot plant environment, we believe that this instrument can successfully measure the viscosity of black liquor in an industrial location if (1) the liquor stream is relatively clear of large suspended particles, (2) an 8-mesh or 12-mesh straining or screening of the liquor is provided (along with the capability of backflushing or cleaning the strainer or screen), (3) the instrument is positioned in a side stream line with provisions for heating the line to prevent freeze-ups, and (4) the instrument is sized for measurements above 10% of maximum torque and is properly calibrated. The Brookfield viscometer is definitely *not recommended* for measurements with liquors containing significant quantities of suspended solid particles of large diameter.

7.3.4 Southwest Research Institute/Quantum Magnetics MR Viscometer

Conclusions

The instrument evaluated was an experimental unit still under development. The MR viscometer is a new and unique method of indirectly measuring viscosity and possibly solids content. Preliminary work has demonstrated that correlations between molecular properties and viscosity is possible. Further work needs to be done to determine if it is possible to develop a model that is independent of black liquor type and if different magnetic-resonance properties may yield improved correlations for viscosity and solids contents.

Recommendations

Given the significant potential of this instrument for black liquor applications, further development of the technology leading to a viable instrument is encouraged.

7.3.5 Cambridge Applied Systems Sliding -Element Viscometer

Conclusions

An on-line prototype viscometer was not delivered for evaluation, but a laboratory bench (*i.e.,* off line) version employing the same principles of viscosity measurement was examined in the flow loop. The Laboratory Cambridge viscometer proved to be a reliable and valuable laboratory instrument during these trials, but its use is limited to Newtonian fluids. When conducting trials with the laboratory instrument there was little question whether the device would properly measure low-to-medium solids black liquor; however, what was not known was the minimum sample residence time for the device, effects of fouling, and interference from line vibrations. Additionally, a prototype on-line instrument would need automated loading and purging of the measuring chamber which would require further evaluation. The laboratory device was installed on-line at the University of Florida pilot plant allowing the assessment of the residence time and sensitivity to vibrations. The instrument demonstrated no adverse affects from line vibration and required an average residence time of 3-5 minutes. Further questions could only be answered with an on-line prototype.

Recommendations

The instrument made available by Cambridge Applied Systems for evaluation studies cannot be recommended for on-line use on-line with black liquor systems due to the shortcomings discussed above. However, the laboratory version of this instrument demonstrated great potential, and it is therefore recommended that the manufacturer pursue further development of an on-line prototype for black liquor applications.

7.3.6 Stevens Institute of Technology Capillary Viscometer

Conclusions

The Stevens Institute of Technology capillary viscometer was not delivered for the evaluation trials. By utilizing a unique method in measuring the pressure drop, the instrument is theoretically capable of measuring very small pressure drops for fully developed flow; however, this technology remains experimentally unproven. Due to difficulties in making this technology reliable, the instrument could not be delivered in time for the evaluation trials. The Stevens Institute of Technology viscometer would have been of use in high viscosity applications where a large pressure drop could be generated across the measuring chamber. At low viscosities the pressure drop may have been below the detection limits for this instrument. This instrument design has the potential of becoming a valuable rheological device for industry.

Recommendations

This instrument cannot be considered for an applications involving black liquor given that no sensor is available in the market.

7.4 Use of On-Line Viscometers in Other Applications

There are many other less demanding applications for these instruments in the pulp and paper, coating, and food industries. One of these applications was tested extensively in the pilot flow loop. A processed food product containing suspended particles was tested. Laboratory studies demonstrated that the slurry behaved as a bulk fluid and that the suspended particle concentration was directly related to the viscosity. This was tested for all three instruments in the pilot flow loop using the processed flood product at different suspended particle concentrations. Based upon the results, the food processor chose and installed viscometers to be used to control suspended particle concentration in the product.

Although this is the only other application tested in our program to date, the instrument manufacturers are now actively pursuing use of these viscometers for other systems.

