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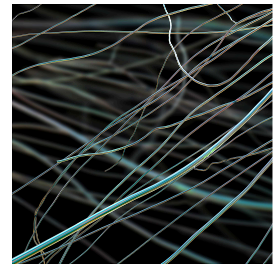
Synthesis of Carbon Nanofibers by Electrospinning



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We use this protocol and it's working

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Disclaimer

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Abstract

This protocol describes the synthesis of aligned carbon nano fiber for adsorbent/photocatalyst in environmental samples, electrochemical energy storage and other industrial applications. The steps for the process are based on Zeng et al [1] summarized in **Figure 1**.

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Materials

Hardware

- Magnetic stirrer
- Laboratory furnace
- Spinbox Electrospinning Instrument with high voltage power supply: 0-30KV (Bionicia Inc., Valencia, Spain) (**link**)

Software

- Spinbox software (Bionicia)

Chemicals, Reagents and Other Materials

- Poly acrylonitrile (PAN) 150,000 Molecular Weight
- N-N Dimethylformamide (DMF)
- Glass ware (adjust based on desired volume)
- Aluminum foil
- 10ml luer lock BD PP/PE syringe
- 20-gauge needle
- Yellow ferrules (Flangeless Ferrule Tefzel™, 1/4-28 Flat-Bottom, for 1/8" OD Yellow)
- Green ferrules (Flangeless Male Nut Delrin®, 1/4-28 Flat-Bottom, for 1/8" OD Green)
- Threaded luer adapter (Luer Adapter 1/4-28 Female to Female Luer, PEEK)
- Tubing, Crack-Resistant Polyethylene Food and Beverage grade (Semi-Clear, 1/16" ID, 1/8" OD)
- Graphite plate 4″ X 4″ size

Troubleshooting

Safety warnings

Eye protection

- DMF can cause serious eye irritation. Wear eye protection at all time when using this chemical (**link to safety data sheet**)

Skin

- DMF is harmful in contact with skin. Care should be taken when working with this chemical. Proper use of PPE should avoid any problems. If any solutions are spilled and encounter skin, immediately rinse under water and wash with soap for at least 5 min (**link to safety data sheet**)

Fumes/aerosols

- DMF may cause respiratory irritation if inhaled. Care should be taken when working with this chemical (**link**
  to safety data sheet)

Heat and Flammable materials

- DMF is a flammable liquid and vapor. Avoid open flame or source of ignition when working with DMF.

Disposal

- Vials of unused polymers and solvent should be discarded in the waste container and not disposed of in the sink.

Before start

- Ensure ample, clean benchtop space is available for reagent preparation
- Ensure access to well ventilated area (e.g., certified hood)
- Use of Safety shut-off switches equipment is encouraged to ensure safety during high voltage operation.
- More safety information on use of electrospinning equipment (**More safety information**)

Note

Critical step:

- Eyewear protection is mandatory
- Butyl or nitrile latex gloves mandatory
- Lab coat is mandatory
- Closed-toed shoes are mandatory

Solution preparation

1d

1 *Step 1)* **Prepare 10% PAN working solutions** (Timing: 24 hours)

1d

- Weigh 3g of polyacrylonitrile and carefully combine in a flask with 27g of dimethyl formamide
  (DMF) under a chemical fume hood.
- Place on magnetic stirrer within chemical hood (**Figure 1**)
- Stir the mixture at 300 to 400rpm for 24 hours.
- Inspect to ensure the solution is completely homogenized (no visible particulate matter) in form of a clear viscous solution.
- The solution may be stored for at least one month when stored at 4C. Care should be taken when storing the solution at room temperature [3]

Figure 1. Preparation of 10% PAN solution under chemical hood

Instrument preparation

25m

2 *Step 2)* **Instrument Set up and preparation** (Timing: 20 minutes)

20m

- Insert a green ferrule followed by the yellow ferrule to each end of the Polyethylene tube (the length of the tube is determined by the distance of the pump to the mounting stage)
- If the PAN solution is not used immediately, stir the already dissolved polymer solution for an additional 10-15 minutes at 400 rpm before use and inspect to ensure a homogeneous solution.
- Decant the polymer solution slowly into a 10ml BD syringe.
- Use a gloved finger to hold the tip or cap on the syringe to prevent spilling.
- Alternatively, slowly use the syringe to draw the polymer solution into the syringe.

