SINCE 1962

VIVEKANAND EDUCATION SOCIETY'S
INSTITUTE OF TECHNOLOGY

ENGINEERING CHEMISTRY

ODD SEM

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LAB 503

1. HARDNESS OF WATER BY EDTA METHOD.

AIM : To determine the hardness of water sample by EDTA method.

APPARATUS: Burette, pipette, conical flask, etc.

REAGENTS : EDTA solution, buffer solution of pH=10, std. $CaCl_2$ solution and solution
REQUIRED of Eriochrome black T.

THEORY :This is a very accurate method based on the fact that when a blue colored Eriochrome back T is added to hard water in alkaline medium, it gives a wine red unstable complex with Ca^{2+} and Mg^{2+} ions. Now if EDTA is added then Ca^{2+} and Mg^{2+} ions form an unstable complex which are attacked by EDTA to form a stable complex of EDTA with Ca^{2+} and Mg^{2+} ions and EBT is given back.

PROCEDURE :

Preparation of Standard Hard Water
Weigh accurately1.1 gm of $CaCl_2$. Transfer the contents to 100 ml beaker Add water to dissolve $CaCl_2$. Transfer the content to a 1000 ml volumetric flask. and dilute up to the mark
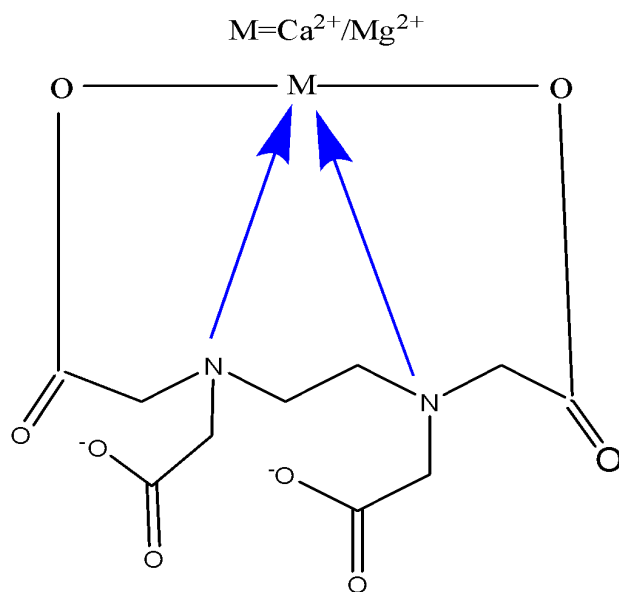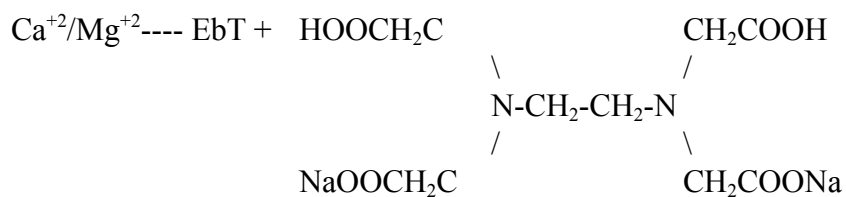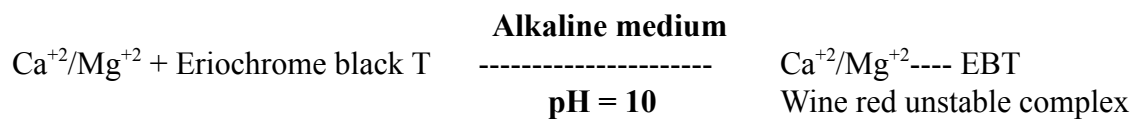
Standardization of EDTA solution.
Rinse and fill burette with an EDTA solution. Pipette out 25 ml of standard $CaCl_2$ solution in a conical flask. Add 1 test tube of buffer solution. Add 5-6 drops of Eriochrome black T till wine red colour is obtained. Then add EDTA solution from the burette till colour changes from wine red to blue.

Titration of unknown hard water sample.
Take 25ml of hard water sample in a conical flask and add 1 test tube of buffer solution and 5-6 drops of indicator. (EbT) Titrate the mixture against EDTA solution till blue color is obtained.

