New Trace Capacity Test Method Needed for Future Activated Carbon Applications

By Henry Nowicki, George Nowicki, Wayne Schuliger, Shib Mookherjea and Barbara Sherman

New test methods can facilitate scientific discoveries and lead to new commercial granular activated carbon (GAC) products. Also, a new or modified paradigm can result when advanced data acquisition is available to base decisions on.

Before new models based on improved data acquisition can be provided, it is critical that we have a complete understanding of the state-of-the-art and history. New working concepts typically build on prior work.

Two historical, recommended readings provide an understanding of how activated carbons work.[1,2] Dr. Milton Manes also connected this early work and brought it up to the present in his writings.[3]

Advanced modern test methods

Two advanced test methods are improving decisions made by activated carbon users and manufacturers. These test methods have been coined Gravimetric Rapid Pore Size Distribution (GRPD) and Trace Capacity Test Method (TCTM). If you are using or plan to apply activated carbon to solve water or air problems, you need to know about these two advanced test methods.

GRPD has been previously covered in additional writings[4-17] providing examples of the usefulness of GRPD. On several occasions, five to 12 different vendor activated carbon samples were analyzed by GRPD and American Society Testing Materials (ASTM) activated carbon test methods to determine the best product for client applications.

Typically the different vendor samples had similar ASTM test method results for iodine and butane activity and other standardized tests. But, upon GRPD advanced testing, significant and relevant differences were revealed, allowing carbon users to select the best GAC for their applications.

GRPD has shown that it is capable of differentiating activated carbons with the same ASTM iodine numbers for drinking water applications. GRPD provides differentiation of these same iodine number samples and a full characterization of the test samples.

GRPD also provides a full characterization of performance and physical properties for the sorbent sample: characteristic and differential adsorption and desorption curves; aqueous- and vapor-phase isotherms; third-degree polynomial equation for adsorption pore volume as a function of specific adsorption potential energies; pore size distribution; total pore volume; cleanliness of tested sample and mid- and high-capacity adsorption values and surface area in meters squared per gram. The process has been run on a wide variety of sorbent types and forms; thus, a new sample can be run to quickly determine its market strengths and weaknesses based on comparison with the prior run collective database.

Trace capacity test method applications

The TCTM process compliments GRPD by providing more detailed information for the tested sample about the high adsorption energy sites in the GRPD test method.[18] The following information will further outline this needed new test method and help set up and/or use the test method to evaluate sorbent materials to solve some pressing real commercial opportunities.

Often, test methods are the difference-makers in commercial product development efforts. Some problems the TCTM are expected to address are: presently difficult-to-remove small adsorbates at trace levels from water and air streams; endocrine disrupter chemicals; carbon dioxide and methane capture and release, and many more.

It is obvious to look at the use of tetrafluoromethane (TFM) as the sorbent challenge gas based on prior use of 1,1,1,2-tetrafloromethane (TFE) as the challenge gas for the GRPD test method.[4-17] These many GRPD applications for a full characterization of sorbent materials will continue to grow as more is learned about the benefits of GRPD.

Use of TFM for the TCTM is a natural extension of TFE, because TFM requires higher energy binding sites to efficiently capture it compared to TFE. Choosing TFM as the challenge gas provides a lower boiling point and lower polarizability of the path to characterize the highest potential adsorption energy sites in a sorbent test sample.

Localized versus mobile graphitic surface binding

Molecules that hit the activated carbon surface (from the vapor-, liquid- or melt-phases) are pictured to glide or hop along the graphitic surface.[2] With activated carbon's heterogenous adsorption energy distribution, the adsorbate speed on the surface and its ability to hold upon the adsorbate and keep it from going back into the bulk phase depend largely on the adsorbent localized adsorption energy.

By designing and manufacturing more high-energy adsorption sites with targeted specificity into the adsorbent, it makes sense that present molecules which are refractory to activated

carbon adsorption may be adsorbable at low, partial pressures and concentrations relative to their saturation concentrations. This is a worthy future goal. A significant trace capacity test method to evaluate present and future emerging sorbents for this market is needed by users and manufacturers.

