

ULTRASONIC NDE RHEOLOGICAL MEASUREMENT TOOLS FOR INDUSTRIAL PROCESS CONTROL

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Abstract: The measurement of various parameters such as viscosity, density, temperature, bulk modulus of materials that are undergoing rheological changes is important for process control in most of the industrial processes. Ultrasonic NDE methods have been employed here to make measurements of these properties under potentially difficult industrial processes. The key ultrasonic parameters that are measured include velocity, attenuation, and reflection-factor of both the shear and longitudinal waves. Several aspects such as temperature gradients, properties gradients, etc., will influence the accuracy of these reading. In this research work, two prototype devices have been constructed and tested. This includes (a) an ultrasonic interferometer, and (b) a rheology cell. Probes to implement the above techniques were fabricated and experiments were conducted to measure the density, viscosity, and bulk modulus of liquids. Glycerin-water mixtures were used as the test material. The application of these techniques may be extended to industrial needs of high temperature process such as glass, metal forming and normal temperature applications such as lubrication oil and soap industries.

Introduction: Density, Adiabatic bulk modulus and Viscosity form a set of important mechanical properties of liquids, especially when they are varying transiently as encountered during many industrial processes such as glass fabrication, in-situ monitoring of slurry and other similar applications. Their quantification provides information regarding the progress of these industrial processes and hence it serves as a useful tool for on-line process control. Various methods have been proposed, particularly frequency based methods ([3]), phase of the reflected wave ([1]), and other shear reflection based methods ([2],[4]-[6], [10]). In the present work, the reflection factor at the solid-liquid interface, the velocity, and the attenuation in the fluid is made use of for determination of mechanical properties of liquids.

The working principle differs significantly between the two methods (ultrasonic interferometer and rheology cell) only for viscosity measurement technique. Density measurement is based on the principle of acoustic impedance mismatch. But prior to this, the ultrasonic longitudinal wave velocity has to be computed, as the velocity is the primary parameter from which we further develop the remaining properties using additional experimental data.

This acoustic impedance of a wave can be mathematically defined as the product of the wave velocity in the medium and its density. As the waves encounter the interface between the solid and the liquid, they sense a change in acoustic impedance, and hence some part of the wave reflects back. This phenomenon is analogous to electrical current where current will divide itself at a junction based on the resistance of different paths, which it has to encounter. Physically it represents the resistance, which the material poses to the propagation of a wave. The longitudinal wave velocity can be easily obtained from the time of flight measurements of the wave passing through a cross-section of the fluid and getting reflected back from the back wall. The density can then be found from the reflection factor measurements of the longitudinal wave. The reflection factor would be the ratio of the difference between the acoustic impedances and their sum. After having calculated the density and velocity the bulk modulus can be found from the formula

$$K = \sqrt{\frac{E}{\rho}} \dots\dots\dots (1)$$

Measurement of Viscosity: The higher the viscosity of a fluid, lesser would be the resistance to the propagation of shear waves. Ideally air offers almost infinite resistance and shear waves do

not propagate in it. The resistance to propagation of shear waves in water is very high (compared to glycerin) and difficult to notice. On the other hand, fluids such as glycerin and honey easily support propagation of shear waves. However solids offer the less resistance to acoustic shear waves. This is expected since less viscous liquids tend to slip when sheared. Hence, by monitoring the reflection factor at the solid-liquid interface (which would depend on viscosity of the liquid), the viscosity of the fluid could be quantitatively as well as qualitatively obtained. The viscosity measurement is performed using two different techniques and the results obtained with each are illustrated and discussed. The range of viscosity for employing a particular method is discussed subsequently.

