B.Sc. Chemistry

Semester-V

17CHU512A POLYMER CHEMISTRY – PRACTICAL 4H 2C

Instruction Hours/week:L:0 T:0 P:4Marks: Internal:40 External: 60 Total:100

End Semester Exam: 3 hrs.

Course objectives

This course enables the student to

1. Have hands on experience to prepare different types of polymers by various methods
2. To do the purification of polymers
3. To characterise the polymers by chemical and instrumental methods.

Course outcome

The students understood

1. To prepare different types of polymers by various methods
2. To purify polymers
3. To characterise the polymers by chemical and instrumental methods.

Polymer synthesis

1. Free radical solution polymerization of styrene (St) / Methyl Methacrylate (MMA) / Methyl Acrylate (MA) / Acrylic acid (AA).
   a. Purification of monomer
   b. Polymerization using benzoyl peroxide (BPO) / 2,2'-azo-bis-isobutylonitrile (AIBN)
2. Preparation of nylon 66/6
   1. Interfacial polymerization, preparation of polyester from isophthaloyl chloride (IPC) and phenolphthalein
   a. Preparation of IPC
   b. Purification of IPC
   c. Interfacial polymerization
3. Redox polymerization of acrylamide
4. Precipitation polymerization of acrylonitrile
5. Preparation of urea-formaldehyde resin
6. Preparations of novalac resin/resold resin.
7. Microscale Emulsion Polymerization of Poly(methylacrylate).

Polymer characterization

1. Determination of molecular weight by viscometry:
   (a) Polyacrylamide-aq.$NaNO_2$ solution
   (b) (Poly vinyl proplylidine (PVP) in water
2. Determination of the viscosity-average molecular weight of poly(vinyl alcohol) (PVOH)andthe fraction of ―head-to-head‖ monomer linkages in the polymer.
3. Determination of molecular weight by end group analysis: Polyethylene glycol (PEG) (OHgroup).
4. Testing of mechanical properties of polymers.
5. Determination of hydroxyl number of a polymer using colorimetric method.

Polymer analysis

1. Estimation of the amount of HCHO in the given solution by sodium sulphite method
2. Instrumental Techniques
3. IR studies of polymers
4. DSC analysis of polymers
5. Preparation of polyacrylamide and its electrophoresis
   *at least 7 experiments to be carried out.

SuggestedReadings

1. Malcohm P. Stevens(1999). *Polymer Chemistry: An Introduction*.3rd Ed. Oxford University Press.
2. Harry R. Allcock, Frederick W. Lampe and James E. Mark, (2003*). Contemporary PolymerChemistry*.3rd ed. Prentice-Hall
3. Fred W. Billmeyer, (1984).*Textbook of Polymer Science*.3rd ed. Wiley-Interscience
4. Joel R. Fried, (2003).*Polymer Science and Technology*.2nd ed. Prentice-Hall.
5. PetrMunk&Tejraj M. Aminabhavi, (2002).*Introduction to Macromolecular Science*. 2nd ed. John Wiley & Sons
6. L. H. Sperling,.(2005). *Introduction to Physical Polymer Science*.4th ed. John Wiley & Sons.
7. Malcolm P. Stevens, (2005).*Polymer Chemistry: An Introduction*. 3rd ed. Oxford UniversityPress.
8. Charles E. Carraher,.(2013). *Seymour/ Carraher's Polymer Chemistry*.9th ed. Jr.

KARPAGAM ACADEMY OF HIGHER EDUCATION

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Coimbatore – 641 021.

