INTERNATIONAL JOURNAL OF CURRENT RESEARCH IN CHEMISTRY AND PHARMACEUTICAL SCIENCES

(p-ISSN: 2348-5213: e-ISSN: 2348-5221)
www.ijcrcps.com

Research Article

VISCOMETIC STUDY ON UBBELOHDE VISCOMETER

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Abstract

Ubbelohde viscometer was used for measurement of viscosity of the polymer solution of a given polymer sample in Di methyl formamide and in Dioxane. The viscometer was washed with chronic acid, then rinsed several times with distilled water and finally with acetone. The viscometer was dried in an oven at 100^0C for 24 hours. It was standardized using solutions of known concentrations of polymer samples of known molecular weight.The clean and dried viscometer was placed in a temperature bath maintained at $35^0 \pm 0.1^0$C. A solvent (5 ml) was carefully introduced into the viscometer. The viscometer containing solvent was allowed to attain the bath temperature. The efflux time was noted following the normal technique. Three independent readings were noted and their average was noted.

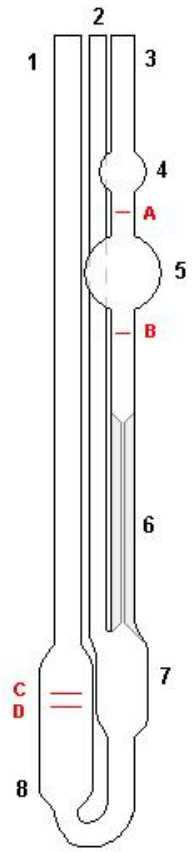
Keywords: Ubbelohde Viscometer, Pippete, Conical Flask, GlassRoad ,Water Bath, Thermometer.

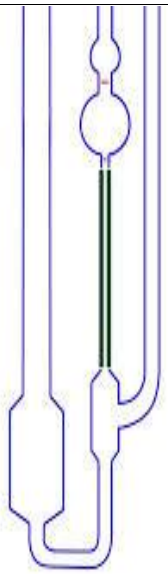
Introduction

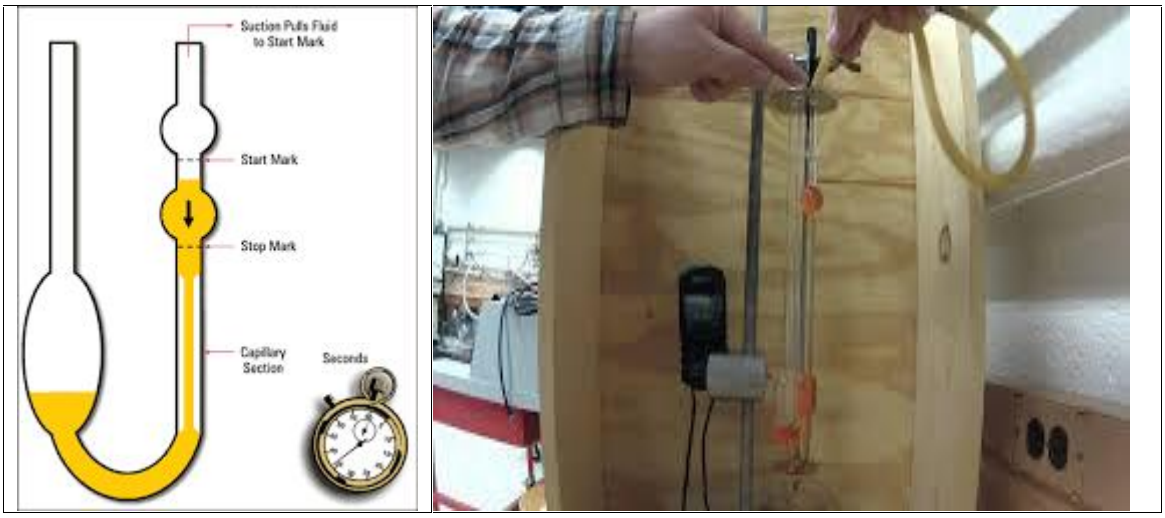
It is recommended for higher viscosity cellulosic polymer solutions. The advantage of this instrument is that the values obtaineAUbbelohde type viscometer or suspended-level viscometer is a measuring instrument which uses a capillary based method of measuring viscosity [1].d are independent of the total volume. The device was invented by the German chemist Leo Ubbelohde (1877-1964).

