

Polymer Characterization: Determination of Viscosity of Different Natural Polymer/Synthetic Polymers

1. Importance of the experiment: Molecular weight of a polymer can be determined by different techniques such as GPC, MALDI-TOF MS, etc. Determination of polymer molecular weight through intrinsic viscosity using Ostwald viscometer is an absolute technique and is also a cost-effective method. Viscosity is the measure of resistance of a fluid to flow. Water has a viscosity of 1 cps (centipoise) at room temperature (25 °C) and is considered as a standard.

2. Concept: Molecular weight of polymer (M) can be derived from intrinsic viscosity data of a polymer solution. But one pre-requirement to determine the molecular weight of a polymer is the knowledge of Mark-Houwink (M-H) coefficients. Upon substitution of intrinsic viscosity and these coefficients in M-H equation, one can find out molecular weight of the polymer. M-H equation $[\eta] = KM_v^\alpha$, where K and α are M-H coefficients and $[\eta]$ = intrinsic viscosity of the polymer solution. In this experiment, we focus on the determination of intrinsic viscosity of the given polymer solution.

3. Applications: Nowadays, polymers have become essential requirements in our day to day activities. The end applications of the polymer depend upon its characteristics such as molecular weight, polydispersity index, thermal stability, crystalline/amorphous nature, stereochemistry, etc. Among these, molecular weight of the polymer is very important. LDPE (low density polyethylene) is used as packing materials and carry bags, whereas UHMWPE (ultra-high molecular weight polyethylene) is used as containers, tubing and other heavy-duty equipments due to its high abrasion resistance, high impact strength and low coefficient of friction. Most of the bottles for carbonated drinks, mineral water, edible oil and personal care products are made of poly(ethylene terephthalate), PET and their intrinsic viscosity values range from 0.7-0.85 dL/g, depending on the length of the polymer chain (the longer the polymer chains, the more entanglements between the chains and therefore the higher the viscosity).

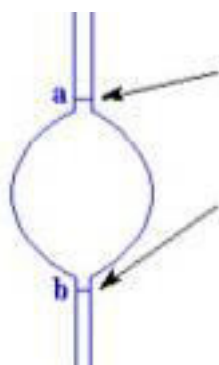


Fig. 1: A portion of Ostwald viscometer showing the upper and lower graduation marks, 'a' and 'b' respectively.

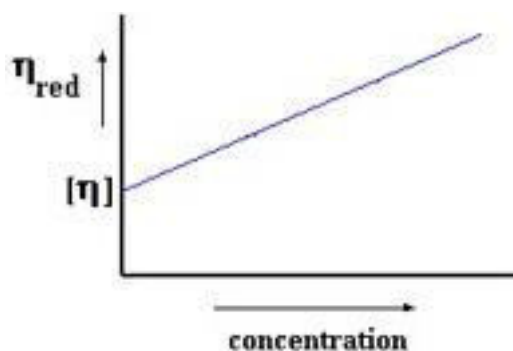


Fig. 2: A plot of reduced viscosity vs. concentration of a polymer solution, y-intercept corresponding to intrinsic viscosity $[\eta]$.

Experiment	Polymer Characterization: Determination of Viscosity of Different Natural Polymer/Synthetic Polymers
Problem definition	Viscosity is the measure of resistance of a fluid to flow. Longer the polymer chains, the more entanglements between the chains and therefore the higher the viscosity. Molecular weight of polymer (M) can be derived from intrinsic viscosity data of a polymer solution.
Methodology	Determination of intrinsic viscosity using Ostwald viscometer. In a particular solvent, concentration of the polymer is directly proportional to viscosity of the solution.
Solution	Determination of intrinsic viscosity and molecular weight of the given polymer sample.
Student learning outcomes	Students will learn to determine intrinsic viscosity and molecular weight of the given polymer solution.