LECTURE PLAN
DEPARTMENT OF CHEMISTRY
III- B.Sc CHEMISTRY

Name of the Staff	:	**Dr. K.Sathya**
Department	:	**Chemistry**
Title of the Paper	:	**Polymer Chemistry Practical**
Paper Code	:	**17CHU512A**
Class	:	**III-B.Sc-Chemistry**
Year and Semester	:	**III Year and V-Semester**
Total Hours	:	**48 Hours**

S. No.	Duration Hours	Name of the Experiment	*Support Material*
1.	4	Procedure Writing	
2.	4	Preparation of phenol-Formaldehyde	
3.	4	Preparation of Urea-Formaldehyde	R1, R2
4.	4	Preparation of poly(methylmethacylate)	R1, R2
5.	4	Preparation of sodium carboxy methyl cellulose	R1, R2
6.	4	Determination of molecular weight by viscometry	R1, R2
7.	4	Determination of Viscosity of Polymer	R1, R2
8.	4	Estimation of Amount of HCHO	R1, R2
9.	4	IR studies of polymers	R1, R2
10.	4	Viva-voice	
11.	4	Viva-voice questions	
12.	4	Model practical examination	

SUGGESTED READINGS:

REFERENCE BOOKS

R1: Aparna G.Sajina, Practical Manual Engineering Chemistry.
R2 : Fred.W.Billmayer, 1984, Text book of Polymer Science, 3rt Edition.

LAB MANUAL
DEPARTMENT OF CHEMISTRY
III- B.Sc CHEMISTRY

CONTENTS

Suggested Readings:

Text Books:

1. Khosla, B. D., Garg, V. C. & Gulati, A.(2011).*Senior Practical Physical Chemistry*. New Delhi :
R. Chand &Co.

Experiment No: 1

PREPARATION OF UREA FORMALDEHYDE RESIN

Aim

To prepare urea formaldehyde and phenol formaldehyde resins.

Apparatus required

Beaker, glass rod, funnel, filter paper and chemical balance.

Chemicals

Urea, formaldehyde sol., conc. H_2SO_4, distilled water.

Theory

Amino resins are condensation products obtained by the reaction of formaldehyde with nitrogen bearing compounds such as aniline, amides for ex:- melamine formaldehyde, urea formaldehyde etc.

Urea formaldehyde is prepared by condensation reaction between urea and formaldehyde in acidic or alkaline medium.

The first product formed during the formation of resin is monomethylol and dimethylol ureas.

Procedure:-

1. Place about 5 ml of 40% formaldehyde solution in 100 mlbeaker.
2. Add about 2.5 g of urea with constant stirring till saturated solution isobtained.
3. Add a few drops of conc. H_2SO_4, with constantstirring.
4. A voluminous white solid mass appears in thebeaker.
5. Wash the white solid with water and dry it in the folds of filterpaper.
6. Weight the yield ofproduct

Precautions:-

1. While adding concentrated H_2SO_4, it is better to stay little away from the

beaker since the reaction sometimes becomesvigorous.

2. The reaction mixture should be stirredcontinuously.

Observations:-

Mass of the beaker (W1) =----------g.

Mass of the beaker with urea formaldehyde (W2) = --------g.

Therefore mass of urea formaldehyde (W2 – W1) = --------g.

Result:-

The yield of urea formaldehyde = -------g

Experiment No: 2

SYNTHESIS AND CHARACTERIZATION OF SODIUM CARBOXY METHYL CELLULOSE

Aim

Synthesis and Characterization of Sodium Carboxy methyl cellulose.

Reagents Required

Name	Amount
Cellulose powder	5 g
NaOH solution (5 wt %) in H_2O	20 mL
i-propanol	100 ml
Sodium monochloro acetate	7 g
1M HCl	10ml
Ethanol (70 wt%)	20ml

Apparatus required

- Conical flask
- Magnetic stirrer
- Beaker
- Glass rod
- Buchner funnel
- vacuum oven

Procedure

To a 250 mL flask equipped with a magnetic stir bar cellulose powder (5 g), NaOHaq.(20 mL, 5 wt%) and isopropanol (100 mL) is added. The cellulose is alkalized at ambient temperature for 1 h. In the following, sodium monochloro acetate (NaMCAc, 7g) is added, the temperature raised to 55 °C and the reaction continued for 3 h. The slurry is converted to the acid form by adding acidified (HCl) isopropanol. Excess acid is removed by washing with a 70 wt% ethanol water solution (until pH is neutral) over a Buchner funnel equipped with a suction flask and the material is dried in the vacuum oven. Weighed samples of the free acid are dissolved in distilled water, containing an excess of standard sodium hydroxide, and the excess base is back-titrated with standard hydrochloric acid (over a burette), using phenolphthalein as pH indicator.