The Ubbelohde viscometer is closely related to the Ostwald viscometer. Both are u-shaped pieces of glassware with a reservoir on one side and a measuring bulb with a capillary on the other. A liquid is introduced into the reservoir then sucked through the capillary and measuring bulb. The liquid is allowed to travel back through the measuring bulb and the time it takes for the liquid to pass through two calibrated marks is a measure for viscosity. The Ubbelohde device has a third arm extending from the end of the capillary and open to the atmosphere. In this way the pressure head only depends on a fixed height and no longer on the total volume of liquid. Some of various physical photo of ubbelohde viscometer shown below,

Viscosity is a unique property that a dilute polymer solution has much higher viscosity than that of the pure solvent. This large difference in viscosity and various function derived there from are usually measured to procedure information about the nature of the micro structure of the polymer under study. It will be proper to define viscosity functions which are being estimated for this purpose.







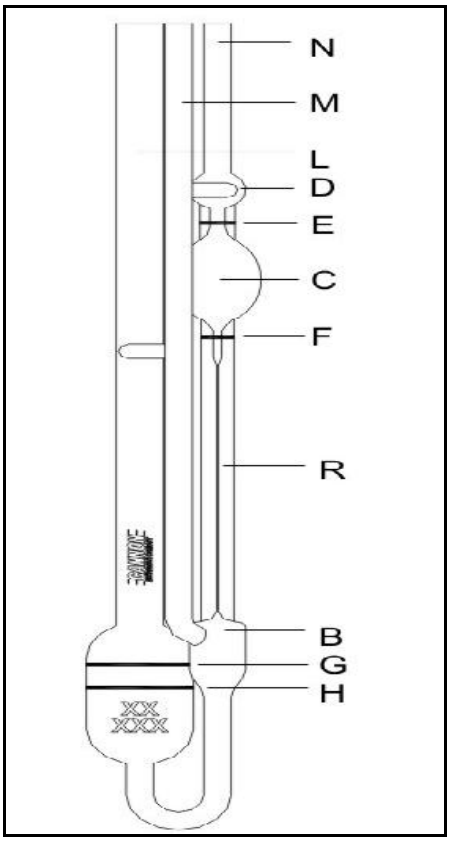
The ratio of viscosity of solution (η) to viscosity of the solvent (η_o) is known as relative viscosity.

$$\eta_r = \eta / \eta_o$$

Instruction for the use and cleaning process of Ubbelohde viscometer.

1. Was cleaned by the viscometer using purified solvents, and by passing clean, dry, filtered air through the instrument to remove the final traces of solvents. Periodically, traces of organic deposits should be removed with chromic acid or non-chromium cleaning solution [16].

2. To remove any lint, dust, or other solid material in the sample was filtered of through a fritted glass filter or fine mesh screen.



3.The viscometer was charge by introducing sample through tube L into the lower Reservoir; introduce enough sample to bring the level between lines G and H.

4.The viscometer was placed into the holder, and insert it into the constant temperature bath. Vertically align the viscometer in the bath if a self-aligning holder has not been used.

5.Allow approximately 20 minutes was allowed for the sample to come to the bath temperature.

6.Finger was placed over tube M and applied suction to tube N until the liquid reaches the center of bulb D. Removed suction from tube N. Removed finger from tube M, and immediately place it over tube N until the sample drops away from the lower end of the capillary into bulb B. Then finger was removed and measure the efflux time.

7.To measure the efflux time, sample was allowed to flow freely down past mark E, measuring the time for the meniscus to pass from mark E to mark F.

8.The sample was calculating by multiplying the efflux time by the viscometer constant.

9.Without recharging the viscometer, make check determinations by repeating

3.3 Method for Solution preparation of Ubbelohde viscometer.

1.The viscometer was checked for clean [17].

2.The viscometer was assembled in the tempering jacket based on the instruction in the manual.

3.A limited quantity (15-20 ml) of the liquid being examined was filled through the filling tube (3) into the storage bulb (4) in such a way that the level is between Min and Max filling marks (10).

4.The temperature was adjust by setting and wait at least 10 minutes after the thermometer in the bath is within 0,2K of the desired temperature in order to reach temperature stability between bath and the liquid being examined.

5.Measurement (figure 3.2):

The venting tube (1) was closed by your finger and apply suction by pipette filler via suction tube (2) until the liquid rises to the half of the pre-run sphere (9) Removed the finger and the pipette filler and measure the time the liquid requires to sink between measurement marks M1 and M2.

Repeat the measurement at least five times.

6.The temperature was adjusted of the bath to the next level according to the test matrix and repeat from point 4.

7.Particles have the potential to stick to the wall; the viscometer was cleaned according to the following method for accurate measurement.

8.A solution consisting of 15 % hydrochloric acid and 15 % hydrogen peroxide was prepared.

9.Here is the cleaning instruction without using a vacuum pump:

The cleaning liquid was filled into the filling tube
The liquid was sucked several times into the measurements sphere
The remaining viscometer parts cleaned by shaking the viscometer
The viscometer was made empty
The cleaning process was repeated two to three times
Acetone was use to rinsing and dried in oven

3.4 Measurement of viscosity by Ubbelohde viscometer or Experimental.

The experimental details for the measurements of viscosity of polymer solutions are presented in this section. The same details have also been followed in the viscometric study in other media.

