Making nylon – the ‘nylon rope trick’

A solution of decanedioyl dichloride in cyclohexane is floated on an aqueous solution of 1,6-diaminohexane. Nylon forms at the interface and can be pulled out as fast as it is produced forming a long thread – the ‘nylon rope’.

Apparatus and chemicals

- Eye protection
- One 25 cm^3 beaker.
- A pair of tweezers.
- Retort stand with boss and clamp.

The chemical quantities given are for one demonstration.

- 2.2 g of 1,6-diaminohexane (hexamethylene diamine, hexane-1,6-diamine, $H_2N(CH_2)_6(NH_2)$ (**Corrosive, Harmful if swallowed or in contact with skin, Respiratory irritant**)
- 1.5 g of decanedioyl dichloride (sebacoyl chloride, $ClOC(CH_2)_8COCl$) (**Corrosive, Harmful if swallowed**)
- 50 cm^3 of cyclohexane (**Highly Flammable andSkin/respiratory irritant**)
- 50 cm^3 of deionised water

Technical notes

2.2 g of hexane-1,6-diamine (hexamethylene diamine,1,6-diaminohexane, $H_2N(CH_2)_6(NH_2)$ (**Corrosive, Harmful if swallowed or in contact with skin, Respiratory irritant**), Refer to SSERC or CLEAPSS Recipe and Hazcards.1.5 g of decanedioyl dichloride (sebacoyl chloride, $ClOC(CH_2)_8COCl$) (Corrosive **Harmful if swallowed**), Refer to SSERC or CLEAPSS Recipe and Hazcards.

50 cm^3 of cyclohexane (**Highly Flammable and Skin/respiratory irritant**), Refer to SSERC or CLEAPSS Recipe and Hazcards.

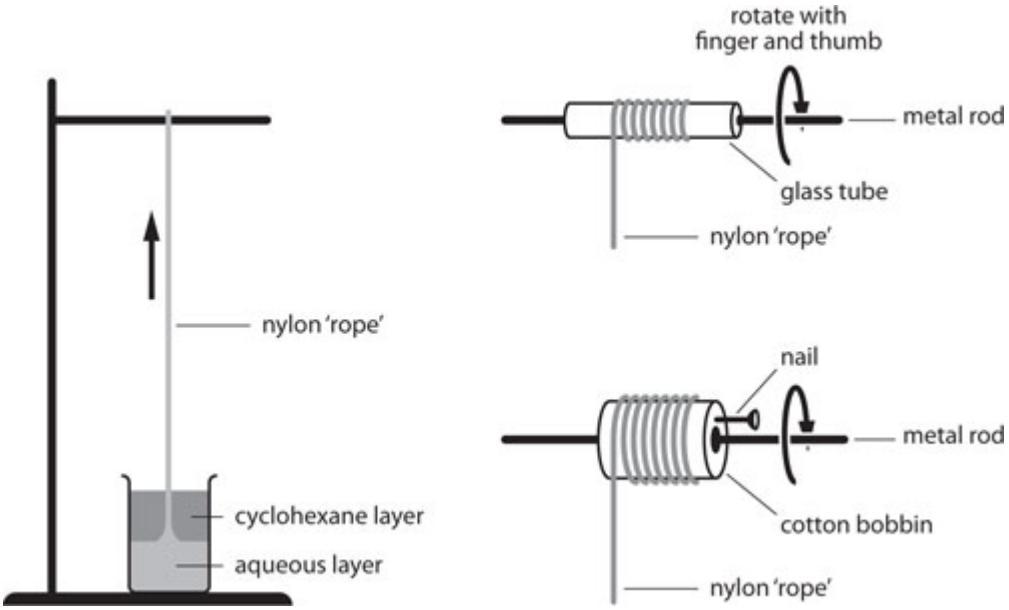
1 Wear eye protection and disposable nitrile gloves when pulling out the thread.
2 The room should be well ventilated and there must be no sources of ignition.
3 Details for waste disposal can be found on ASE[1], SSERC[2] or CLEAPSS[3] websites.
4 This demonstration has been described in many sources using chlorinated solvents for the acid chloride. These are no longer considered safe and will soon become unavailable. Cyclohexane is less dense than water whereas chlorinated solvents are denser. The layers are therefore inverted compared with the old method.
5 Cyclohexane is preferred to hexane as it is less harmful.
6 Hexanedioyl dichloride (adipoyl chloride) can be used as an alternative to decanedioyl dichloride, but it does not keep as well.
7 Decanedioyl dichloride reacts with moisture in the air to produce decanedioic acid which forms nylon much less readily than the acid chloride. Ensure that the bottle is restoppered carefully after opening and consider storing it in a desiccator. The dichloride is also available in 5 cm^3 sealed ampoules. The cyclohexane solution will still make nylon for a couple of days after being made up

