AGGREGATION BEHAVIOR OF POLYETHYLENE GLYCOL IN THE PRESENCE OF ELECTROLYTES AND NON-ELECTROLYTES

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Abstract

Polyethers are the class of organic substances prepared by joining together simpler monomer units by establishing ether links between them. Polyether is also called as Poly(ethylene oxide) or Poly(oxyethylene) based on source and structure respectively. Polyethylene glycole-8000 (PEG), which is highly biocompatible and processable, less toxic and has the greater affinity to couple due to its low molecular weight, as these polyethers are more viscous and colorless. As we know like dissolve like, they are extremely soluble in aqueous and organic solvents. In current work, we measured an exhaustive set of viscosities of Polyethylene Glycol (PEG) having equivalent molecular weight but varying salt concentration in both single as well as double phase section at constant temperature of 28 degree Celsius. As the more and more amount of salt is added, the solution elapsed time is increased and, therefore, the viscosity of solution is also increased. This increment of viscosity is due to the aggregation of PEG as a result of increased salt concentration which occupied the voids in Polyethylene Glycol making it more viscous. The different electrolytic and non-electrolytic salts which we have added in this particular study are sodium chloride (NaCl), sodium dodecyl sulfate (SDS), cetrimonium bromide (CTAB) and starch, glucose gelatin respectively which measures viscosity of PEG by means of Ostwald's model or Power law model using Ostwald's viscometer. In the midst of above work we observed that the viscosity of linearly fashioned PEG has been appreciably increased on increasing the concentration of the different salt and vice-versa.

Keywords: Polyether; Bio-compatible; Viscosity; Ostwald's model.

Introduction

Poly ethylene glycol(PEG) is among the most widely used synthetic polymers because of its hydrophilicity and biocompatibility in nature. PEGis an oligomer or polymer of ethylene oxide and its structure is commonly expressed as $H-(O-CH_2-CH_2)_n-OH$.The high molecular weight PEG is also known as polyethylene oxide (PEO) or polyoxyethylene (POE).[1]It is soluble in water and other organic solvents. The physical states (i.e., liquids or solids)of PEG and PEO depend on their molecular weights. The viscosity of the aqueous solution of PEG is the function of chain length whereas their chemical properties are nearly identical. PEG finds a wide range of applications in medicines, automobiles, petroleum, textile, food industries*etc.*[2]

Polyethylene glycol plays a vital role in the formation of aqueous two-phase system (ATPSs), which provides a powerful technique for the separation, purification of biological materials such as proteins, enzymes, plants, viruses, chloroplast, nucleic acids,*etc*. PEG based aqueous two phase system provides an environmentally safe process for the purification of biological materials and at the same time doesn't affect their activity.[3]Despite the success of ATPSs separation technique, data on physical properties of PEG at various concentrations and temperature are necessary for fundamental understanding of phase-forming ability and also for the development

of theoretical model for the prediction of partitioning behavior of two phase systems. During the last few years numerous studies have been carried out on ATPSs containing PEG. ATPSs have been numerously used in the fields like: biotechnology, chemical purification and extractive crystallization of organic salts.[4]

Both thermodynamic and transport properties of aqueous polymer salts are very important in chemical engineering and in other disciplines that contribute in understanding the fundamentals of separation process, fluid transport, waste-water treatment, *etc.* viscosities of aqueous solution are useful for pipe sizing, pump calculation, material balance calculation, and evaporator. They are useful in determining the nature of solute-solvent and solute-solute interaction. While viscosity data of aqueous PEG + saltsystem are available, correction and prediction of model with thermodynamic basis are scarce.[5]

PEG 8000is water-soluble, waxy solid that is used extensively in the several industries such as rubber, textile, paper, and metal, wood, pharmaceutical, cosmetics and coating. It provides enhanced solvency, lubricity, hygroscopicity, and other important functional properties.[6-7] It is highly compatible to various kinds of organic compounds, having high boiling points, degree of condensation and hygroscopic properties can easily be controlled, less toxic, with less skin irritation. It does not cause damage to skin or lipsin case of any contact.[1-2]

Viscosity is an important polymer property while analyzing liquid behavior and fluid motion near solid boundary. Viscosity of a polymer is a measure of its resistance of gradual deformation by shear stress or tensile stress. The shear resistance in a polymer is caused by inter-molecular friction exerted when a layer of fluid attempts to slide on one another.[8]

Absolute viscosity (coefficient of viscosity) is a measure of its internal resistance during flow. Dynamic viscosity is defined as the tangential force per unit area required to move one horizontal plane with respect to another plane at a unit viscosity when maintaining an unit distance apart in polymer.[8]

An Ostwald Viscometer is a device used to measure the fluid viscosity under one type of flow condition. This fluid viscosity represents the fluids resistance to flow, it can be thought of as the fluids friction, often referred to as internal resistance or thickness. Ostwald's viscometer is also known as an U-tube viscometer or capillary viscometer, the device utilizes the direct or reverse flow of the test fluid through a U-tube and the measurement is determined by noticing the line required for fluid to flow a certain distance through tubing of specific diameter.[9-10]

Mixed polymer-surfactant systems have broad applications. Depending on theidentity of the components the macroscopic properties can vary widely. The properties have their basis in the intermolecular interactions between the different components.An overview of these interactions will be provided and, with this is as background, different aspects such as phase separation phenomena, rheological properties, and interfacial behavior will be described.

