

## Accepted Manuscript

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PII: S1466-8564(18)31312-2  
DOI: <https://doi.org/10.1016/j.ifset.2019.05.003>  
Reference: INNFOO 2164

To appear in: *Innovative Food Science and Emerging Technologies*

Received date: 28 October 2018  
Revised date: 29 March 2019  
Accepted date: 2 May 2019

Please cite this article as: N. O'Shea, T.F. O'Callaghan and J.T. Tobin, The application of process analytical technologies (PAT) to the dairy industry for real time product characterization - process viscometry, *Innovative Food Science and Emerging Technologies*, <https://doi.org/10.1016/j.ifset.2019.05.003>

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**The application of Process Analytical Technologies (PAT) to the dairy industry for real time product characterization - Process Viscometry.**

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ACCEPTED MANUSCRIPT

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**Abstract:**

The ideal PAT tool is an inline instrument that can monitor and measure process parameters simultaneously in real time while operating in a highly automated environment. Instruments must be of sanitary design, operate robustly within the full process cycle (production and cleaning). Inline determination of the rheological properties of moving fluids (i.e. dairy concentrates) is one of the process parameters where PAT tools can add real value in terms of optimising process control. Measurement of process viscosity is crucial in the monitoring and control of a variety of concentration processes in the dairy industry. Continuous monitoring of the rheological behaviour of the fluid can allow for optimisation of the process e.g. pumping (avoid pump blockage and failure), evaporation (limit fouling and maximise water removal) and spray drying (avoidance of nozzle fouling). This review concentrates on the state of the art developments being made in the area of process viscometry.

**Keywords:** Process viscosity, PAT, Inline, Dairy, Skim milk.

**1. Introduction:**

The application of process analytical technologies (PAT) encompasses three main areas – process analysers, multivariate analysis and process control. Synergy between all three components ensures a greater process understanding, optimum process control and enhanced final product quality (Munir, Yu, Young, & Wilson, 2015; Munir et al., 2017). In general, PAT tools work through inline, real-time monitoring of process variables, giving the manufacturer accurate and instantaneous information to facilitate modification of process parameters for optimal process efficiency (Munir, Yu, Young, & Wilson, 2015). PAT tools have been widely integrated in pharmaceutical and chemical manufacturing processes; however, up until recently PAT tools have been underutilised in the food industry (Munir et

al., 2017). In food manufacturing processes, PAT tools can be used to monitor and control multiple inline process parameters through continuous monitoring of composition, temperature, pH, pressure, flow, and density. Process viscometry or the study of viscosity; described as the resistance of a fluid to flow (O'Connor, McEntee, & O'Callaghan, 1995), is an essential tool for monitoring the flow properties of solutions both in dilute and concentrated formats. Authors have previously published studies relating to real time monitoring of viscosity in the production of food products i.e. the manufacture of certain condiments (Cullen, Duffy, & O'Donnell, 2001) and for the production of skim milk powder (O'Donnell, Herlihy, & McKenna, 1994).

The adoption and correct positioning of PAT tools in a dairy process along with interpretation of the outputs ensures an efficient process with the following benefits (Munir et al., 2017):

- Optimisation of raw material usage.
- Optimisation of process conditions for maximum efficiency.
- Reduction of laboratory testing.
- Production of consistent high quality end products.
- Reduction of product recalls or rework.
- Reduction of operational expenditure.
- Avoidance of any harmful effects on the environment.

Overlooking the importance of measuring process viscosity can place limitations and risks on many dairy processes. Ignoring process viscosity can have a negative impact on major unit operations i.e. the mobility of the process concentrate resulting in pump failure, (Bakshi & Smith, 1984), thermal process heat exchangers and evaporator fouling (Nema & Datta, 2005; Westergaard, 2004) and deposit formation on spray drying lances (beard formation) (Straatsma, Van Houwelingen, Steenbergen, & De Jong, 1999). Also, importantly, the

physical and functional characteristics, and hence quality, of the subsequent dairy powders are influenced by the viscosity of the concentrate prior to spray drying.

*1.1. Influence of concentrate viscosity on the major processing steps involved in the production of dairy powders:*

As viscosity of dairy concentrates influences process performance and the quality of finished products, the locations where viscosity can be monitored and hence controlled in the factory is of interest to dairy processors. Taking a generic overview on the typical unit operations involved in the manufacture of skim milk powder (Figure 1), which are ubiquitous across many other dairy and nutritional products. The logical points at which it would be desirable to measure the viscosity of the medium in real time within the process can be inferred based on the changes in solution behaviour as it transitions from liquid milk to a dried powder. Typically raw liquid milk is preheated to  $\sim 50^{\circ}\text{C}$  using a plate heat exchanger, and separated into cream and skim milk using a centrifugal separator and both streams are typically pasteurised before further processing. A high temperature short time (HTST) heat treatment is applied to the skim milk; (typically  $\sim 73^{\circ}\text{C}$  for 15 seconds) and in addition to being a legal requirement is also used to ensure a reduction in the microbial load of the milk before further processing.

Following pasteurisation the skim milk may be stored ( $4^{\circ}\text{C}$ ) for a period of time before delivery of the liquid skim milk to the evaporator where it is again heat treated prior to concentration to specific total solids content. The predefined total solids target for the evaporator is usually chosen based on historical knowledge of the specific dairy process concentrate characteristics and end use application (Kelly & Fox, 2016). Often the predefined total solid content targeted by dairy processors does not reflect the levels achievable within

the process, if the actual limitations related to product viscosity were considered. The viscosity of a dairy concentrate can vary based on total solids, temperature, flowrate, shear history, storage of the concentrate, concentrate composition (seasonal changes to the composition of the milk), heat load applied before concentration and physico-chemical behaviour of the material (age thickening) (Bloore & Boag, 1981). Thus measurement of process viscosity offers a more informative indicator than either total solids content or density measurements, ensuring conveyance of optimal process concentrate dry matter content to the spray dryer.

Previously process viscometers have been installed to monitor the viscosity of dairy concentrates post evaporation (Figure 1 – L1) (O'Donnell, Herlihy, & McKenna, 1994). Installation of an inline viscometer can ensure the evaporator is operating efficiently and that the product has been concentrated to the maximum achievable dry matter content, prior to spray drying (Kelly & Fox, 2016). Typically a high pressure positive displacement pumps, are used to feed dairy concentrates to the atomiser of the spray dryer, whereby the concentrate is sprayed into the drying chamber and a powder is produced (Kelly & Fox, 2016). Installing an inline viscometer in the high pressure line leading to the atomizer is the optimal location, as it is the last point before the concentrate is delivered to the atomizer and offers the best representation of the properties of the concentrate at the point of atomisation (Figure 1 – L2).

The high pressures (10 – 30MPa) required to deliver the concentrate to the dryer, has presented a challenge for the implementation of PAT, as few inline viscometers are rated to operate at such high pressures. It is accepted practice to locate high pressure pumping systems feeding concentrate to spray dryers at ground level. However, injection of the concentrate into the drying chamber occurs at the top of the drying tower often at vertical distances of 40 – 50 m above the high pressure pump. In practise this means that hot concentrate (a time dependent fluid) has a significant residence time within the concentrate feed system of the spray drier.

