Experimental Methodology
CHAPTER III

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REFERENCES
3.1 Techniques for Solution Preparation:

In the present investigation the ultrasonic studies and associated thermodynamic parameters were evaluated for the following liquid mixture:

1. Urea + Water
2. Gelatin + Water and
3. Urea + Gelatin + Water

The above mentioned liquid mixtures of various concentrations in mole fraction were prepared by taking purified AR grade sample at 303 K. In the entire mixtures gelatin from BDH were used in this study. A standard solution of 10% gelatin was prepared. To enhance the dissolution, the mixture was kept in warm water bath during the preparation. From the above standard solutions of concentrations 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10% were prepared.
Urea has been used to prepare inorganic salt solutions. For urea the concentrations were 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 mole/lit.

For measuring the ultrasonic velocity and density, for the protein and salt mixture were prepared, by adding 20ml. of protein solutions of ideal concentration has mixed with 10ml. of urea salt solutions and different concentrations specified above and stirred for 48 hours and then the measurements were performed (protein paper).

3.2 Techniques for measurement of ultrasonic velocity:

There are different methods for the measurement of ultrasonic velocity. Among many few methods are discussed here......

1. Optical Method
2. Acousto-optical Method
3. Pulse Method
4. Sing-around method
5. Interferometer Method
3.2.1. Optical Method:

Principle –

To study the velocity and absorption of ultrasonic waves the optical method is based on the diffraction of light. The diffraction of light occurs as it passes through a medium in which ultrasonic waves are being transmitted (2-6).

In this optical diffraction method measurements can be made of the velocity of sound in a liquid, at temperature starting at its melting point up to close to its critical temperature. This method was also used successfully, for measuring the velocity of sound in superheated and saturated vapors and in the critical region of liquid vapors system.

3.2.2. Acousto-optical Method:

Principle –

An ultrasonic wave passing through a medium creates a periodic change in the density of the medium and consequently in its refraction index. The spatial variations in the refraction index at a given moment form a phase grating.
The light passing through such a grating gets diffracted in one or more directions. It is easy to show that in the Raman–Nath region of diffraction, the frequency of the diffracted light is shifted by an amount which is multiple of the frequency of the ultrasonic wave passing through the crystal (7). This shift in the frequency makes it possible to use optical heterodyning for the detection of the diffracted light in the Raman–Nath region. As the +1 or -1 order diffracted light is heterodyned with the zero order-diffracted light. One obtains photoelectrical signal at the photo multiplier tube placed in the Fresnel region of the diffraction. This signal contains the two parameters, which are amplitude and phase velocity of ultrasonic waves (8). The phase velocity of ultrasonic wave is measured by scanning the sample by a laser beam along the direction of propagation of ultrasonic wave and measuring n cycles of 2π phase changes of heterodyne signal.

Ultrasonic velocity is calculated by

\[ u = \frac{df}{\lambda} \]

where

\[ u = \text{ultrasonic velocity} \]
3.2.3 Pulse Method:

Principle –

In pulse method, time required for a pulse to travel forth and back, in the space between source and refractor is measured.

Pulse method is used to measure ultrasonic velocity in solids and liquids (9-11). The pulse oscillator supplies short radio-frequency pulses to the quartz crystal. The electric pulse is changed into an ultrasonic one, which after going through the medium under investigation is then reconverted into an electric pulse by the quartz transducer. The ultrasonic pulse, after passing through the medium under investigation and after being converted into an electric pulse, is amplified by the receiver and displayed as a vertical pulse on the oscillator at the chosen time after the transmitter pulse.

The pulse generated triggers the time base of the oscilloscope. The change in the path length for the ultrasonic
pulse causes a change in its time delay, which is observed on the oscilloscope. The delay time is used for determining the ultrasonic velocity, and is measured on the calibrated time based of oscilloscope. The variation of the amplitude of the electric pulse at the receiver input is used to determine the absorption. The precision of this method is few parts per thousand.

Several pulse techniques with high degree of precision have been developed for ultrasonic studies (9,12-13). They obtained better accuracy by the use of sing around technique. The reverberation method was developed for the measurement of ultrasonic velocity (14).