Polymer molecules have low translational entropy and, therefore, the solubility is typically limited. The situation is different for polyelectrolyte because of the large entropy contribution from the counter ions.[11]

Conjugation with polyethylene glycol (PEG), known as PEGylation, has been widely used to improve the bioavailability of proteins and low molecular weight drugs. The covalent conjugation of PEG to the carbohydrate moiety of a protein has been mainly used to enhance the pharmacokinetic properties of the attached protein while yielding a more defined product.PEG is an amphiphilic polymer consisting of repeating units of ethylene oxide which may be assembled in linear or branched structures to give a range of PEGs with different shapes and molecular weights.[12] PEG must be activated for further conjugation by substitution of terminal OH by a functional group that could react with an appropriate site in the molecule to be conjugated, maintaining its biological activity.

Polyethylene glycol (PEG)-sugar compound has been investigated as cost effective shape-stabilized phase change materials for thermal energy storage. PEGs form internal hydrogen bonds stabilizing their chains at solid state. [13] However low molecular weight PEGs are liquid due to short chains as high molecular weight PEGs have too little concentration of hydroxyl groups. Therefore, glucose, fructose, and lactose are used as hydrogen bond source in this study.Number of hydroxyl chain ends which is responsible for the hydrogen bonds in PEGs is an important parameter because hydrogen bonding interactions stabilize PEGs. Low molecular weight PEGs are too short chains with very low melting temperatures and enthalpy and using external hydrogen bonding agents in low molecular weight PEGs decreases the melting temperature and enthalpy due to cryoscopy effect.

The number of hydroxyl chain ends is low at high molecular weight PEG .Thus using external agents to interact with PEG chains causes PEGs to stabilize in solid state.In this work sufficiently high molecular weight PEGs have been used and hydrogen bonds are supplied by the addition of sugar derivatives. The resultant system is high molecular weight PEGs stabilized with hydrogen bonds caused by external OH groups from sugar derivatives.

Methodology

Materials

Polyethylene glycol (PEG) was obtained from Central Drug House, New Delhi. It was used as received with average molecular weight of 8000, also known as PEG-8000 having melting point in range of (55-60 ^{0}C) and boiling point of ~ 200 ^{0}C. The density of polyethylene glycol was 1.1g cm^{-3}and appearance was white. NaCl, Gelatin, glucose, starch were purchased from CDH, New Delhi and were used as received.The surfactants of sodium dodecyl sulfate (SDS) and cetrimethylammonium bromide (CTABr) were procured from Central Drug House, New Delhi.Double distilled water (DMW) was used throughout the experimental work. The stock solution of PEG8000 was prepared in 100 ml standard flask using DMW. The stock solutions of SDS, CTABr, gelatin, glucose, starch were prepared in 100 ml standard flask of 0.1 weight percent. Also, a stock solution of NaCl of 2 M in 250 ml standard flask was taken.

Apparatus

An analytical balance with accuracy limit of$\pm$0.1 mg was to weigh the polymer, surfactants and electrolytes and non-electrolyte. Standard laboratory thermometer having the range from 0 to 100^{0}C was used to measure the temperature of the solutions. A three neck Ostwald's viscometer was used to measure the viscosity of the solution and was kept in water bath during the experiment.[14] The requisite temperature in the water bath was maintained within the temperature range of $\pm$ 0.5 ^{o}C. The other apparatus used were glass stirrer, beakers, conical flasks, funnels, pipettes and burettes. The time required to flow the solution through the Ostwald viscometer was measured using Stopwatch.[14] All the viscometric measurements were made at 28 ^{0}C.

Procedure

The viscosities of DMW, aqueous solutions of PEG8000 and PEG8000 with electrolyte/surfactants/non-electrolytes were determined using an Ostwald's viscometer at constant temperature of 28 degree Celsius in water bath. The flow time was measured by using the stopwatch for different weight percentage of aqueous PEG solutions. Thereafter, the same procedure was adopted to measure the flow time for the mixture of aqueous PEG with salt, surfactant or non-electrolytes. Suction pump was used to lift the solution level in the viscometer. The flow time was measured thrice for each solution and the observed results were found to be within the error limits of $\pm$ 5%.[15]

In this experiment, we have calculated the relative viscosity of the fluid by using the following relationship given in equation (4). The required concentrations of PEG8000, PEG8000 + NaCl, PEG8000 + surfactant and PEG8000 + non-electrolyte were obtained by mixing the required amount of materials from the stock solutions using the simple relation given in equation (5).

$$N_1V_1 = N_2V_2 \tag{5}$$

Where, $N_1$and V_1 are the concentration and volume of the required strength, respectively. $N_2$and V_2 are the respective concentration and volume of the stock solutions.