This residence time is dependent on a number of factors that include the dimensions of the dryer, the sizing of the pipework and the mass flow rate of the concentrate within the high pressure pumping system. As a result, the fluid properties and hence behaviour of the viscous concentrate may be different in the moments before atomization, compared to those that are typically measurable prior to the high pressure pump. Hence there is a desire within the dairy processing industry to install robust high pressure rated PAT to monitor the behaviour of dairy concentrates at the latest possible point within the process flow scheme to ensure a true reflection of concentrate characteristics prior to stabilisation in the dry form. The value of this data in complex control philosophies, would include feedback control of upstream unit operations to ensure that optimal solution properties are obtained at the point of atomization to maximise the quality and functionality of the subsequent dried product. However, the delivery of such a PAT solution has presented a challenge, as few inline viscometers are rated to operate at such high pressures (up to 30MPa).

Monitoring and controlling the process viscosity of the concentrate prior to spray drying is essential to ensure optimal dryer efficiencies. Increased viscosity can result in the formation of larger droplets during atomisation, which may slow down the drying process and in extreme cases could wet the drying chamber if effective atomization cannot be achieved for a given nozzle geometry (De Souza, 2010). This will result in the spray dryer using higher energy consumption and decreasing the efficiency of the process (De Souza, 2010). In extreme cases the high viscosity of the concentrate may cause fouling of the nozzle/lances, creating a build-up of poorly atomized concentrate/powder at the leading nozzle causing blockages, this “bearding” also poses the risk of ignition and subsequent fires in the dryer. In general nozzle fouling requires more frequent lance changes and can reduce cleaning intervals which necessitate the plant to come to a complete stop to facilitate cleaning, resulting in reduced operational utilization and increased operational expenditure (De Souza, 2010).

Finally, a sub-optimal process can cause a deleterious effect on the quality of the subsequent powder with stickiness issues and loss in product functionality, possibly leading to the powders requiring re-working or downgrading (Vignolles, Jeantet, Lopez, & Schuck, 2007). Thus, installation of a process viscometer inline could allow the manufacturer to make process control interventions, at a point before the process has entered an out of control state, allowing for the avoidance of some or all of the challenges outlined above. The installation location of any inline viscosity monitoring tool and its design robustness are key considerations for selection of a process viscometer, while the potential benefits in terms of enhanced process control make this an area of significance which will be explored within this review.

### *1.2. Criteria required prior to installation:*

Before a viscometer can be purchased or installed it is necessary to ascertain whether the instrument is fit for propose and complies with both product and process criteria.

These criteria have been discussed in detail previously by Cullen et al. (2000), whereby the PAT tool must be:

- **Cost effective.**
- **Design appropriate:** must be a robust and stable sanitary design; ideally meeting European Hygienic Engineering Design Group (EHEDG) standards or 3A design, without moving parts or seals.
- **Compatible with the product or the process stream:** hygienic and risk-free without the contribution of dead spots/legs where microbial growth may occur.
- **Antifouling:** Surface characteristics should not promote fouling of the product contact surfaces.
- **Clean-in-place (CIP):** able to tolerate standard CIP procedures.



- **Highly sensitive:** to measure subtle changes in the process concentrate.
- **Rapid response time:** capable of giving a series of points covering the relevant range of shear rate.
- **Output signal:** continuous or intermittent signal.
- **Plant operating environment:** independence of the measurement relative to plant vibrations and at high operating temperatures and pressures (up to 30MPa) (Cullen, Duffy, O'Donnell, & O'Callaghan, 2000).

Finally, the location of where the viscometer is installed is of great significance, some viscometers can be installed inline (directly in the process), online (a bypass loop is required) and at line (viscometers that operate alongside the process).

This review will discuss the advancements which have been made to date in the area of process viscometry since that of Cullen et al. (2000). Examples illustrated in this review of each measuring principle mainly focus on applications from the food industry, with the objective of this work being to critically assess the process viscometers currently available on the market and compare them to the criteria outlined above to facilitate identification of the most suitable process viscometers for dairy applications.

## 2. Existing inline/online process viscometers:

The type of process viscometer installed is often dictated by the flow properties of the material. An ideal liquid e.g. water, can be described as a Newtonian fluid (the flow properties are independent of shear rate). Dairy liquids e.g. skim milk concentrate are described as non-Newtonian (shear-thinning), whereby as the product is exposed to shear forces throughout the process it exhibits a shear thinning behaviour which resolves itself as a decrease in viscosity of the liquid (Schramm, 1994). Hence viscometers such as capillary flow viscometer (pressure drop), falling body viscometers, orifice-type viscometers, paddle

viscometers and the hot wire are more suited to measuring Newtonian fluids or fluids in a stationary position (Abbas, Abdulkarim, Saleh, & Ebrahimian, 2010). While viscosity measurements of dairy concentrates (non-Newtonian fluids are taken as the material is in transit) flowing to the spray drier. Currently adopted process viscometry methods can be split into three categories: rotational viscometry, vibrational viscometry and tube viscometry/velocity profile. Velocity profiles can be measured using a number of different operating principles but the most frequently discussed method in literature is ultrasound in combination with pressure differential (O'Connor, McEntee, & O'Callaghan, 1995).

### *2.1 Rotational viscometers:*

Rotational viscometers are commercially available as an inline measurement system and are based on the conventional lab based rheometers. The measuring principle is based on a drag force placed on a rotating body (cylinder) enclosed in another immobile cylinder, whereby the fluid being measured, is pulled into the moving cylinder (O'Connor, McEntee, & O'Callaghan, 1995). It operates at a predefined shear rate (provided by the rotating body) and the torque (the drag force) measured represents the shear stress (Cicchese, 2013). This type of technology has been utilised by Lamy Rheology (RM100 L Touch), however, to the authors knowledge there is no record of it being adopted in a dairy process.

Brookfield engineering is widely known for developing rotational viscometers (STT-100 in-Line viscometer (Couette type) (Figure 2)). A similar model the TT100 inline process viscometer (Brookfield Engineering) was implemented in an Irish dairy plant to prevent evaporator fouling and optimise steam consumption, through continuous monitoring of the viscosity of skim milk concentrate (SMC) exiting the evaporator. Based on the recommended maximum viscosity that could potentially block an evaporator (100cP at 100/s shear rate), it was demonstrated that the TT100 inline viscometer could successfully monitor the viscosity

of the SMC concentrate during typical production scenarios (O'Donnell, Herlihy, & McKenna, 1994). Although there are no moving parts in this viscometer and it is compatible with cleaning in place (CIP), the narrow measuring gap can be susceptible to clogging or blocking at higher dry matter contents, potentially reducing the accuracy of the instrument.

## 2.2. *Vibrational viscometers:*

Vibrational viscometers consist of a probe inserted into the dairy concentrate to which a vibrational force is applied. The dampening of this vibration is calculated relative to the properties of the concentrate as it flows past the probe and is proportional to the viscosity (O'Connor, McEntee, & O'Callaghan, 1995). The measuring geometry typically comes in the form of a cylinder of varying diameter, with multiple commercial examples available (**Table 1**).

