EXPERIMENT 20
PREPARATION OF NYLON 66 - A CONDENSATION POLYMER

Structure

20.1 INTRODUCTION

In the previous experiment you have learnt about preparation and use of azo dyes. In this process you have learnt about diazotisation reaction.In the present experiment you are going to prepare a sample of nylon 66 as an example of yet another important class of organic compounds viz. polymers.

In past few decades the developments in the field of polymers have dramatically changed our way of living. The way we dress ourselves, the way we pack our things, the way we decorate/furnish our household, everything has changed. Owing to their wide variety and versatility they have entered into practically every sphere of our day to day life. Polythene, used in carry bags; polyesters, the synthetic fabrics ; poly vinyl chloride (PVC) used in water proof coverings ; melamines, the unbreakable crockery ; plastics, used for a whole range of materials have become part of our day to day vocabulary. The large number of available monomers and the ways in which they can combine together to form polymers provides for such a wide range of polymeric molecules,

In the preparation of nylon 66 you will learn about how small molecules " condense " together to make larger molecules i.e. condensation polymerisation. In the next section we have included a brief introduction to polymers, in general and nylon, in particular. In the next experiment, you will learn about another important class of organic compounds namely cosmetics and you will be preparing a sample of a face cream.

Objectives :

After performing these experiments you should be able to :

- prepare a sample of nylon 66
- explain condensation polymerisation, and
- list various applications of the polymer prepared.

20.2 POLYMERS : AN INTRODUCTION

The term 'POLYMERS' has been derived by the synthesis of two Greek words, "poly" meaning "many" and "meros" meaning "part".i.e. a molecule made up of many

parts. The number of small molecules that makes the polymer are called Monomers (monos meaning single and meros meaning part). The polymers can be of natural origin or synthetic.Natural polymers like proteins, nucleic acids and cellulose are the basic building blocks of living organisms.

The synthetic polymers, used in day to day life, may be classified on the basis of their properties as elastomers, fibres or plastics. Elastomers get elongated under stress and reform their shape on removing the stress. Fibres are thread like polymers which can be woven into fabrics e.g. nylon, dacron etc. These are light and have high tensile strength. Plastics, on the other hand are intermediate between elastomers and fabrics. They can have variety of properties from highly flexible thin sheets to tough solid material of polypropyline.

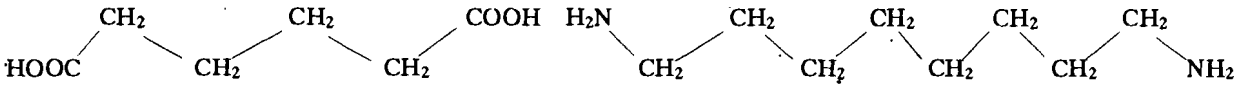
Another classification of polymers is based on the way the monomers are joined together to make a polymer. In addition, polymers the monomers are normally unsaturated and simply add on by 'opening up' their double bonds where as in condensation polymers the two monomers join together and eliminate a small molecule (like H_2O).

Nylon was the first completely synthetic fibre developed by a Carruthers of Du Pont laboratories in 2030. It is basically a polyamide formed by the condensation of monomers containing -- NH_2 and --COOH groups, respectively.

Now a days there are a whole range of nylon polymers. These are named according to the numbers of carbon atoms in the monomers making the polymer. For example the polymer you are going to prepare, nylon 66, has 6 C atoms in each of the monomers. While nylon 6 10 has 6 C atoms in the diamine and 10 carbon atoms in the diacid. In the next section you will learn about the reactions involved in the preparation of nylon 66 .

20.3 PRINCIPLE

As said above nylon 66 is a polyamide obtained by condensation polymerisation. The monomers of nylon 66, are adipic acid (I) and hexamethylene diamine (II). Adipic acid is a dicarboxylic acid with 6 carbon atoms while hexamethylenediamine is a diamine with same number of carbon atoms.



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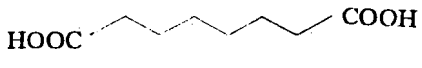
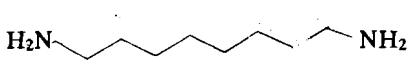


(I)

Or



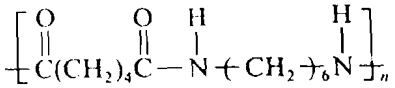
(II)

Adipic acid contributes toward the sharp taste of beets.

Let us see how do we get the polymers from the monomers. The polymerization reaction can be visualised as follows. First of all one molecule each of the monomers react to produce a dimer (dimer is a molecule made from 2 monomers). A carboxylic acid group condenses with an amine by eliminating water molecule to produce an amide.

The resulting dimer has got two functional groups one carboxylic acid and the other amino group. This dimer can further combine with diamine and diacid on the acidic and amino functional group respectively or with another dimer molecule. This process continues to generate the polymer.

The proteins in our body also contain such linkages formed by condensation of amino and carboxylic acid groups of different amino acids.