8. APPENDIX

8.1 Detailed Description of the Pilot Flow-Loop Plant

The experimental system consists of a piping loop, viscometers, and a computerized data acquisition system. Figure 8.1.1 shows the process and instrumentation diagram of the University of Florida pilot flow loop used for the study [Fricke and Crisalle, 1994; Gibbs, 1994; Dutka, 1996]. This figure is identical to Figure 2.2, and it is reproduced here for convenience of exposition. Table 8.1.1. defines key symbols used in the figure. Black liquor from the main tank is pumped through a 2" (0.05m), schedule 40, 304L stainless steel piping loop labeled *main stream* in Figure 8.1.1. At the sampling point (valve V20 in Figure 8.1.1) the main stream branches into a 1" (0.0254 m) piping loop labeled *sample stream*, where the on-line viscometers being tested and other auxiliary equipment are located. The black liquor entering the sample stream passes through a heater (E1), a series of on-line viscometers (VIS 1 - VIS 4), and a cooler (E2), before rejoining the main stream at the return point (valve V16). Up to four on-line viscometers and one refractometer are installed in series on the sample stream. The physical location of the Torsional-Oscillatory viscometer in the pilot flow loop is indicated by the tag VIS 1 in Figure 8.1.1.

Depending on the controlled static pressure at the sampling point, a portion of the flow in the main stream is drawn into the sample loop and enters the heater. If desired, the liquor can be made to bypass the viscometer section of the sample stream, and flow directly through the cooler before returning to the main loop. The temperature, flow rate, and solids content can be adjusted in the flow loop to emulate conditions in a pulping mill. In addition to the testing of viscometers, the design enables heat transfer studies to be conducted on the exchangers, and also allows for the on-line determination of other important properties of the flowing fluid, such as refractive index and density. The feed tank and pipes are steam-traced in order to preheat the system and to maintain a constant temperature during operation. All the equipment and pipes in the pilot flow loop are thoroughly insulated to avoid heat losses. An elaborate network is also in place for supplying steam at different pressures to various points in the flow loop.

