3.1 INTRODUCTION

Polymer blends have a great market value now a days because of their peculiar property profiles, cost/performance and processing advantages. Their superiority over homo-polymers can be compared to those of alloys over metals. While many of the polymer blends may not be alloys, they may be considered as polymer mixture having various degrees of compatibility\(^1-^3\).

Some extent of compatibility is essential in a polymer blend for various reasons: so as to achieve mechanical integrity and interface adhesion, better processing and optimum property gains, etc. Being a key parameter in the tailoring of polymer blends, compatibility is calculated theoretically\(^3\) as well as estimated experimentally by dynamic mechanical\(^4\) and thermal\(^5,^6\) methods (DMA, DSC), electron microscopy\(^3\) (SEM/TEM), etc. which are sophisticated in nature. Since large variations in compatibility of polymer blends are possible, [e.g. by varying the size\(^3\) of the dispersed (elastomer) phase, by grafting of phases\(^3\) (ABS, HIPS) or by the addition of a compatibilizer\(^7\) (usually a block copolymer\(^8\))], there is a need to find a simple and quick method for determining compatibility.

The viscosity method\(^9,^11\) is simple, requires no expensive equipment, yet offers a classification of the compatibility of polymer blend constituents into incompatible, semi-compatible and compatible. The basis for using dilute solution, viscosity as a parameter for compatibility characterization lies in the fact, while in solution macromolecules of both types may exist in a molecularly dispersed state and undergo mutual attraction or repulsion, thereby rendering
positive or negative influences on viscosity. Interactions among polymer blend constituents are reflected in the solution viscosity of polymer blend, because polymer-polymer interactions usually dominate over polymer-solvent ones. The viscosity method derives its versatility from the following facts.

- Thorough mixing in molecular dimensions is possible only in polymer solutions. Besides, solvation in a mutual solvent screens out extremely incompatible polymer mixtures.

- Results are not affected by the crystallinity or the morphological states of polymer blend.

- Phase inversions, if present, can be detected in polymer blends from viscosity vs. composition plots$^{9,10}$

- No sophisticated equipment is necessary.

- The compatibility assessed by this method in fact relates to the compatibility of polymer blends in highly viscous or solid forms inferred$^6$ by DSC, DMA, SEM, etc. Because of these advantages, the polymer blend compatibility was studied by viscosity method by many workers.

3.2 THEORY

3.2.1 Solution viscosity

The viscosity measurements of polymer solutions constitute an extremely valuable tool for the molecular characterization of polymers. Polymer solution viscosity is basically a measure of the size or extension in space of polymer molecule. Parameters derived from measurements of dilute solution viscosity can also be empirically related to the molecular weight and to the interaction between polymer and solvent. They may also be used in studies of chain stiffness, chain branching, polydispersity of polymers and association of polymers in solution.
3.2.2 Dilute polymer Solutions

Conventionally, polymer solutions can be considered dilute, if the molecules of the dissolved polymer practically have no polymer-polymer interactions. Owing to the length of chain and flexibility of polymer molecules, the volume per molecule within which it may meet other molecules, increases with increasing molecular weight. Therefore, the boundary between concentrated and dilute polymer solutions depends on the molecular mass i.e. higher the degree of polymerization of the polymer the lower will be the limiting concentration. In practice, this limiting concentration is usually less than one per cent.

The viscosities of even very dilute solutions of high polymers are always greater than those of solvents. As said earlier, this is a consequence of the large size of the polymer molecules, relative to those of solvents and their extension in space.

If \( \eta \) is the viscosity of a dilute polymer solution (concentration of polymer not more than about 1 g/dl and \( \eta_0 \) that of the solvent, the ratio \( \eta/\eta_0 \) is called relative viscosity or viscosity ratio and is denoted as \( \eta_r \).

The increment of viscosity due to the polymer may be represented by the specific viscosity \( \eta_{sp} \).

\[
\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} \tag{3.1}
\]

The ratio \( \eta_{sp}/C \) where \( C \) is the concentration of the polymer, is called the reduced viscosity or viscosity number. The value of \( \eta_{sp}/C \) at the limiting concentration \( C \to 0 \) is the limiting viscosity number \([\eta]\), more usually, called the intrinsic viscosity.
The limiting viscosity number is almost invariably expressed in terms of volume per unit weight of the polymer (i.e. ml/g or dl/g). The limiting viscosity number $[\eta]$ is dependent on the nature of polymer, solvent and average molecular weight ($M$) of the polymer.