Results and discussions

Influence of the varying in the concentration of PEG8000 on the relative viscosity of

the solution.

The concentration of PEG8000 was increased from 2% to 20% in DMW by weight and the following change in relative viscosity was observed (Graph 1).
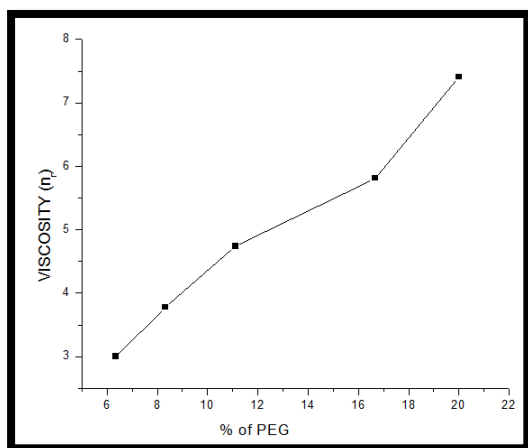
Graph 1. Plot of η_r vs PEG 8000 Solutions at $28 \pm 0.5\ ^0$C ($t_0 = 27$ s)

From the graph, it can be seen that at Conc. 11.11% there is a slight variation in the viscosity of PEG 8000. This is due to the variation in temperature which resulted into more mixing of PEG 8000 with DMW.

Influence of the varying in the concentration of gelatin at different [PEG8000] on the

relative viscosity of the solution

It is observed from Graph 2-4 that the viscosities of the solution containing gelatin and fixed concentration of PEG8000 (6% by weight) increases[16] with the increase in [gelatin] from 7.81×10^{-2} to 9.61×10^{-2} mol dm^{-3}. The plot of η_r *versus* [gelatin] is presented in Graph 2.
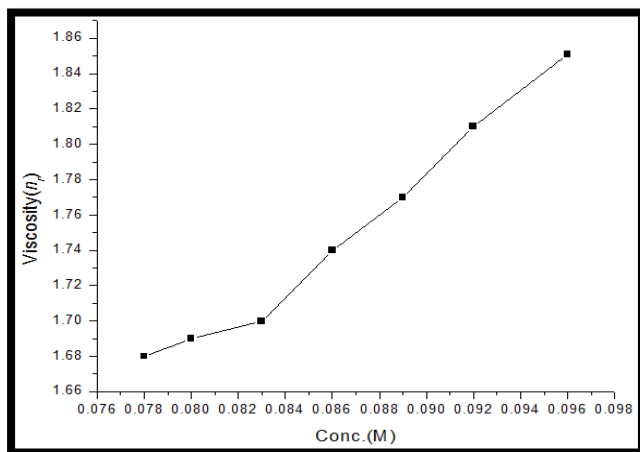
Graph 2. Plot of η_r *versus* [gelatin] for 6% PEG8000 solution at 28 ± 0.5^0C.(t_0=27 s)

From the above graph, it is seen that at Conc. 8.3×10^{-2} mol dm^{-3} there is a slight variation in viscosity. This is due to the structural complexation in solution because of the aggregation of Gelatin with PEG 8000.
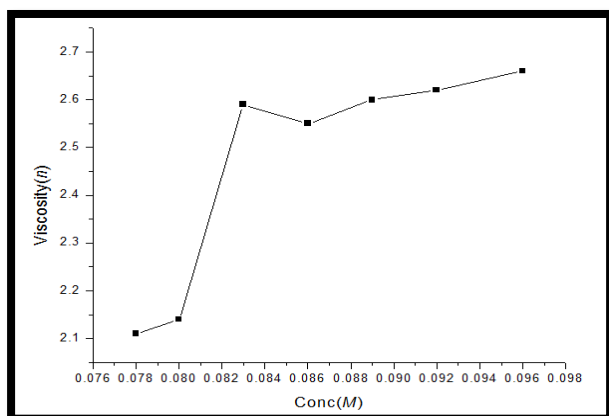
Graph 3. Plot of η_r *versus* [gelatin] for 8% PEG8000 solution at 28 ± 0.5^0C. (t_0=27 s)