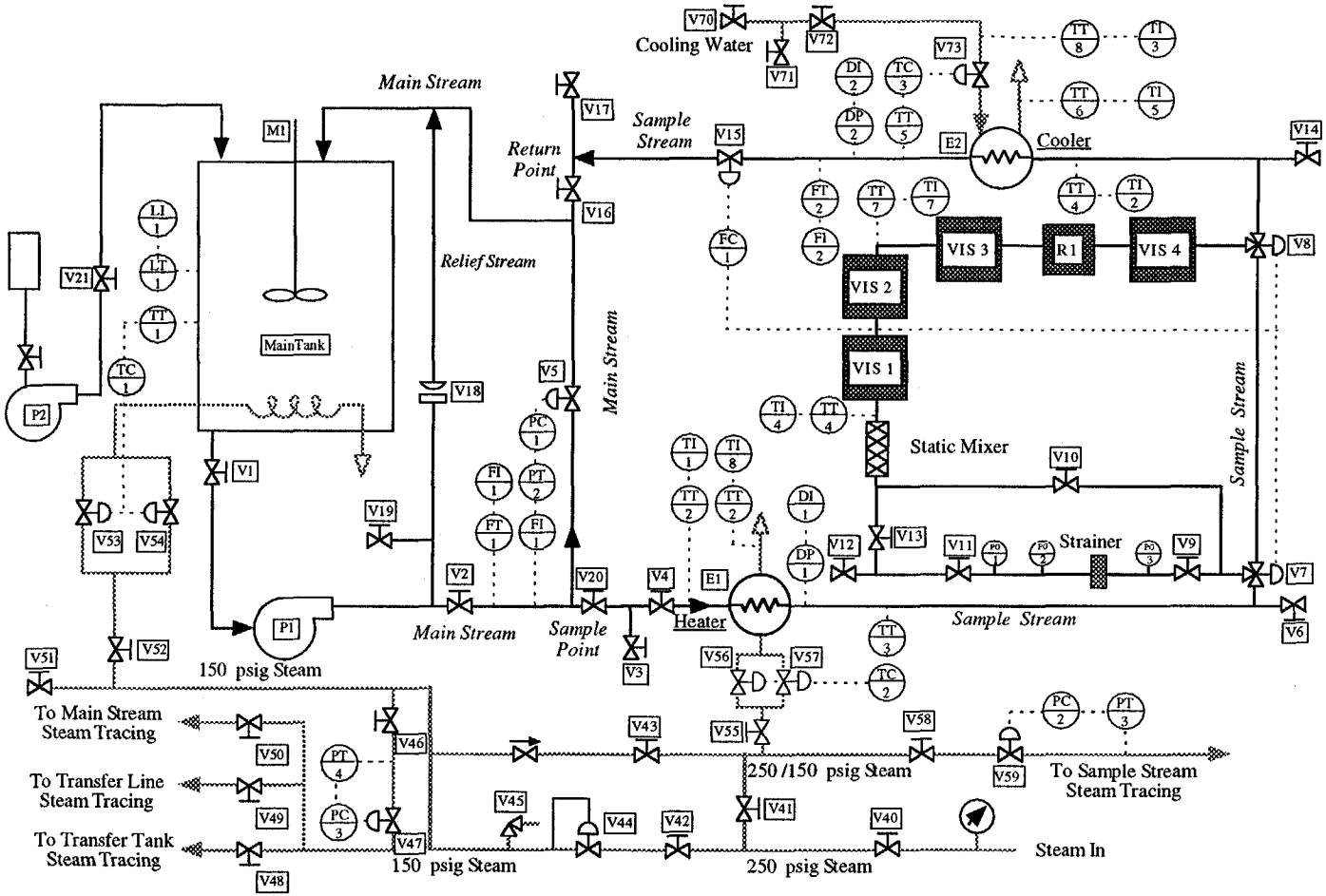


Figure 8.1.1. Process and instrumentation diagram for the flow loop used in the University of Florida pilot plant to evaluate on-line viscometers. The progressive cavity pump P1 takes black liquor from the main tank and circulates it through the closed loop labeled main stream. Valve V20 diverts a fraction of the flow into the by-pass loop labeled sample stream where the on-line viscometers are installed in positions labeled VIS 1 through VIS 4. Relevant symbols are defined in Table 8.1.1.

Table 8.1.1. Instrumentation symbols used in Figure 8.1.1

VIS	Viscometer	TC	Temperature controller
PT	Pressure transducer	R1	Refractometer
PC	Pressure controller	P1	Main pump
PG	Pressure gauge	P2	Transfer pump
FT	Flow transducer	E1	Heat-exchanger (heater)
FI	Flow indicator	E2	Heat-exchanger (cooler)
FC	Flow controller	DP	Differential pressure cell
TT	Temperature transducer	V	Valve
TI	Temperature indicator	M1	Mixer

8.1.1 Main Stream

The main stream is designed to accommodate corrosive materials such as black liquor, at pressures up to 300 psi (2.068 10^6 Pa), flow rates up to 55 gpm (3.47 10^{-3} m^3/s), and temperatures up to 110 °C (383 °K). A pulsation free, positive displacement flow is delivered by a Moyno progressive cavity pump with stainless steel internals and fitted with a Viton stator rated for discharge pressures up to 150 psig (1.135 10^6 Pa) and a maximum flow rate of 55 gpm (3.47 10^{-3} m^3/s). The Viton stator became hard and brittle with continued exposure to black liquor, and had to be replaced with an EDPM (Ethylene Propylene Dimer material) stator that proved to be durable. The 300-gallon (1.136 m^3) feed tank acts as both the source and the sink for black liquor circulating in the loop. Since black liquor can possibly undergo thermal degradation with increased exposure time at elevated temperatures, most liquors were maintained at temperatures well below the liquor boiling point in the main loop. The black liquor in the feed tank is stirred and maintained at a constant temperature for thermal and physical homogeneity. In case of excessive pressure at the pump outlet, a pressure relief valve (V18) protects the instruments in the pilot flow loop by returning the flow into the tank. The liquor flow rate and the tank level are continuously measured and displayed in the main stream. The tank temperature and static pressure are feedback controlled.