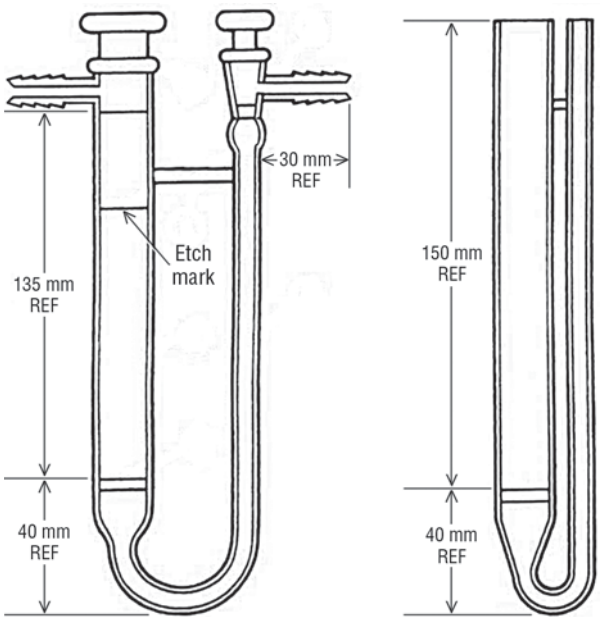
In order to help solve some real-world problems, this new trace-capacity test method will improve capture and release of low-concentration, hard-to-remove organics from water, carbon dioxide, hydrogen, methyl bromide, ethylene and endocrine disrupters. Other applications are also beneficiaries of this test method. TCTM provides a way to better determine the high-energy binding site total pore volume per mass or volume of the tested sorbent.

New application for ASTM butane activity apparatus

The test apparatus for the TCTM can be the standard ASTM butane activity test apparatus. The test method number is D 5742-95 and is titled *Standard Test Method for Determination of Butane Activity of Activated Carbon*.

The above sample tube has 16.7 mL (plus or minus 0.05 mL) of volume for the sample. The sample packing density in the sample tube (carbon weight in grams/16.7 mL) must be equal to at least 94 percent of its dry apparent density obtained in ASTM method *D 2854: Test Method for Apparent Density of Activated Carbon*.

Butane tube schematic



Butane gas is passed through the sample at 25°C (77°F). The butane is thermostated in a water bath before it is sent through the sample tube, containing the sample, in a water bath at 25°C. The sample tube is weighed until constant weight for butane capacity is obtained. The result is presented in grams of butane per 100 grams of sorbent tested or 100-mL of sorbent volume.

Using this ASTM butane test stand, TCTM total pore volume can be obtained by using TFM in place of butane gas. TCTM is done like a butane number, but TFM gas is used in place of butane.

Since TFM is a much more difficult challenge for the sorbent, only high-adsorption potential energy binding sites adsorb TFM. Again, test results can be expressed in grams of TFM adsorbed per 100 grams of sorbent or per 100 mL of sorbent volume, just as is done with the standard butane test.

Unused and used GAC

Granular activated carbons have a determinable limit on how much pollutants they can hold before breakthrough. You need to know that all activated carbons are not the same and will have different pollutant capacities before breakthrough.

GRPD is being used to determine what GAC pore size adsorption spaces

are needed for different applications. This needed adsorption space is based on a comparison of GRPD runs with the unused and used GAC for the application. The difference in the used and unused GRPD runs provides the pore structure needed for specific applications.[19]

It is important to clearly understand the relatively recent ASTM recommended terms, unused and used GAC.[20] The ASTM recommends (via personal discussions with Dr. Amos Turk) use of these words in place of virgin (unused) and spent or exhausted (used) carbon. The starting carbon in your application is the unused GAC and the used GAC is what you change out to start a new treatment.

Completely used GAC occurs when influent and effluent through a GAC column is the same. We suspect that we will continue to see virgin, fresh, spent, exhausted and other terms to describe the status of GAC; but we should all try to use the ASTM recommended glossary of terms in order to understand each other better and be more productive.

ASTM method approval process

Getting GRPD formally through the ASTM process as a listed method will not happen in the near future. There are only a few instruments available to provide this sample-specific, full characterization for adsorption information package; the ASTM process takes years.

One way around the small number of GRPD type instruments is to apply the ASTM butane activity test equipment. There are many laboratories with butane activity test stands and well qualified equipment operators. By selecting a suite of challenge gases and applying the butane activity test stand, it is possible to approach the GRPD test package of information.