(a) A very important empirical relationship between $[\eta]$ and average molecular weight ($M$) is the well-known Mark-Howink equation.

$$[\eta] = K \cdot M^a$$  \hspace{1cm} \text{(3.3)}

where 'K', and 'a' are constants for particular pair of solvent and polymer. Thus, the intrinsic viscosity $[\eta]$ is a measure of the contribution of individual polymer molecules to the viscosity.

(b) Intrinsic viscosity data have been used to determine chain dimensions of polymer molecules in solution using the following relationship \(^{12}\) (3.4)

$$[\eta] = \frac{\Phi (r^2)^{3/2}}{M}$$  \hspace{1cm} \text{(3.4)}

where, $\Phi$ is a constant, 'r' is root mean square of end-to-end distance of the coiling chain.

The unperturbed dimensions of polymer chains under theta conditions of solvent and temperature have been obtained by the relationship\(^ {13}\) (3.5)

$$[\eta] = \frac{\Phi (\bar{r}_0^2)^{3/2}}{\bar{M}^{1/2} \cdot \alpha^3}$$  \hspace{1cm} \text{(3.5)}
where

$$\overline{r_0^2} = \text{unperturbed linear chain dimension, root mean square of end to end distance of coiling chain}$$

$$\alpha = \text{Expansion factor.}$$

The relationship between dilute solution viscosity and polymer concentration has been described by various functions, all of which have been used to obtain intrinsic viscosity $[\eta]$ extrapolation to limiting zero concentration $C$. The most extensively used equation is that due to Huggins $^{13}$ (3.6)

$$\eta_{sp} \frac{1}{C} = [\eta] + K'H[\eta]^2C \quad 3.6$$

where, $K'H$ Huggins constant is said to be a constant for a given polymer-solvent pair. Its value usually lies in between 0.3 (in a good solvent) and 0.5 (in a poor solvent). Thus it is also a measure of solvent power for a polymer.

For flexible chain polymer, an alterative expression (3.7)$^{14}$

$$\frac{\ln (\eta/\eta_0)}{C} = [\eta] - \beta[\eta]^2C \quad 3.7$$

is sometimes employed. The plot of $[\ln(\eta/\eta_0)/C]$ against concentration $C$, is less steep. The two plots can be combined together facilitating extrapolation, since both lines have the same ordinate intercept.

The constant $K'H$ and $\beta$ are related by

$$K'H + \beta = 0.5$$

Equations (3.6) and (3.7) permit evaluation of intrinsic viscosity from measurement of $\eta_{sp}$ at a single concentration, if values of $K'H$ and $\beta$ for polymer-solvent pair are known.
A generalized dependence of the viscosity of dilute as well as moderately concentrated polymer solutions has been obtained by Budtoa\textsuperscript{15}, Kuwahara\textsuperscript{16}, Solomon and Ciuta\textsuperscript{17} and Narr et al\textsuperscript{18}. The most general expression for $\eta_{sp}/C$ as a function of $C$ is the power series.

$$\frac{\eta_{sp}}{C} = a_1 + a_2 C + a_3 C^2 + \cdots \quad 3.8$$

from hydrodynamic theory $a_1 = [\eta]$ and $a_2 = K [\eta]^2 \quad 3.9$

where, $K$ is a hydrodynamic interaction constant arising from hydrodynamic interaction between separate polymer chains. For dilute solution of low and moderate molecular weight polymers $K$ is usually identified with $K'_H$. For high molecular weight polymers in dilute solution, chain entanglement leads to higher value of $K'_H$, which will then exceed that of $K$.

