I. PREPARATION OF PHENOL - FORMALDEHYDE (P-F) RESIN (NOVOLAC)

Chemicals required: Phenol (2g), 40% aq formaldehyde solution or formalin (2.5 cc), glacial acetic acid (5 cc) and conc. HCl (8cc). 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 (Novolac) or a higher crosslinked polymer (Bakelite) A highly cros 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 hexamethylene tetramine is added during synthesis.

Procedure

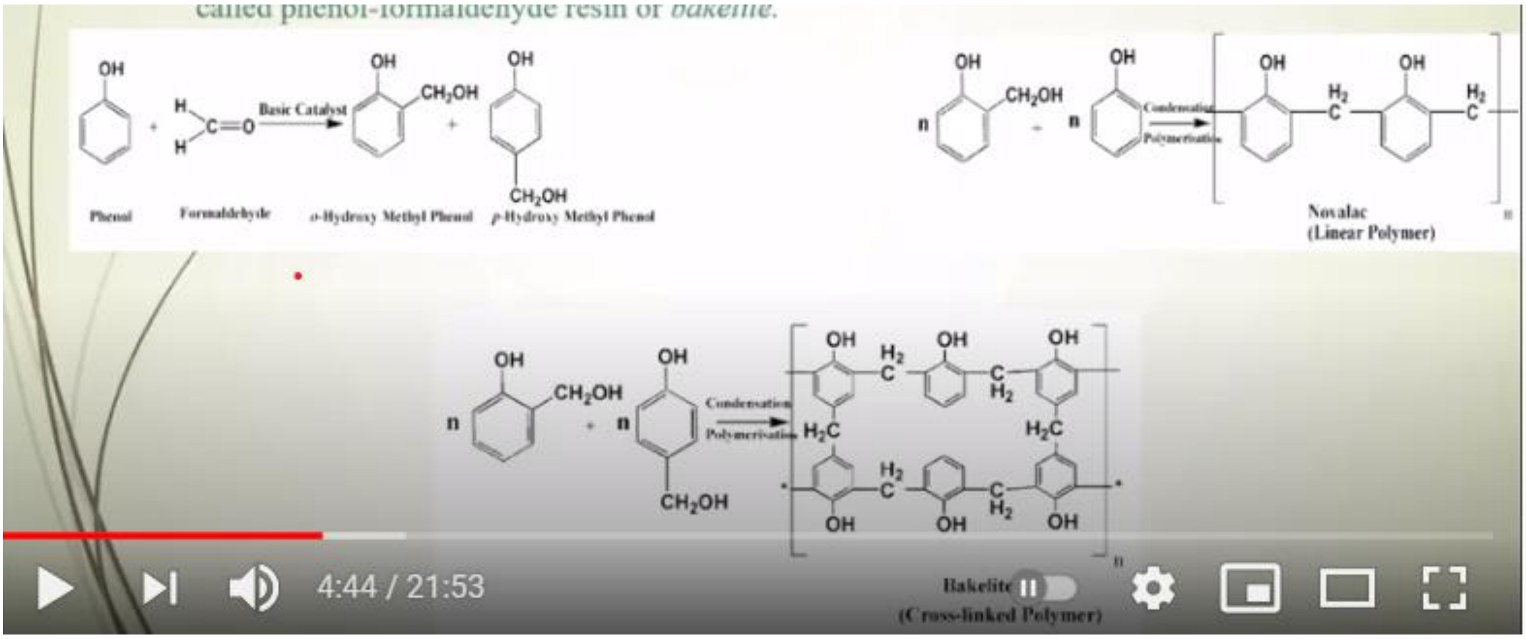
1. Place 5 cc of glacial acetic acid and 2.5 cc of 40 % aq formaldehyde solution in a 100 cc beaker. Add 2 g phenol safely.

2. Add conc. HCl drop wise with vigorous stirring (Preferably in a magnetic stirrer), and wrap it by a glass rod till a pink coloured gummy mass appears.

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

4. 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 g Weight of watch glass + poymer formed = W2 g Weight of polymer formed = W2 – W1 g Result Weight of phenol formaldehyde resin = W g



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II. PRECIPITATION POLYMERIZATION OF ACRYLONITRILE WITH A REDOX SYSTEM IN WATER.

Procedure:

Into 250 cc round-bottomed flask 175 cc water is added and nitrogen is babbled for about 30 min. Then add 15 cc acrylonitrile, 0.5 cc of $Na_2S_2O_5$ solution (5% in water) and 2.5 cc of $FeSO_4.7H_2O$, solution (10 mg in 100 cc water+2 cc conc. H_2S0_4) are introduced and subsequently 2.5 cc of $K_2S_2O_8$ solution (5% in water) is added and mixed briefly at 20°C. The mixture is stirred at 20°C for about 1 h, the precipitated polymer is filtered washed with water then with methanol and dried at 50°C in vacuum

III. PREPARATION OF NYLON 66

Requirements:

Apparatus: Round bottom flask (50 cc), Air condenser, Beaker (100 cc), Beaker (400 cc)

Chemicals: Adipic acid, Hexamethylene diamine, Thionyl chloride, Dimethyl formamide, Carbon tetrachloride, Sodium hydroxide, Alcohol

Procedure:

Preparation of adipoyl chloride:

1. Set up the apparatus as shown in the figure. For this take a 50 cc round bottom flask and fit an air condenser on to it. Take a glass funnel and inset its sleen into a piece of rubber tubing. Insert a glass tube in the other end of the rubber tube and fit it on the air condenser. Put the inverted funnel into a beaker containing water or NaOH solution.

