

Amorphous Cellulose Preparation

Introduction

This protocol will prepare a relatively non-crystalline amorphous cellulose that is used throughout various other protocols listed.

Reagents

- 🔗 250 mL Milli-Q H₂O
- 🔗 25 g NaOH pellets
- 🔗 5 g dried cellulose (BC or MCC)
- 🔗 Glacial acetic acid

Equipment

- 🔗 1 L Erlenmeyer flask
- 🔗 Magnetic stir plate
- 🔗 Large magnetic stir bar
- 🔗 pH meter
- 🔗 Büchner funnel
- 🔗 Paper filter
- 🔗 Filtration flask
- 🔗 Vacuum line
- 🔗 Oven

Procedure

Cold Stir

1. Fill a graduated cylinder with 250ml Milli-Q H₂O and empty into a 1L Erlenmeyer flask.
2. Place a large stir bar in the flask and set it to a moderate speed without heat.
3. Quickly measure out 25 g of NaOH pellets and add them to the Erlenmeyer flask.
 - a. Take care not to add the pellets all at once or too slowly. If you add all of the pellets at once, you risk rapidly heating the water. If you add it too slowly, the NaOH will be exposed to H₂O in the air that will be absorbed, throwing off your weight calculations.
4. Weigh out 5 g dried cellulose (either MCC or BC) and dump into the flask while stirring.
5. Move mixture to a -5°C cold room and let stir overnight on moderate speed.

Pre-oven Preparation

6. Prepare the pH meter by calibrating using pH buffer solutions.
7. Take the previously cold-stirred solution and adjust the pH to 10 using small additions of glacial acetic acid to precipitate amorphous cellulose.
 - a. *It may be helpful to place the solution into a smaller container without the stir bar.*
 - b. *Less is more! Remember that the pH may swing wildly when adding too much acid.*

Amorphous Cellulose Preparation Cont.

8. Vacuum filter the solution, which should now look chunky, with a vacuum filtering setup consisting of a Büchner funnel, paper filter, and filtration flask connected to a vacuum line.
 - a. *We recommend using a liquid catch to prevent potential damage to the vacuum pump.*

Pre-oven Preparation

9. Scoop the now gel-like amorphous cellulose onto a Pyrex glass petri dish and place it in an oven at 105°C for 7-8 hours.