Experimental variables affecting the values of intrinsic viscosity and $K'_H$ for a given polymer-solvent combination include the rate of shear and temperature at which viscosities are measured. Since, the value of intrinsic viscosity depends on the size and shape of the polymer chains (chain dimensions), it will vary with the molecular weight of the polymer and with the solvent. Other factors such as chain stiffness, polydispersity of polymer, chain branching, aggregation of polymer molecules etc. also affect the intrinsic viscosity and $K'_H$. Recently, Kashyap, Reddy and Kalpagam\textsuperscript{19} have observed variation of $K_H$ with composition for acrylonitrile methyl methacrylate copolymer in three different solvents, viz., 2-butanone, acetonitrile and N,N,-dimethyl Formamide. $K'_H$ is found to depend on both composition of copolymer and nature of the solvent.
3.2.3. Solution of Polymer blends

(a) Numerous attempts have been made to correlate the viscosity of a binary polymeric mixture to the viscosities of the components. Baker\textsuperscript{20} has expressed the viscosity of a single component as a function of its concentration. The relationship is known as a power law (3.10).

\[
\eta_r = \left[ \frac{[\eta]}{1 + \frac{C}{8}} \right]^8 \quad \text{3.10}
\]

(b) Hess and Philippoff\textsuperscript{21} have used the following relation for a binary polymeric system.

\[
[\eta]_{\text{mixture}} = \sum [\eta]_i x_i \quad \text{3.11}
\]

where, \(x_i\) = weight fraction of the \(i^{th}\) component.

They have found that the above modification gives better fit than those obtained by any of the mixture laws previously proposed.

(c) A simpler expression obtained by expanding power law for mixture of two components can result into the following relation \textsuperscript{22} (3.12)

\[
\eta_{sp\text{ mixture}} = [\eta]_2 C_2 + [\eta]_3 C_3 + 0.43 \left[ [\eta]_2^2 C_2^2 + 2 [\eta]_2 [\eta]_3 C_2 C_3 + [\eta]_3^2 C_3^2 \right] \quad \text{3.12}
\]

It is effective for fairly concentrated binary mixtures.

(d) The following empirical additivity law (3.13) gives satisfactory results to a mineral oil mixture of a methacrylate copolymer and polyisobutylene to a concentration up to 4 per cent (w/v)\textsuperscript{31}
Where $C_t = C_1 + C_2$

(e) Recently, Rudin, Hoegy and Johnston\textsuperscript{24} have studied the viscosities of mixed polymer solutions by applying a semi-empirical model to relatively concentrated solutions of mixtures of polymers in a common solvent. However, this model does not appear to be adequate for mixtures in which an extensive hydrogen bonding interaction occurs.

(f) Dimov, Dilova and Stoyanov\textsuperscript{25} have correlated (i) intrinsic viscosities of dilute solutions, and (ii) viscosity parameters such as ‘$K$’, ‘$\eta$’ in concentrated solutions of multicomponent blends, with composition of polymer mixtures by means of mathematical equations where, ‘$K$’ and ‘$\eta$’ have been defined by following relationship

$$\tau = K \cdot \gamma^n \quad 3.14$$

$\tau$ = shear stress
$\gamma$ = shear rate
$n$ = the deviation of concentrated solution from Newtonian behavior
$K$ = related to the viscosity index-indicating the interactions of supra molecular structure formation.

Similarly, physico-mechanical properties of a blend have been correlated to reduced viscosity of solutions of blend at different composition\textsuperscript{26}
Chee method

To quantify the miscibility of the polymer blends, Chee suggested the general expression for interaction parameter when the polymers are mixed in weight fractions $W_1, W_2$ as

$$
\Delta b = \frac{b - \bar{b}}{2 \omega_1 \omega_2} \quad (3.15)
$$

Where $b = \omega_1 b_{11} + \omega_2 b_{22}$, $b_{11}$ and $b_{22}$ are the slopes of the viscosity curves for the components, and $b$ is related to Huggin's coefficient $K_H$ as

$$
b = K_H [\eta]^2 \quad (3.16)
$$

for a ternary system. It is also given by

$$
b = \omega_1^2 b_{11} + \omega_2^2 b_{22} + 2 \omega_1 \omega_2 b_{12} \quad (3.17)
$$

Where $b_{12}$ is slope for the blend solution.