(a) **Measurement Using Rheology-meter Cell:** This method makes use of reflection factor at the solid-liquid interface of a normally incident shear wave for measurement of viscosity of liquid that is contained in the measurement cell. The experiments are performed using a rectangular container, which has three sides made of plexi-glass and one side made of steel. Three ultrasonic transducers for (a) generating shear waves (3

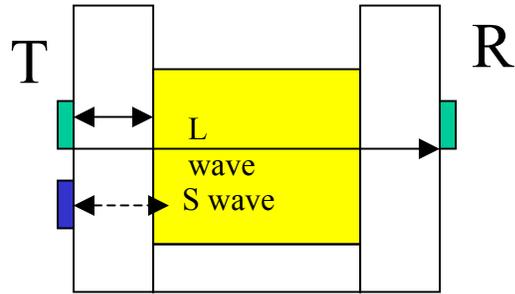


Figure 1: The rheology-meter Cell

MHz) (using pulse-echo mode), (b) generating and (c) receiving Longitudinal waves (5 MHz.) (using through-transmission and pulse-echo mode), were bonded on the outer side of steel surface as shown in the figure 1. A Panametrics PR 500 pulser-receiver was used to excite the crystal and the signals are received, digitized using a 100 ms/s Analog-digital converter and analyzed using software. S-waves are generated using the crystal and a reference signal is obtained with air as a fluid medium inside the container. Next, the container is filled with the specimen liquid and another signal is recorded. The reflection factor obtained from the two signals at the plexiglass-liquid interface was used for determination of viscosity. Liquids do not support shear waves; consequently, the reflection factor at the interface is very close to unity. The deviation of reflection factor from unity is noticeable after sixth echo. Hence, to obtain observable difference in voltages, reflection factor corresponding to the seventh echo was measured. A similar method can be found in [8-10] where pitch-catch techniques were used for viscosity measurements.

The acoustic impedance offered by light liquids to the propagation of shear waves is given by

$$z = \sqrt{0.5\rho\eta\omega} *(1 + j) \dots\dots\dots (2)$$

where ρ is the Density, η is the viscosity of the liquid and ω is the circular frequency of shear waves

The reflection coefficient can be expressed as

$$R^* = \frac{Z_L - Z_S}{Z_L + Z_S} \dots\dots\dots (3)$$

$$= -R \{ [\text{Cos}(\beta)] - j[\text{Sin}(\beta)] \}$$

Solving for Z_L and assuming β to be small, we get,

$$Z_L = Z_s \frac{(1-R)}{(1+R)} + j Z_s \frac{(2R \sin \beta)}{(1+R)^2} \dots\dots\dots (4)$$

Equating real parts of (2) and (4),

$$\sqrt{\rho\eta} = \rho_s V_s \sqrt{\frac{2}{\omega}} \left(\frac{1-R}{1+R} \right) \dots\dots\dots (5)$$

Where ρ_s is the density of solid, V_s is the speed of shear wave in solid

Thus, by measuring the reflection coefficient, the shear impedance comprising of a product of density and viscosity is measured. Using the density measurement from the L wave reflection factor, the viscosity of the fluid can be determined.

One of the difficulties of this technique involves the rather small sensitivity of the reflection factor to fluid property changes, particularly for viscosity in the range less than 200 Poise. Hence, in the rheology-meter cell technique described here, the multiple reverberation echoes inside the Plexiglas are used to improve the sensitivity and the results will be described in more details in the later sections. An alternate method for the improvement of the sensitivity will be to develop an ultrasonic interferometer.