Principle:

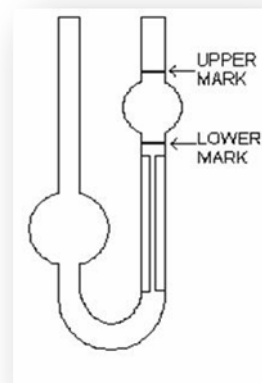
When a polymer is mixed with a solvent, the solvent enters into the polymer matrix and swelling of polymer coils takes place. This expanded polymer coil disintegrates and moves out of polymer matrix and dissolves in the solvent. The apparent volume occupied by the expanded coil is referred to as the 'hydrodynamic volume' of the polymer molecule in the solution under flow. Viscosity of a polymer solution is a direct measure of hydrodynamic volume of the polymer, which in turn, is a measure of its molecular weight. Viscosity of a polymer is more in a good solvent than in a poor solvent.

Reagents: PEG (polyethylene glycol) solution of different concentrations, Distilled water.

Apparatus: Ostwald viscometer, stop-clock, 50 mL standard flasks

Procedure:

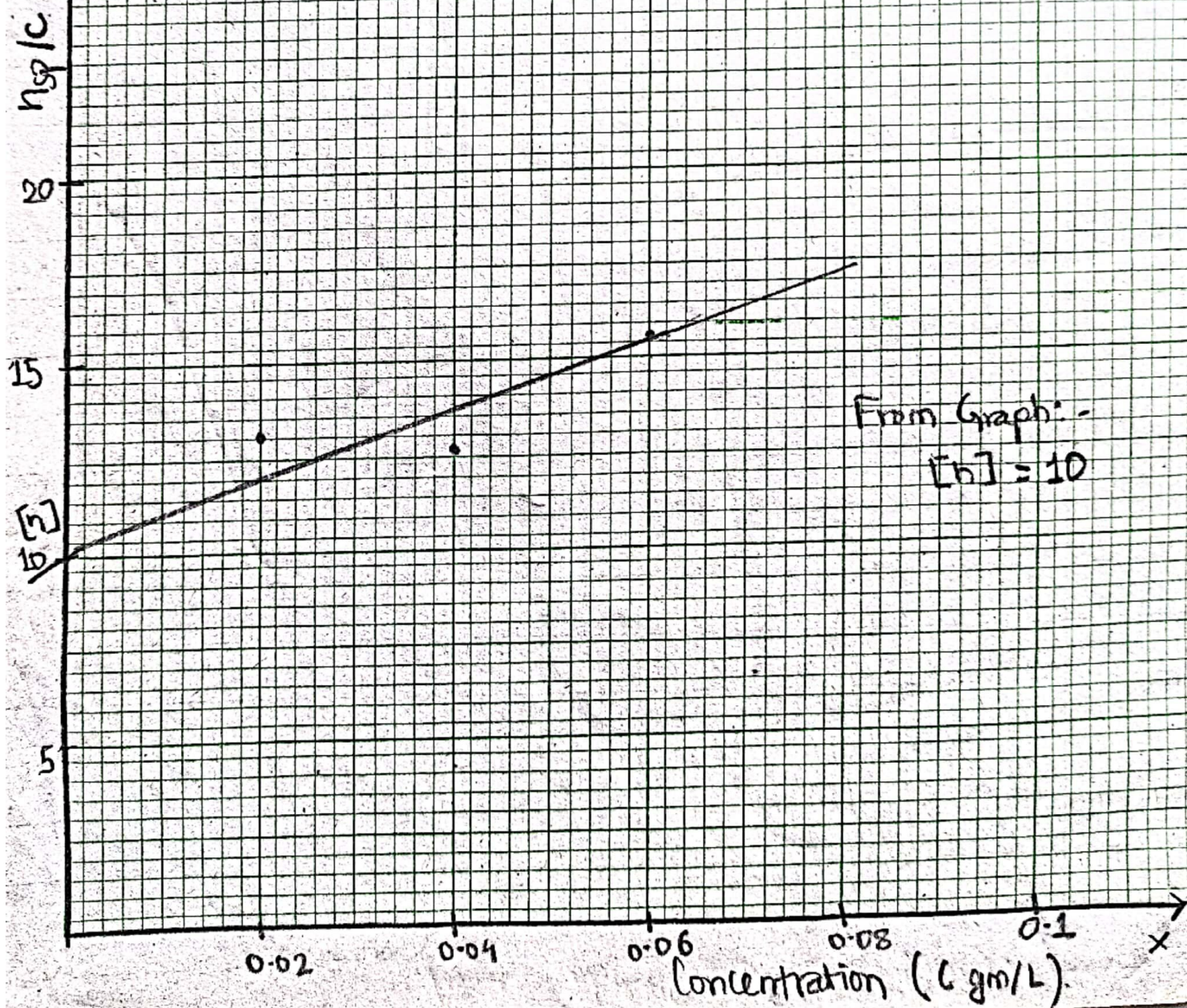
Prepare at least 3 different diluted concentrations of PEG (1 to 5%) in water using 10% PEG stock solution (10 g/100 mL). Initially, rinse the Ostwald viscometer with a little amount of water. Fill it with 20 mL pure water and use a rubber filler to suck the water above the upper mark. By keeping the upper mark of the small reservoir of viscometer parallel to eyes, allow the solvent to flow down to the lower mark and note down time in seconds. This is known as the E_{flux} time. Repeat the same experiment for 2 times to get the average E_{flux} time for water (t_0). Apply the same procedure to determine the flow rate for remaining 2 diluted solutions and note down their flow time in seconds. Calculate relative viscosity, specific viscosity and reduced viscosity as shown in **Table 1**. Plot the graph between polymer concentrations (C g/mL) vs η_{red} . The value of intercept at $C = 0$ will give intrinsic viscosity of the polymer solution (see **Fig. 1**).