Note

Note:
- Depending on the spinning rate and spinning duration, the volume of polymer suspension may be modified.

- Turn the syringe upside down and agitate slowly so that any bubbles rise to the top of the solution.
- Push the plunger slowly to remove any air bubbles made during the process.
- Use a Kimwipe or adsorbent cloth to clean any small droplets of polymer solution from the syringe.
- Attach the syringe to the tube and connect to the spinning head.
- Insert electrospinning needle into the spinning head.
- Push the plunger slowly to transport solution into the tubing.

3 *Step 3)* **Load the syringe into electrospinning instrument** (Timing: 5 minutes) 5m
- Load the spinning head into the feeding stand of the machine (**Figure 2**).
- Load the syringe in the feeding pump.
- Lock the screw to hold it slightly tight.

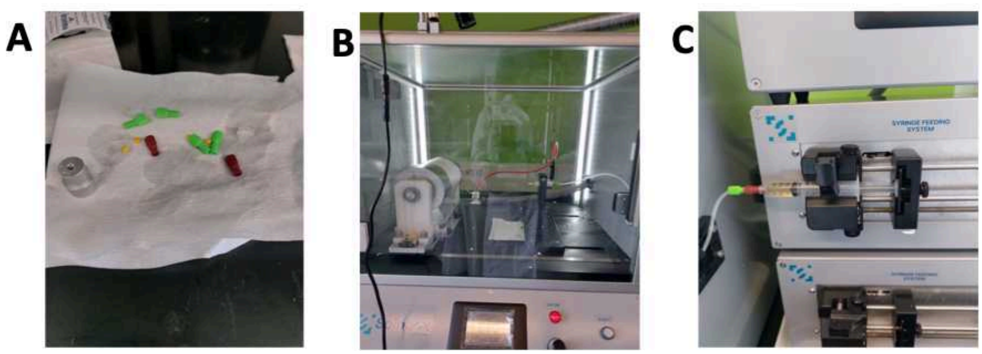

Figure 2. Electrospinning set up. **A)** Electrospinning accessories **B)** Spinning set up containing spinning needle and collector **C)** Feeding pump

4 *Step 4)* **Connect electrical system** (Timing: 2 minutes) 2m
- Connect the voltage line of the machine to the feeding stand on the electrospinning instrument.
- Set the voltage line on the feeding stand
- Lock the screw.

5 *Step 5)* **Load the collector** (Timing: 5 minutes) 5m
- Load the fiber collector with clean aluminum foil.
- Use tape to secure it the foil on the fiber collector

Process and Collect Fiber

10h

6 *Step 6)*

Electrospinning nanofiber (Timing: 5 hours)

5h

- Turn on the computer for the electrospinning instrument
- Open the spinning software and set up the parameters accordingly:

Note

1. syringe diameter = 10ml BD syringe
2. feeding rate = 1ml/hour

- Switch on the rotating drum collector and adjust the rotating speed appropriately:

Note

1. 2000rpm for aligned fibers
2. 400rpm for random fibers

- Set the spinning voltage by adjusting the knob to the required voltage (Voltages between 12KV-18KV are appropriate for this protocol)
- Start the process and run the instrument for 5 hours (or more depending on the parameter setting)

Note

Critical step:

- After the process is complete, turn off the power source, rotating drum and feeding pump.

7 *Step 7)* **Collect electro-spun nanofibers** (Timing: 5 hours)

5h

- After turning off the power source, drum and feeding pump, remove the spun fibers from the aluminum foil
- Store fibers in fume hood
- Clean the ferrule and other accessories with (DMF). Capture any excess DMF in a glass beaker or other appropriate waste container
- Dispose of the waste DMF/PAN in the chemical disposal area

Stabilize and Carbonize Fibers

9h

8 *Step 8)* **Stabilize electro-spun fibers** *(Timing: 8 hours)* 8h

- Cut the spun fiber into desired sizes of available graphite plate
- Place the cut fibers on graphite plate
- Place graphite plate and fibers into furnace
- Bake the electro spun fibers in air at 280 °C for 6 hours at a heating rate of 1°C per minute.
- Inspect the material after stabilization (baking).
- A brown colored fiber should be achieved after stabilization (**Figure 3**).