(b) Measurement using Ultrasonic Interferometric techniques: In this technique, the shear wave is generated at mono-frequencies and propagated on two rods kept on opposite sides. At the end of one of the rods, a liquid of known viscosity is placed and the other end of the rod is in contact with the fluid that is been measured (See Figure 2). The lengths of the rods are adjusted such that the two shear waves reflected are allowed to interfere at the crystal. The two waves coming from either side of the crystal will interfere and be sensed by the crystal. Each crystal, though designed for a particular frequency, can well operate outside its range to a certain extent. Now the frequency can be adjusted so that the total amplitude of the interfering waves change. Initially, both sides of the probe are in contact with the reference fluid and the frequency of the input sinusoidal excitation to the shear crystal is continuously changed till at a particular frequency maximum destructive interference takes place. This is called **NULLING** of the probe. Hence, any phase change that occurs at the reflection interfaces due to changes in the shear impedance (which contains viscosity) will be recorded as an increase in the signal magnitude. Using calibration fluids, we have to correlate the amplitude to change in viscosity. The frequency can be varied within the bandwidth of the crystal. For a 2.25 MHz crystal the bandwidth would be probably ranging from say 1 MHz to around 3.5 MHz. One of the points of maximum destructive interference was found by trial and error to be 1.542 MHz (there may be more than one frequency corresponding to the point of maximum destructive interference. This was the lowest of those frequencies). Now as the fluid is filled on one side of the container, the amplitude at the point of complete destructive interference changes. The fluid on the other side must be the same throughout the experiment and is set as the standard. Water was used as reference fluid and it was unchanged throughout the experiment

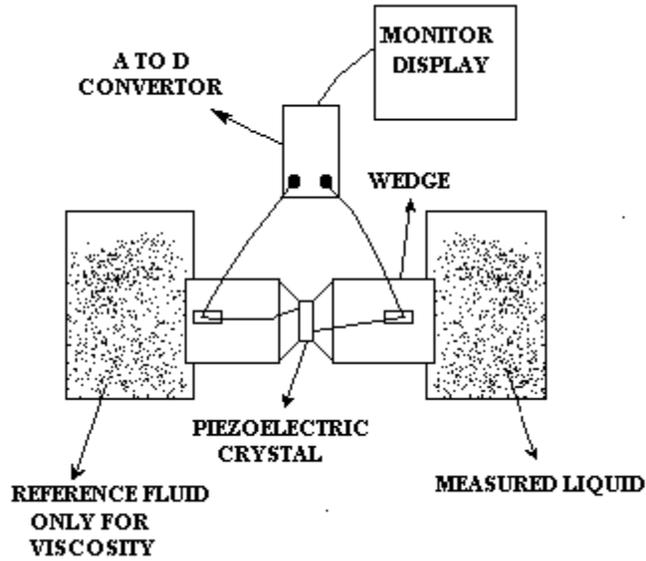
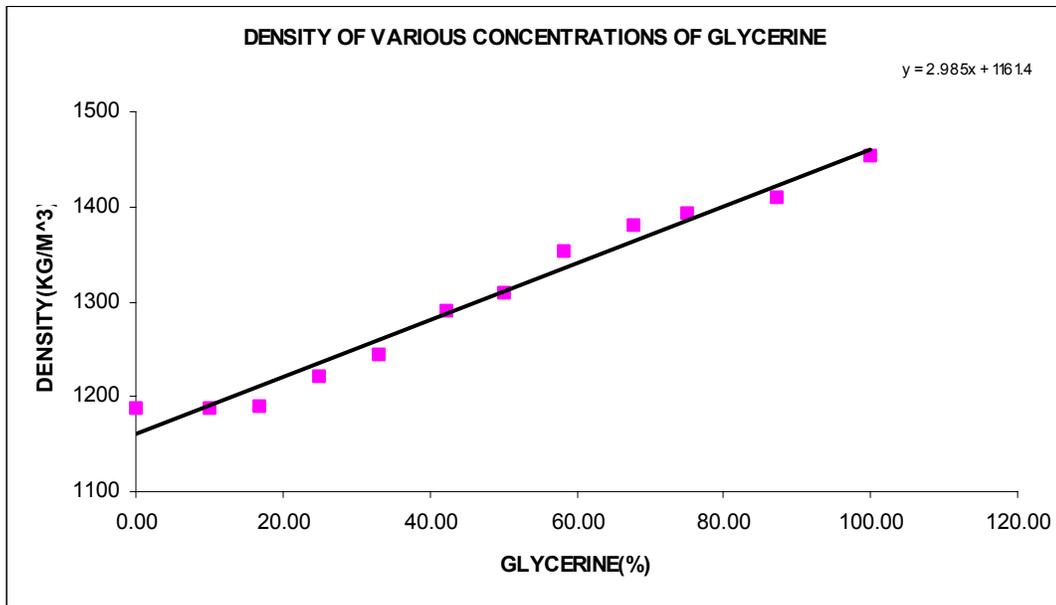