RESULT : Hardness of given water sample is 400 ppm.

REACTIONS :

Alkaline medium

Ca^{+2}/Mg^{+2} + Eriochrome black T   ---------------------- Ca^{+2}/Mg^{+2}---- EBT

                                                                    pH = 10   Wine red unstable complex

Ca^{+2}/Mg^{+2}---- EbT + $HOOCH_2C$ CH_2COOH

                                                                   \   /

                                                          $N-CH_2-CH_2-N$

                                                                   /   \

                                           $NaOOCH_2C$ CH_2COONa

$M=Ca^{2+}/Mg^{2+}$

O ——————— M ——————— O

M-EDTA

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OBSERVATION :

PART I

Solution in burette   : EDTA solution
Solution in conical flask: 25ml of $CaCl_2$ solution + 1 test tube buffer solution+5-6drops indicator
Indicator   : Erichrome black T
Endpoint   : Wine red to blue
Pilot Reading   : **25** ml to **26** ml.

READINGS	I	II	III	CONSTANT V1(ml)
INITIAL (ml)	0.0ml	0.0ml	0.0ml	
FINAL (ml)	25ml	25ml	25ml	**25ml**
DIFFERENCE	25ml	25ml	25ml	

PART II

Solution in burette   : EDTA solution
Solution in conical flask: 25 ml of hard water + 1 test tube of buffer solution.
Indicator   : Erichrome black T
Endpoint   : Wine red to blue
Pilot Reading   : **10** ml to **11** ml.

READINGS	I	II	III	CONSTANT V2(ml)
INITIAL (ml)	0.0ml	0.0ml	0.0ml	
FINAL (ml)	10ml	10ml	10ml	**10ml**
DIFFERENCE	10ml	10ml	10ml	

CALCULATIONS :

1ml of standard CaC1$_2$ solution   1 mg CaCO$_3$

25ml of standard CaC12 solution   25 ml EDTA

i.e. 25ml EDTA   25mg CaCO$_3$ equivalent

Hence, 1ml EDTA   25/25 mg CaCO$_3$ equivalent

25ml of unknown hard water   10ml EDTA
                                                    10* 25/25 mg CaCO$_3$ equivalent.

Hence, 1000ml of unknown hard water   (25/ 25) *10*(1000/ 25) mg of CaCO$_3$ equivalent
                                                                            =400 mg CaCO$_3$ equivalent

Hence, hardness of water = **400 ppm**

2. CHLORIDE CONTENT OF WATER

AIM : To determine the chloride content of water.

APPARATUS: Burette, conical flask, measurement cylinder, etc.

REAGENTS : Silver nitrate solution and potassium chromate.
REQUIRED

THEORY : When silver nitrate is titrated against chlorides present in water using Potassium chromate as indicator, silver chloride is formed. When all the Chlorides in water are consumed, the slight excess of silver nitrate imparts Brick red colour due to the formation of silver nitrate to silver Chromate.

PROCEDURE:

Preparation of 0.01N KCl
Weigh accurately 0.0745 gm of KCl. Transfer the contents to 100 ml beaker Add water to dissolve KCl. Transfer the content to 1000 ml volumetric flask. and dilute up to the mark.
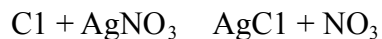
Standardization of Ag NO$_3$
Fill the burette with approx 0.01N AgNO$_3$. Pipette out 10 ml of 0.01N KCl solution in conical flask. Add 3/4 drops of 5% K$_2$CrO$_4$ indicator. Titrate the yellow solution with AgNO$_3$ adding drop wise with stirring. When solution shows brick red ting stop titration and record the burette reading.

Estimation of Chloride in water sample.
Pipette out 10 ml of sample water in a conical flask and add a 3/4 drop (1ml) of Potassium chromate to it. Titrate the contents of conical flask against approx 0.01NAgNO$_3$ solution from burette till brick red colouration appears.

RESULT :The chloride content of water is **218.4** ppm.