Fork type viscometers are also available for the measurement of fluid viscosity and commercially they are available from Emerson (Micro Motion 7827 Digital Viscosity Meter). The tines of the fork are vibrated piezo-electrically at its natural frequency, the bandwidth of which is dampened proportional to the viscosity of the flowing liquid (De Souza, 2010; Lin, De Souza, & Young 2009). O'Callaghan et al. (2001) investigated torsional and transverse-mode vibrating probes, installed at two locations (ex-evaporator (before the balance tank) and pre-atomiser (before concentrate heater) within a dairy process. The authors concluded that the torsional vibration probe performed the best, as it was found to operate independently of in-process equipment vibration, required low maintenance, comprised of no moving parts and could be cleaned effectively during CIP (O'Callaghan et al., 2001). A vibrational process viscometer based on the torsional measuring principle has been installed commercially to monitor the viscosity of tomato based pizza sauces. This viscometer was successfully used to identify sauces that fell outside the specified formulation limits and was found to be capable

of accurately measuring the viscosity of sauces that contained particulates, without affecting the accuracy of the instrument (Cullen, Duffy, & O'Donnell, 2001).

The Hydramotion XL7/100 (torsional probe) and Solartron 7827 (vibrating tuning fork) were both used to measure the viscosity of skim and whole milk concentrate in a test skid (De Souza, 2010). These authors compared their findings to a laboratory rheometer and found on average the difference between the measurements to be 0.33cP. While, the authors conclude that it was difficult to replicate the exact same conditions for the inline versus laboratory based measurements (difference between technologies (vibration versus rotational) and sample size), the results were deemed sufficiently accurate for process control (De Souza, 2010).

Vibrational process viscometers have been described as stable, robust, have no moving parts and are easily cleaned by CIP processes. They are also easy to install, can operate at high pressures and can be installed inline removing the requirement for a flow by-pass loop. As outlined in the aforementioned studies vibrational viscometers have good repeatability and are sensitive to small changes in solution properties. However, vibrational viscometers are challenged when it comes to calculation of rheological values such as yield stress, as they do not have well-defined shear rates. Additionally sources of error within the measurements can be exacerbated by fouling of the probe by the medium, or if entrained air is associated with the surface of the probe ( Cullen, Duffy, & O'Donnell, 2001).

### *2.3. Tube Viscometry / Velocity profile:*

Tube viscometers measure viscosity, based on the flowrate of a fluid and its pressure drop when flowing within a known length of pipe. The process concentrate must be in laminar flow for the method to be successful (O'Connor, McEntee, & O'Callaghan, 1995).

The benefits associated with this method include, continuous flow operation, and the implementation of the technology is relatively easy and inexpensive. However, there are considerable disadvantages associated with this methodology in that it constantly requires a fully laminar flow, meaning it would often necessitate a by-pass loop. Good knowledge of the concentrate flow properties and characteristics of the fluid are essential to designing an appropriate tube viscometer for dairy (Cullen, Duffy, O'Donnell, & O'Callaghan, 2000). Process concentrates that have a high level of particulates can be susceptible to wall slip. Wall slip occurs in fluids that contain a high concentration of small particles. The particles in the fluid form an attraction with the solid surface resulting in the particles collecting at the pipe wall, this can lead to inaccurate viscosity values that do not represent the true viscosity of the fluid as some of the particles are no longer flowing. Tubes with a larger-diameter are required to prevent wall slip (Cullen, Duffy, O'Donnell, & O'Callaghan, 2000). Temperature fluctuations can also occur in long straight pipes which will affect the viscosity readings of the concentrate; this must be accounted for when reliant upon pressure drop measurements. Using volumetric calculations, this method only gives one data point for viscosity at a single pressure and flow rate measurement (Roberts, 2001). The technology has in recent years undergone a series of revisions to eliminate or reduce such limitations.

Advanced technologies such as ultrasound and magnetic resonance imaging have been used to determine a velocity profile (using Doppler optical coherence tomography) of a specified cross-section of a pipe. This has been used in combination with the pressure drop of a specified length of pipe to calculate the rheological properties of the process concentrate. This is usually carried out by measuring the Doppler shift in the frequency from the reflected ultrasound and the corresponding time delay (Haavisto, Koponen, & Salmela, 2014). Using this method of measurement provides multiple shear viscosity and shear-rate data points from

one combined viscosity profile and pressure drop measurement (Cullen, Duffy, O'Donnell, & O'Callaghan, 2000).

### *2.3.1. Magnetic resonance imaging:*

A velocity profile can be created using magnetic resonance imaging (MRI) from a fluid flowing through a non-metallic pipe. Using the determined velocity profile, viscosity flow curves can be constructed that are comparable to flow curves generated from an off-line rheometer. The velocity profile is developed from two magnetic field gradients. The first magnetic field is positioned in the direction of flow and measures the position of protons at two different times. The second magnetic field is positioned perpendicular to flow and portrays the location of the displacement as a function of tube radius; this is fulfilled using pulsed gradient spin-echo procedures. Once the time and displacement is derived the velocity can be calculated (Roberts, 2001). Generally the shear rate is calculated from the velocity profile and the shear stress is calculated from the pressure difference.

To the best of the author's knowledge this technology has not been utilised in the dairy industry, even though it is currently available commercially (Aspect Imaging – Flowsan).

While, this technology is not currently active in the dairy industry, it has been utilised in scientific literature e.g. in the pulp, paper and biomass processing industries. It was utilised in a study to determine the yield stress of de-lignified commercial cellulose. Results from this study were compared to results from an off-line rheometer. This study demonstrated experimental yield stress values comparable to off-line measurements (Lavenson, Tozzi, McCarthy, & Powell, 2011). An additional study investigated the effects of fibre concentration, flow rate on the velocity profile of the suspension in the pipe. Whereby, it was proposed that large changes in velocity asymmetry, flatness and pressure drop occur at higher

concentrations of cellulose, dependent upon the length of the fibres in suspension (Tozzi, Lavenson, McCarthy, & Powell, 2013).

There are many advantages associated with this technology such as, it is non-destructive, non-invasive and measurement can be carried out in a number of seconds (Arola, Barrall, Powell, McCarthy, & McCarthy, 1997). However, it also has many disadvantages including a high capital cost associated with purchase and installation of the system, the potential for installation in a by-pass loop, with associated implications surrounding true representation of the full fluid flow. Additionally a significant amount of time may be required for signal processing, reducing its ability to produce results in real-time. Finally, it requires more highly skilled engineers to operate the system and software than alternative methods (Roberts, 2001).

### *2.3.2. Ultrasonic-pulse-Doppler (UPD) with Pressure difference (UVP & PD):*

Essentially, this method applies an ultrasonic impulse (of known frequency) into the flow of fluid. The impulse is then reflected back from the moving particles present in the flow. Measurement of this reflected wave is achieved via the Doppler effect, whereby the frequency that is reflected, is shifted proportional to the fluid velocity. The radial spatial position of particles in flow can then be calculated based on the time to send and receive the frequency wave and the known speed of sound in the fluid (Roberts, 2001).

This method is heavily reliant on reflective solid surfaces within the fluid such as fat globules, but can perform additional rheological measurements such as yield stress calculations. The disadvantages associated with this equipment include high purchase and associated installation costs and while measurements can be performed in a short timescale, data processing challenges are similar to MRI technologies, requiring a highly skilled technician for operation and data processing. The UPD also requires pressure drop measurement across

the flow tube, and is challenged from an accuracy perspective if there is entrained air (bubbles) within the fluid.