Experiment No:3

PREPARATION OF PHENOL FORMALDEHYDE RESIN

Aim:-

To prepare phenol formaldehyde resin.

Apparatus:-

Beaker, glass rod, funnel, filter paper, and chemical balance.

Chemicals:-

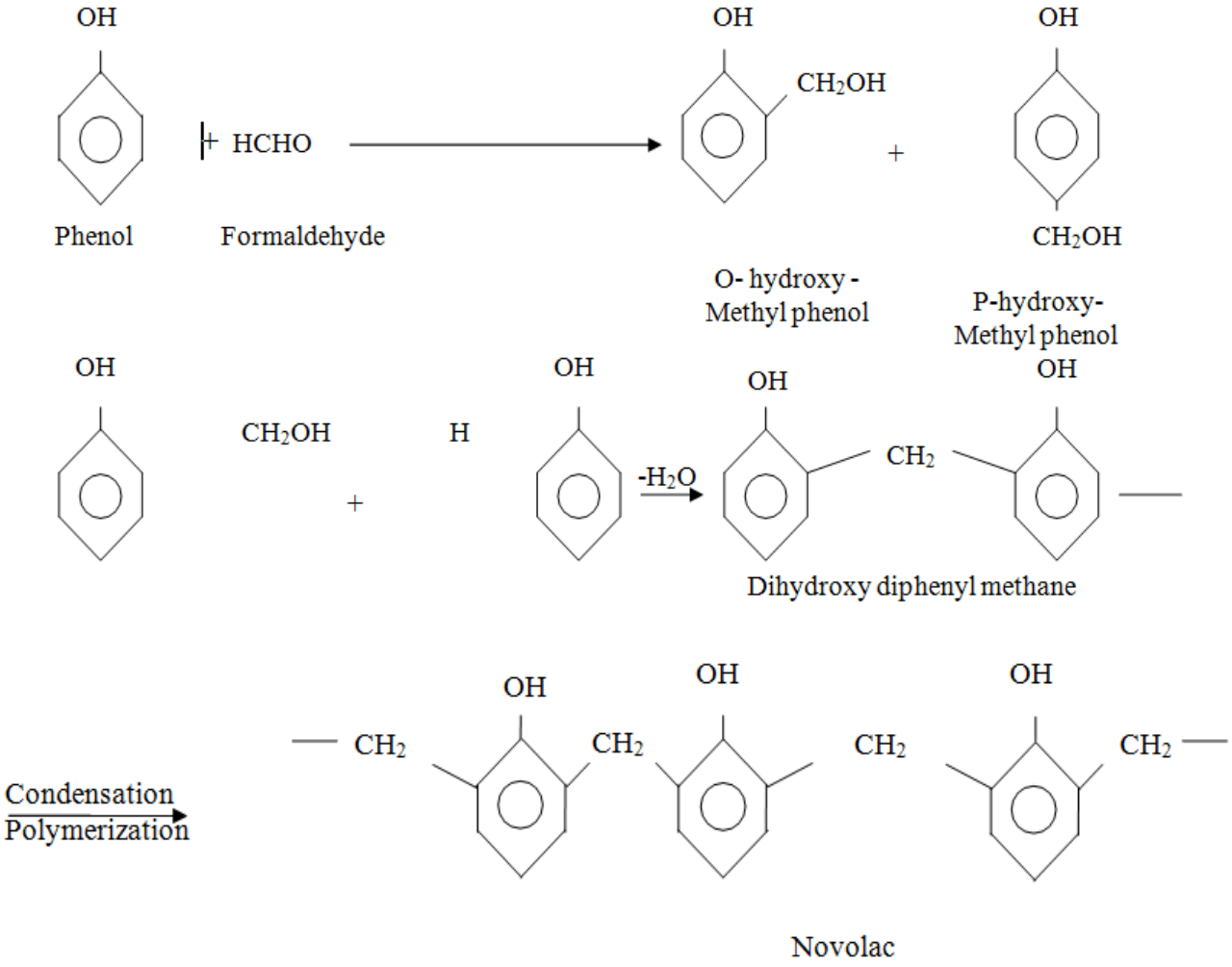
Phenol formaldehyde, conc. HCl, glacial acetic acid, distilled water.