From the above Graph, it can be seen that at Conc.8.0×10^{-2} mol dm^{-3}and 8.3×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is due to the structural complexation of solution because of the aggregation of Gelatin with PEG 8000.
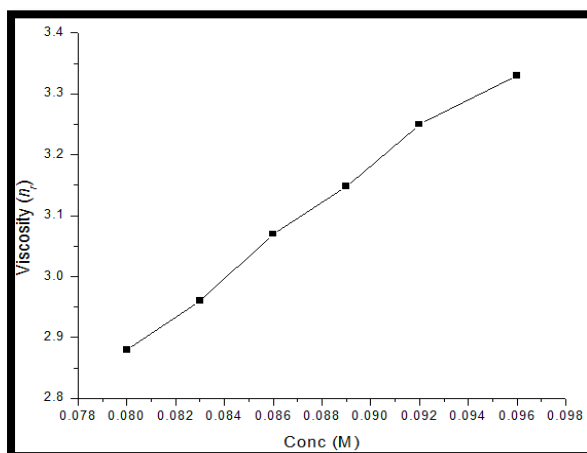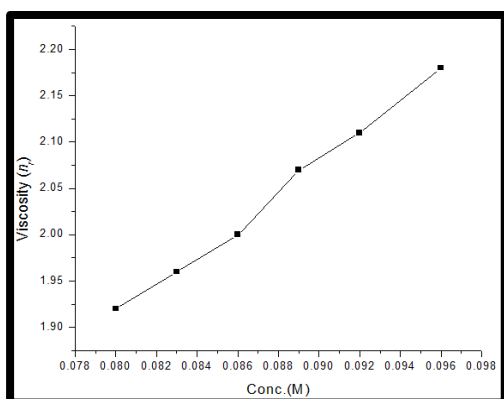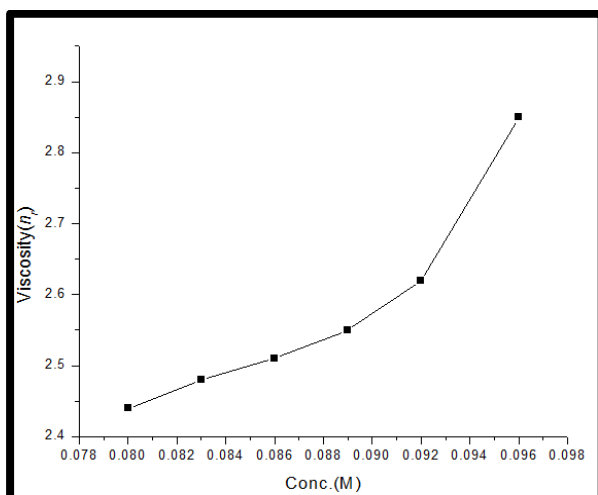

Graph 4. Plot of η_r *versus* [gelatin] for 11% PEG8000 solution at 28 ± 0.5^0C. (t_0=2 7s)

From the Graph above, it is shown that there is a linear change in viscosity of the solution because absence of additional aggregation of gelatin with PEG 8000.Due to which no structural complexation is form, so viscosity is linearly fashioned with conc.

Influence of the varying in the concentration of glucose at different [PEG8000] on the

relative viscosity of the solution.

It is observed from Graphs 5-7 that the viscosities of the solution containing glucose and fixed concentration of PEG8000 (6% by weight) increases with the increase in [glucose] from 7.81×10^{-2} to 9.61×10^{-2} mol dm^{-3}. The plot of η_r *versus*[glucose] is presented in Graph.
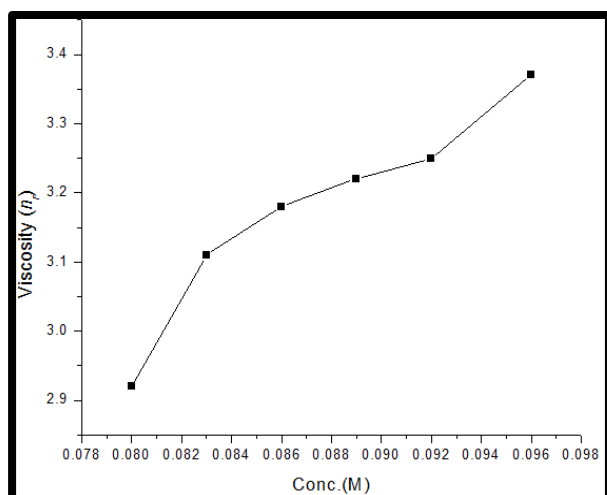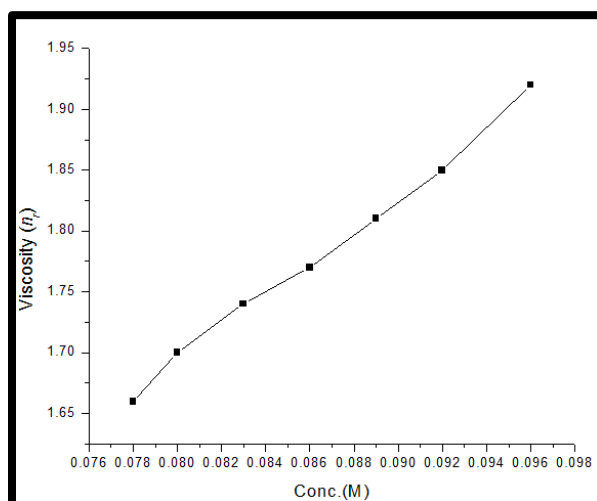
Graph 5. Plot of η_r *versus* [glucose] for 6% PEG8000 solution at 28 ± 0.5^0C.(t_0=27s)

From the above Graph, it is showed that at conc.8.6×10^{-2} mol dm^{-3}there is a slight variation in viscosity of the solution. This is due to the hydrogen bonding between the glucose and hydroxyl group of PEG 8000.