A number of studies have been completed to assess the potential of this technology as outlined in **Table 2**. It has been applied to measure the rheological properties of food/industrial suspensions including; tomato concentrates, chocolate suspensions, starch suspensions and cement grout. This technology is also commercially available from the following companies, Met-flow (UVP-duo) and Flow-Viz.

### **3. Emerging Technologies:**

#### *3.1. Microacoustic viscosity sensors:*

The basic operation of the sensor focuses on measuring a response to an input stimulus (physical, chemical, biological or electrical) resulting in an altered electrical output signal, intrinsically linked to the input stimulus (Ferrari & Lucklum, 2009). A viscosity measurement is achieved after the sensor face is placed in a fluid, the acoustic wave comes in contact with the input stimulus (fluid), and a change in resonance frequency is observed. As the viscosity of a fluid increases, the damping of the acoustic wave also increases at a similar rate. Correlations between the increase in fluid viscosity and the damping of the measured acoustic wave electrical parameters can then be generated (Drafts, 2001).

The acoustic waves associated with these types of sensors are defined by their velocities and displacement directions. Waves used in these sensors include Rayleigh, Lamb, Bleustein-Gulyaev and Love waves (Drafts, 2011).

Acoustic wave devices commonly used and described in the literature include surface acoustic waves (Rayleigh wave) (SAW), Shear-horizontal surface acoustic wave (SH-SAW), Bulk acoustic waves (BAW) and Shear-horizontal acoustic plate mode (SH-APM). These devices



differ either by the type of acoustic wave used or in the configuration of the device (Drafts, 2011).

SAW sensors are based on the propagation of acoustic waves across the surface of a substrate and consequently have been described as not being suited to aqueous environments. Authors have described the returning wave as becoming completely dampened, as a result of energy losses in the liquid and thus are more suited to gas environments (Ferrari & Lucklum, 2009). The sensor uses separate input and output transducers for propagation and detection of the surface acoustic wave, which allows the sensor to differentiate between different signal measuring parameters (Durdag, 2008). Acoustic transmission has been linked as an indirect measurement of viscosity, as the acoustic transmission decreases the viscosity of a process medium increases linearly. This wave type is used in commercial instruments used to measure volume flow and temperature of liquids (Burkert Fluid Control Systems (FloWave)) (van Loon & Klingler, 2015). This instrument is low cost compared to other commercially available instruments and gives instantaneous results. It is made of stainless steel so there are no CIP issues; it has a sanitary design with no moving parts. The sensing part of the instrument is around the pipe so it non-invasive; as a result it does not interrupt the flow of concentrate.

Bulk acoustic wave (BAW) sensors propagate a wave through the interior of a substrate and are more suited to operating in fluid environments. This wave is generated from a piezoelectric disc containing a quartz crystal that is excited by a high frequency oscillator. The piezoelectric disc vibrates in the thickness shear mode (the entire disc vibrates) releasing the wave into the substrate. The BAW produced propagates through the interior of the process medium, resulting in the process medium coupling to the sensors vibrating surface. This type of sensor usually resonates between 5-30 MHz. The reduction in resonant frequency occurs from the added process medium (input stimulus) placed on the sensor from

the fluid (Figure 3) damping the vibration and also the frequency, it is essentially measuring acoustic impedance. Increases in viscosity of the process media result in an increase in the damping of the BAW, which also decreases the BAW frequency (Ferrari & Lucklum 2009). Currently applications for this type of microacoustic viscometer are mainly seen in the oil industry, where this sensor offers significantly lower costs compared to the other technologies described in this review. Benefits of this sensor include, it can be used inline and produces real time continuous results while having no moving parts. As a result of the high frequencies used by the sensor it is not affected by plant vibrations, or flow conditions of the liquid. The major disadvantage associated with this sensor revolves around the availability of a sanitary designed commercial system for food, in addition to a low operational pressure range (max 0.3MPa).

Agoston et al. (2005) demonstrated the use of a microacoustic sensor (using the thickness shear mode and a quartz resonator operating at a frequency of 6 MHz) to measure the viscosity of deteriorated engine oils.). Using mineral oils of known viscosities these authors successfully plotted the viscosity results of the microsensors versus kinematic viscosity (results produced from the same oils using a laboratory instrument) resulting in a linear correlation (Agoston, Ötsch, & Jakoby, 2005).

SH-APM can be described as having elements similar to both the SAW and BAW devices. It combines the input and output transducers (required for distinguishing signal measurements) similar to SAW devices. It also employs a sensor crystal used as a physical barrier between the electronics and the sensing medium that is usually used in BAW device allowing the device to be used in fluids (Durdag, 2008).

Finally, one consideration associated with these sensor types is the unit the result is measured in, which is acoustic viscosity; this result needs to be correlated with apparent viscosity.

Hence, a conversion table is required prior to installation of the sensor to take into account the conversion of acoustic viscosity into the apparent viscosity; this may be time consuming and include a level of uncertainty in the final result.

### *3.2. Coriolis flow measurement:*

Coriolis based mass flow meters are installed commercially across the food industry. The operating principle of the sensor is based upon two tubes vibrating at their natural resonant frequency (in phase). Upon entry of the process concentrate into the tubes, natural Coriolis forces are applied to the tubes resulting in the tubes falling out of synchronicity and a phase shift or time delay is developed. From this time delay the mass flow can be calculated (Wang & Baker 2014). The following parameters can be measured from one device - mass flow, density, temperature and volume flow.

Viscosity measurements have now been added to the parameters that can be measured from a Coriolis meter. Using a straight tube mass flowmeter, a pendulum is attached to the middle of the measuring tube. The measuring tube oscillates at its resonance frequency in lateral mode (measuring flow), whereas with the attachment of a pendulum a torsional oscillation is induced. The torsional oscillation is performed at a higher frequency and is used to indirectly measure viscosity. It operates on a similar principle to a vibrational process viscometer as discussed in section 2.2 (**Figure 4**). An excitation current is required to maintain the tube in oscillation. This excitation current is dampened as a result of the shear forces occurring in the process concentrate. Hence, the viscosity of the process concentrate can be determined from the excitation current (Drahm & Bjonnes 2003). An example of a commercial meter that uses this principle is available from Endress + Hauser (Proline Promass I 300 / 83I). It is a

commercial instrument with no moving parts, does not need to be installed in a bypass loop it has EHEDG certification and was manufactured to meet a sanitary design.

The disadvantages of this system include measurement accuracy in the presence of entrained air, and while the meter is fabricated out of a durable metal (titanium).

### *3.3. Mixer viscometry:*

This technology is an extension of a method designed by Metzner and Otto, The Metzner and Otto method relates the impeller speed in a mixer, to the shear rate for a non-Newtonian fluid in laminar flow. It is assumed an average mixer shear rate near the impeller, can be related to the torque exerted on the impeller this can be measured as the power consumption of the impeller. If this method is measured in reverse, viscosity of a concentrate can be ascertained. In the past this method has been successfully utilised in measuring the viscosity of samples in a vessel (La Fuente et al., 1998). An extension of this method can be applied to measuring the dynamic viscosity of process fluids in a pipe. This is possible by relating the shear rate in a pipe to the pressure drop as a consequence of two consecutive static mixers (of known geometry) in combination with flowrate (Arzate, Reglat, & Tanguy, 2004).