REACTIONS :

$Cl + AgNO_3 \quad AgCl + NO_3$

$2AgNO_3 + K_2CrO_4 \quad Ag_2CrO_4 + 2KNO_3$

Brick red

OBSERVATION :

Standardisation of AG NO$_3$

Solution in burette : 0.01 N(approx.) of AgNO$_3$

Solution in conical flask: 10 ml of 0.01N KCl

Indicator : Potassium chromate solution

Endpoint : Yellow to reddish orange

Pilot Reading : **10** To **11** ml

READINGS	I	II	III	CONSTANT
INITIAL (ml)	0.0ml	0.0ml	0.0ml	
FINAL (ml)	10.3ml	10.4ml	10.4ml	**10.4ml**
DIFFERENCE	10.3ml	10.4ml	10.4ml	

Estimation of Chloride in water sample.

Solution in burette : 0.01 N of AgNO$_3$

Solution in conical flask: 10 ml of sample water

Indicator : Potassium chromate solution

Endpoint : Yellow to reddish orange

Pilot Reading : **6** To **7** ml

READINGS	I	II	III	CONSTANT
INITIAL (ml)	0.0ml	0.0ml	0.0ml	
FINAL (ml)	6.4ml	6.3ml	6.4ml	**6.4ml**
DIFFERENCE	6.4ml	6.3ml	6.4ml	

CALCULATION :

Standardisation of Ag NO$_3$

$$
\begin{array}{cc}
\textbf{KCl} & \textbf{AgNO}_3 \\
N_1 V^1 & N_2 V_2 \\
0.01 \times 10 \quad = & N_2 \times [\;] \\
\end{array}
$$

$$\frac{0.01 \times 10}{10.4} = 9.6153 \times 10^{-3} \; N$$

Normality of AgNO$_3$ = 9.6153 x 10-3 N

Estimation of Chloride in water sample.

1000 ml of 1 N AgNO$_3$ 35.5g of Chlorine

Hence, [6.4] ml   of 9.6153 x 10^{-3} N AgNO3 $\dfrac{35.5 \times 9.6153 \times 10\text{-}3 \times 1000 \times [6.4]}{1000}$

$$= 2.184 \text{ mg of Cl}$$

10ml of sample water contains of   2.184 mg of Cl

Hence, 1000 ml of sample water contains $\dfrac{[2.184] \times 1000}{10}$ mg of Cl

$$= 218.4 \text{ mg}$$

Hence, chloride content of Water = **218.4 ppm.**

3. Determination of pH

Aim : To Determine the pH of the given concentrations of Hydrochloric acid   solution .
Introduction :   The term pH refers to the measure of hydrogen ion concentration in a solution and defined as the negative log of H^+ ions concentration in water and wastewater. The values of pH O to a little less than 7 are termed as acidic and the values of pH a little above 7 to 14 are termed as basic. When the concentration of H^+ and OH^- ions are equal then it is termed as neutral pH.

Principle : The pH electrode used in the pH measurement is a combined glass electrode. It consists of sensing half cell and reference half cell, together form an electrode system. The sensing half cell is a thin pH sensitive semi permeable membrane, separating two solutions, viz., the outer solution, the sample to be analyzed and the internal solution, enclosed inside the glass membrane and has a known pH value. An electrical potential is developed inside and another electrical potential is developed outside, the difference in the potential is measured and is given as the pH of the sample.

Apparatus Required : pH meter, Standard flasks, Funnel, Beaker, Wash Bottle, Tissue Paper, Forceps.

Chemicals Required : Buffers solutions of pH 4.01, 7.0 and 9.2, Potassium Chloride, Distilled water, HCl solution of different concentrations.

Procedure : Three major steps are involved in the experiment. They are …
1] Preparation of Reagents.
2] Calibrating the Instrument
3] Testing of Sample.

Preparation of Reagents :
i] Buffer solution of pH 4.0 : Take 100 ml standard measuring flask and place a funnel over it. Using the forceps carefully transfer one buffer tablet of pH 4.0 to the funnel. Add little amount of distilled water, crush the tablet and dissolved it.   Make up the volume to 100 ml using distilled water.

ii] Buffer solution of pH 7.0 : Take 100 ml standard measuring flask and place a funnel over it. Using the forceps carefully transfer one buffer tablet of pH 7.0 to the funnel. Add little amount of distilled water, crush the tablet and dissolved it. Make up the volume to 100 ml using distilled water.
iii] Buffer solution of pH 9.2 : Take 100 ml standard measuring flask and place a funnel over it. Using the forceps carefully transfer one buffer tablet of pH 9.2 to the funnel. Add little amount of distilled water, crush the tablet and dissolved it. Make up the volume to 100 ml using distilled water.