The use of pulse technique has two main advantages over the continuous wave technique (interferometer and optical diffraction). (1) Elimination of standing waves.

(2) Minimization of local heating effects.

3.2.4 Sing-around method

This is an automated method of measuring ultrasonic velocity to a moderately high accuracy and for monitoring changes in ultrasonic velocity to high accuracy. It is good,
but not necessarily an ideal method for absolute measurements, but useful for certain types of relative measurements. The measurement must be made through transmission with two transducers.

In this method, the principle applied is the signal from output transducer is taken to trigger the input signal and generate ultrasonic pulses repeatedly. The transit time of ultrasonic wave propagating across the sample is determined by the repetition time of pulse sequence. Each pulse sequence includes many transit time which enhances the accuracy of velocity measurement.

The sing around velocity method of Satyabala et. al.[15] has moderate accuracy whereas high speed ultrasonic sing around system of Whitehead and Palmer [16] is capable of measuring velocity of sound in a sample to an accuracy of few parts in $10^4$ in period of 10 microsecond. Performance of studies of high speed ultrasonic sing around system has been carried out by Jiles et al.[17]. They have used this method to calculate ultrasonic velocity in electrolytic solutions. As discussed in the principle if pulse repetition
time is $T$ (called sing around time) and external delay is $T_e$, then sampled delay is

$$T_s = T - T_e$$

Knowing $T_s$ and measuring length of sample $L$, ultrasonic velocity through the sample can be found from

$$V = \frac{L}{T_s}$$

### 3.2.5 Interferometer Method:

**Principle** –

This method is based upon the formation of standing waves in the liquid, between the transducer and a flat receiver. By measuring wavelength of these waves, according to a formula

$$U = f \cdot \lambda$$

And by knowing frequency $f$, the velocity ‘$u$’ can be determined.

Ultrasonic waves of known frequency are produced by an X-cut quartz crystal which is fitted at the bottom of the cell. These waves travel through the liquid and get reflected from a movable flat metallic plate, kept parallel to the crystal
surface. When distance between the transducer and reflector, is an integral multiple of half wavelength, stationary waves are formed, in the medium. The reflected waves received back at the crystal are out of phase. The resulting decrease in the amplitude of crystal oscillations is accompanied by an alternating current through the crystal. The driving oscillator is loosely coupled with the LC circuit having quartz crystal in parallel with the condenser. Both LC circuit and an oscillator are tuned to the resonating frequency of the crystal and the crystal current is measured when the position of the reflector is changed, there is variation in crystal current (fig 3.1 & 3.2). The distance between the successive maxima or minima is half of the wavelength in the medium, which can be measured with the help of screw movement on the scale along with the reflector and by knowing \( \lambda \) and frequency \( f \), velocity of waves can be calculated.

(a) Interferometer Assembly –

An interferometer used in the present work was supplied by Messers Mittal Enterprises, New Delhi (F81). The line diagram is shown in fig.4.3. It consists of
1. Quartz crystal,
2. R. I. Oscillator,
3. The cell and the measuring assembly and
4. Thermostat

1. Quartz Crystal

Crystalline form of silicon dioxide (SiO₂) is called as quartz crystal. It is a crystal of the trigonal class possessing three polar axes at 60° to each other. These are the X-axes of the crystal and all are equivalent, Y-axes lie in the same plane but are perpendicular to this plane. In this case of quartz, the piezoelectric modulus sensor has fixed components, only two of which are independent. A widely used transducer is the X-cut crystal whose flat faces are perpendicular to the X-axis. For such a cut, the application of stresses in X direction produces a polarization component in the same direction, similarly an application of electric field in the X direction gives rise to the strain in X direction. Thus, such a cut of crystal is of use in producing and detecting compressional waves in the X direction.

A transducer for providing ultrasonic waves is a circular plate of diameter 1cm cut from X-cut crystal. This
plate is coated with gold from both sides to act as electrodes when a P.D. 'V' is applied between them, the thickness changes by

\[ u = d \times V \]

where, \( d = 2 \mu \mu /V \).