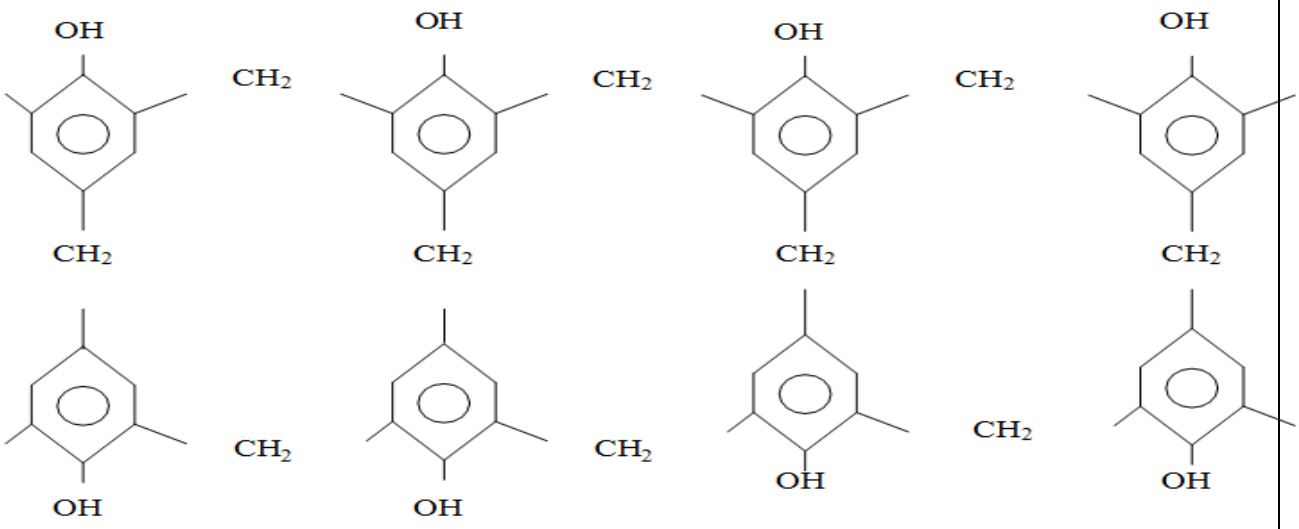
Theory:-

Phenolic resins are condensation polymerization of phenolic derivatives (like phenol, resorcinol) with aldehyde (like formaldehyde, furfural). Most important member of this class is bakelite or phenol formaldehyderesin.

Phenol formaldehyde is prepared by condensing phenol with formaldehyde in presence of acidic or alkaline catalyst. The initial reaction result in the formation of o- and p-hydroxy methyl phenol, which react to form linear polymer navalac.

During moulding hexamethyline tetramine [$(CH_2)_6N_4$] is added which convert the fusible novalac in to hard infusible and insoluble solid of cross – linked structure known as Bakelite.


OH
+ HCHO
CH2OH
Phenol
Formaldehyde
O- hydroxy - Methyl phenol
P-hydroxy- Methyl phenol
CH2OH
H
-H2O
CH2
Dihydroxy diphenyl methane
Condensation Polymerization
Novolac



BAKELITE

Procedure:-

1. Place 5 ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a 100 ml beaker .
2. Add 2 g of phenol toit.
3. Wrap a cloth loosely round the beaker. Add a few ml of conc. HCl in to the mixture carefully and heat itslightly.
4. A large mass of plastic pink in colour isformed.
5. A residue is washed with water andfiltered.
6. The product dried and yield isweighed.

