

Nanocellulose



Center for Nanotechnology Education

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Based on a work at www.nano-link.org.

Nanocellulose

Abstract

This module presents the nanotechnologies, processes, and methods being developed to produce strong and stiff cellulose nanofibers. The module is designed to take the student through processing a material (called feed stock or substrate) to break down and extract cellulose fibrils. A pulp is created from recycled newspaper, a special enzyme is added, and then the substance is processed further by spinning, screening, and curing, to produce a cellulose nanofiber paper with varying fiber orientations and changes in properties such as rigidity and stiffness. Hands-on activities include processing and creating cellulose nanofiber paper and observing samples for changes in rigidity and /stiffness.

Outcomes

Students will produce observable enhancements and changes in material properties using simulated nanotechnologies and processing that effect changes at the nanoscale.

Prerequisites

- Basic background in materials, material properties, and chemical reactions (such as hydrolysis) is required.
- This module builds on Material Science topics/content already in use at the MatEdU National Resource Center for Materials Technology Education. It is suggested to review the related MatEdU modules prior to teaching this module. The following related modules may be downloaded from <http://materialseducation.org>: Properties of Fibers and Fabrics.

Science Concepts

- Cellulose is a naturally occurring polymer.
- Cellulose fibers make up many common items, such as paper and fabric.
- Cellulose can be altered physically and chemically to enhance its properties.

Nanoscience Concepts

- Forces that act at the nanometer scale lead to observable macroscopic phenomena.
- Changes in the surface area to volume ratio will change the physical properties of a material or system.

Background Information

Nanocellulose. Cellulose is among the most abundant naturally occurring and renewable resources on Earth. It is a biological polymer consisting of long chains of glucose, and is found in the cell walls of plants and bacteria. Cellulose is responsible for the structure of all plants. It is not surprising that cellulose has played a major role in our lives; it can be found in our food and products we use every day. It's the main component in textiles such as fabric and paper.

Why cellulose nanotechnology? Ever since humans began dyeing fibers (China, 2600 B.C.), we've applied nanotechnology to make practical goods such as textiles. People didn't know it back then, but they were practicing the science of surface manipulation of a fiber to impart a particular color. Today, with more advanced tools and understanding, we are using nanotechnology to manipulate fabric structures at the nanoscale, and processing fibrous materials to create new nanocomposites. One of those materials of interest is cellulose fiber. Cellulose nanotechnology is focusing on a couple of categories for research (*note: the bolded item is demonstrated in the module activities*):

- Fabrication of nanofibers: the primary industrial use of cellulose has been the manufacture of paper and fibers for fabric. Commercial research is currently focused on **isolating and characterizing cellulose in the form of nanofibers found in feedstock materials such as wood pulp or recycled paper.**
- Nanoparticle composite fibers: Cellulose fibers are being used as fillers or substitutes for material components such as aggregates in concrete. Materials scientists are also looking at using lignin, a protein found within cellulose, as a constituent in concrete.

What is nanocellulose? Nanocellulose consists of cellulose fibers extracted from the cell structure of plants. Cellulose fibers created from a bulk material, usually wood or paper pulp, can be disintegrated into individual nanoscale fibrils or fibril bundles (cellulose nanofibrils, or CNF), the main constituent of cellulose fibers. The dimensions of CNF can be from 5 to 20 nm across, and range from 10 nm to several micrometers in length; this is about 1,000 times smaller than conventional cellulose fibers. When cellulose nanofibers are liberated, they possess a high surface area to volume ratio; this can produce a particle that can bind more readily due to a greater number of hydrogen bonds. Further, fibrils can self-assemble creating alignments and orientations that can change the properties of the material.

Material scientists hope to be able to use cellulose nanofibers as the building blocks for future high-performance biomaterials and textiles, and to create new lightweight materials with high mechanical strength. Soon we may see cars, houses, and even bridges made from cellulose-based products.