However, Chee's theory fails because experimental data are in conflict with the theoretical predictions. Using these values, Chee defined a more effective parameter

$$
\Delta B = \frac{\mu}{[\eta_1]^2 - [\eta_2]^2} \quad (3.18)
$$

Where $[\eta_1]$ and $[\eta_2]$ are the intrinsic viscosities for the pure component solutions. The polymer blend is miscible if $\mu \geq 0$ and immiscible when $\mu < 0$. 
Sun method

Recently Sun et al.\textsuperscript{28} suggested a most satisfactory new equation for the determination of polymer miscibility as

\[
\alpha = \frac{K_m - K_1 [\eta]^2 \omega_1^2 + K_2 [\eta]^2 \omega_2^2 + 2\sqrt{K_1 K_2 [\eta]^2 \omega_1 \omega_2}}{[\eta_1 \omega_1 + [\eta_2 \omega_2]^2}
\]

Where \( K_1, K_2 \) and \( K_m \) are the Huggin’s constants for individual components 1 and 2 and the blend respectively. The long range hydrodynamic interactions are considered while deriving the equation. The polymer blend is miscible if \( \alpha \geq 0 \) and immiscible when \( \alpha < 0 \).

3.2.4. Polymer-Polymer Interaction parameter and Polymer blend Compatibility

The miscibility of polymer mixtures is usually studied in bulk through various methods such as differential scanning calorimetry, dynamic mechanical analysis and electron - microscopy. The decision regarding the miscibility character of a mixture depends on the method of examination, mixing history, solvent choice when films are cast from solution and on the intended use of the mixture\textsuperscript{29}. Many investigators have studied polymer miscibility in solution in mutual solvents of blend components, because whether a bulk mixture is actually in an equilibrium state or not, may sometimes be uncertain\textsuperscript{27}. Thorough mixing in molecular dimensions is possible only in solution. Besides solvation in a mutual solvent screens out extremely immiscible polymer mixtures. Miscible components form, a single, transparent phase in the solution, while immiscible polymers exhibit phase separation\textsuperscript{30}. So, dilute solutions of mixtures of two polymers have been examined by numerous authors using viscosity method to know the compatibility of the polymer blends. As per this viscosity method, it is well known\textsuperscript{9,10} that plots of absolute viscosity vs blend composition deviate from linearity according to the degree of compatibility of polymer blends. Plots for
compatible systems are linear whereas for incompatible systems, plots are nonlinear, indicating two-phase formation with reversal of phases at intermediate composition.

The interaction parameter between the two polymers characterizes intermolecular interaction. These interactions include all kinds of physical interactions that are possible in solution. It can also give valuable information on the compatibility of the two polymeric components. The different methods to evolve the polymer-polymer interactions in polymer blend solutions and their approaches to explain the polymer compatibility are given in literature. The method used in the present study is given below.

(a) Additive Rule of Mixture Method

The compatibility or incompatibility of polymer blends can also be explained by using the additive rule of mixture proposed by Williamson and Wright. They studied a number of mixed polymer systems and concluded that the deviation of the observed viscosity from the ideal mixing viscosity are larger in the case of strongly interacting polymers and smaller in the case of weakly interacting polymers. The reduced viscosity follows the simple additive rule of mixtures when polymers are miscible. The calculated reduced viscosity ($\eta_{\text{red (cal)}}$) is given by the relation

$$\eta_{\text{red (cal)}} = \left[ \left( c_1 \eta_{sp1} / c \right) + \left( c_2 \eta_{sp2} / c \right) \right]$$

where $c = c_1 + c_2$; $c$ is the total concentration of the blend, $c_1$ and $c_2$ are the concentrations of components 1 and 2 respectively; $\eta_{sp1}$ and $\eta_{sp2}$ are the specific viscosities of the components 1 and 2 respectively.

If the plot of experimentally determined reduced viscosity versus composition at a fixed concentration of the blend solution is exactly coinciding with the calculated reduced viscosity values (using the additive rule of mixture), then such blends are considered as "Miscible blends". On the other hand, if it
deviates from the additive rule of mixture, it is considered as an immiscible blend. The interactions are said to be strong, if the deviation is positive and weak if the deviation is negative.

Singh et al., to explain the compatibilities of the polymer blends from solution viscosity measurements, have successfully used this additive rule of mixture phenomenon.

Viscometric study of interactions and miscibility has been successfully applied to the water-soluble polymers, grafted copolymers, electrolytes and even to the mixtures of polymer solutions and micro-emulsions.