[1] https://www.ase.org.uk/home/
[2] http://www.sserc.org.uk/
[3] http://www.cleapss.org.uk/

even if left unstoppered. A solution kept in a stoppered bottle is still usable after two weeks. The solution can be stored over anhydrous sodium sulphate or calcium chloride to keep it dry.
8 Solid 1,6-diaminohexane can be difficult to get out of the bottle. The easiest way to manipulate it is to heat the bottle gently in warm water until it melts at 42 °C and dispense the liquid using a dropping pipette.

Procedure

Health & Safety: Work in a well-ventilated laboratory. Wear eye protection and disposable nitrile gloves when pulling out the thread.



Before the demonstration

a Make up a solution of 2.2 g of 1,6-diaminohexane (**Corrosive, Harmful if swallowed or in contact with skin, Respiratory irritant**) in 50 cm^3 of deionised water. This solution is approximately 0.4 mol dm^{-3}.
b Make up a solution of 1.5 g of decanedioyl dichloride (**Corrosive Harmful if swallowed**) in 50 cm^3 of cyclohexane (**Highly Flammable and Skin/respiratory irritant**). This solution is approximately 0.15 mol dm^{-3}.

The demonstration

Pour 5 cm^3 of the aqueous diamine solution into a 25 cm^3 beaker. Carefully pour 5 cm^3 of the cyclohexane solution of the acid chloride on top of the first solution so that mixing is minimised. Do this by pouring the second solution down the wall of the beaker or pour it down a glass rod. The cyclohexane will float on top of the water without mixing. Place the beaker below a stand and clamp as shown (see figure).

A greyish film of nylon will form at the interface. Pick up a little of this with a pair of tweezers and lift it slowly and gently from the beaker. It should draw up behind it a thread of nylon. Pull this over the rod of the clamp so that this acts as a pulley.

Continue pulling the nylon thread at a rate of about half a metre per second. It should be possible to pull out several metres. Take care, the thread will be coated with unreacted monomer and may in fact be a narrow, hollow tube filled with monomer solution. Wearing disposable gloves is essential.

Visual tips

The beaker is rather small so allow the audience as close as possible consistent with comfort and safety.

Disposal

Dispose of the mixture as follows: First shake the reaction to mix the two layers. A lump of nylon will be produced which can be removed with tweezers, rinsed well with water, and disposed as solid waste. The remaining liquids can be mixed with detergent and washed down the sink. Failure to do this may result in the polymerisation taking place in the sink, leading to a blockage. The organic liquid can be disposed of in a hydrocarbon waste bottle stored in a flammable cabinet.

Teaching notes

Point out that this demonstration is different from the industrial method of making nylon which takes place at a higher temperature. Molten nylon is then forced through multi-holed 'spinnerets' to form the fibres.

Theory

The reaction is a condensation polymerisation

$nH_2N(CH_2)_6\ NH_2 + nClOC(CH_2)_8COCl \rightarrow H_2N[(CH_2)_6NHCO(CH_2)_8]_nCOCl + nHCl$

The nylon formed is nylon 6 –10 so called because of the lengths of the carbon chains of the monomers. Nylon 6 – 6 can be made using hexanedioyl dichloride (adipoyl chloride).

The diamine is present in excess to react with the hydrogen chloride that is eliminated. An alternative procedure is to use the stoichiometric quantity of diamine dissolved in excess sodium hydroxide solution.

Extensions

There are many ways of conveniently winding the nylon thread – for example using a windlass improvised from a cotton bobbin or a short length of glass tube slid over the rod of a clamp (see Fig).

Reference

This experiment has been adapted from *Classic Chemistry Demonstrations*, Royal Society of Chemistry, London, p.159-161

Credits

Health & safety checked January 2018

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