Arzate et al. (2004) investigated the reliability of using the static mixer system to measure dynamic viscosity of three different fluids that had different rheological properties (starch suspensions of increasing concentrations, cooked starch at 40°C and paper coating colour formulations) at an industrial scale. The authors found the inline static mixer system successfully determined the viscosity of starch suspensions of increasing concentrations. Results from a second trial demonstrated the ability of the mixing system to identify the shear-thinning effects of cooked starch. Finally, the viscosity results of the paper coating colour formulation (with a solids content of 57% w/w) from the inline static mixer system were compared to rotational rheometer results with both measurement systems yielding

similar results. It was proposed by the authors that this demonstrated the ability of the mixing system to be sensitive to measure fluids containing different solids content (Arzate, Reglat, & Tanguy, 2004).

An extension of this static mixer system is commercially available from Krohne (Viscoline). It was designed for non-Newtonian fluids, can be installed inline and provides continuous measurement of fluid viscosity in a sanitary designed measurement system. Unfortunately, no studies are available as which demonstrate the robustness of the system when measuring the viscosity of dairy concentrates.

#### *3.4. Dynamic Fluid Pressure:*

This method is based on dynamic fluid pressure which is proportional to the viscosity of the fluid. It operates via a partially opened tube that contains a rotating cylindrical shaft. A constant flow of the process concentrate is drawn into the inline viscometer and enters between the shaft and the tube wall. As the process concentrate travels through the system the gap between the shaft and tube wall becomes gradually smaller. This results in the spring-like tube wall being deflected by the pressure of the process concentrate. Viscosity is calculated from the length of deflection path which is measured via an electrical sensor (Figure 5). The manufacturer (Anton Paar) describes this sensor as having such benefits as working in continuous flow i.e., not requiring a by-pass loop. This instrument is robust, requires low maintenance and is not affected by plant vibrations. The viscosity of the process concentrate can also be monitored in real time. The major disadvantage associated with this sensor, it is not of sanitary design, nor has it EHEDG certification (Anton Paar, 2016).

#### 4. Final Remarks:

The aim of this review was two-fold, the identification of process viscometers currently available for implementation in the dairy industry. Secondly, to discover new technologies or highlight existing technologies in other industries that could have potential to be used by the dairy industry. Process viscometry offers many benefits to the producer through monitoring the viscosity of a process in real time. Employment of process viscometers as a process analytical tool is currently under-utilised in the dairy industry for control of downstream processes, especially when considering optimisation of concentration processes such as evaporation and spray drying. It is clear that better interaction between sensor designers and end users in the dairy industry could promote adoption of these tools, by addressing the specific challenges on design and implementation of sensors which can be exposed to high pressure environments, a fluid material with a high fouling propensity and aggressive cleaning protocols all within an instrument which must be manufactured to meet sanitary design guidelines.

Advancements in new vibrational methods (Micro Motion 7827 Digital Viscosity Meter from Emerson & Proline Promass I 300 / 83I from Endress + Hauser) and new technologies (e.g. L-Vis 510 from Anton Paar, viscoline from Krohne) have emerged since their view of Cullen et al. 2000.

While these measurement systems are attractive, as they are based upon well-established principles, alternative solutions such as the FloWave instrument (Burkert) could be a potential novel cost-effective solutions once validated by robust product specific studies

Advancements have also been made in vibrational methods, in particular the Proline Promass I300 / 83I instrument from Endress + Hauser. Producers may be more willing to introduce this meter into their process. In particular if it offers the convenience of swapping their

current mass flow/density meter for the new advanced viscometer that offers additional measuring variables (viscosity, mass flow and density).

In conclusion significant progress has been made since the review carried out by Cullen and others (2000), but there are still hurdles that need to be overcome before the ideal viscometer is developed e.g. high pressure rated viscometers. However, the desired and practical technological gap between the standard process viscometers currently available and the ideal cost-effective process viscometer is getting smaller.

### **5. Acknowledgements:**

This work is supported by the Irish State through funding from the Dairy Processing Technology Centre research programme - Grant Number TC/2014/0016.

### **6. References:**

- Abbas, K., Abdulkarim, S., Saleh, A., & Ebrahimian M. (2010). Suitability of viscosity measurement methods for liquid food variety and applicability in food industry-A review. *Journal of Food, Agriculture & Environment* 8(3&4), 100-7.
- Agoston, A., Ötsch, C., & Jakoby, B. (2005). Viscosity sensors for engine oil condition monitoring—Application and interpretation of results. *Sensors and Actuators A: Physical* 121(2), 327-32.
- Anton Paar. Inline Viscometer: L-Vis 510. Available from: <http://www.anton-paar.com/corp-en/products/details/inline-viscometer-l-vis-510/> Accessed 2016 November 15.

- Arola, D. F., Barrall, G. A., Powell, R. L., McCarthy, K. L., & McCarthy, M. J. (1997). Use of nuclear magnetic resonance imaging as a viscometer for process monitoring. *Chemical engineering science* 52(13), 2049-57.
- Arzate, A., Reglat, O., & Tanguy, P. (2004). Determination of in-line process viscosity using static mixers. *Flow Measurement and Instrumentation* 15(2), 77-85.
- Bakshi, A. & Smith, D. (1984). Effect of Fat Content and Temperature on Viscosity in Relation to Pumping Requirements of Fluid Milk Products<sup>1</sup>. *Journal of Dairy Science* 67(6), 1157-60.
- Bloore, C. & Boag, I. (1981). Some factors affecting the viscosity of concentrated skim milk. *New Zealand Journal of Dairy Science and Technology*.
- Cicchese S. (2013). Inline Viscosity Measurements. *Chemical Engineering* 120(5), 34.
- Cullen, P., Duffy, A., & O'Donnell, C. (2001). In-line consistency monitoring of tomato based products using vibrational process viscometry. *Journal of food processing and preservation* 25(5), 337-51.
- Cullen, P., Duffy, A., O'Donnell, C., & O'Callaghan, D. (2000). Process viscometry for the food industry. *Trends in Food Science & Technology* 11(12), 451-7.
- De Souza G. (2010). Viscosity measurements for milk powder process control. ResearchSpace@ Auckland.
- Dogan, N., McCarthy, M. J., & Powell, R. L. (2003). Compariosn of in-line consistency measurment of tomato concentrates using ultrasonics and capillary methods. *Journal of food process engineering* 25(6), 571-87.
- Dogan, N., McCarthy, M. J ., & Powell, R. L. (2005). Applcation of an in-line rheological characterization method to chemically modified and native corn starch. *Journal of Texture Studies* 36(3), 237-54.