8.1.2 Sample Stream

The sample stream loop is constructed mostly of 304 stainless steel tubing of 1" OD (0.0254 m) and 0.035" (8.89 10^{-4} m) wall, with dual ferrule compression fittings that ensure flexibility for modifications. However, to balance cost with flexibility, sections of the sample loop that are least

likely to be modified are constructed of 1" (0.0254 m) NPS, schedule 40, 304L stainless steel pipe. The main purpose of the sample loop is to maintain a controlled environment for the operation of the viscometers and other equipment. The temperature and flow rate are controlled in order to ensure nearly constant conditions for testing the viscometers. The black liquor entering the sample loop passes through the heater where the temperature is raised to the desired level. A 40-mesh strainer protects the instruments from damage and plugging that could be caused by large particles. Provision is also made to bypass the strainer section, if necessary or desired. Thermal homogenization of the heated liquor is ensured by a static mixer in the sample loop. In order to prevent thermal degradation in the main tank, the liquor leaving the sample loop passes through a cooler before returning to the main stream. The on-line viscometers tested are installed in series in the sample loop, permitting the comparison of sensors under identical conditions. Since the effect of temperature on the viscosity of black liquor is significant, high accuracy RTD probes were installed at various points in the sample loop. This redundancy of measurement also served as a check for the temperature measurement produced by the internal probes of the viscometers. The sample loop is also provided with valves for cleaning the viscometers with water, steam, or hot caustic solutions, with forward or back flushing, as required.

8.1.3 Electronic Signals in the Pilot Flow Loop

The pilot plant is equipped with a state-of-the-art computerized data acquisition system (DAQ) that captures signals originating from RTDs, thermocouples, pressure transducers, flow transducers, viscometers, and other auxiliary instruments. Geffran controllers and Eurotherm indicators respectively control and monitor the pressure, temperature, flow, and the liquid level in the tank. In addition to providing readings for visual inspection, the controllers and indicators also retransmit process measurements as 4-20 mA signals. All of the 4-20 mA signals are converted to 1-5 Vdc signals by 250 Ω biasing resistors, and are fed into the DAQ system. The DAQ set-up can also communicate with RS232C and/or RS485 interfaces available in some viscometers. The output from the controllers to the actuators are in the form of 4-20 mA signals. The data-acquisition computer also serves as a digital control system which permits the use of more advanced control algorithms than those employed in standard industrial PID controllers.

8.1.4 Data Acquisition System

The main components in the data acquisition system are (*i*) a computer based on a Pentium processor, (*ii*) a hardware interface connecting the computer with the controller/process, and (*iii*) driver software. A National Instruments Corporation multi-function 12 bit plug-in AT-MIO-16E10 DAQ board receives data from a 32-channel SCXI-1100 multiplexer with a maximum sampling

rate of 100 KHz. The DAQ system has the ability to do front-end signal conditioning, amplification, and filtering. The driver software is LabVIEW, a graphical programming language from National Instruments Corporation. In-house software has been developed to automatically read data from the cards and write it to files, hence minimizing human intervention in the data acquisition process.

Though the pilot flow loop was designed for the on-line study of black liquor viscosity, the flexibility of the test bed makes it possible to study various other materials, including slurries, pastes, suspensions, food products, etc.

8.2 Detailed Description of Experimental Design Followed for Evaluations in the Pilot Flow Loop

This section describes the matrix of experiments that are carried out to evaluate the performance of the prototype viscometers in terms of (*i*) instrument variables such as accuracy and response time, and (*ii*) process variables such as flow rates and temperature. A list of relevant variables considered are shown in Table 8.2.1.

Table 8.2.1. Process and instrument variables of interest.