Graph 6. Plot of η_r *versus* [glucose] for 8% PEG8000 solution at 28 ± 0.5^0C.(t_0=27 s)

From the Graph above, it can be seen that at conc. 9.2×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is because of the formation of hydrogen bond between the glucose molecule and hydroxyl group of PEG 8000.

Graph 7. Plot of $\eta_r \, versus$ [glucose] for 11% PEG8000 solution at 28 ± 0.5^0C. $(t_0=27$ s).

From the above Graph, it is showed that at conc. 8.25×10^{-2} mol dm^{-3} and 9.2×10^{-2} mol dm^{-3} there is an abrupt change in the viscosity of solution. This is because of the formation of hydrogen bond between the glucose molecule and hydroxyl group of PEG 8000.

Influence of the varying in the concentration of starch at different [PEG8000] on the relative viscosity of the solution.

It is observed from Graphs 8-10 that the viscosities of the solution containing starch and fixed concentration of PEG8000 (6% by weight) increases with the increase in [starch] from 7.81×10^{-2} to 9.61×10^{-2} mol dm^{-3}. The plot of $\eta_r \, versus$ [starch] is presented in Graph.

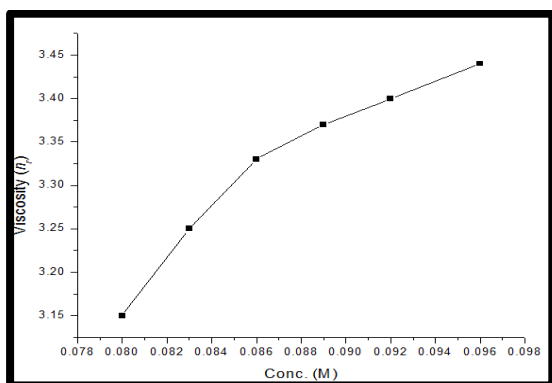**Graph 8.** Plot of $\eta_r \, versus$ [starch] for 6% PEG8000 solution at 28 ± 0.5^0C. $(t_0 =27$ s).

From the above graph, it is showed that there is a linear variation in viscosity of solution resulting from the interaction of starch with PEG 8000.
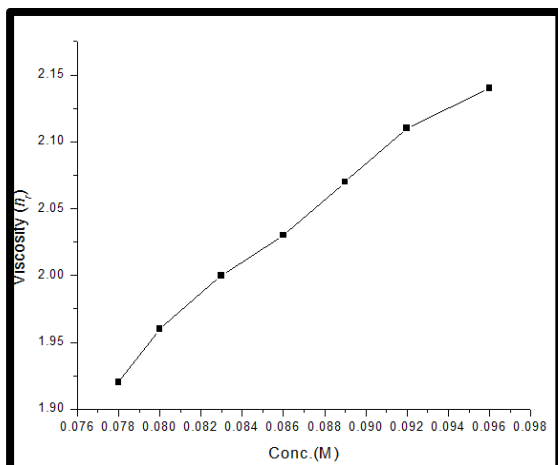
Graph 9. Plot of $\eta_r versus$[starch] for 8% PEG8000 solution at 28 ± 0.5^0C. (t_0 =27 s).

From the above Graph, it is seen that there is a slight variation in viscosity of solution. This is due to the formation of hydrogen bonding between starch and PEG 8000.
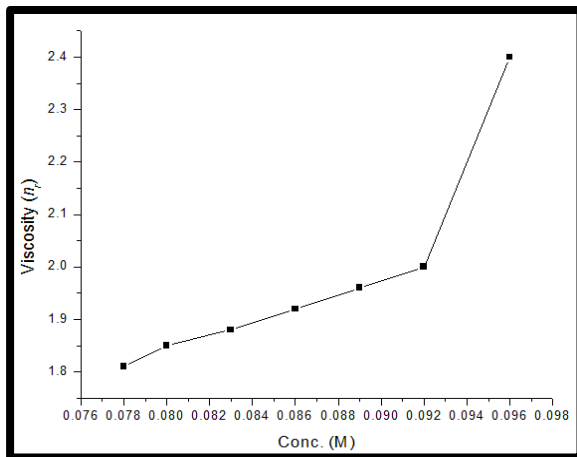

Graph 10. Plot of $\eta_r versus$[starch] for 11% PEG8000 solution at 28 ± 0.5^0C.(t_0=27 s).