Calibrating the instrument : Using the buffer solutions calibrate the instrument.

Step I :
In a 100 ml beaker take pH 9.2 buffer solution and place it in a magnetic stirrer insert the teflon coated stirring bar and stir well. Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter. If the instrument is not showing pH value of 9.2, using calibration knob adjust the reading to 9.2 Take the electrode from the buffer wash it with distilled water and then wipe gently with soft tissue.

Step II :
In a 100 ml beaker take pH 7.0 buffer solution and place it in a magnetic stirrer, insert the teflon coated stirring bar and stir well. Now place the electrode in the beaker containing the stirred buffer and check for reading in the pH meter. If the instrument is not showing pH value of 7.0 using the calibration knob adjust the reading to 7.0. Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Step III :
In a 100 ml beaker take pH 4.0 buffer solution and place it in a magnetic stirrer, insert the teflon coated stirring bar and stir well. Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter. If the instrument is not showing pH value of 4.0 using the calibration knob adjust the reading to 4.0. Take the electrode from the buffer wash it with distilled water and then wipe gently with soft tissue.
Now the instrument is calibrated.

Testing Of Sample :
In a clean dry 100 ml beaker take the oxalic acid solution sample and place it in a magnetic stirrer, insert the teflon coated stirring bar and stir well.
Now place the electrode in the beaker containing the water sample and check for the reading in the pH meter. Wait until you get a stable reading

Take the electrode from the water sample, wash it with distilled water and then wipe gently with soft tissue.

Observation Table :

Sr. No.	ml of Initial Concentration of HCl acid	Dilution to -- ml	pH	Final concentration of HCl acid solution
1	50 ml of 1 M solution	500 ml	2.2	6.3×10^{-3}
2	50 ml of 10^{-1} M solution	500 ml	3.2	6.3×10^{-4}
3	50 ml of 10^{-2} M solution	500 ml	5.25	5.62×10^{-6}
4	50 ml of 10^{-3} M solution	500 ml	7.6	2.51×10^{-8}

Calculations :

Solution 1 : pH = 2.2

$$= \frac{1}{Antilog[2.2]}$$

$$= 6.3 \times 10^{-3} M$$

Solution 2 : pH = 3.2

$$= \frac{1}{Antilog[3.2]}$$

$$= 6.3 \times 10^{-4} M$$

And so on.

Result : It is confirmed that pH of the solution increases as the hydrogen ion concentration (Molarity) decreases.

4. ESTIMATION OF COPPER BY COLORIMETRIC METHOD

AIM: Estimation of Copper by Colorimetry

APPARATUS: - Colorimeter, Test tubes, Burettes.

PRINCIPLE: - Colorimeter measures the optical density of an absorbing substance where optical density (O.D) is defined as $O.D = \log \frac{I_o}{I} \to 1$ Where I_o= Intensity of incident light I = Intensity of transmitted light

As per beers law, optical density of an absorbing substance is related to the concentration by the equation. $OD. = ECl. . OD. = (El). C \to 2$ Where 'C' is the concentration of the substance, l is the path length, which represents the width of the cell used and is constant for a given cell used, E is the molar absorption coefficient and is a constant for given substance. Equation 2 may be written as $O.D. \alpha C \; 3 \to$

Equation 3 represents the quantitative form of Beer's law. if the optical density of a substance is determined at varying concentration. A plot of O.D.vs C gives a straight line.

PROCEDURE:- Take the sample solution of CuSo4 and prepare the following 10 sample solutions
in test tubes as 1 to 10

1. 1 ml 1 m $CuSO_4$ + 9 ml Distilled water
2. 2 ml 1 m $CuSO_4$ + 8 ml Distilled water
3. 3 ml 1 m $CuSO_4$ + 7 ml Distilled water
4. 4 ml 1 m $CuSO_4$ + 6 ml Distilled water
5. 5 ml 1 m $CuSO_4$ + 5 ml Distilled water
6. 6 ml 1 m $CuSO_4$ + 4 ml Distilled water
7. 7 ml 1 m $CuSO_4$ + 3 ml Distilled water
8. 8 ml 1 m $CuSO_4$ + 2 ml Distilled water
9. 9ml 1 m $CuSO_4$ + 1 ml Distilled water
10. 10 ml 1 m $CuSO_4$ + 0 ml Distilled water

The ten sample solutions prepared above have a varying concern from 0.1m to1m, choose the filter in the colorimeter with maximum absorbance. Tabulate the result of filter and O.D with a given CuSo4 sample solutions.