Figure 2: The experimental setup for the ultrasonic interferometric method.

Results: Using the two experimental methods described earlier, the density, bulk modulus and viscosity of fluids mixtures of glycerin and distilled water were obtained. The longitudinal wave was employed to measure the velocity and the reflection factor from which the bulk modulus and the density were obtained. The results are shown in Figure 3a and 3b.



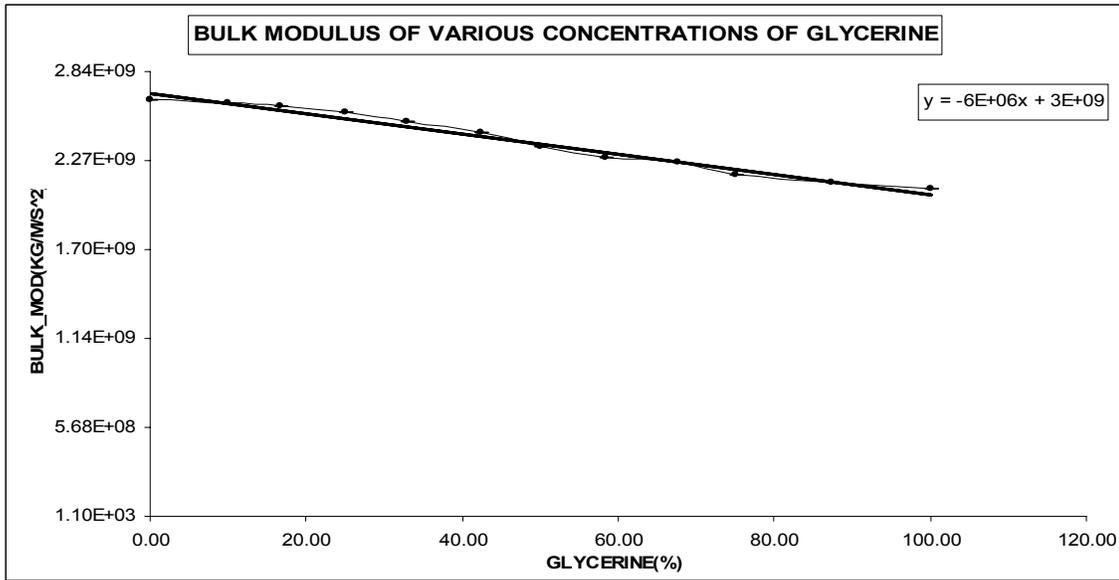


Figure 3: Results of the (a) density and (b) bulk modulus measurements using longitudinal wave velocity and reflection factor measurements. The points are measured values and solid lines are the calculated values.

Viscosity Measurements using the Rheology-meter Cell: The viscosity measurements are performed for *Glycerin-Water* solutions with varying concentration of Glycerin by weight. The results for viscosity measurement using the Rheology-meter cell technique are shown in Table 1. Corresponding to Seventh Echo, the reflection factor can be written as

$$R = \left(\frac{V_{Liquid}}{V_{Air}} \right)^{\frac{1}{7}} \dots\dots\dots(6)$$

Medium	Voltage(V)	$R_{Measured}$	$R_{Predicted}$	% Error
Air(Reference)	4.10	1.0000	1.0000	0.00
Water	3.89	0.9925	0.9967	0.42
30% Glycerin	3.85	0.9910	0.9948	0.37
50% Glycerin	3.80	0.9892	0.9918	0.26
80% Glycerin	3.76	0.9877	0.9770	-1.09
100%Glycerin	3.64	0.9831	0.9040	-8.75

Table 1: Reflection factors using L wave that is measured and predicted.

