Making Activated Carbon by Wet Pressurized Pyrolysis

Raw materials other than the traditional ones can now be used.

Ames Research Center, Moffett Field, California

A wet pressurized pyrolysis (wet carbonization) process has been invented as a means of producing activated carbon from a wide variety of inedible biomass consisting principally of plant wastes. The principal intended use of this activated carbon is room-temperature adsorption of pollutant gases from cooled incinerator exhaust streams.

Activated carbon is highly porous and has a large surface area. The surface area depends strongly on the raw material and the production process. Coconut shells and bituminous coal are the primary raw materials that, until now, were converted into activated carbon of commercially acceptable quality by use of traditional production processes that involve activation by use of steam or carbon dioxide.

In the wet pressurized pyrolysis process, the plant material is subjected to high pressure and temperature in an aqueous medium in the absence of oxygen for a specified amount of time to break carbon-oxygen bonds in the organic material and modify the structure of the material to obtain large surface area. Plant materials that have been used in demonstrations of the process include inedible parts of wheat, rice, potato, soybean, and tomato plants. The raw plant material is ground and mixed with a specified proportion of water. The mixture is placed in a stirred autoclave, wherein it is pyrolyzed at a temperature between 450 and 590 °F (approximately between 230 and 310 °C) and a pressure between 1 and 1.4 kpsi (approximately between 7 and 10 MPa) for a time between 5 minutes and 1 hour.

The solid fraction remaining after wet carbonization is dried, then activated at a temperature of 500 °F (260 °C) in nitrogen gas. The activated carbon thus produced is comparable to commercial activated carbon. It can be used to adsorb oxides of sulfur, oxides of nitrogen, and trace amounts of hydrocarbons, any or all of which can be present in flue gas. Alternatively, the dried solid fraction can be used, even without the activation treatment, to absorb oxides of nitrogen.

This work was done by John W. Fisher of Ames Research Center and Suresh Pisharody, K. Wignarajah, and Mark Moran of Lockheed Martin Corp.

This invention is owned by NASA and a patent application has been filed. Inquiries concerning rights for the commercial use of this invention should be addressed to the Ames Technology Partnerships Division at (650) 604-2954. Refer to ARC-14929-1.

Composite Solid Electrolyte Containing Li⁺-Conducting Fibers

Li⁺-ion conductivities are greater than those achieved before.

John H. Glenn Research Center, Cleveland, Ohio

Improved composite solid polymer electrolytes (CSPEs) are being developed for use in lithium-ion power cells. The matrix components of these composites, like those of some prior CSPEs, are high-molecular-weight dielectric polymers [generally based on polyethylene oxide (PEO)]. The filler components of these composites are continuous, highly-Li⁺-conductive, inorganic fibers.

PEO-based polymers alone would be suitable for use as solid electrolytes, were it not for the fact that their room-temperature Li⁺-ion conductivities lie in the range between 10⁻⁶ and 10⁻⁸ S/cm — too low for practical applications. In a prior approach to formulating a CSPE, one utilizes nonconductive nanoscale inorganic filler particles to increase the interfacial stability of the conductive phase. The filler particles also trap some electrolyte impurities. The achievable increase in conductivity is limited by the nonconductive nature of the filler particles.

These Arrhenius Plots were derived from Li⁺-ion conductivity measurements on two CSPEs that were identical in the proportions of all ingredients (including the La₀.₅₅Li₀.₃₅TiO₃ filler), except that in one CSPE, the filler was in particle form, and in the other CSPE, the filler was in fiber form.
In another prior approach — the one leading to the present improved CSPEs — one utilizes a highly-Li+-conductive inorganic filler material to increase the effective Li+-conductivity of the solid electrolyte. In prior CSPE formulations following this approach, the highly-Li+-conductive fillers have been in the form of particles. It has been found that Li+-ion conductivity can be increased to about $10^4$ S/cm by use of particles, but that the potential for any further increase is limited by the inherently restrictive nature of contacts between particles.

In contrast, in a CSPE of the present type, interparticle contact or the lack thereof is no longer an issue. In a typical application, a CSPE is formed as a film. The highly-Li+-conductive fibers can penetrate the entire thickness of the film and can thereby effectively constitute a relatively-long-distance Li+-transfer tunnel. The Li+-ion conductivity of the film as a whole is thus increased substantially beyond that achievable by use of particles. For example, the figure presents results of conductivity measurements on two CSPEs made from a PEO-LiN(SO$_2$CF$_2$CF$_3$)$_2$ polymer electrolyte filled with 20 weight percent of the highly-Li+-conductive compound La$_{0.5}$Li$_{0.5}$TiO$_3$.

An additional advantage of using filler fibers is that mechanical properties of the resulting CSPEs are superior to those attainable by use of particle fillers. One disadvantage — at the present state of development — is that relative to particles, fibers are less effective for interface stabilization and trapping of impurities. In contemplated further development, it may be possible to overcome this disadvantage by reducing fiber diameters to the order of nanometers. Other avenues of development could include selection of fiber materials having greater Li+-ion conductivities and finding ways to arrange fibers in velvety mats to maximize through-the-thickness conductivities.

This work was done by A. John Appleby, Chunsheng Wang, and Xiangwu Zhang of Texas A&M University for Glenn Research Center. Further information is contained in a TSP (see page 1).

Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Commercial Technology Office, Attn: Steve Fedor, Mail Stop 4–8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17470-1.

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**Electrically Conductive Anodized Aluminum Surfaces**

These coatings are highly adherent, transparent, and relatively inexpensive.

*Marshall Space Flight Center, Alabama*

Anodized aluminum components can be treated to make them sufficiently electrically conductive to suppress discharges of static electricity. The treatment was conceived as a means of preventing static electric discharges on exterior satin-anodized aluminum (SAA) surfaces of spacecraft without adversely affecting the thermal-control/optical properties of the SAA and without need to apply electrically conductive paints, which eventually peel off in the harsh environment of outer space. The treatment can also be used to impart electrical conductivity to anodized housings of computers, medical electronic instruments, telephone-exchange equipment, and other terrestrial electronic equipment vulnerable to electrostatic discharge.

The electrical resistivity of a typical anodized aluminum surface layer lies between $10^{11}$ and $10^{15} \, \Omega \cdot \text{cm}$. To suppress electrostatic discharge, it is necessary to reduce the electrical resistivity significantly — preferably to $\lesssim 10^9 \, \Omega \cdot \text{cm}$. The present treatment does this. The treatment is a direct electrodeposition process in which the outer anodized surface becomes covered and the pores in the surface filled with a transparent, electrically conductive metal oxide nanocomposite. Filling the pores with the nanocomposite reduces the transverse electrical resistivity and, in the original intended outer-space application, the exterior covering portion of the nanocomposite would afford the requisite electrical contact with the outer-space plasma.

The electrical resistivity of the nanocomposite can be tailored to a value between $10^7$ and $10^{12} \, \Omega \cdot \text{cm}$. Unlike electrically conductive paint, the nanocomposite becomes an integral part of the anodized aluminum substrate, without need for adhesive bonding material and without risk of subsequent peeling. The electrodeposition process is compatible with commercial anodizing production lines.

At present, the electronics industry uses expensive, exotic, electrostatic-discharge-suppressing finishes: examples include silver impregnated anodized, black electroless nickel, black chrome, and black copper. In comparison with these competing finishes, the present nanocomposite finishes are expected to cost 50 to 20 percent less and to last longer.

This work was done by Trung Hung Nguyen of EIC Laboratories for Marshall Space Flight Center. Further information is contained in a TSP (see page 1). MFS-32092-1