(i) Process variables	(ii) Instrument variables
Temperature	Accuracy and repeatability
Solids content	Range of applicability
Liquor source	Response time
Flow rate	Sample acquisition and conditioning
Contaminants	Reliability and maintenance

Of the process variables listed on the first column of Table 8.2.1, the temperature and the solids content of the liquor have a dominant effect on the viscosity of black liquor are [Fricke, 1987; Zaman, 1993; Zaman and Fricke, 1994]. Therefore, the experimental plan for evaluating the prototype viscometers includes subjecting the prototype viscometers to operation regimes at various temperatures and solid concentrations that are representative of pulping-mill conditions. Black liquors obtained from different sources—such as a liquor produced at the Palatka site of Georgia Pacific in Florida, or the liquor from Chesapeake Paper Company produced at the West Point site in Virginia—have different viscosity behavior because the sources use different wood species and different pulping recipes. The experimental program includes testing black liquors provided by three different sources of different geographical locations. It is now well known that black liquor behaves mostly as a Newtonian fluid [Zaman and Fricke, 1991; Zaman and Fricke,

1994], hence, the on-line viscometers should produce viscosity measurements that are independent of flow rate. Therefore, it is important to evaluate the viscometers under different flow rate regimes in order to determine if the design of the instruments permits them to effectively cope with the effects that a varying flow may have on the probes and other sensing elements. Finally the presence of contaminants and additives in the liquor may adversely impact the performance of an on-line viscometer. Contaminants such as fly ash augment the concentration of particles in the flow, and hence increase the likelihood of fouling in the sensors, as well as of causing malfunctions due to obstructions and interactions between the solid particles and the sensors.

The instrument variables shown in the second column of Table 8.2.1 include prominently the accuracy and the repeatability of the instrument. Accuracy refers to the agreement between the viscosity measurement produced by the on-line instrument and the viscosity measurement produced by a reliable instrument under laboratory conditions. On the other hand, repeatability (or precision) refers to a situation where the on-line instrument produces the same measurement after repeated readings under the same process conditions. Ideally one would want the viscometers to be both accurate and precise; however, often in processing operations it is sufficient to obtain a precise measurement because a repeatable measurement permits the deployment of effective process control strategies. Another instrument variable of interest is the range of applicability of the viscometers. For example, it may be possible that the principle of operation of a viscometer makes it reliable at low viscosities, but not reliable at high viscosities. Hence, the range of applicability is a critical variable of interest. Different viscometers acquire black-liquor samples from the flow stream using different techniques, such as passing the flow through a rotating cup, through a pair of bent capillary tubes, or through a sampling chamber. Furthermore, conditioning of the flow may also be done, such as adjustment of the temperature. The experimental evaluation study includes an assessment of the suitability of the different sampling methods used by the prototype viscometers considered. Finally, a primary concern with all prototype viscometers is their reliability and their maintenance requirements. Obviously, an instrument that operates without malfunctions and without need for maintenance for a long period of time is highly desirable.

8.2.1 Experimental Plan

The testing plan was designed to examine factors affecting performance of the prototype viscometers under various process conditions. Three different types of black liquor have been acquired for the evaluation phase from the following pulping-mill sources:

- Canadian Forest Products LTD., Prince George, Canada
- Chesapeake Paper Products Company, West Point VA, USA

- Georgia Pacific Corporation, Palatka FL, USA

A forth type of black liquor type was generated at the University of Florida by combining Georgia Pacific and Canadian Forest black liquors in an approximate 50%/50% mix. This mixture was made with the objective of obtaining a liquor with high solids content.

Five hundred gallons each of Canadian Forest and Chesapeake Paper black liquors were shipped to the pilot flow loop with a concentration of approximately 50% solids. A total of 700 gallons of Georgia Pacific black liquor was received at concentrations of approximately 34% and 42% solids. It was necessary to have on site such large samples of black liquor so that they could be concentrated to higher solids contents using the University of Florida evaporation system.

For each of the black liquor types, a range of solids concentrations was chosen to represent different locations where the viscometers might be placed within a pulp and paper mill. Once a black liquor type at a fixed solids concentration was loaded into the pilot flow loop, experiments were performed at various temperatures. Typically the temperature was progressively increased in the main tank from run to run so that the loop was thermally equilibrated; however, since the main holding tank is at atmospheric pressure, the highest temperature was limited to 10-20°C below the boiling point of the black liquor. At least one run was performed using the heater in the sample loop so that temperatures above the atmospheric boiling point of the black liquor could be tested. Higher temperatures are more likely to represent the operating conditions of a pulp and paper mill.

