Rapid Production of a Porous Cellulose Acetate Membrane for Water Filtration using Readily Available Chemicals

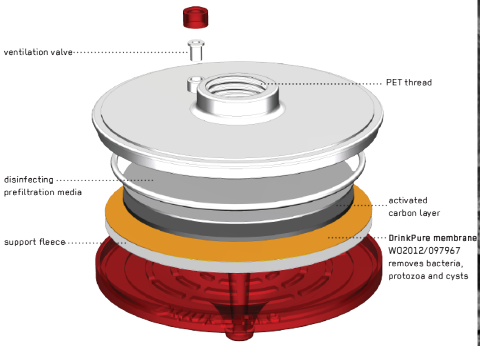
Handout for Students

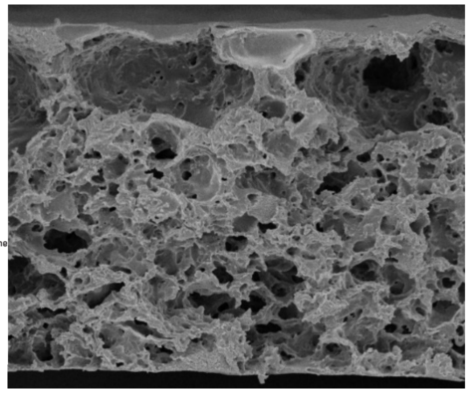
General

Access to clean drinking water is still a global problem. In developing countries around 80% of diseases are linked to poor drinking water supplies and sanitary conditions. The largest health risks of impure drinking water include pathogenic bacteria and viruses. For the removal of such microbial contamination several water treatment technologies have been developed.

The problem of most systems lies in their relative complex installation and the urge of periodical maintenance. For application in developing countries it is inevitable that the installation of the device is straightforward and is able to operate independently of a power grid. Portable solutions on the market today include ultra- and microfiltration membranes, ceramic filters, activated charcoal filters or chemical disinfection with halogens.

The commercially available *DrinkPure*™ filter was developed in collaboration with the ETH spinoff company Novamem at the Functional Materials Laboratory of ETH Zurich. The key part of this filter is a microfiltration membrane that is produced by pore formation via dissolvable calcium carbonate nanoparticles.





The industrial production of this membrane only utilizes low-cost processes and materials, but it relies heavily on sophisticated machinery. The scope of this laboratory experiment is to produce a drinking membrane that resembles the production stages of the commercially available membrane, but is accomplished by using daily commodities. In a second step, the effectiveness of the filter is tested with watercolor dispersions and food color solutions. In addition to the microfiltration membrane (which filters bacteria and solid contaminants) the *DrinkPure*™ filter product also includes an activated charcoal filter, which mostly filters dissolved impurities out of the feed water.

Safety

Put on safety goggles and a lab coat. Wear gloves when contact with liquids (acids and dispersions) is possible or necessary. Handle all employed chemicals with care. In case of skin contact wash immediately with water and soap and rinse for at least 15 minutes. In case of eye contact rinse with water for at least 15 minutes.

Equipment and Chemicals

For the production of approximately 25 membranes in ½ page size each one needs:

Chemicals:

- 20 g Cellulose acetate (Sigma-Aldrich No. 180955)
- 41 g Calcium carbonate (Sigma-Aldrich No. 21069)
- 17.6 g Glycerol
- 5 liters 0.24 M HCl (prepared by Instructor from concentrated HCl solution)
- 200 g Acetone
- Ethanol
- 10 L water (demineralized)
- Watercolor (e.g. Artists Loft Fundamentals Watercolor Pan Set from amazon.com)
- Food coloring (e.g Brilliant Blue FCF E133)

Equipment:

- Two 250 ml Schott flasks
- Kitchen blender with at least 800 W power (e.g Philips HR 2195/04)
- 50 small beakers
- Mirror or glass plate (148 x 210 mm or larger area)
- Scotch tape
- Ruler with stainless steel cutting edge or aluminum profile
- Two plastic basins for diluted hydrochloric acid and water bath
- Spatula
- Pair of scissors
- Filter flask
- Rubber ring
- Büchner funnel
- Büchner funnel filter
- Permanent marker
- Magnet stirrer
- Magnet stir bar
- Water and ethanol safety wash bottles
- Safety goggles

- Nitrile safety gloves

Preparation:

Preparation of Cellulose Acetate Polymer Solution

1. In a Schott flask mix 200 g acetone with 20 g of cellulose acetate.
2. Close the flask, shake it shortly and add a magnetic stir bar.
3. Mix the polymer solution by means of a magnetic stirrer until the solution becomes colorless (duration: approximately 1 hour)
4. The polymer solution can be stored over a long time period, but needs to be well protected from heat and evaporation.