From the above Graph, itis showed that at conc. 8.57×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is because of the formation of hydrogen bond between the starch   molecule and hydroxyl group of PEG 8000.

Influence of the varying in the concentration of CTAB at different [PEG8000] on
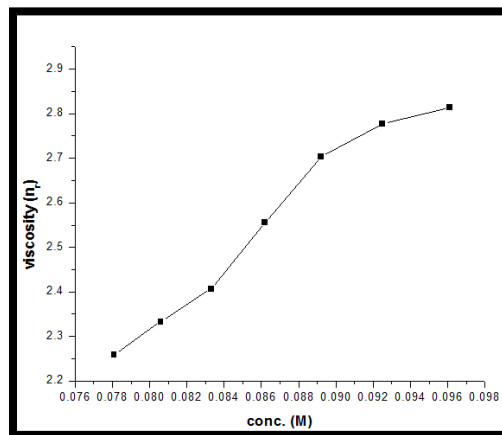
the relative viscosity of the solution.

It is observed from Graphs 11-13 that the viscosities of the solution containing [CTAB] and fixed concentration of PEG8000 (6% by weight) increases with the increase in [CTAB] from 7.81×10^{-2} to 9.61×10^{-2} mol dm^{-3}. The plot of $\eta_r versus$ [CTAB] is presented in Graph.
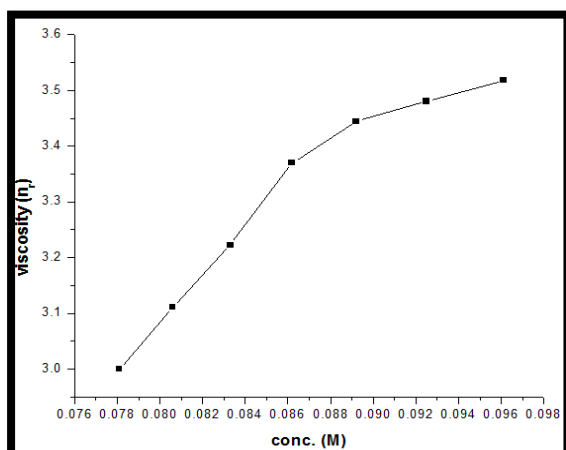
Graph 11. Plot of η_r *versus* [CTAB] for 8% PEG8000 solution at 28 ± 0.5^0C.(t_0=27 s).

From the above Graph, it is showed that at conc. 9.2×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is because of the electrolytic interaction that takes place due to which surfactant CTAB aggregates with PEG 8000 and due this structural complexation of mixture show sudden increment of viscosity.

Graph 12. Plot of η_r *versus* [CTAB] for 8% PEG8000 solution at 28 ± 0.5^0C.(t_0=27 s).

From the above Graph, it is shown that at conc. 8.3×10^{-2} mol dm^{-3}and8.9×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is because of the electrolytic interaction that takes place due to which surfactant CTAB aggregates with PEG 8000 anddue this structural complexation of mixture show sudden increment of viscosity.
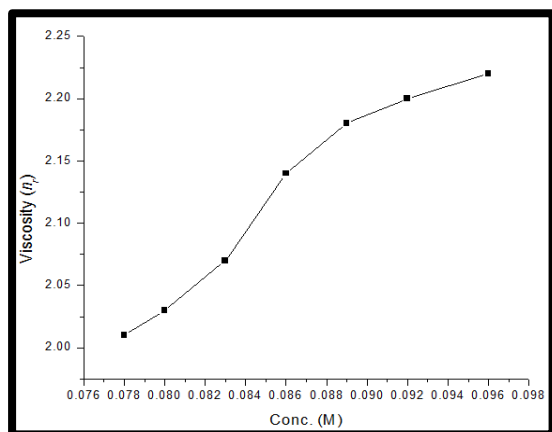
Graph 13. Plot of $\eta_r versus$[CTAB] for 11% PEG8000 solution at 28 ± 0.5^0C.(t_0=27s).
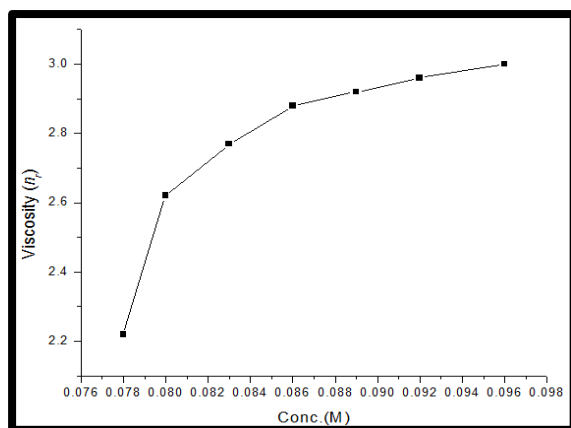
From the above Graph, it is shown that there is a slight variation in viscosity resulted due to the electrolytic interaction between the CTAB and PEG 8000 molecules.