At each temperature, several runs at different flow rates were made. These included one batch (or zero flow) run in which the viscometer line was isolated from the flowing stream. The flow rate range used depended upon the limits of the pilot flow loop. In general, the flow rates in a run were set in a sequence of steps. Typically, the first step was a run at 0.5 gpm. The second and subsequent steps consisted of an increase in the flow rate by 0.5 gpm, as shown in Table 8.2.2, until a maximum sustainable flow rate was attained. Finally, the zero-flow run was made.

Table 8.2.2. Flow rate steps examined in experimental runs

Step	Flow rate (gpm ± 0.25)
1	0.5
2	1.0
3	1.5
4	2.5
5	0.0

Overall, five flow rate runs were performed within each temperature run. The upper limit of flow

rate for these runs were dependent upon the output of the main progressive cavity pump at its maximum operational limit of 150 psig. In general, however, the main line pressure was kept at 70 psig so as not to stress the main pump to its maximum.

Two modes of operation were also examined: Steady state and transient. A majority of the experimental runs were at a thermal and flow steady state. Several thermal transients were examined to determine response times from the instruments. Thermal transient experiments were done by introducing step changes in the opening of the steam control valve of the heater to induce a viscosity step change resulting from a step change in temperature.

Once the experiments for a given black liquor type and solids concentrations were completed, the black liquor was off-loaded from the loop into transfer tanks. Then the Viscometer Line was cleaned first with low-pressure steam, and then with circulating hot water. The black liquor in the transfer tanks was taken to the evaporator and loaded into the evaporator feed tank. The black liquor was concentrated to the next solids concentration of interest using a wiped-film evaporator operating at a jacket pressure of 115 psig of saturated steam and process pressure of 0.5 atm. Under these conditions, the average evaporation rate is approximately 15 lbm_{water}/hr. Once the evaporation was completed the black liquor was then loaded back into the pilot flow loop for further experiments.

8.2.2 Experimental Method

During the evaluation phase, we used a comprehensive experimental plan to examine various black liquor solids concentrations ranging from 34% to 73% with four different types of black liquor (Table 8.2.3). Of the four black liquor types, the Georgia Pacific was the easiest to handle and work with because of its low level of air entrainment and ease of evaporation; therefore, more experiments were performed with this black liquor. Although the Canadian Forest and Chesapeake Paper liquors were not evaporated to high solids contents, two runs were made at low temperatures to simulate the high-viscosity behavior that these liquors would have displayed at higher solids concentration.

A main problem experienced with the handling of the Canadian Forest and Chesapeake Paper black liquors was the formation of crystalline particles. Crystals formed in stagnant flow portions in the Main Holding Tank T1 (*cf.* P&ID diagram in Figure **8.1.1), and were eventually pumped throughout the loop where they collected and created blockages in the lines. There is a strainer with a 40 mesh screen installed in the Viscometer Line to protect the viscometer from larger particles. The viscometers tested were not affected by particles smaller than 40 mesh.

Table 8.2.3. Black liquor types and solids concentration examined.

Georgia Pacific	Canadian Forest	Chesapeake Paper	GP & Canadian Forest Mix
34.14%	47.96%	49.39%	67.71%
42.24%	57.78%	59.28%	71.38%
48.29%			73.40%
55.84%			
65.51%			
68.37%			

The Chesapeake Paper black liquor created more difficulties due to air entrainment, which made laboratory characterization, especially density correlations, extremely difficult to perform. The air entrainment in the loop is normally insignificant due to the high operating pressures and would not be a problem in actual mill operation. The effects of air entrainment can be determined by examining the density measurement as a function of flow rate with the Micro Motion instrument at a constant temperature. Black liquor can be assumed to be an incompressible fluid, therefore its density is independent of pressure. Since the flow rate in the Viscometer Line is a function of the Main Line pressure, the density of the black liquor is independent of flow rate. Significant aeration of the black liquor would give an artificially low density measurement with the Micro Motion device, and the density would increase as the flow rate increases; however, the density measurement of the Chesapeake Paper black liquor was unaffected by flow rate and remained constant at a constant temperature.