In the present study the viscosity results are discussed from the point of view the following ways.

(i) Absolute viscosity Vs Concentration variation method.

(ii) Chee, Sun methods for the interaction parameters.

3.3 RESULTS AND DISCUSSION

Polymer-Polymer miscibility is generally considered as a result of the specific interactions between polymer segments. The specific interactions include donor-acceptor, dipole-dipole, H-bonding, ion-ion, acid-base and ion-dipole interactions. Basing on the constituent polymers in a polyblend it will be discussed for the following polyblend systems.

Chitosan (CS)-Poly (vinyl alcohol) (PVA) blends in 2%acetic acid

Chitosan and PVA are very useful polymers which are capable of forming inter polymer complexes through secondary bonding forces and often exhibit unusual properties. Chitosan is a cationic and rigid polymer used in water treatment, agriculture, food, cosmetics and biopharmaceutical etc., PVA finds numerous applications in adhesive, binders, paper and textile industries, emulsifying, dispersing films, ceramics in reprography and photography molded
products and cosmetics etc.,\textsuperscript{42} Hence the miscibility studies of CS/PVA blend might be important to enhance some of their performance properties in view of preparing more useful and products.

The densities ($\rho$) and absolute viscosity ($\eta$) for CS -PVA blends in 2\% acetic acid solution for different concentration of various compositions of the Blends (0\%CS +100\%PVA, 20\%CS +80\%PVA, 30\%CS +70\%PVA, 50\%CS +50\%PVA, 70\%CS +30\%PVA, 100\%CS +0\%PVA) at 30\°C, are measured and the results are presented in the Table.3.3.1.

The absolute viscosity Vs concentration curves for different composition of CS-PVA blends at 30\°C are shown in the Fig.3.3.1 from these figs. It is observed that the absolute viscosity varies linearly with increase in concentration of blend for all the blend composition. It was well established earlier by many workers,\textsuperscript{43, 44} that the variation of viscosity Vs composition plots are linear for compatible blends and non linear for incompatible blends. On this basis in the present study, the linear variation of the absolute viscosity with blend concentration in the case of CS-PVA in 2\% acetic acid has been attributed of the miscible nature of chitosan -gelatin blend in buffer solution was reported by Basava Raju \textit{et.al} \textsuperscript{45} from their dilute solution viscosity studies.

From Fig 3.3.1 it is observed that as the concentration of blend increases the absolute viscosity also increases. At the higher concentration of the blend, PVA can interact with several chains of CS and can from highly cross-linked structure, which causes high solution viscosity. Further, with the increase in composition of CS in the blend along with cross linking effect, the effect of hydrogen bonding between the hydroxyl groups of CS and PVA also increases. These effects at higher contents of Cs of these blends to enhance the cross linking effect as well as hydrogen bond effect leading to high viscosity of the blend which is observed in the present study.
Chee and Sun methods

In the present study the values of Chee’s differential interaction parameters $\Delta B$ and $\mu$ are calculated with the help of equations 3.15 to 3.18 (as explained in section 3.2.3) for the CS-PVA blend systems in 2% acetic acid solution at 30°C and presented in the Table 3.3.2.

It is observed from the Table 3.3.2 that the values of $\Delta B$ and $\mu$ are positive which indicate the miscibility nature of CS and PVA blends. Sun et al., has suggested new parameter $\alpha$ (given by the equation 3.19 explained in the section 3.2.3) for the determination of miscibility of polymer blends. The values of $\alpha$ calculated for the present systems are presented in Table 3.3.2. According to him a blend will be miscible if $\alpha$ is $\geq 0$ and immiscible if $\alpha$ is $< 0$. In the present study, the values of $\alpha$ are found to be positive indicating that PVA and CS are miscible blends due to the H-bond formation (see Scheme I).

![Scheme I. Schematic representation of the hydrogen bonding between CS and PVA](image)
This supports the conclusions drawn regarding the miscibility of CS and PVA blend in 2% acetic acid from the variation of absolute viscosity Vs composition of the polyblends explained above.

