New technique produces highly selective filter materials

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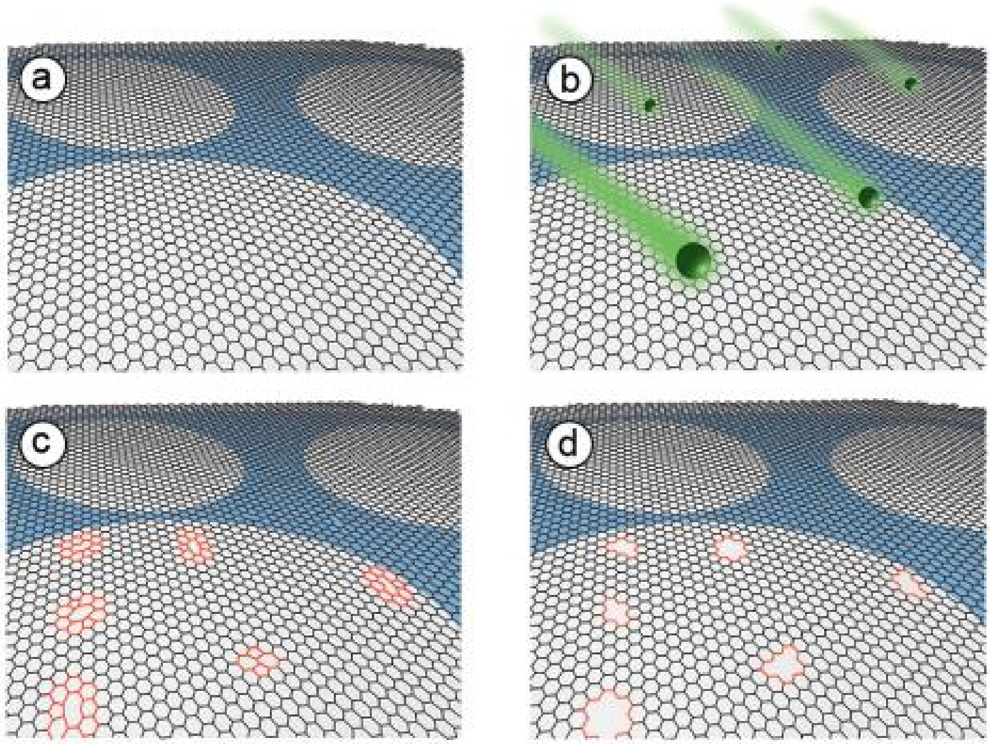

The MIT researchers used a four-step process to create filters from graphene (shown here): (a) a one-atom-thick sheet of graphene is placed on a supporting structure; (b) the graphene is bombarded with gallium ions; (c) wherever the gallium ions strike the graphene, they create defects in its structure; and (d) when etched with an oxidizing solution, each of those defects grows into a hole in the graphene sheet. The longer the material stays in the oxidizing bath, the larger the holes get.

Researchers have devised a way of making tiny holes of controllable size in sheets of graphene, a development that could lead to ultrathin filters for improved desalination or water purification.

The team of researchers at MIT, Oak Ridge National Laboratory, and in Saudi Arabia succeeded in creating subnanoscale pores in a sheet of the one-atom-thick material, which is one of the strongest materials known. Their findings are published in the journal *Nano Letters*.

The concept of using graphene, perforated by nanoscale pores, as a filter in desalination has been proposed and analyzed by other MIT researchers. The new work, led by graduate student Sean O'Hern and associate professor of mechanical engineering Rohit Karnik, is the first step toward actual production of such a graphene filter.

Making these minuscule holes in graphene—a hexagonal array of carbon atoms, like atomic-scale chicken wire—occurs in a two-stage process. First, the graphene is bombarded with gallium ions, which disrupt the carbon bonds. Then, the graphene is etched with an oxidizing solution that reacts strongly with the disrupted bonds—producing a hole at each spot where the gallium ions struck. By controlling how long the graphene sheet is left in the oxidizing solution, the MIT researchers can control the average size of the pores.

A big limitation in existing nanofiltration and reverse-osmosis desalination plants, which use filters to separate salt from seawater, is their low permeability: Water flows very slowly through them. The graphene filters, being much thinner, yet very strong, can sustain a much higher flow. "We've developed the first membrane that consists of a high density of subnanometer-scale pores in an atomically thin, single sheet of graphene," O'Hern says.

This experimental setup was used to test the properties of the graphene filters. A red dye in the water on left was used to demonstrate the filter's ability to block passage of the dye molecules.

For efficient desalination, a membrane must demonstrate "a high rejection rate of salt, yet a high flow rate of water," he adds. One way of doing that is decreasing the membrane's thickness, but this quickly renders conventional polymer-based membranes too weak to sustain the water pressure, or too ineffective at rejecting salt, he explains.

With graphene membranes, it becomes simply a matter of controlling the size of the pores, making them "larger than water molecules, but smaller than everything else," O'Hern says—whether salt, impurities, or particular kinds of biochemical molecules.

The permeability of such graphene filters, according to computer simulations, could be 50 times greater than that of conventional membranes, as demonstrated earlier by a team of MIT researchers led by graduate student David Cohen-Tanugi of the Department of Materials Science and Engineering. But producing such filters with controlled pore sizes has remained a challenge. The new work, O'Hern says, demonstrates a method for actually producing such material with dense concentrations of nanometer-scale holes over large areas.

"We bombard the graphene with gallium ions at high energy," O'Hern says. "That creates defects in the graphene structure, and these defects are more chemically reactive." When the material is bathed in a reactive oxidant solution, the oxidant "preferentially attacks the defects," and etches away many holes of roughly similar size. O'Hern and his co-authors were able to produce a membrane with 5 trillion pores per square centimeter, well suited to use for filtration. "To better understand how small and dense these graphene pores are, if our graphene membrane were to be magnified about a million times, the pores would be less than 1 millimeter in size, spaced about 4 millimeters apart, and span over 38 square miles, an area roughly half the size of Boston," O'Hern says.

With this technique, the researchers were able to control the filtration properties of a single, centimeter-sized sheet of graphene: Without etching, no salt flowed through the defects formed by gallium ions. With just a little etching, the membranes started allowing positive salt ions to flow through. With further etching, the membranes allowed both positive and negative salt ions to flow through, but blocked the flow of larger organic molecules. With even more etching, the pores were large enough to allow everything to go through.

Scaling up the process to produce useful sheets of the permeable graphene, while maintaining control over the pore sizes, will require further research, O'Hern says.

Karnik says that such membranes, depending on their pore size, could find various applications. Desalination and nanofiltration may be the most demanding, since the membranes required for these plants would be very large. But for other purposes, such as selective filtration of molecules—for example, removal of unreacted reagents from DNA—even the very small filters produced so far might be useful.

"For biofiltration, size or cost are not as critical," Karnik says. "For those applications, the current scale is suitable."

Bruce Hinds, a professor of materials engineering at the University of Kentucky who was not involved in this work, says, "Previous groups had tried just ion bombardment or plasma radical formation." The idea of combining these methods "is nice and has the potential to be fine-tuned." While more work needs to be done to refine the technique, he says, this approach is "promising" and could ultimately help to lead to applications in "water purification, energy storage, energy production, [and] pharmaceutical production."

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