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Graph:- Concentration (g/L) Vs. n_{sp}/C

Scale:- Along X-axis, 10 divisions = 0.02 g/L.
Along Y-axis, 10 divisions = 5 L/gm



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Table 1: Viscosity measurement data

S. No.	Concentration, C (g/mL)	E _{flux} time, t (sec)			$\eta_r = t_s/t_0$	$\eta_{sp} = \eta_r - 1$	$\eta_{red} = \eta_{sp}/c$
		t_1	t_2	$t_s = t_1 + t_2 / 2$			
1	0.02	95	94	94.5	1.26	0.26	13
2	0.04	114	112	113	1.51	0.51	12.75
3	0.06	146	145	145.5	1.94	0.94	15.67

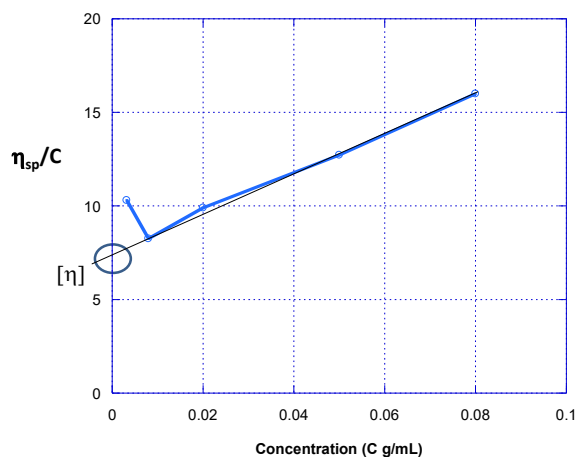


Fig. 1. Concentration (C g/mL) Vs η_{sp}/C

Calculations:

$$[\eta] = KM_v^a$$

∴ Molecular weight of the given polymer (M_v) =

$$M_v = \text{Anti ln} \left(\frac{\ln [\eta] - \ln K}{a} \right)$$

Constants for PEG in water $K = 0.0428$ and $a = 0.64$

Result:

(a) E_{flux} time for pure water (t_0) = 75 sec.

(b) Intrinsic viscosity of the polymer (η) = 10

(c) Molecular weight of the given polymer (M_v) = 5077.41