- Drafts, B. (2001). Acoustic wave technology sensors. *IEEE Transactions on Microwave Theory and Techniques* 49(4),795-802.
- Drahm, W. & Bjonnes, H. (2003). A Coriolis meter with direct viscosity measurement. *Computing and Control Engineering* 14(4), 42-3.
- Durdag, K. (2008). Solid state acoustic wave sensors for real-time in-line measurement of oil viscosity. *Sensor Review* 28(1), 68-73.
- Endress + Hauser. Proline Promass 83I Coriolis flowmeter. Available from:  
<http://www.ie.endress.com/en/Field-instruments-overview/Flow-measurement-product-overview/Product-Coriolis-flowmeter-Proline-Promass-83I> Accessed 2016 November 17.
- Ferrari, V., & Lucklum, R. (2009). Overview of acoustic-wave microsensors. *Piezoelectric transducers and applications*: Springer. p. 39-62.
- Haavisto, S., Koponen, A. I., & Salmela, J. (2014). New insight into rheology and flow properties of complex fluids with Doppler optical coherence tomography. *Frontiers in chemistry* 2.
- Jin Choi, Y., Milczarek, R. R., Fleck, C. E., Casey Garvey, T., McCarthy, K. L., & McCarthy, M. J. (2006). In-line monitoring of tomato concentrate physical properties during evaporation. *Journal of food process engineering* 29(6), 615-32.
- Kelly, A. L., & Fox, P. F. (2016). Manufacture and Properties of Dairy Powders. In: McSweeney HPL, O'Mahony AJ, editors. *Advanced Dairy Chemistry: Volume 1B: Proteins: Applied Aspects*. New York, NY: Springer New York. p. 1-33.
- Köseli, V., Zeybek, Ş., & Uludağ, Y. (2006). Online viscosity measurement of complex solutions using ultrasound Doppler velocimetry. *Turkish Journal of Chemistry* 30(3), 297-305.

- Krohne. Viscoline – New Generation Rheometer. Available from <http://us.krohne.com/en/products/viscosity-measurement/viscoline/>. Assessed 2016 November 17.
- La Fuente, E. B. D., Nava, J. A., Lopez, L., Medina, L., Ascanio, G., & Tanguy, P. A. (1998). Process viscometry of complex fluids and suspensions with helical ribbon agitators. *The Canadian Journal of Chemical Engineering* 76(4), 689-95.
- Lavenson, D. M., Tozzi, E. J., McCarthy, M. J., & Powell, R. L. (2011). Yield stress of pretreated corn stover suspensions using magnetic resonance imaging. *Biotechnology and bioengineering* 108(10), 2312-9.
- Lin, T. I., De Souza, G., & Young, B. (2009). Towards a Viscosity and Density Correlation for Dairy Fluids-A Soft Sensor Approach. In *Computer Aided Chemical Engineering* (Vol. 27, pp. 1371-1376). Elsevier.
- Munir, M. T., Yu, W., Young, B. R., & Wilson, D. I. (2015). Industrial Information, and Control Centre. "The current status of process analytical technologies in the dairy industry. *Trends in Food Science & Technology* 43(2), 205-218.
- Munir, M.T., Wilson, D. I., Depree, N., Boiarkina, I., Prince-Pike, A., & Young, B. R. (2017). Real-time product release and process control challenges in the dairy milk powder industry. *Current Opinion in Food Science* 17, 25-9.
- Nema, P. K., & Datta, A. K. (2005). A computer based solution to check the drop in milk outlet temperature due to fouling in a tubular heat exchanger. *Journal of Food Engineering* 71(2), 133-42.
- O'Callaghan, D., Schulz, D., O'Donnell, C., Duffy, M. A., Hade, M. J., & Howard, M. V. (2001). Improved Control in Dairy Processing.
- O'Connor, W., McEntee, S., & O'Callaghan, D. (1995). In-line viscometry in the dairy processing industry. *International Journal of Dairy Technology* 48(2), 44-9.

- O'Donnell, C., Herlihy, N., & McKenna, B. (1994). Use of an In-Line Viscometer in the Manufacture of Skim Milk Powder. *Developments in Food Engineering*: Springer. p. 927-9.
- Ouriev, B., & Windhab, E.. (2002). Rheological study of concentrated suspensions in pressure-driven shear flow using a novel in-line ultrasound Doppler method. *Experiments in fluids* 32(2), 204-11.
- Ouriev, B., Windhab, E., Braun, P., & Birkhofer, B. (2004). Industrial application of ultrasound based in-line rheometry: From stationary to pulsating pipe flow of chocolate suspension in precrystallization process. *Review of scientific instruments* 75(10), 3164-8.
- Roberts, I. (2001). 14 - In-line and on-line rheology measurement. *Instrumentation and Sensors for the Food Industry (Second Edition)*: Woodhead Publishing. p. 760-75.
- Schramm, G. (1994). A practical approach to rheology and rheometry: Haake Karlsruhe.
- Straatsma, J., Van Houwelingen, G., Steenbergen, A. E., & De Jong, P. (1999). Spray drying of food products: 1. Simulation model. *Journal of Food Engineering* 42(2), 67-72.
- Tozzi, E., Lavenson, D., McCarthy, M., & Powell, R. (2013). Effect of fiber length, flow rate, and concentration on velocity profiles of cellulosic fiber suspensions. *Acta Mechanica* 224(10), 2301-10.
- Tweedie, R. J. (2015). Process analytical instrumentation, the challenges for in-situ characterization of complex particulate materials. *Procedia Engineering* 102,1714-25.
- van Loon, J., and Klingler, M. (2015). Revolutionary flow measurement concept for the Pharma industry [White paper]. Retrived September 5, 2017 from Burkert: <https://www.burkert.com/en/Company-Career/What-s-New/Press/Technical-Reports/Technical-Reports-.hygienic/Revolutionary-flow-measurement-concept-for-the-Pharmaceutical-industry>

- Vignolles, M-L., Jeantet, R., Lopez, C., & Schuck, P. (2007). Free fat, surface fat and dairy powders: interactions between process and product. *A review. Le Lait* 87(3),187-236.
- Wang, T., & Baker, R. 2014. Coriolis flowmeters: a review of developments over the past 20 years, and an assessment of the state of the art and likely future directions. *Flow Measurement and Instrumentation* 40, 99-123.
- Wassell, P., Wiklund, J., Stading, M., Bonwick, G., Smith, C., Almiron-Roig, E., & Young, N.W. (2010). Ultrasound Doppler based in-line viscosity and solid fat profile measurement of fat blends. *International journal of food science & technology* 45(5), 877-83.
- Westergaard, V. (2004). Milk powder technology: evaporation and spray drying: Niro A/S.
- Wiklund, J., Birkhofer, B., Ricci, S., Meacci, V., & Stading, M. (2015). An integrated in-line fluid characterization system for industrial applications.
- Wiklund, J., Johansson, M., Shaik, J., Fischer, P., Windhab, E., Stading, M., & Hermansson, A-M. (2002). In-line ultrasound based rheometry of industrial and model suspensions flowing through pipes. Trans. Third International Symposium on Ultrasonic Doppler Methods for Fluid Engineering, EFPL, Lausanne, Switzerland. p. 69-76.
- Wiklund, J., Shahram, I., & Stading, M. (2007). Methodology for in-line rheology by ultrasound Doppler velocity profiling and pressure difference techniques. *Chemical Engineering Science* 62(16), 4277-93.
- Wiklund, J., & Stading, M. (2008). Application of in-line ultrasound Doppler-based UVP-PD rheometry method to concentrated model and industrial suspensions. *Flow Measurement and Instrumentation* 19(3), 171-9.
- Wiklund, J., Stading, M., & Trägårdh, C. (2010). Monitoring liquid displacement of model and industrial fluids in pipes by in-line ultrasonic rheometry. *Journal of Food Engineering* 99(3), 330-7.