Viscosity Measurements involving Ultrasonic Interferometric Techniques: The ultrasonic interferometer was evaluated for the viscosity (in Poise) versus the Glycerin concentration as shown in Figure 4. The calibration curve obtained for reflection factor as a function of density-viscosity product is shown in Figure 5.

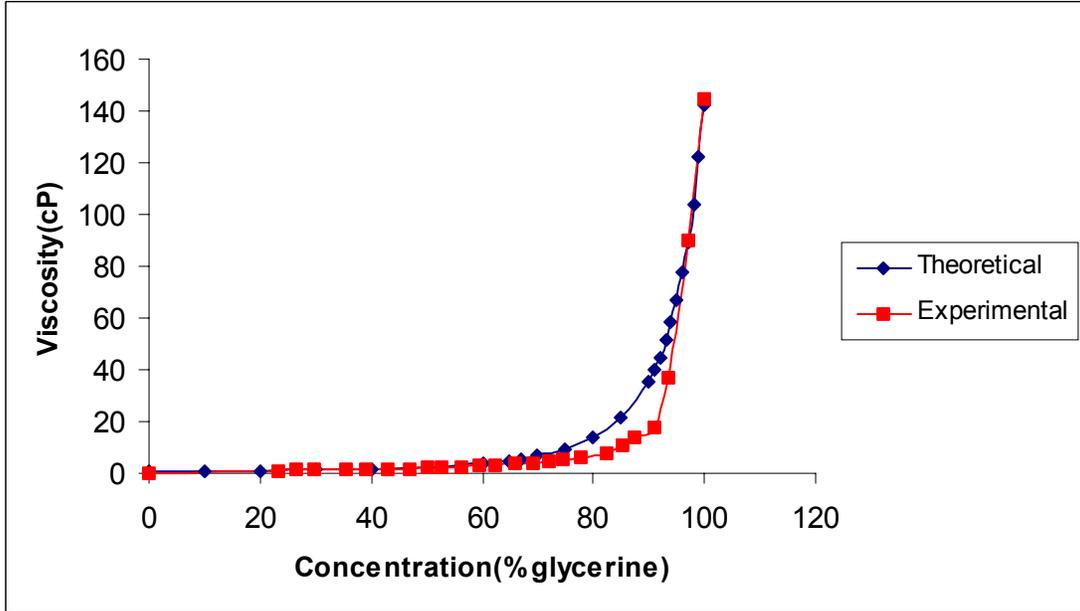


Figure 4: The measurement using the ultrasonic interferometer technique.