Note

Note:

- If preferred, a cell phone app may be used to detect the color of the sample. For example, Color Name AR is a useful tool that is available for both iPhone and Android (as well as tablets) (https://apps.apple.com/us/app/color-name-ar/id906955675)

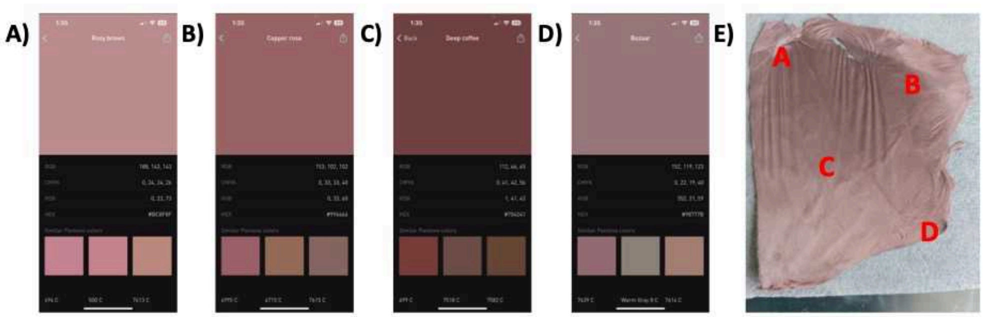

Figure 3. Color analysis of stabilized PAN fibers. Naked eye analysis (panel E) may be used, or camera applications (Color Name AR app for iPhone shown here). Various shades of "brown" are shown in panel A to D denote specific locations on the PAN sample shown in D. See note below for link to mobile phone app colorimeter.

9 *Step 9)* **Carbonize fibers** *(Timing: 1 hour)* 1h

- While the carbon nano fiber is still in the oven, carbonize in nitrogen at 1200 °C for 1 hour at a heating rate of 5 °C per minute.

Note

Note:
- The oven requires few hours to heat up to 1200 temperature an d few hours to cool down after the process.

- Remove carbonized material from the oven.
- A black colored material should be achieved after the carbonization step (**Figure 4**).

Note

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A black colored material should be achieved after the carbonization step (**Figure 4**).

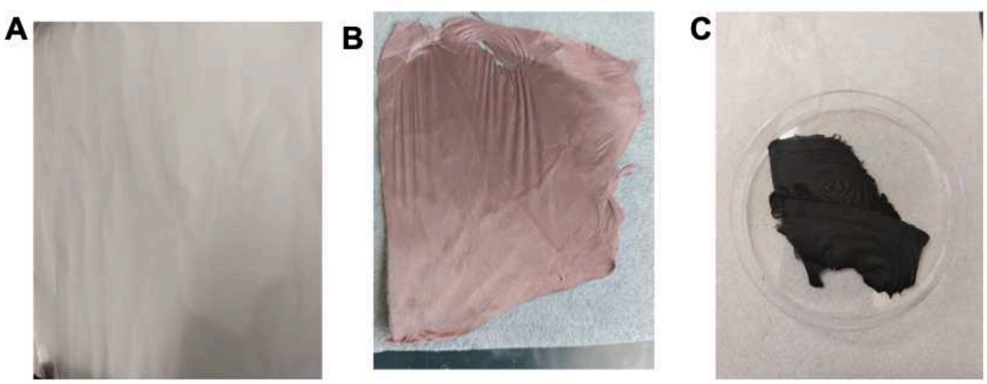

Figure 4 A) Carbon nano fiber before stabilization **B)** Stabilized nano fiber **C)** Carbonized nano
fiber.

Clean up

10m

10 *Step 10) Clean up laboratory (**Timing**: 10 min)*
- Clean workstation/benchtop
- Ensure electrospinning instrument is turned off
- Ensure furnace is turned off
- Vials of unused polymers and solvent should be discarded in the waste container and not disposed of in the sink.
- All waste DMF/PAN should be discarded in the satellite chemical disposal area

Protocol references

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