Ubbelohde viscometer was used for measurement of viscosity of the polymer solution of a given polymer sample in Di methyl formamide and in Dioxane. The viscometer was washed with chronic acid, then rinsed several times with distilled water and finally with acetone. The viscometer was dried in an oven at 100^0C for 24 hours. It was standardized using solutions of known concentrations of polymer samples of known molecular weight.

The clean and dried viscometer was placed in a temperature bath maintained at $35^0 \pm 0.1^0$C. A solvent (5 ml) was carefully introduced into the viscometer. The viscometer containing solvent was allowed to attain the bath temperature. The efflux time was noted following the normal technique. Three independent readings were noted and their average was noted.

In the same clean viscometer 5 ml of the solution of the given polymer was noted at 35^0C. Two ml of the

solvent was introduced into viscometer. A slow stream of air was passed through the solution to ensure uniform mixing. The efflux time of the diluted solution was measured after 10 minutes, during which time the solution in the viscometer attained the bath temperature.

Subsequent dilutions were made by successive additions of 2 ml of solvent. The efflux time of each of these solutions was measured. Measurements were carried out at 4-5 different dilutions. The quantities measured in the viscometric study are;

i. to – the efflux time in seconds for the solvent (average of three readings).
ii. t – the efflux time in seconds of the solution of concentration C, g dl^{-1} at the same temperature (average of three readings)
iii. Such measurements were carried out using 4 to 5 solutions of decreasing concentrations.
iv. The viscosity functions n_{sp} were evaluated for solutions of different concentrations

$$\eta_{sp} = \eta-\eta_o / \eta_o = t - t_o / t_o$$

v.The values of reduced viscosities (η_{sp}/C) were evaluated and plots of reduced
viscosity against concentration were made.

The results of such a study of solutions of polymer samples and fractions are illustrated in table 3.1 and 3.2.

Table 3.1 Viscosity functions of Salicylic acid-formaldehyde polymer and its 1st fraction

Polymer sample: *AI – conglomerate and 1st fraction*
Solvent: *Di methyl formamide (DMF)*
Efflux time of pure DMF (to): *202.5 seconds*
Temperature: *35 ± 0.1^{0}C*

Polymer sample	Concentration (C) g. dl^{-1}	Efflux time: (t) of solutions in seconds	($t-t_o$) Seconds	Specific viscosity $n_{sp} = t-t_o/t_o$	Reduced viscosity $\eta_{red} = \eta_{sp}/C$ dlg^{-1}
AI-Conglome-rate	3.00	235	30.2	0.1491	0.0497
	2.145	227.8	21.9	0.1081	0.0504
	1.670	223.3	16.8	0.0830	0.0497
	1.360	219.9	14.2	0.0701	0.0515
	1.150	219.6	14.1	0.0696	0.0605
AI-1st fraction	3.00	238.5	33.9	0.1674	0.0558
	2.145	230.5	25.5	0.1259	0.0587
	1.670	226.3	20.5	0.1012	0.0606
	1.360	223.0	16.6	0.0819	0.0602
	1.150	222.8	15.9	0.0785	0.0682

Table 3.2 Reduced viscosity polymer sample: AI and BI conglomerates
Medium: *DMF-water or Dioxane-water mixture*
Temperature: *35 ± 0.1^{0}C*

Letter / Number	Significance
A	Salicylic acid – formaldehyde polymer
B	4-hydroxy benzoic acid – formaldehyde polymer
C	Resorcilic acid – formaldehyde solution
I	Aqueous 40% H_2SO_4
II	(1:1 conc. HCl - Water) – oxolic acid mixture
III	6.0% H_2SO_4 in glacial acetic acid

Table 2.1 Hyroxybenzoic acid- formaldehyde polymers

Polymer Sample	Medium	Reduced viscosity (η_{red} dlg^{-1})at concentration, C g-dl^{-1}				
		3.00	2.145	1.670	1.360	1.150
AI – conglomerate	85-15(V/V) DMF-H_2O Mixture	0.0496	0.0490	0.0496	0.0503	0.0506
BI – conglomerate	-do-	0.0342	0.0347	0.0349	0.0369	0.0376
AI – conglomerate	75-25(V/V) Dioxane-H_2O Mixture	0.0473	0.0468	0.0474	0.0490	0.0498
BI – conglomerate	-do-	0.0365	0.0342	0.0339	0.0352	0.0353

Thus a designation of AI suggests that it is a salicylic acid – formaldehyde polymer prepared in presence of aqueous 40% H_2SO_4.

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