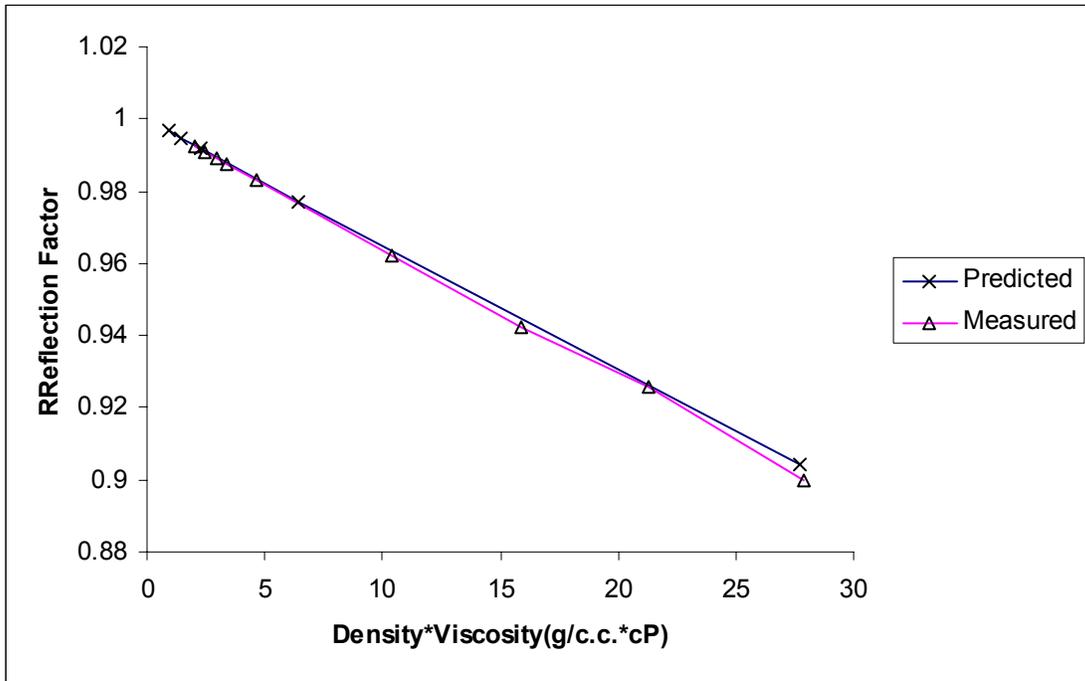


Figure 5: Comparison for Reflection factor .vs. Density-Viscosity product obtained using ultrasonic interferometer method.

Discussions: The density data in Figure 3 that were obtained shows a more or less linear profile, which is to be expected. Similar results are observed for Bulk Modulus measurement, also obtained by employing longitudinal waves. For the viscosity measurements, both the techniques explored here show good promise. For solutions with low percentage of Glycerin, the reflection factor is close to unity. This is expected, as low viscosity fluids do not support shear waves. To get observable change in reflected amplitude, the amplitude corresponding to seventh echo was measured. The results are compared with the predicted values, which are deduced using standard data available for *Glycerin-Water* solutions. The results in Table 1 show that the viscosity measurement is within 5% up to 80% concentration of Glycerin. However, the change in viscosity was found to be small at low concentration but shoots up at higher concentrations. Beyond this range the viscosity of the solution increases radically and subsequently, larger errors are encountered. In this region, the assumption of small value for β is violated and hence the technique requires modification. However, the method can be successfully used for quantification of viscosity for light liquids that are often encountered in many Industrial processes. The small errors may be expected due to the fact that plexiglass has a relatively higher value of acoustic impedance among commonly used materials. The higher the acoustic impedance of the wedge, higher will be the impedance mismatch and hence, prone to error. The sensitivity at lower range of viscosity is about 200 cP for a unit change in magnitude. Also the size of the probe is very small and can be used for measurements in-situ. The wedge also provides for self-calibration regarding changes in temperature, pressure, etc.

The ultrasonic interferometer method was found to be very reliable over the entire region of the experiment including the high concentrations of glycerin. However, this method involves calibration and nulling that may create difficulties in practice.

Conclusions: Two ultrasonic methods for the measurement of Density, Bulk Modulus, and Viscosity, were evaluated using glycerin-water mixtures. Both methods used longitudinal waves for measuring the first two parameters and the shear wave for the viscosity measurement. It was observed that the rheology cell had some difficulties for higher concentrations of glycerin which was not the case using the interferometric method. In general the advantages of this combined probe can be summarized as follows:

- (a) It is able to predict a number of material properties such as density, viscosity and bulk modulus.
- (b) If both the probes are used in tandem, a wide range of the properties, significantly the viscosity could be quantitatively obtained with a very little loss in terms of sensitivity or error.
- (c) The portability and the very minimal requirement for the probes (voltage source and a computer with suitable A to D cards) tend to make the methods more robust and hence increase their ability to cater to wider applications.

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