Precautions:-

1. While adding conc. HCl, it is better to stay little away from the beaker since the reaction sometimes becomesvigorous.
2. The reaction mixture should be stirredcontinuously.

Observations:-

Mass of the beaker(W1)=---------- g.

Mass of the beaker with phenol formaldehyde(W2)= ---------- g.

Therefore mass of phenol formaldehyde (W2 –W1) = --------- g.

Result:-

The yield of phenol formaldehydeis= ----------g

Properties:-

1. Phenol formaldehyde moulding resins have excellent heatresistance.
2. These have high dimensionalstability.
3. Phenolic resins have good dielectricproperties.
4. They have hard, regid and scratchresistant.

Uses:-

1. They are used for making electric insulator parts like switches, plugs, switch board, heater, handles etc.
2.
3. These are also used in varnishes, paints and protective coatings.
4. These are used in the protection of ion exchange resins for watersoftening.
5. Phenolic resins are used for improving impregnating paper, wood and otherfillers.

Experiment No: 4

SYNTHESIS OF ISOTACTIC AND SYNDIOTACTICPMMA

Aim

Synthesis of Isotactic and SyndiotacticPMMA

Reagents and Materials

Name
Methyl methacrylate (MMA), dry
n-BuLi (1.6M in hexane)
Dimethoxyethane (DME), dry
Toluene, dry
Dichloromethane
Methanol
Pyridine, dry
dry ice/ethanol, dry ice/acetone

Procedure

The initiator mixture is prepared by mixing $6.4x10^{-3}$ mol of n-BuLi (solution in hexane) with 1.5 equivalents of dry pyridine in a well dried, nitrogen flushed Schlenk tube closed with a septum, using a syringe.

15 mL of dry DME is added into one of two completely dried, nitrogen flushed 25 mL Schlenk flask (NS 14, equipped with a magnetic stir bar) (**1.2.1.1**) and 15 mL of dry toluene into the other (**1.2.1.2**). The tubes are closed with septa under a slight nitrogen stream.

Inject about 0.5mL of the initiator to each of the tubes and cool them down to -78°C (**1.2.1.2**), -50 °C (**1.2.1.1**). Under vigorous stirring, add $9.39x10^{-3}$ mol MMA to each tube. The reaction mixture should turn red. If there is no change in color, there must have been some moisture in the reaction mixture. Try to add another 0.5 mL of the initiator mixture. After 20 min the tubes are replaced from cooling and the polymerizations are stopped by slowly adding about 1 mL methanol.

Work up:

The polymer from **1.2.1.1** (DME) is precipitated by dropping into 75 mL of stirred methanol (beaker equipped with magnetic stir bar), whereas the polymer from **1.2.1.2** (toluene) is diluted with 10

mL DCM prior to precipitation into 150 mL methanol. The polymers are filtered with suction, washed with methanol and dried overnight at 50 °C in a vacuum oven. The next day determine the yield and the melting points of the polymers.

Evaluation

Store the polymers for IR and 1H-NMR analysis for the determination of their tacticity.

The table below gives the wave numbers of the IR spectra which are sensitive to the tacticity of the polymers (+ indicates the relative intensity).

cm-1	**677**	**790**	**808**	**945**	**1035**	**1100**	**1212**	**1240**	**1338**
syndiotactic	+++ +	+++ +	+++ +	+++ +	++++	++++	++++	++++	++++
atactic	+++	+++ +	+++	+++	+++	+++	+++	+++	+++
isotactic	0	+++ +	++	0	+	0	0	+	++

CLASS : I B.Sc CHEMISTRY
COURSE NAME : STATES OF MATTER AND IONIC EQUILIBRIUM -PRACTICAL
COURSE CODE : 18CHU112SEMESTER : I BATCH-2018-2020

VISCOSITY MEASUREMENTS

Experiment No 5

DETERMINATION OF CO-EFFICIENT OF VISCOSITY OF AN UNKNOWN AQUEOUS SOLUTION

Aim:

To determine the viscosity of a given unknown liquid with respect to water, at laboratory temperature, by viscometer.