(Nylong 66)

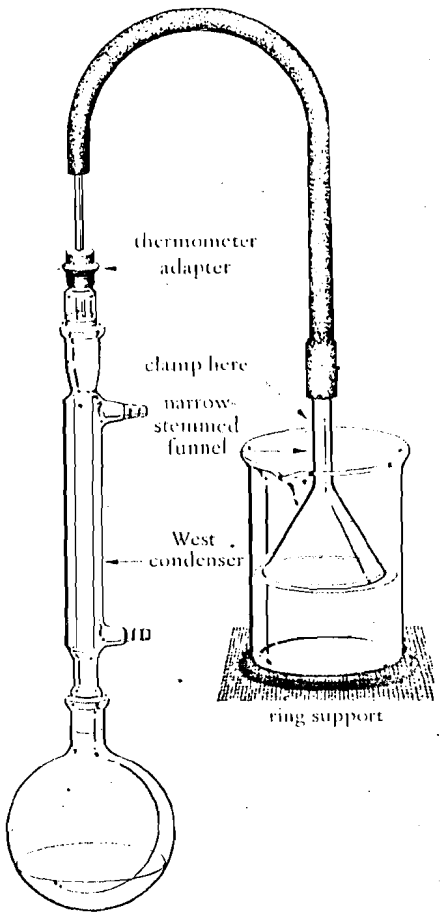
The condensation of monomers to form the polymer can be done in a number of ways. You would be using the method of interfacial condensation. In this technique the polymerization is made to take place at the interface of an organic and an aqueous medium. This technique is good for the reactants with quite reactive functional groups, which is true in our case.

The diamine is dissolved in water and the diacid chloride is dissolved in an organic solvent like chloroform or carbon tetrachloride. The aqueous layer is carefully poured on the top of organic layer. Diamine being soluble in both the solvents diffuses from aqueous to organic layer and react with the reactive diacid chloride at interface and produces the insoluble polymer. This polymeric layer checks further diffusion of the diamine and the reaction stops. The by-product, HCl,formed during condensation diffuses back into the aqueous phase and gets neutralized by the base. The polymer can be removed with the help of a forcep or a glass rod and the process continues. The polymer is removed continuously till the reactant are exhausted.

At industrial scale production the polymer is not pulled out, as stated above , but the two solution are thoroughly agitated so as to form an emulsion. This leads to large increase in interfacial surface area. As a consequence the rate of polymerization increases and a good amount of polymer is obtained in short time.

20.4 REQUIREMENTS :

apparatus		chemicals
Round bottom flask 50 cm^3	1	Adipic acid
Air condenser	1	Hexamethylene diamine
Beaker 100 cm^3	2	Thionyl chloride
Beaker 400 cm^3	1	Dimethyl formamide
		Carbon tetrachloride
		Sodium hydroxide
		Alcohol



20.5 PROCEDURE :

Preparation of adipoyl chloride :

1. Set up the apparatus as shown in the figure. For this take a 50 cm^3 round bottom flask and fit an air condenser on to it. Take a glass fannel and inset its sleen into a piece of rubber tubing. In sent a glass tube in the other end of the rubber tube and fit it on the air condenser as shown in the Fig. Given in martgin. Put the inverted funnel into a beaker containg water or NaoH solution.

2. Take 1g of adipic acid in 3-4 drops of dimethyl formamide in the flask and add 1 cm^3 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 30cm^3 of CCl_4 to it. Mix thoroughly to dissolve the product obtained.

5. Transfer this solution to a 100 cm^3 beaker. Wash the flask throughly with additional 20 cm^3 of CCl_4 and transfer the washings to the beaker.

6. In a separate 100 cm^3 beaker take 1.1 g of hexamethylene diamine and 0.75 g (6-7 pallets) of NaOH in 25 cm^3 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 a copper wire into the solution and pull out the polymer formed. You may even use a forceup to do so.

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

CAUTION ! Avoid handling the polymer with hands at this stage because the reactants specially adipoyl chloride and the solvent (CCl_4) both are harmful.

Do not pour the unreacted polymerization mixture in the sink. Stir it with a glass rod till there is no more polymerization. You may recover this crop of the polymer also and discard the rest of the solvent as usual.

10. Wash the polymer thoroughly with water. For this place the test tube, with polymer wrapped around, under the running tap water for about 5 minutes. Alternatively you may first wash the polymer with 50 % aqueous alcohol (alcohol:H_2O ::1:1) followed by tap water.

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

12. Submit a sample of the polymer so prepared to your counsellor.

Put 1-2 crystals of azobenzene, if available, to this solution and mix to dissolve. This will enhance the visibility of the interface of two liquids.

OR

You may add a drop of phenolphthalein to the aqueous layer containing hexamethylenediamine.

You should add the aqueous solution slowly along the walls of the beaker. Taking care, not to allow mixing of the solvents.

20.6 RESULT

..... g of nylon 66 was obtained from ..... g of adipic acid.