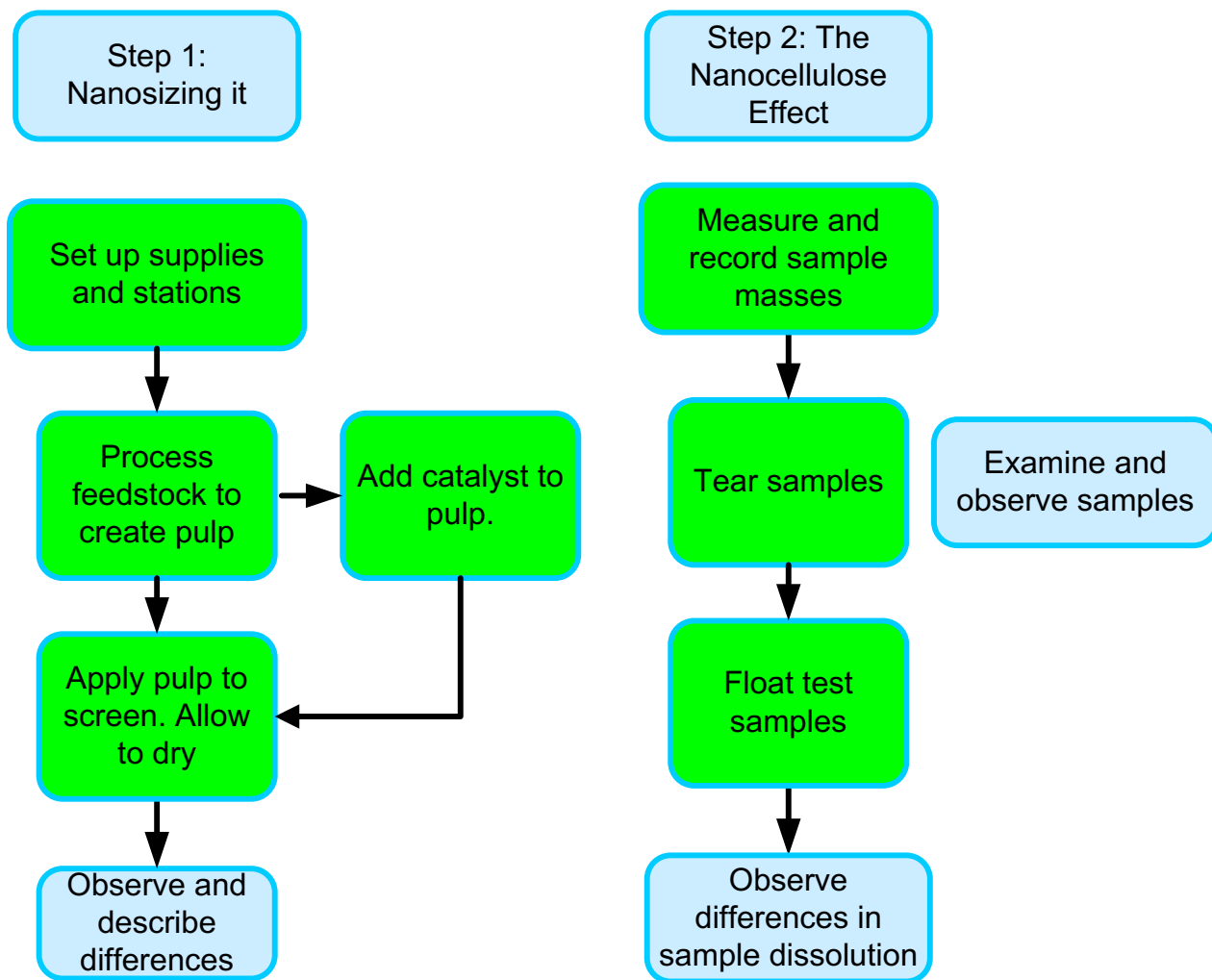
Current and Future Applications

Lightweight, stiff, durable materials are in high demand. The nanotechnology and processes studied in this module offer promising routes to such materials. Areas for nanofiber exploration include:

- *Fabrication of nanofibers:* Commercial/Industrial cellulose use has been primarily in the manufacture of paper and fabric. Research is currently focused on isolating and characterizing cellulose in the form of nanofibers found in the bulk materials such as wood pulp or recycled paper used as feedstocks in papermaking.
- *Surface modification of fibers and fabrics:* The hydrophobic (water repelling) and hydrophilic (water attracting) properties of cellulose are being modified to create materials that can resist water and oil. In other cases, surface modifications can prevent combustion and even impart antibacterial properties.
- *Nanoparticle composite fibers:* Cellulose fibers are being used as fillers or substitutes for material components such as aggregates in concrete. Materials scientists are also looking at using lignin, found with cellulose, as a constituent in concrete.

Learning Activity: Nanocellulose

Activity Flow Chart



Nanocellulose

Step 1: Nanosizing It

Supplies

Per person/team:

- Newspaper
- 3 – 5 gal bucket
- Screened frame, 6" x 6" minimum
- Unframed screen (same size as screen frame)
- Pan, must be larger than the screened frame and deep enough to submerge the screen
- Measuring spoon (tablespoon size)
- Fibrillation medium/agent (enzyme): Alpha amylase
 - Available at www.milehighdistilling.com, about \$15.00/pound
- Water (use water from soaking, must be about 75°C)
- Blender
- Digital scale, tabletop type such as a kitchen/food scale
- Sponge
- 2 test tubes
- Pipette
- Paper towels or other absorbent material to lay sample on to dry
- Latex gloves and safety glasses, aprons optional

Additional supplies if monitoring pH or temperature:

- pH meter, or
- pH test strips, available from pool supply stores. (Lignin-degrading enzymes generally function best under slightly acidic or neutral pH, but the most common pulping reactions and recycled fiber processes are alkaline.)
- Thermometer - the type used in cooking (ideal temperature is room temp).