Wunderlich, T., & Brunn, P. (1999). Ultrasound pulse Doppler method as a viscometer for process monitoring. *Flow Measurement and Instrumentation* 10(4), 201-5.

Zhang, L., Schultz, M. A., Cash, R., Barrett, D. M., & McCarthy, M. J. (2014). Determination of quality parameters of tomato paste using guided microwave spectroscopy. *Food Control* 40,214-23.

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**Figure Legends:**

**Figure 1:** Simplistic representation of skim milk powder process: L1: usual location for process viscometer, L2: ideal location for a process viscometer. Location: ■ ■ ■.

**Figure 2:** Brookfield STT-100 in-line viscometer (Roberts, 2001).

**Figure 3:** Representative image of BAW sensor measuring a fluid (Durdag 2008).

**Figure 4:** Operation of the Proline Promass I 300 / 83I (Endress + Hauser, 2016).

**Figure 5:** Representative of dynamic fluid pressure principle (Anton Paar, 2016).

**Table 1:** Vibrational Process Viscometers.

<b>Manufacturer</b>	<b>Sensor Name</b>	<b>Principle</b>
SoFraser	MIVI Process Viscometer	Low Frequency transverse vibration
Nametre	Visco-tech	Torsional Oscillation
Marimex Industries GmbH & Co. KG	ViscoScope	Torsional Oscillation:
Hydramotion	XL7-100	Torsional vibration
Fuji Ultrasonic Engineering Co.,Ltd.	Ultrasonic Viscosity Meter	Ultrasonic torsional vibration
Brookfield Engineering	AST100	Torsional vibration
Vaf Instruments	ViscoSense	Torsion vibration (pendulum)

**Table 2:** Studies completed to date using Ultrasonic-pulse-Doppler & Pressure Difference.

<b>Process Technology</b>	<b>Purpose</b>	<b>Reference</b>
Ultrasonic-pulse-Doppler (UPD)	To determine UPD as a viable technology to be used as a method for quality control (compared to rotational concentric cylinder).	(Wunderlich & Brunn, 1999)
	To show the inline applications of UPD and pressure difference (PD) in line to analysis the flow behaviour of non-transparent and highly concentrated suspensions.	(Ouriev & Windhab, 2002)
	To investigate the application of UPD + PD on industrial surfactant solutions (e.g. shampoo) and model cellulose fibres in water suspensions.	(Wiklund et al., 2002)
	To quantify the flow behaviour of tomato concentrates, using velocity profiles.	(Dogan, McCarthy, & Powell, 2003)
	To use UPD+PD to determine the flow properties of a chocolate suspension (recrystallization process) in stationary to pulsating flow.	(Ouriev, Windhab, Braun, & Birkhofer, 2004)
	To measure shear viscosity of starch suspensions and gels using UPD + PD.	(Dogan, McCarthy, & Powell, 2005)
	To measure the fluid viscosity of tomato concentrate using UPD + PD and correlate the values with physical properties of the tomato concentrate i.e. total solids and density.	(Jin Choi et al., 2006)
	To demonstrate how the inline UPD + PD can be used to measure the flow properties of non-Newtonian fluids in a wide range of shear rates and in a short time.	(Köseli, Zeybek, & Uludağ, 2006)
	To upgrade the UPD equipment and validate it using rheological measurements of different fluids (e.g. Newtonian fluids, concentrated opaque non-Newtonian, and fluids containing particles of different shapes and sizes from (µm-cm))	(Wiklund, Shahram, & Stading, 2007)
	To characterise the rheological properties of complex a model (model e.g. glass beads suspended in glycerol) and industrial suspensions (industrial e.g. mineral slurries) of different size distributions and made up of soft and hard particles.	(Wiklund & Stading, 2008)
To study and monitor liquid (model and industrial non-Newtonian fluids) displacement in pipes.	(Wiklund, Stading, & Trägårdh, 2010)	



To compare new upgraded UPD+PD technology using solid fat content as the test material compared with results from older UPD+PD technology using the same test material. (Wassell et al., 2010)

To measure rheological flow properties of industrial models e.g. ketchup, cement grouts and waste water using UPD +PD (Wiklund, Birkhofer, Ricci, Meacci, & Stading, 2015)

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**Highlights:**

- Process viscosity is crucial for monitoring of dairy concentration processes
- The application of current sanitary process viscometers are assessed
- This review evaluates the potential of viscometers employed in other industries
- Recommendations on viscometers from other industries are presented

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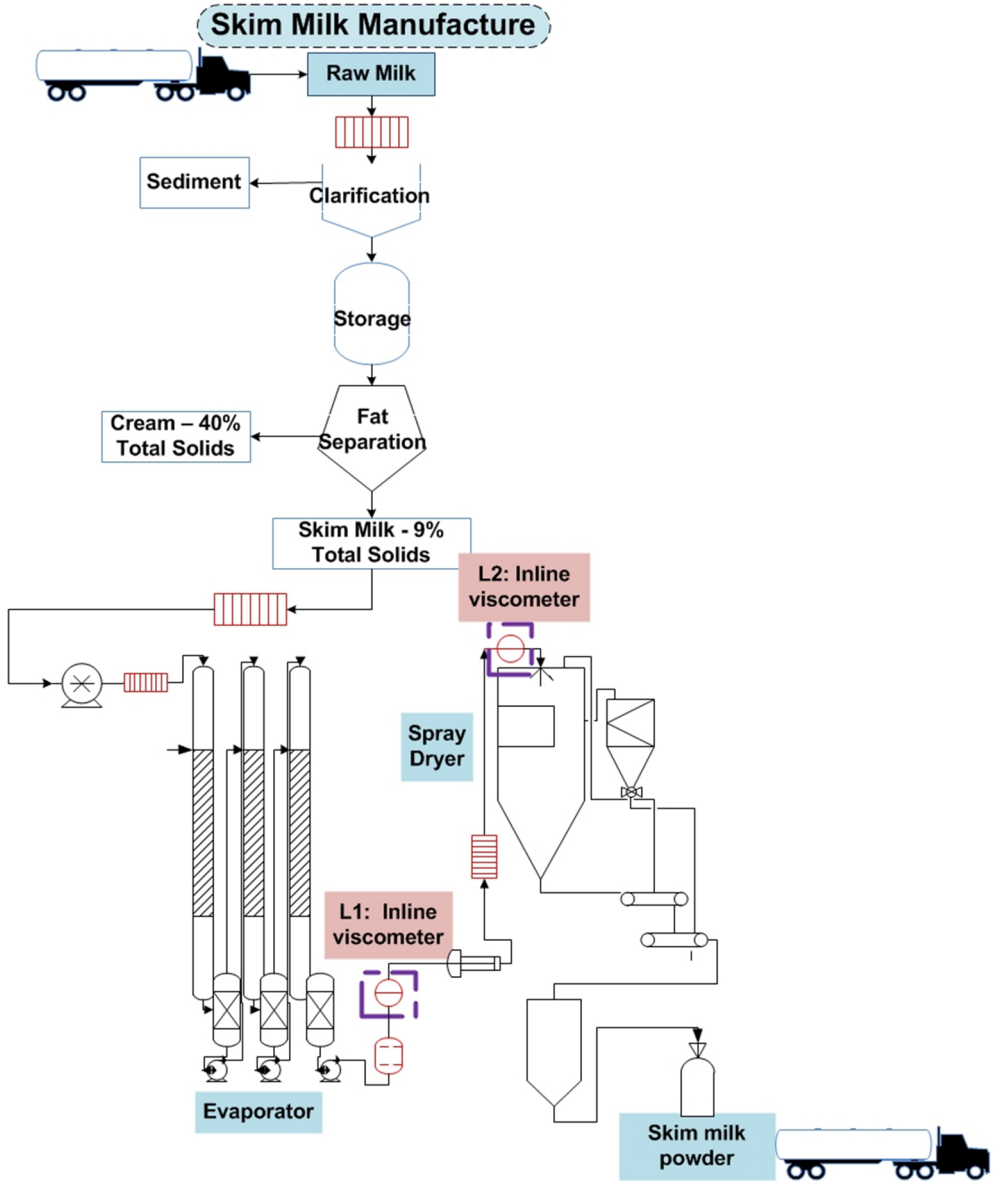