On the other hand, if the plate is compressed by a force \( F \), the electrodes acquire electric charge.

\[ q = d \times F \]

where,

\( d \) is the same value as before, or expressed in a slightly different through equivalent way, is approximately 2 \( \mu \mu \) coul /N.

If the voltage is reversed, the strain changes sign i.e. changes from extension to contraction or vice-versa. So when alternating voltage is applied across such transducer, it vibrates and when the natural frequency of vibration of the crystal is equal to the frequency of applied voltage, resonance takes place and amplitude of oscillation becomes very large, great strains are produced which may crack the quartz plate. Hence a. c. voltage applied across the crystal is kept low.
2. R.I. Oscillator

This is a fixed frequency oscillator, the frequency of which is controlled by quartz crystal in the power amplifier of the circuit (fig 3.2). When R.I. power is drawn, the micrometer shows constant current, due to d. c. voltage applied across it. An oscillator provides the voltage of about 400 volts across the crystal. R.I. generator is used to excite the X-cut quartz, crystal fitted at the bottom of the measuring cell.

3. Cell and the measuring assembly

Different parts of an interferometer can be described as follows. These parts can be separated from each other.

i) The Base:

It is a rectangular metallic heavy base having a space at the center equal to the outer diameter of the cell. So that it can be placed in the base. At its center, there is an electrode isolated from the base and pointed upwards to make an electrical contact with the crystal at the bottom of
the cell. Electrical contact of crystal is made with an oscillator from the side of the base by a shielded cable. Cell can be tightly fitted into the base by a side screw present in the base.

ii) The cell:

It is specially designed double walled. Cylindrical vessel made of stainless steel having outer diameter 3.96 cm, inner diameter 0.87 cm and height 9.25 cm. It has a capacity of 12 ml of liquid. For maintaining temperature of the liquid constant, water can be circulated through the annular space between the walls. It has an X-cut quartz crystal of diameter 10 mm fixed at its bottom exactly at its center.

Upper surface of the crystal plate is covered with a diagram to prevent the liquid coming in direct contact with the crystal surface. The inner wall of the cell is corrugated to prevent the wall reflections.

The cell has got a small hole in its bottom from lower side upto the crystal so as to fit it, in its bottom from lower side upto the crystal so as to fit it, in the base and to have contact of an electrode with the crystal.
iii) **Measuring Assembly**:

The reflector is connected with the plunger (E) which slides through a tightly fitting cylindrical bore (F), ensuring a self parallel displacement of the reflector. Upper end of the plunger is connected to the lower end of the screw by means of a locking ring. The screw forms the part of the screwguage, which has a least count of 0.001 cm. Hence the position of the reflector can be accurately, measured up to the fourth place of decimal. The distance of 2.5 cm is available for the movement of the micrometer screw. The whole assembly can be screwed on the top of the cell as a cap.

(b) **Thermostat Assembly** –

Velocity of ultrasonic waves in liquid is extremely sensitive to changes in temperature. Hence it becomes necessary to obtain a good stability of temperature. Thermostat of temperature stability of ± 0.5°C with a pump was used for this purpose.
The ultra thermostat incorporates the following main units: the thermostat bath, thermostat relay, the contact thermometer, the control thermometer, thermostat pump and valve relay.

i) Thermostat Bath:

The thermostat bath comprises a rectangular reservoir with a bath capacity of approximately 0.5 liters. The cover plate is provided with a bath opening and the filling aperture. Between both the openings, there is an adjustable lifting platform able to take an insert pot, the useful capacity of pot being 5 liters. The filling aperture is closed by the lid covering bath opening is possible optionally with the insert lid or water bath rings. The cover plate is provided with sockets for accommodating the contact thermometer (0°-100°) as well as protecting the tube and a rotary magnet for the contact thermometer.

ii) Thermostat Relay:

The thermostat relay is fixed on the cover plate of the thermostat bath. The seven contact switch serves for
switching on the instrument and adjusting the required degree of heating power. The signal lamp indicates the connection state of contact thermometer.

The connecting cable provides the connection between the thermostat relay and the contact thermometer. The mains cable connects the thermostat to mains supply through a safety socket.