The absolute viscosity data for different concentration of various blends of 1% (w/v) (Chitosan/PVA) in 2% acetic acid at 40°C and 50°C are also measured and given in Tables 3.3.3 and 3.3.4 respectively. The variation of absolute viscosity with concentration for different blend composition of 1% (w/v) (Chitosan/PVA) blends in solution at 40°C and 50°C are shown in figs.3.3.2 and 3.3.3 respectively. From this data (Table 3.3.3 and 3.3.4) and figures 3.3.2 and 3.3.3 it is clearly observed that the variation of absolute viscosity with blend concentration and composition they exhibit linearly. This is a clear indication that this blend shows miscibility at higher temperatures also.
Table 3.3.1. Viscosity data for different concentrations of various blends of (1% w/v) Chitosan/PVA in 2% Acetic acid at 30° C

<table>
<thead>
<tr>
<th>Concentration of (CS/PVA) blends (g/dl)</th>
<th>Density ρ (g/cm³)</th>
<th>Absolute Viscosity η (cp)</th>
<th>Relative Viscosity η_r=η/η₀</th>
<th>Reduced Viscosity (η_r-1)/C (dl/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(0% CS + 100 %PVA)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.2</td>
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<td>(20% CS + 80 %PVA)</td>
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<td>21.8420</td>
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<td>29.8884</td>
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</tbody>
</table>
Fig 3.3.1. Plots of Absolute Viscosity (η (cp)) Vs Concentration (C) for different compositions of 1% w/v Chitosan/PVA blend solutions at 30°C

Table 3.3.2 Interaction Parameters μ and α of Chitosan /PVA Blend in 2% Acetic acid at 30°C

<table>
<thead>
<tr>
<th>Concentration of (CS / PVA) blends (g/dl)</th>
<th>ΔB</th>
<th>μ</th>
<th>α</th>
</tr>
</thead>
<tbody>
<tr>
<td>70/30</td>
<td>43.5733</td>
<td>0.07079</td>
<td>0.02101</td>
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<tr>
<td>50/50</td>
<td>15.0464</td>
<td>0.03710</td>
<td>0.01700</td>
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<tr>
<td>30/70</td>
<td>6.03706</td>
<td>0.01096</td>
<td>0.26281</td>
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<tr>
<td>20/80</td>
<td>2.07093</td>
<td>0.00375</td>
<td>0.41073</td>
</tr>
</tbody>
</table>
Table 3.3.3 Absolute Viscosity (cp) data for different concentrations of various blends of (1% w/v) Chitosan/PVA in 2% Acetic acid at 40°C.

<table>
<thead>
<tr>
<th>Concentration of (CS/PVA) blends (g/dl)</th>
<th>Absolute Viscosity (cp)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0% CS + 100% PVA</td>
</tr>
<tr>
<td>0.2</td>
<td>0.8683</td>
</tr>
<tr>
<td>0.4</td>
<td>1.4005</td>
</tr>
<tr>
<td>0.6</td>
<td>1.5025</td>
</tr>
<tr>
<td>0.8</td>
<td>1.5506</td>
</tr>
<tr>
<td>1.0</td>
<td>1.7410</td>
</tr>
</tbody>
</table>

Fig. 3.3.2 Plots of Absolute viscosity (cp) Vs Concentration (C) for different compositions of 1%(w/v)Chitosan/PVA blend solutions at 40°C.
Table 3.3.4 Absolute Viscosity (cp) data for different concentrations of various blends of (1% w/v) Chitosan/PVA in 2% Acetic acid at 50°C.

<table>
<thead>
<tr>
<th>Concentration of (CS/PVA) blends (g/dl)</th>
<th>Absolute Viscosity (cp)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(0% CS + 100% PVA)</td>
</tr>
<tr>
<td>0.2</td>
<td>0.8274</td>
</tr>
<tr>
<td>0.4</td>
<td>1.2523</td>
</tr>
<tr>
<td>0.6</td>
<td>1.3356</td>
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<tr>
<td>0.8</td>
<td>1.4140</td>
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<tr>
<td>1.0</td>
<td>1.4217</td>
</tr>
</tbody>
</table>

Fig. 3.3.3 Plots of Absolute viscosity (cp) Vs Concentration (C) for different compositions of 1% (w/v) Chitosan/PVA blend solutions at 50°C.
REFERENCES