After selecting filter, determine the O.D. of the above mentioned ten sample solutions and tabulate the results.
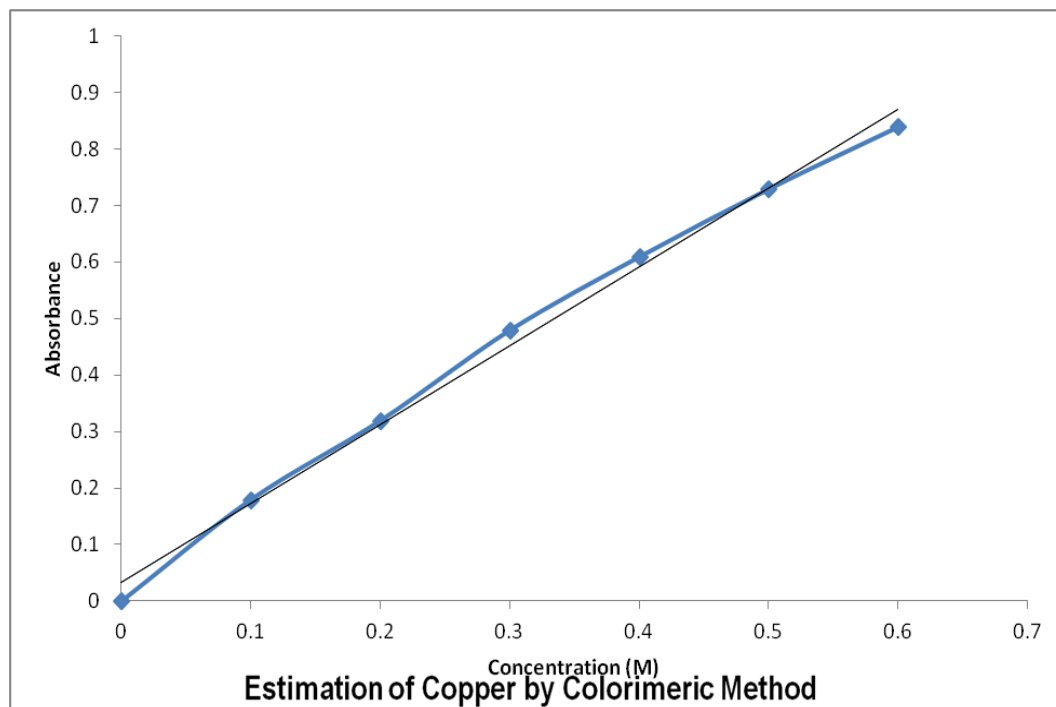
Observation:

Table-1

Filter No.	ג Range (nm)	Optical Density (OD)
1	400	0.19
2	450	0.12
3	490	0.04
4	520	0.04
5	540	0.13
6	570	0.30
7	620	0.46
8	680	0.74

Table II   **Selected Filter : 680nm**

Sl. No.	Volume Of CuSO4 (ml)	Volume of H2O (ml)	Concentration of CuSO4	Optical Density
1	1	9	0.1	0.18
2	2	8	0.2	0.32
3	3	7	0.3	0.48
4	4	6	0.4	0.61
5	5	5	0.5	0.73
6	6	4	0.6	0.84
7			Unknown	0.54

GRAPH: - Plot a graph of O.D Vs [CuSo4]. A straight line passing through origin is obtained the slope of which gives El, E is the molar absorption coefficient. (sample graph)

Estimation of Copper by Colorimeric Method

RESULT: **-** Amount of copper present in given CuSo4 solution is **22.86 g/lit.**

5. TO PREPARE PHENOL FORMALDEHYDE (P-F) RESIN.

Aim: To prepare Phenol formaldehyde (P-F) resin.