Procedure

1. Taking a handful of soaked newspaper, squeeze it to release excess water.
2. Place one unit of the wet newspaper feedstock on a scale.
3. Place the feedstock in blender.
4. Add water, using a 1:9 volume mix ratio (feedstock to water). You need to make enough pulp to fill the frame. For example, a 9x9 inch screen requires 2 fluid oz. (57ml) of feedstock mixed with 18 fluid oz. (475 ml) of water.
5. Turn on blender to high, and grind/crush/pulverize until pulp is fluid enough to be able to see fibers and it holds no clumps.
6. Place screen frame in a large flat pan of water; submerge briefly.
7. With screen frame floating in the water and while moving the screen gently in a circular motion, pour paper pulp to fill the screen frame area evenly.

8. Remove the entire frame assembly from the water and place on paper towels or flat newspaper.
9. Place a piece of unframed screen over the paper pulp.
10. Gently and evenly press the sponge to remove water. Squeeze excess water out of the sponge, and repeat several times. As the paper dries it will begin to adhere to the unframed screen; this is a sign that it is drying properly.
11. Continue drying. Leave the paper in the frame and set in a safe place to dry. NOTE: You may be able to lift the paper out of the frame at this point, if it holds its shape without tearing. If so, set the paper in a safe place to dry.
12. For the enzymatic catalyst steps, repeat the entire procedure but after step 5, add a fibrillation medium (catalyst) in a mix ratio (feed stock:water:fibrillation agent) of 1:9:1. Mix gently, just enough to disperse the particles, then continue with steps 6-11. Repeat with the following mix ratios to get varying degrees of material properties: 1:9:2, 1:9:4, 1:9:6.

NOTE: Units used in this module to describe mixing ratios are dry oz: liquid g: dry. That is, a 1:9:1 mix ratio uses 1oz (28g) feed stock, 9 oz (30ml) water, and 1 oz (28g) of catalyst (enzyme).

Safety

There are no known dangers with the pulp mixture as described. However, take any necessary precautions and supervision is recommended. Read all labels on any of the fibrillation agents or other things added to the pulp.

Step 2: The Nanocellulose Effect

Supplies

- Dried samples from Step 1
- Test tubes
- Water
- Worksheet “Classifying Nanomaterials” (can be printed from the slideshow accompanying this activity)
- Digital scale, tabletop type such as a kitchen/food scale
- Latex gloves and safety glasses, apron optional

Procedure

1. Measure and record the mass of the dried samples before testing.
2. Examine each sample, describe and record observations.
3. Tear the samples, observe, and discuss observations.
4. Float test the samples:
 - a. Cut equal sections from each sample and place in a test tube, add equal amounts of water and swirl the tube.

- b. Observe: the sample with the enzyme catalyst mix should dissolve (they've become more permeable) while the sample with no enzyme should hold its "sponge" shape.

Discussion Questions

This is meant to be a measure of understanding of the material presented; it could be written or oral.

1. Describe the general nature of cellulose.

Answer: A biological polymer that consists of long chains of glucose and is found in the cell walls of plants and bacteria.

2. Describe the nanotechnologies that are benefiting paper/cellulose materials science.

Answers: Grinding to reduce size, disintegration to separate constituents, emulsification to homogenize (and decrease viscosity).

3. What are the nanomechanics that explain how fibrils are liberated in cellulose.

Answer: Enzymatic Hydrolysis is a degradation of a compound into its constituent parts. In this case: lignin and glucose molecules.

4. Explain the nanoprocess, fibrillation and its effect on fibrils.

Answer: Fibrillation is a process used to disintegrate a material into its constituent parts. Through the fibrillation process, the nature of the cellulose is influenced by enzymatic degradation: the nanofibrils become more visible (they are liberated) and, like the process of papermaking, the fibers also align themselves (self-assemble) and appear to give the paper a "grain."

5. Explain the nature of nanoscale materials and how they differ from other materials.

Answer: Size; nanoscale is 1-billionth of a meter 10^{-9} , and surface area available for reaction.

6. Describe the effects of nanoprocessing on the microstructure of materials.

Answer: Makes the material smaller, there's more surface area to volume, enhance bonding.

7. How can the nanoprocesses used affect material properties?

Answer: Make the material more stiff/brittle. Liberate fibers.

Contributors

- This module was developed and written by Kim Grady, M.Ed., BehaveHeuristics LLC, Phoenix AZ, with assistance from
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Resources

Articles

This module builds on Material Science topics/content already in use at the MatEdU National Resource Center for Materials Technology Education. Depending on the audience, it could benefit them to do and/or review the information in: The following related modules may be downloaded from <http://materialseducation.org>: Properties of Fibers and Fabrics

- HQ Papermaker: www.hqpapermaker.com/paper-history/
- Nanocellulose: www.azonano.com/article.aspx?ArticleID=3139
- Using nanocellulose to create novel composite material: www.empa.ch/plugin/template/empa/3/113491/---/l=2
- Hydrodynamic alignment and assembly of nanofibrils resulting in strong cellulose filaments: www.nature.com/ncomms/2014/140602/ncomms5018/full/ncomms5018.html
- Cellulose nanocrystals possible 'green' wonder material www.purdue.edu/newsroom/releases/2013/Q4/cellulose-nanocrystals-possible-green-wonder-material.html