Practical part

Equipment (for groups of two)

Two 250 ml Schott flasks, kitchen blender with at least 800 W power (e.g Philips HR 2195/04), 50 small beakers, mirror or glass plate (148 x 210 mm or larger area), scotch tape, ruler with stainless steel cutting edge or aluminum profile, two plastic basins for diluted hydrochloric acid and water bath, spatula, pair of scissors, suction flask, rubber ring, Büchner funnel, Büchner funnel filter, permanent marker, magnetic stirrer, magnet, water and ethanol safety wash bottles, safety goggles and nitrile safety gloves.

Chemicals

Cellulose acetate polymer solution (20 g cellulose acetate dissolved in 200 g acetone), calcium carbonate (Sigma-Aldrich No. 21069), glycerol, hydrochloric acid bath, water bath.

Procedure for the Production of the Membrane

Preparation of Cellulose Acetate Dispersion for the whole class (performed by two students)

1. Transfer the whole cellulose acetate polymer solution into the glass of a kitchen blender. Add 41 g of calcium carbonate and 17.6 g of glycerol.
2. Mix the suspension for three minutes at the highest setting of the kitchen blender. Transfer the whole mixture into a fresh Schott flask and close the flask to avoid solvent evaporation.
3. Fill the kitchen blender glass immediately with water to precipitate the remaining dispersion. The precipitated polymer waste can be disposed into the household waste.

Production of membrane sheet (in groups of two)

1. Four layers of scotch tape are applied to two parallel sides of a glass or mirror plate. Take care that no air bubbles are trapped within the tape layers.
2. Rinse the surface of the mirror or glass plate with a paper towel and some ethanol.
3. Transfer approximately 10 g of the Cellulose-acetate polymer dispersion) from the Schott flask (stock for whole class) into a small beaker glass for direct usage.

4. Subsequently poor this amount of dispersion onto the mirror as a 1 inch wide stripe. Distribute the stripe of dispersion from the top to the bottom of the glass plate using the edge of a stainless steel ruler.
5. Dry the final membrane sheet at ambient air for a total of five minutes.

Removal of Calcium Carbonate Particles

1. Fill one plastic basin with 5 L water and one basin with 5 L of 0.24 M HCl (prepared by your instructor) in a fume hood. The mirror or glass plate should fit into the plastic basin. Clearly label the basins.
2. Put on nitrile gloves and submerge the mirror or glass plate with the membrane on top cautiously into the diluted hydrochloric acid bath. The membrane should automatically loosen itself from the surface. If not, put a small spatula underneath the membrane and manually loosen the membrane. Keep the membrane in the diluted hydrochloric acid bath for approximately ten minutes. Write down your observations:

   Observation:

   Interpretation (write down the chemical reaction formula):

3. Cautiously transfer the membrane into the second plastic basin containing only water. Leave the membrane in the water bath for an additional five minutes. The easiest way to transfer the membrane is by holding it with two hands at the upper edge. Take care that the membrane does not tear!
4. Transfer the membrane out of the water bath and place it on top of a paper towel. Use a second paper towel to dry the membrane.

Functional Testing of the Membrane

1. Produce a colorful mixture using ca. 100 mg of solid watercolor and a minimal amount of 20 ml water.
2. Put the Büchner funnel filter onto the membrane and transfer the shape of the filter with a permanent marker. Cut out the round piece of membrane with a scissor.
   Set up the filtration apparatus (suction flask, Büchner funnel and rubber ring) and connect it to a vacuum hose. Carefully place the membrane on top of the Büchner funnel. Filter half of the watercolor mixture by applying vacuum.

   Observation:

3. Prepare a 20 ml mixture of food coloring (e.g Brilliant Blue FCF E133), using 1 mg of the solid color.
   Filter around 10 ml of the food coloring solution through the same membrane.

   Observation:

4. Remove the membrane and clean the Büchner funnel and suction flask.
5. Mount a standard Büchner funnel filter (standard from laboratory supplies) on the Büchner funnel and repeat the experiment with the remaining 10 ml of watercolor and food coloring solutions.

   Observation:

   From your observations, what conclusions can you make regarding the chemical nature of the watercolor?

From your observations, what conclusions can you make regarding the chemical nature of the food coloring solution?

What can you say about the pore size of the commercial filter paper?

Can you think of three drinking water contaminants that will behave like the watercolor and be removed by an advanced membrane filter with pore sizes in the 1 μm range (similar to the filter you made)?

Can you think of three drinking water contaminants that will behave like the food coloring and pass through the filter?

What additional methods/devices could be used so that these contaminants can also be removed from the drinking water?