2. Take l g of adipic acid in 3-4 drops of dimethyl formamide in the flask and add 1 cc of thionyl chloride dropwise with constant stirring.

3. Heat the flask on water bath for about 15 minutes. By this the evolution of gas ceased and the solid will disappear.

4. Allow the flask to cool a little and then add about 30cc of $CC1_4$ to it. Mix thoroughly to dissolve the product obtained.

5. Transfer this solution to a 100 cc beaker. Wash the flask thoroughly with additional 20 cc of CCl_4, and transfer the washings to the beaker.

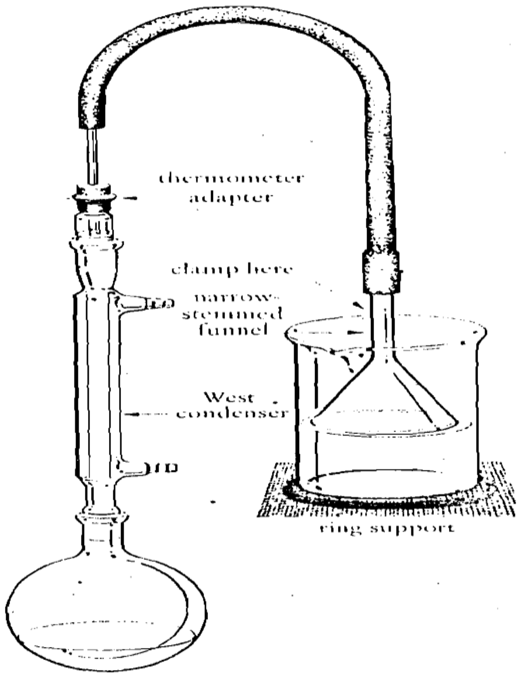
6. In a separate 100 cc beaker take 1.1 g of hexamethylene diamine and 0.75 g of NaOH in 25 cc of water and mix to dissolve them.

7. Carefully transfer the aqueous solution of hexa methylene diamine to the beaker containing adipoyl chloride.

8. You will observe the formation of a film of nylon 6,6, at the interface of the two liquids. Carefully insert a glass rod or forceps into the solution and pull out the polymer formed.

9. Wrap the polymer around a clean test tube. Rotate the test tube to pull more and more of nylon.

11. Dry the polymer in air or in the folds of filter paper. Weigh it and report the yield.



Reaction link : https://youtu.be/mIKdQG5Mp9k
Preparation Link: https://youtu.be/HTh_5CWMSoQ

****Difference between Nylon 6 and 66

Both Nylon 6 and Nylon 66 are polyamides, which means they are molecules whose repeating units are linked by amide bonds. Some polyamides, such as silk, can be found naturally, but nylons are made in a lab. There are several types of nylons, but Nylon 6 and 66 are two of the most popular ones thanks to being relatively lightweight while also strong and durable.

Chemical Differences

Though Nylon 6 and Nylon 66 share some physical properties, their chemical structures are different. Nylon 6 is made from a single type of monomer, called caprolactam. Caprolactam's formula is (CH2)5C(O)NH. Since its discovery in the 1800s, global demand of caprolactam has grown to more than 5 million tons per year, almost all of which goes toward making Nylon 6.

Nylon 66 is made up of two monomers, adipoyl chloride and hexamethylene diamine. The strong chemical bond between the two forces gives Nylon 66 a more crystalline structure, making it slightly stiffer and better equipped to handle more heat than Nylon 6.

Practical Applications

The first popular commercial use of nylon in the United States began in the early 1940s when the material was used to produce stockings for women. When World War II began and many of the country's resources were geared toward helping the war effort, scientists took to the lab to manufacture new, stronger materials. The result was the creation of nylon varieties like Nylon 6 and Nylon 66, which are far more durable than the nylon used for stockings.

Nylon 6 is used in all kinds of products including hammerheads, plastic cutting boards, rope and circuit breakers. One of its greatest strengths is its flexibility, which makes it a suitable metal replacement in products such as car parts. It's also a bit more lustrous than Nylon 66, so it's more commonly used in items such as radiator grilles, stadium seats or firearm components where manufacturers want an attractive surface finish.

Nylon 66 has a higher melting point and is usually more durable than Nylon 6, so it's a good choice for high-performing products that must withstand heat or wear and tear. That characteristic makes it a popular choice for items such as friction bearings, battery modules, luggage and conveyor belts.

Both Nylon 6 and 66 are used in household items. Nylon 66 tends to be more commonly used to make products like durable carpeting, whereas Nylon 6 is often found in places like the bristles of a cleaning brush.