Figure 1

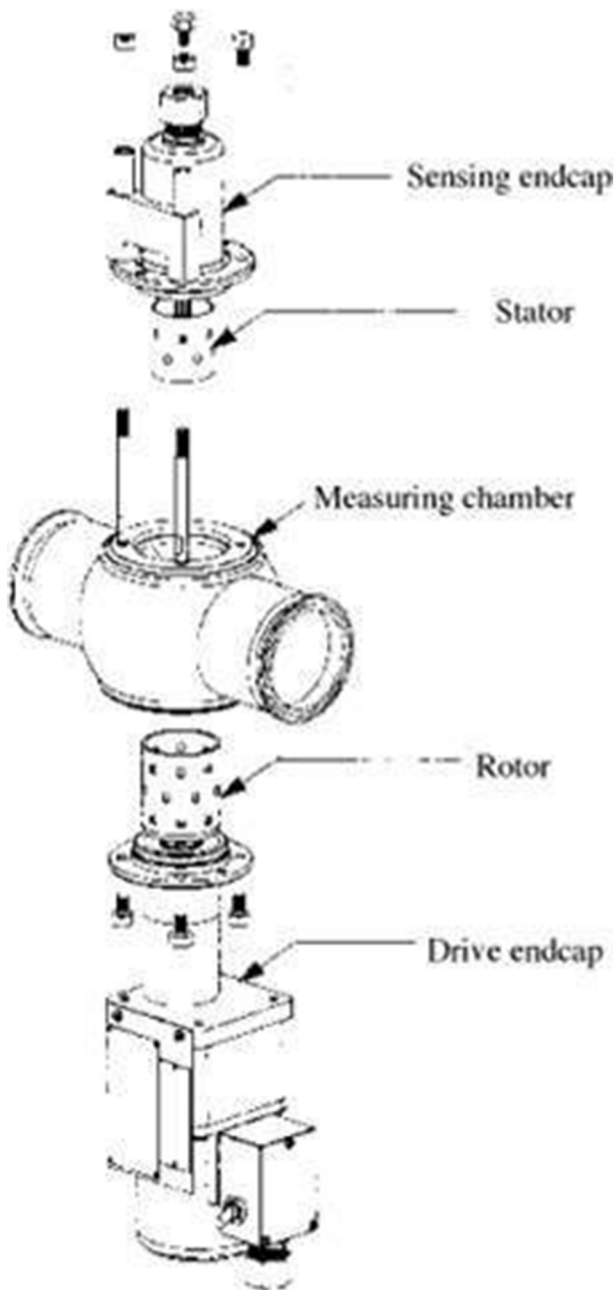


Figure 2

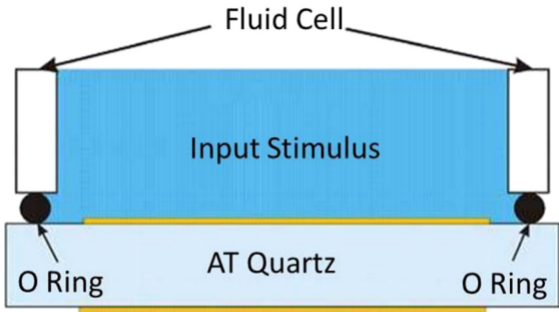
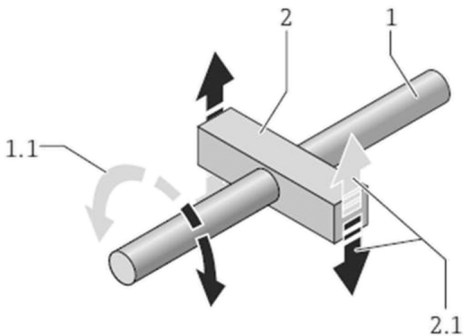
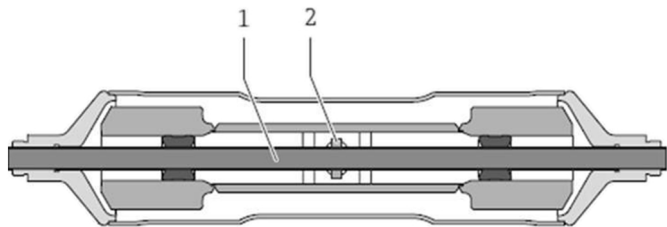


Figure 3



1 *Measuring tube*

1.1 *Rotational movement of the measuring tube*

2 *Torsion bar*

2.1 *Rotational movement of the torsion bar*

Figure 4

New measuring principle: Only available from Anton Paar

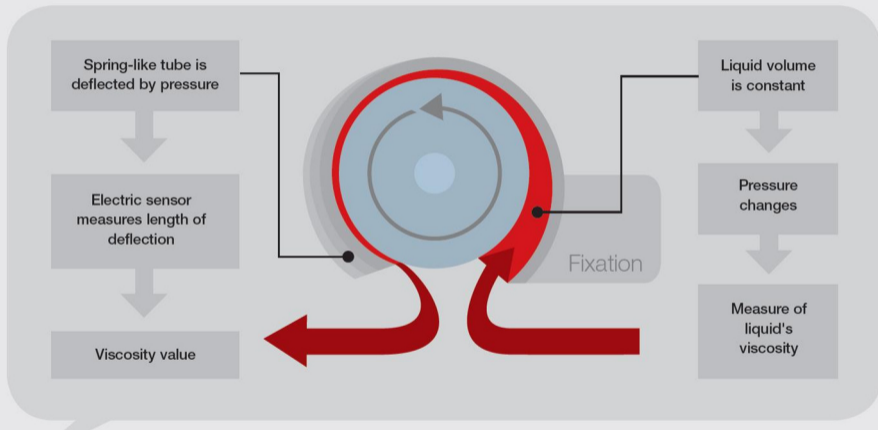


Figure 5