Once the Pilot Flow Loop was loaded for the current black liquor run, the Main Holding Tank and the flow loop were kept at a nominal temperature for several hours to ensure that the black liquor was thoroughly mixed and homogeneous. Flow in the loop was stopped briefly and a one-liter sample was withdrawn for laboratory characterization. This procedure was followed to prevent flashing of the liquor when the laboratory sample was withdrawn, since the sample point was upstream of the heater, and the main tank was well below the boiling point at this point. Flashing is an undesirable effect because the loss of water and organics makes the sample appear more highly concentrated. If possible, the experimental runs were delayed until laboratory characterizations were completed so that an immediate check could be performed on the viscometers before testing.

In preparation for an experimental run, various instruments and sensors needed to be zeroed or calibrated.

- The flow loop was turned off momentarily and the Main Line Flow Meter FT1 (*cf.* PI&D diagram in Figure **8.1.1) and the Sample Line Flow Meter FT2 (*cf.* PI&D diagram in Figure **8.1.1) were zeroed at zero flow.

- Valves V4 and V16 were closed so the differential pressure cells (DP1 and DP2) across the Heater E1 and Cooler E2 could be zeroed.
- The Viscometer Line is bypassed by directing flow through the bypass using the Viscometer Line Isolation Valves (V7 and V8) so that the Brookfield and Micro Motion viscometers could be zeroed.

Once the preliminary equipment and instrumentation checks and calibrations were completed, the main line flow was set to approximately 30-40 gpm at a pressure of 70 psig and the flow was passed through the sample and viscometer lines to establish thermal equilibrium. In the control room, the National Instruments LabVIEW software and Micro Motion Prolink package were turned on concurrently with the DAQ computer and the data files were initialized. The values recorded by LabVIEW and Prolink were verified against the displayed values of indicators and controllers on the control panel. For the final step in preparing the data acquisition system, the Southwest Research Institute/Quantum Magnetics computer was then setup to acquire its own data.

For the first flow rate run at the first temperature of interest, the Sample Line Controller FC1 (cf. P&ID diagram in Figure **8.1.1) was manually adjusted until the desired flow rate was attained. The Viscometer Line was allowed to achieve steady state, with the Cooling Water Controller TC2 (*cf.* P&ID diagram in Figure **8.1.1) set to manual at 99.9% open. Once at steady state, the Southwest Research Institute/Quantum Magnetics data acquisition system is turned on first, followed by Prolink and then LabVIEW. For each flow rate run, three different Brookfield rotational speeds were usually examined. The LabVIEW program paused while the Brookfield rotational speed were changed. In the meantime, the other systems were left running. LabVIEW was restarted after the rotational speed had been changed and the new calibration factors were entered into the LabVIEW program. Once all the data for the current flow rate had been acquired, all the DAQ systems were turned off, and flow through the Viscometer Line was increased to the next flow rate of interest. After steady state was achieved once again the data acquisition process was repeated.

The final flow rate run was always the zero-flow (or batch run) condition which was handled in two steps. In the first step, the same procedure was followed as in the other flow rate runs except that the Southwest Research Institute/Quantum Magnetics system was turned off. On the second step, the Southwest Research Institute/Quantum Magnetics instrument was run alone in the batch mode, since the Southwest Research Institute/Quantum Magnetics DAQ system can control the Viscometer Line Isolation Valves (V7 and V8). The Southwest Research Institute/Quantum

Magnetics instrument in the automatic mode first opened the viscometer line for a predetermined time, and then closed the Viscometer Line and analyzed the black liquor sample. When the test was completed, the isolation valves were opened and a new sample was loaded into the line. This was repeated until the Southwest Research Institute/Quantum Magnetics DAQ system was turned off.