Recent comparison of ASTM butane activity method and working capacity and butane working capacity determined by GRPD indicates both GRPD and ASTM butane activity test *D 5742-95* and *ASTM D 5228-96, Test Method for Determination of the Butane Working Capacity of Activated Carbon*, yield the same results.[21] This is another testimonial to the power and ruggedness of GRPD testing.

The trace capacity method is designed to compliment the GRPD full-characterization test method by focusing on the binding sites with the highest adsorption forces. All adsorption binding sites in activated carbons are not the same.

Selecting the best activated carbon for a specific application depends on determination of the sorbent's pore structure. The TCTM and GRPD test methods are powerful aids in pore structure determinations. Being able to better measure the distribution of high-energy adsorption sites with TCTM is expected to help facilitate development of new emerging sorbents designed to help solve some important problems.

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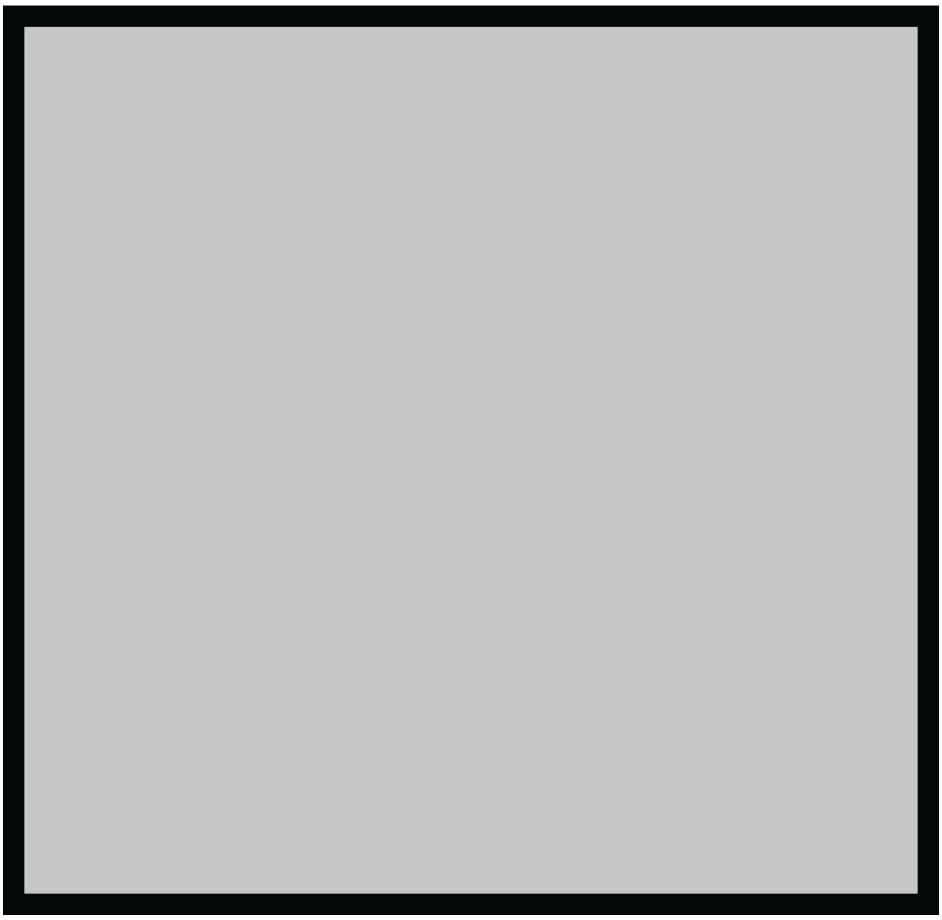

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♦ *Professional Analytical and Consulting Services, Inc. (PACS) is celebrating its 26th year of business and its major division, Activated Carbon Services, is in its 25th year. The firm provides products and services to enhance its clients' position in their market places. Products and services are described at www.pacslabs.com.*

If you would like to hear the inventors talk about the new Trace Capacity Test Method, plan to attend their session in Pittsburgh, PA, Oct. 6 at 9:15 a.m. at IACC-24. If you know of other new test methods capable of aiding better sorbent selection decisions, let us know through Henry@pacslabs.com.