The contact thermometer lies in the grid circuit of an electric valve. Interpolated into the anode circuit of this valve is the coil of a mercury relay, controlling the heating power of the thermostat.

iii) Thermostat pump:

The double acting pump is fitted into an appropriate hole on the cover plate of the thermostat bath and fixed by means of quick acting fastening devices. The pump keeps the temperature regulating liquid in a constant flow and feed it, in case of necessity, to an external user. The pressure pump pushes the temperature regulating liquid, from the bath to the external consumer where as the suction pump gets it from the external consumer back into the bath.
Interferometer has two advantages:

1. It is very simple technique,
2. It requires a small amount (i.e. 12ml) liquid.

Due to design, the effect of reflection of ultrasonic waves from wall of the cell can be minimized. As a result of design the Interferometer technique seems to have more accuracy than the other techniques.

In view of the above merits of Interferometer technique of ultrasonic velocity measurement, it has been used in present investigation for the study of ultrasonic velocities in different solutions.

3.3 Density measurements:

While applying different theories to the mixtures of liquids, it is observed that volume factor is the most important one in case of mixture. Hence stress was given to measure densities of different liquid solutions very accurately. Density is measured with the help of density bottle or pyknometer.
3.3.1 Measurement of density using density bottle

Density bottle is a small bottle of 5-25 ml capacity with a capillary stopper fitted in its mouth. The experimental liquid is filled in the bottle up to the mouth and the stopper is fitted tightly. A small amount of the liquid flows out of the capillary and the liquid fills the bottle and the capillary right up to the upper end. After carefully cleaning the empty bottle, experimental liquid is filled in the density bottle. Density bottle is immersed in a water bath, at a required constant temperature with a neck of bottle above the level of water in water bath.

When the bottle has attained the equilibrium temperature, it is removed from the water bath and its outer surface is wiped carefully by means of a cloth. Weight of the bottle is measured carefully, and then weight of the experimental liquid is calculated. For weighing a monopan digital balance having least count as 0.0001gm is used.

3.3.2 Measurement of density using Pyknometer

The pyknometer is a vessel of accurately defined volume as shown in (Fig 3.2).
Fig. 3.2: Pyknometer

A pyknometer is a U-tube with a cylindrical bulb and two capillary arms A and B. One end of the arm is drawn into a point and the other arm carries a mark, B so as to define the volume of the apparatus.

Initially the pyknometer is thoroughly cleaned with chromic acid followed by with water and drying of it. It is hanged upto the stand before it is weighed. Then the pyknometer is filled with air free given liquid by dipping the capillary end A in liquid while keeping the vessel in it's inverted position and sucking gently through a rubber tubing attached to the arm B. By means of a hook, wire, suspended
the pyknometer in a thermostat (not the capillary end) at the required temperature for 15-20 minutes, so that temperature of liquid and thermostat become in equilibrium. The amount of water in the pyknometer is adjusted so that it fills the vessel from the mark B to the tip of the arm A. Then the pyknometer is removed from the thermostat and wiped carefully its outer surface by a cloth to see it is completely dried. The pyknometer is then weighed. The same process is repeated for distilled water.

By using the following relation (1) the density is calculated:

\[ \text{Density (} \rho \text{)} = \frac{\text{Mass (m)}}{\text{Volume (v)}} \quad \text{--------- (1)} \]

In the present work “A” grade density bottles were used for the determination of density of the solutions.

3.4 Viscosity Measurement:

The viscosity is an important physical quantity. A large number of instruments have been developed to measure it (18). A viscometer provides means of measuring the rate of flow in the liquid and the force exerted or work done. In
rotating cylinder viscometers a thin film of liquid is sheared between inner and outer concentric cylinders.

In the coquette viscometer the outer cylinder is rotated at constant speed and the force exerted on the inner cylinder is determined by the steady deflection of the torsion wire on which the inner cylinder is suspended.