Influence of the varying in the concentration of SDS at different [PEG8000] on the relative viscosity of the solution.
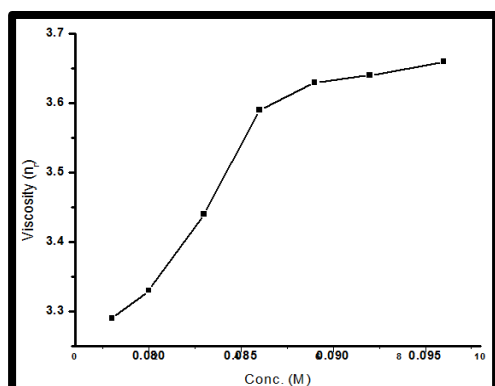
It is observed from Graphs 14-16 that the viscosities of the solution containing SDS and fixed concentration of PEG8000 (6% by weight) increases with the increase in [SDS] from 7.81×10^{-2} to 9.61×10^{-2} mol dm^{-3}. The plot of $\eta_r versus$ [SDS] is presented in Graph.

Graph 14. Plot of $\eta_r versus$ [SDS] for 6% PEG8000 solution at 28 ± 0.5^0C. (t_0 = 27 s).

From the above Graph, it is shown that there is a slight variation in viscosity resulted due to the electrolytic interaction between the SDS and PEG 8000 molecules.

Graph 15. Plot of $\eta_r versus$ [SDS] for 6% PEG8000 solution at 28 ± 0.5^0C. (t_0 = 27 s).

From the above Graph, it is shown that at conc. 7.85×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is because of the electrolytic interaction that takes place due to which surfactant SDS aggregates with PEG 8000 and due to which structural complexation of mixture, show sudden increment of viscosity.
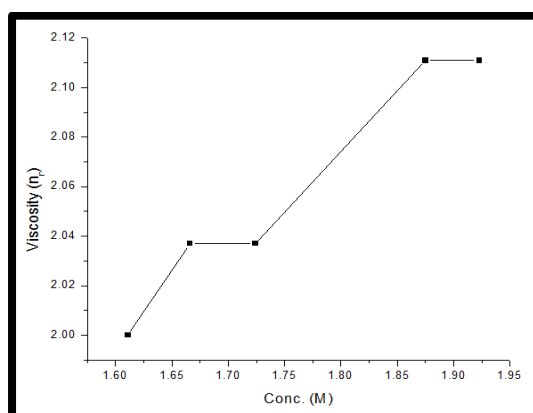
Graph 16. Plot of η_r *versus* [SDS] for 6% PEG8000 solution at 28±0.5⁰C. (t₀ = 27 s).

From the above graph, it is shown that at conc. 8.7×10^{-2} mol dm^{-3}there is an abrupt change in the viscosity of solution. This is because of the electrolytic interaction that takes place due to which surfactant SDS aggregates with PEG 8000 and show very rich phase behavior.

Influence of the varying in the concentration of NaCl at different [PEG8000] on

therelative viscosity of the solution.

It is observed from Graphs 17-19 that the viscosities of the solution containing NaCl and fixed concentration of PEG8000 (6% by weight) increases with the increase in [NaCl] from 1.562 to 1.923 M. The plot of η_r *versus* [NaCl] is presented in Graph.
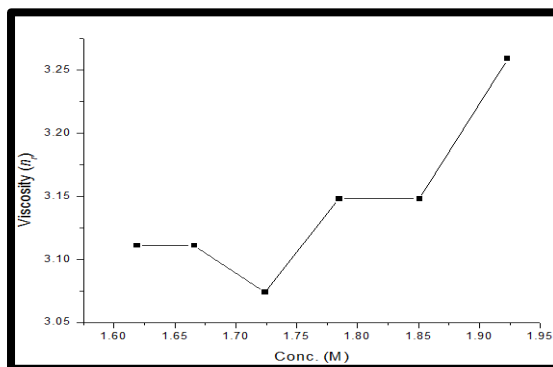

Graph 17. Plot of η_r *versus* [NaCl] for 6% PEG8000 solution at 28 ± 0.5^0C.(t₀=27 s).

From the above Graph, it is shown that at conc. 1.65 M, 1.725M and 1.86 M,there is an abrupt change in the viscosity of solution. This is because of the electrostatic interaction that takes place due to which salt NaCl shows distributive character with PEG 8000 and show decrement of viscosity.
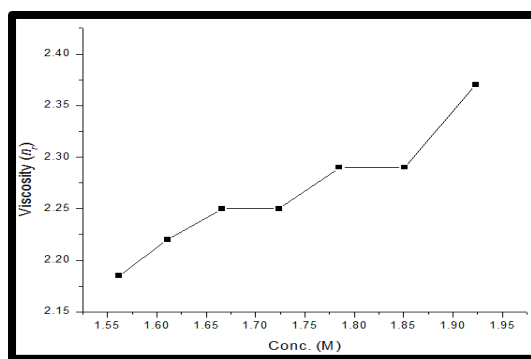
Graph 18. Plot of η_r *versus* [NaCl] for 6% PEG8000 solution at 28 $\pm0.5^0$C.(t_0=27 s).