When the first temperature run was completed, the temperature in the main holding tank was increased using the Tank Temperature Controller TC1. After the flow loop has reached a new thermal equilibrium, the flow rate step runs were duplicated as described above. The procedure was repeated for the later temperature runs until the last run, in which the sample line heater was utilized to raise the temperature up past the boiling point of the black liquor sample. The flow rate runs were handled in the same manner, except that more care was needed to maintain a constant flow rate and temperature. The Process Heater Output Controller TC2, the Sample Flow Rate Controller FC1, the Main Line Pressure Controller PC1, and the Main Pump Speed Control were adjusted manually until the desired temperature and flow rate were achieved. At this point, The Sample Line Steam Tracing Controller PC2 is set to approximately to 25 psig to help offset heat losses in the sample line. Once steady state was attained, the flow rate run was initiated. This procedure was followed for the remaining flow rate runs.

For the transient heater runs, the flow through the viscometer line was fixed with the Process Heater Output Controller TC2 set manually to 0% open. Only the LabVIEW software was used for transient experiments, since LabVIEW preempts Prolink when multitasking with the DAQ computer. Although Prolink works well in the background of LabVIEW under steady state conditions, it does not respond quickly enough to rapidly changing variables. The LabVIEW software was turned on and then the Process Heater Output Controller TC2 was set to manually open the valve by small increments. The sample line flow rate could not be held to an absolutely constant value, but an approximate value could be maintained by adjusting the Motor Speed Controller. Once the viscometers had reached steady state, TC2 was stepped open to a new value. This procedure was repeated until the process output was saturated.

The set of data were collected as ANSI files from three locations; LabVIEW, Prolink and the Southwest Research Institute/Quantum Magnetics. The data were then transferred into Excel 5.0 files for analysis. Later, the data is divided for analysis of individual instrument and then analyzed.

8.3 Description of Laboratory Characterizations

Well mixed representative black liquor samples from the pilot flow loop were characterized In the laboratory to develop viscosity, density, and heat capacity correlations. The laboratory characterization included measurements and correlations of the following properties:

- Viscosity
- Density
- Heat Capacity
- Solids Content
- Boiling Point Elevation

The viscosity for each black liquor sample was measured over the operating temperature range of the pilot flow loop using, at a minimum, three laboratory viscometers. The laboratory models (the Lab Cambridge, the Lab Nametre, and the Lab Brookfield) operated with the same principles and specifications of their on-line counterparts. The Haake Rotovisco RV12 Rotational Viscometer is a standard concentric cylindrical rheometer. In the laboratory, two types of units are employed: One is the open cup viscometer for use at low temperature for high viscosity samples, and the other is a pressure cell that was modified unit for use at high temperature and high pressure.

The viscosity data for each laboratory viscometer is gathered as a function of temperature and fitted to the form

$$\eta(cP) = \exp\left(a + \frac{b}{T(K)} + \frac{c}{T(K)^2}\right)$$

where a, b, and c are the correlated parameters [Zaman, 1993; Zaman and Fricke, 1994]. Afterwards, the smoothed correlations from at least three viscometers are compared with one another over the operational range to provide a separate and independent verification of the data, and to determine the upper and lower ranges of the laboratory reference. Generally, these correlations are within 5% of one another, which is the normally experienced agreement between instruments in the laboratory. Even though the black liquor samples were expected to be Newtonian at the solids content and viscosity ranges that were being tested, the viscosity was determined as a function of shear rate to provide verification.

Similar procedures are utilized in developing correlations of density and heat capacity as functions of temperature, as well as boiling point rise as a function of the solids content of each black liquor type. Density correlations of black liquors as a function of temperature are determined

using the AccuPyc 1330 Pycnometer to measure the density of the sample at a fixed temperature and dilatometer to measure thermal expansion of the black liquor sample. The density correlation have the form of a second order polynomial

$$\rho(cP) = a + bT(°C) + cT^2(°C)$$

where a, b, and c are calibration constants [Fricke, 1987; Fricke, 1990].

8.4 Contact information for project participants

This list below provides key contact information for relevant instrument manufacturers, pulp mill operations, and instrumentation companies that have participated in the viscometer development project for black liquors.

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