Requirements:

Ostwald viscometer, rubber tube with screw pinch cock, stand, beaker, unknown liquid, distilled water. specific gravity bottle

Theory:

The force of friction which one part of the liquid offers to another part of the liquid is called viscosity. For measuring the viscosity coefficient, Ostwald viscometer method is used which is based on Poiseuille's law. According to this law, the rate of flow of liquid through a capillary tube having viscosity coefficient, , can be expressed as

$$\eta = \pi \cdot r^4 tP/8vl$$

where, v= vol. of liquid (in ml)
t= flow time (in sec.) through capillary r= radius of the capillary (in cm)
l= length of the capillary (in cm)
P= hydrostatic pressure (in dyne/sq.cm)
η= viscosity coefficient (in poise).

Since, the hydrostatic pressure (the driving force) of the liquid is given by P = dg h (where h is the height of the column and d is the density of the liquid); $\eta \propto Pt$; or, $\eta \propto d\ g\ ht$

If, η1 and η2 are the viscosity coefficients of the liquids under study, d1, d2, are their densities and t1 and t2 are their times of flow of *equal volume* of liquids through the same capillary respectively, then

η1 ∝ d1 g h t1 and
η2∝ d2 g h t2

$\eta_1 = \underline{d_1 t_1}$

Hence, $\eta_2 = d_2 t_2$

Here, usually the viscosity of given liquid is measured with respect to water whose viscosity is known very accurately at different temperatures. The SI physical unit of viscosity is the pascal-second (**Pa·s**), (i.e., $kg \cdot m^{-1} \cdot s^{-1}$). This means if a liquid with aviscosity of one Pa·s is placed between two plates, and one plate is pushed sideways with a shear stress of one pascal, it moves a distance equal to the thickness of the layer between the plates in one second. The cgs unit for the same is the poise (P), (named after J. L. Marie Poiseuille). It is more commonly expressed, as centipoise (cP). [1 cP = 0.001 Pa·s]. Water at 20°C has a viscosity of 1.0020 cP.

Procedure:

1. Note the laboratory temperature.
2. Wash the specific gravity bottle with distilled water anddry.
3. Take the weight of the empty & filled (with distilled water) specific gravity bottle (with stopper). Then, weigh the filled with specific gravity bottle h unknown given liquid. Use the data for measuring thedensities.
4. Clean and rinse the viscometer properly with distilled water. Fix the viscometer vertically on the stand and filled with specific amount (say 20ml) of mixture (every time take the samevolume).
5. Time of flows were recorded for each solutions (water and the given liquid).
6. Take 3 to 4readings.

Observations:

Laboratory temperature=….□C

Density measurement:

Weight of empty R.D.bottle (w1) =…g.
Weight of R.D.bottle with water (w2) =…g.
Weight of R.D. bottle with liquid (w3) =…g. So,
weight of water (ww) = (w2-w1) =…g.

l no.	**Flow times (sec)**			
	t1	t2	t3	mean
1				
2				
3				
4				

Calculations:

Determination of the density of the liquid(dl):

Density of liquid (dl) / Density of water (dw) = Weight of liquid (wl) / Weight of water (ww)

Density of liquid (*dl*) □*wl* /*d w ww*

(Take density of water =1.0g/ml at 25

Determination of the viscosity of the liquid (□l)

$$Viscosity of the liquid, \eta_l = \frac{t_1 d_1}{t_w d_w}$$

Result:

The viscosity of the given liquid with respect to water atlaboratory temperature was foundtobe cP.

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