All the other methods depend on the flow relationship derived from the fundamental equation viz. Poiseuille’s equation for flow (J.L.M. Poiseuille in 1844)

$$
\eta = \frac{\pi pr^4t}{8Vl} \quad \text{(2)}
$$

where,

\[ V = \text{Volume of fluid with viscosity } \eta, \text{ passing through a tube of length } l, \text{ radius } r, \text{ in time } t. \]

When a pressure difference \( p \) is maintained between ends of capillary tube. This equation holds accurately for ‘streamline flow’ but not for the “turbulent flow”, which sets in at high velocities.

Another well-established accurate hydro-dynamic law is Stokes’s law of the terminal rate of fall (v) of a sphere (radius \( r \), density \( \rho_1 \)) through a liquid density \( \rho_2 \) namely –
In the falling sphere viscometer a ball bearing is timed in falling a measured distance through a cylindrical tube of liquid. This method is particularly suitable for viscous oils. The simple apparatus developed by Gibson and Jacob (19) is suitable.

Other methods of measuring viscosity depend on more complicated system of flow for example, the damping of torsional oscillations of a disc suspended in a liquid on a torsional wire (20) for a hollow sphere (21) or hollow cylinder (22) counting the liquid.

The viscosity measurement is usually done with the help of Ostwald viscometer (10). The principle involved can be easily understood from Fig. 3.3 which represents a simple viscometer (W. Ostwald);
It consist essentially of bulb A with a mark above (x) and below (y), attached to a capillary tube B and a storage bulb C. sucked into A, and the time $t$ is observed for the liquid to flow between the marks $x$ and $y$; the experiment is repeated with another liquid. The pressure at any instant driving the liquid through the capillary B is equal to $h \rho$, where $h$ is the difference in height between the levels of the liquid in the two limbs; this varies during the experiment, but as the initial and final values are the same in every case, it is evident that the applied pressure is proportional to the density $\rho$ of the liquid. It follows, therefore, from Poiseuilles
equation since the same capillary tube is employed, i.e. r and 1 are constant, and the same volume v of liquid flows through it in each case, that for two liquids 1 and 2,

The ratio of viscosity of two liquids is given by —

\[
\frac{\eta_1}{\eta_2} = \frac{\pi p_1 r^4 t_1 / 8 L v}{\pi p_2 r^4 t_2 / 8 L v} = \frac{p_1 t_1}{p_2 t_2} \quad \text{-----------------(4)}
\]

\[
\frac{\eta_1}{\eta_2} = \frac{h \rho_1 g t_1}{h \rho_2 g t_2} \quad \text{-----------------(5)}
\]

\[
\frac{\eta_1}{\eta_2} = \frac{\varrho_1 t_1}{\varrho_2 t_2} \quad \text{-----------------(6)}
\]

Knowing \(\varrho_1\), \(\varrho_2\), \(\eta_1\) measuring \(t_1\) & \(t_2\) we can able to find \(\eta_1\) i.e. viscosity of liquid another consideration.

By keeping the viscometer in water bath, at a desired temperature and allowing it to 15 to 20 minutes to attain thermal equilibrium, we can find out viscosity of liquid at destined temperature.
The ultrasonic velocity of the mixture was measured by using the continuous wave variable path interferometer with accuracy 0.03%. The density of the mixture was measured by using a specific gravity bottle with the accuracy of ± 0.0001 gm/cm³. The viscosity was measured using Ostwald's viscometer with the accuracy of 0.1%.

3.5 Methods of measurements of properties in the present work and calibration of instruments.

A) Density

For the measurement of density of the liquids/liquid mixtures that are under investigation in the present work, the specific gravity bottles are used. They are calibrated by reproducing the available data (i.e. literature values) of density of water at different temperatures as tabulated in Table 3.1

B) Viscosity

The viscosity measurement of samples is carried out using Ostwald's capillary viscometer, and the data of
viscosity of water at different temperatures reproduced for the calibration is given in Table 3.2

C) Ultrasonic velocity

The ultrasonic velocity measurements of liquids that are under investigation in the present work are made by using ultrasonic interferometer. The instrument being calibrated by reproducing the data for ultrasonic velocity of water at different temperature as given in Table 3.3
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Fig. 3.2 Experimental set up for ultrasonic study of liquid mixtures