From the above Graph, it is shown that at conc.1.65 M and 1.755 M, there is an abrupt change in the viscosity of solution. This is because of the electrostatic interaction that takes place due to which salt NaCl shows distributive character with PEG 8000 and show decrement in viscosity.

Graph 19. Plot of η_r *versus* [NaCl] for 6% PEG8000 solution at 28 $\pm0.5^0$C.(t_0 =27 s).

From the above Graph, it is shown that at conc. 1.65 M, 1.725M and 1.86 M,there is an abrupt change in the viscosity of solution. This is because of the electrostatic interaction that takes place due to which salt NaCl shows distributive character with PEG 8000 and show decrement of viscosity.

Discussion

The viscosity of various PEG aqueous solutions with different salt concentrations are shown in as plotted in the Graphs 2-19. It is observed that the viscosities of PEG8000 at different concentrations increased with an increase in additives. A close look on the behavior of change in the viscosity of PEG8000 with increase in [additives] demonstrates in an abrupt change. The break in slope of the curve shows the structural transformation of the PEG8000 aggregates in aqueous solution. Usually, in dilute aqueous solutionin the absence of any additives PEG8000 exist in the highly hydrated state whereas in the presence of additives, the PEG aggregates to form spherical or cylindrical structure with different degrees of hydration and therefore a break in viscosity curve is observed. The degree of hydration and structural change in PEG aggregates depend upon the concentration of PEG, nature and concentration of additives used.

Polyethylene Glycol 8000 when treated with Gelatin,Glucose and Starch (a polysaccharide), shows two types of interaction i.e., Hydrogen bonding and PEGylation. The former takes place between the electronegative oxygen and hydrogen of PEG and Gelatin depending on their nature. Due to hydrogen bonding, complexation of the polymer with starch and gelatin takes place which increases the density of the molecule and thereby the viscosity also increases. The latter i.e., PEGylation is the conjugation of PEG to the carbohydrate moiety of a protein which has been mainly used to improve the bioavailability of proteins and low molecular weight drugs. PEGylation results in the structural complexation which increases the density andthereby increases the viscosity of the polymer.

Polyethylene Glycol 8000 when treated with surfactants such as CTAB and SDS, results into the complexation of the polymer with the surfactants. This occurred due to two types of interactions that take place between PEG

8000 and the surfactants i.e., Electrostatic interaction and Hydrophobic interaction, respectively. The former takes place between ionic surfactants such as CTAB and SDS which results into the complexation of the polymer and hence the viscosity increases. The latter i.e., Hydrophobic interaction takes place between the hydrophobic moiety of the polymer and the surfactants resulting into the structural change, which therefore increases the density and thereby increases the viscosity of the polymer.

Conclusions

As we have performed the entire experiment at a constant temperature of $28 \pm 0.5\ ^0C$, we measured the variations in viscosity by using Gelatin, Glucose, Starch, CTAB, SDS and NaCl salts. Among these electrolytes and Non electrolytes, both CTAB and SDS are surfactants. From the experiment performed, it is concluded that Polyethylene Glycol 8000 when reacted with these above chemical , showed aggregation in its behavior and the viscosity of Polymer varied with different conc. of different Electrolytes and Non - electrolyte. This happened because of interactions such as hydrogen bonding, hydrophobic interaction, electrostatic interaction and PEGylation (of PEG with carbohydrates) between the Polyethylene Glycol 8000 and different Electrolytes and Non– electrolytes salts.

Apart from measuring viscosity, this experiment can also lead to interesting and more fascinating results. Also, after seeing the current work progress in this particular research area we can also study the variations in density as well by using the pycnometer. We can understand the variations at different concentrations and temperatures of this long chain polyether. Further, we too can find the viscosity of the various salts used in the experiment using the Ostwald's Viscometer. In this way, this area of research is of utmost importance in various fields of Chemistry, Biology, Bio-chemistry, Bio-technology and Polymer Chemistry as the main.

ACKNOWLEDGEMENT

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LIST OF ABBREVIATIONS

Abbreviation	Full form
PEG	Polyethylene Glycol
SDS	Sodium Dodecyl Sulfate
CTAB	Cetyltrimethyl ammonium bromide
NaCl	Sodium Chloride
KCl	Potassium Chloride
$NaNO_3$	Sodium Nitrate
CDH	Central Drug House
ATPS	Aqueous Two Phase System
VOL	Volume
CONC	Concentration
DIST	Distilled