Chemicals required: Phenol (2g), 40% aq formaldehyde solution or formalin (2.5 mL), glacial acetic acid (5 mL) and conc. HCl (8mL).
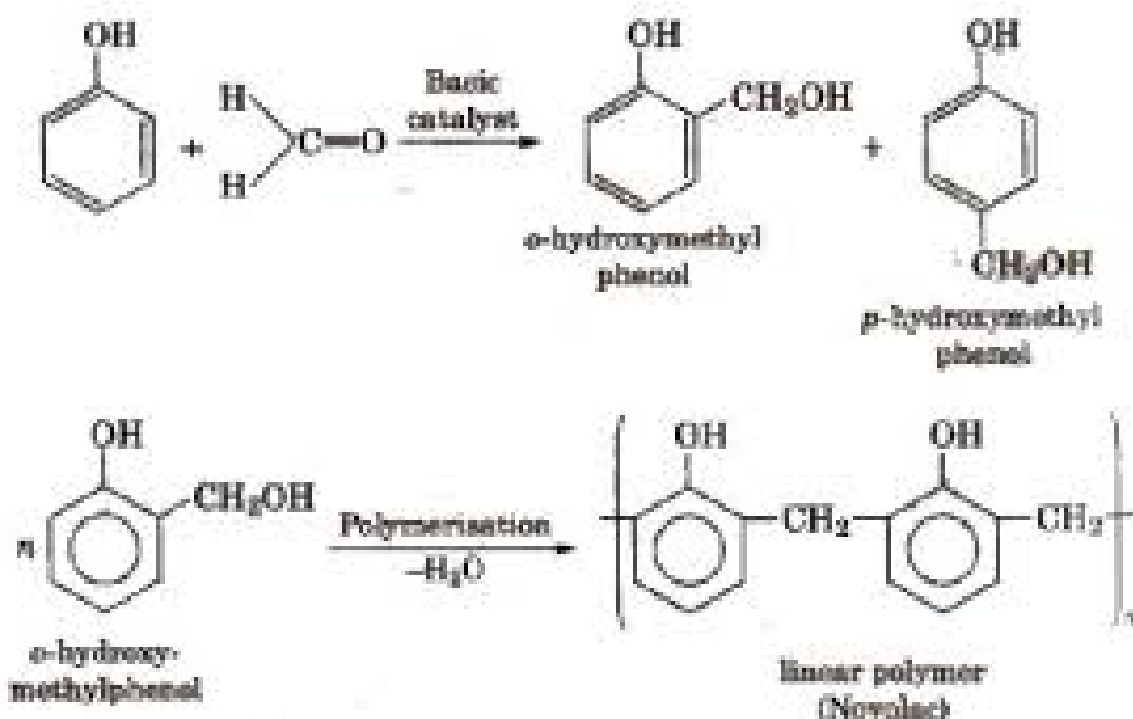
Theory: Phenol formaldehyde resin or P-F resin or phenolic resins (also called phenoplasts) are important class of polymers which are formed by condensation polymerization of phenol and formaldehyde in acidic or alkaline medium. Following steps are involved:

Step 1: Formation of methylol phenol derivative
Initially the monomers combine to form methylol phenol derivative depending upon phenol to formaldehyde ratio.
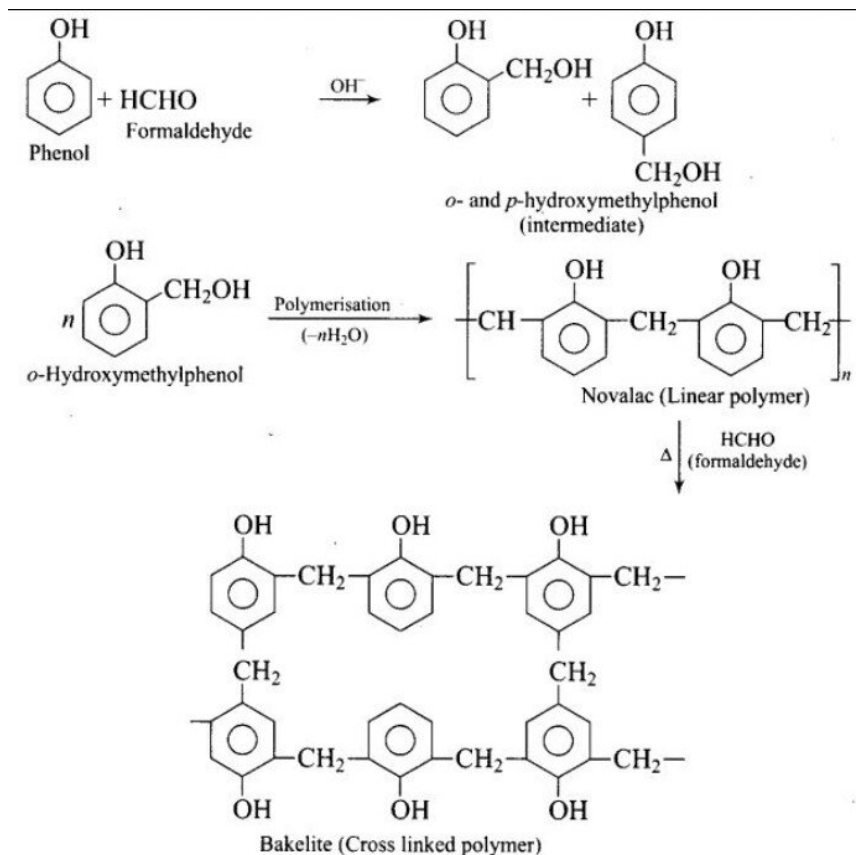
Step 2: The phenol formaldehyde derivatives react among themselves or with phenol to give a linear polymer or a higher cross linked polymer.
   (a) Linear polymer (Novolac)

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(b) Cross linked polymer (Bakelite)

A highly cross linked thermosetting polymer called Bakelite may be formed by further condensation of novolac or methylol derivative. It was first prepared by Backeland. It is easily formed if curing agent hexamethylenetetramine is added during synthesis

o- and p-hydroxymethylphenol
(intermediate)

Novalac (Linear polymer)

Bakelite (Cross linked polymer)

.

Procedure:

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40 % aq formaldehyde solution in a 100 mL beaker. Add 2 g phenol safely.

2. Wrap the beaker with a wet cloth or place it in a 250 mL beaker having small amount of water in it.

3. Add conc. HCl drop wise with vigorous stirring by a glass rod till a pink coloured gummy mass appears.

4. Wash the pink residue several times with to make it free from acid.

5. Filter the product and weigh it after drying in folds of a filter or in an oven. Report the yield of polymer formed.

Observation:

Weight of empty watch glass= W1 = **68.55** g
Weight of watch glass + polymer formed=W2 = **71.15** g
Weight of polymer formed = W2 – W1 g = **2.6** g

Result:

Weight of phenol formaldehyde resin = **2.6** g

6. TO PREPARE UREA FORMALDEHYDE (U-F) RESIN.

Aim: To prepare Urea formaldehyde (U-F) resin.

Chemical required: Urea (2g), 40% aq formaldehyde solution or formalin (5 mL), conc. H2SO4 (3-4 drops).

Theory:
Urea formaldehyde resins are formed by condensation of urea and formaldehyde in acidic medium in following steps:

Step 1. Formation of methylol urea derivative
Initially urea and formaldehyde react to form methylol urea derivatives depending upon forsmaldehyde (U/F ratio).

Step 2: Polymerization of methylol urea
Several molecules of methylol urea derivatives condense with loss of water molecules to form a highly cross linked urea formaldehyde resin.

Procedure :
Take a 5 mL of 40% aqueous formaldehyde solution in a 100 mL beaker. To this add 2 g urea powder. Stir with a glass rod to make a saturated solution. Add a few drops of conc. H2SO4 and stir vigorously till a white solid mass is formed. Filter the residue and wash it several times with distilled water to remove any acid. Dry the residue in folds of filter paper or in an oven and weigh. Report the yield of urea formaldehyde polymer formed.

Observation :

Weight of empty watch glass =W1= **72.99** g
Weight of watch glass + polymer formed = W2= **81.2** g
Weight of polymer formed = W2 – W1 g = **8.21** g

Result : Weight of urea formaldehyde resin = **8.21** g