Testing Tips for Activated Carbon Users: Part 1

By Henry Nowicki, PhD, George Nowicki, Wayne Schuliger, Christopher Brunning, Gary Pittman and Barbara Sherman

Series introduction

Activated carbon (AC) is an ambiguous term applied to a broad range of end products that satisfy an amazingly long list of commercial markets. In most of the world, charcoal refers to the precursor material used to manufacture activated carbons, but in Europe, charcoal is used to mean activated carbon. It is important to build yourself a good vocabulary on activated carbon and understanding of ASTM test methods.1, 2 We will focus on water purification, but the informational value is not limited to this market sector. Some of the topics we will cover in this article are shown in Table 1.

Technically, activated carbon is complex and evolving; AC users only learn about this material on the job and through continuing education. Activated carbon products can be manufactured from a wide variety of raw materials and conversion processes. With currently accepted, state-of-the-art methods, heating almost any carbon-containing material to a certain temperature in the absence of oxygen will result in black char, formed by pyrolysis. The raw material changes form, resulting in two very different useful products, charcoal and synthetic gas. These charcoals can be activated by replacing some dense charcoal carbon atoms with open spaces to increase the surface area, from a few squared meters per gram to 900 to 1,200 squared meters per gram, to provide the physical adsorption phenomenon.

Char is a low-value, dense material that can be made porous or activated, resulting in higher economic value and performance. Activation is any process whereby a substance is treated to develop adsorptive properties; water is the predominant form of activation. This conversion can be done with oxygen, water or carbon-dioxide activation; activation with steam enlarges micropores as burnout proceeds, shifting them to mesopores (the water-gas reaction: $\text{H}_2\text{O} + \text{C} = \text{CO} + \text{H}_2$). Gaseous steam attacks a solid carbon that has been heated to 954°C (1,750°F), an endothermic reaction. Carbon monoxide and hydrogen reaction products can be converted to carbon dioxide and water by carefully adding small amounts of oxygen into the furnace to generate free internal furnace heat. Activation with oxygen and carbon dioxide proceed differently; oxygen is hard to control and carbon dioxide should be used to make micropores. Laboratory testing is necessary to match the best activated carbon for specific applications and monitor its life-cycle performance to maximize use rate and gallons treated per pound, before replacement with unused AC. Part 1 of this series introduces some important characteristic physical test information and useful tips for AC users.

On-site physical tests and observations

Activated carbon users need to examine incoming material to ensure it does not have bad odor, feel wet or oily, have excess dust or be friable; i.e., granular AC should not disintegrate when moderate pressure is applied between the fingers. These are easy observations that can be accomplished with incoming AC. Users should also do heat-of-immersion (HOI) testing on incoming material, which uses safe, simple equipment: a container with mineral oil and a thermometer. This testing is based on exothermic principles and measures the adsorption heat in mineral oil.3 These tests can be done quickly at low cost to evaluate incoming activated carbon.

Representative sampling

Sampling begins in the field from fresh, unused or used AC from operating adsorbers. Knowing the characteristics of the installed AC is important in order to select the best AC for your applications. An AC that lasts two to three times or longer is more valuable, even if initial cost per pound is more than shorter performing AC. Every activated carbon can and should be tested prior to its installation and use; a planned performance testing schedule should be a part of initial AC installation. A representative sample should be obtained while it is being installed, and saved in a protective, air-tight container. The container should be labeled with complete sample identification, including date, supplier source, manufacturer’s lot number, carbon type and vendor specifications. Each test method requires a specific amount of material. Labs ask for more material than needed because it helps in acquiring a representative and retained sample. A retained sample is the unused field sample that can be used to recommend additional testing after initial testing is completed and the results are evaluated. Laboratories should provide requesting engineers advice on proper sampling, offer turnkey sampling for clients on request and have a retained sample standard operating procedure (SOP) on unused received samples. Clients should become familiar with the SOP.

On-site AC installation sampling provides a preserved representative retained sample, which makes it possible for comparison should subsequent operational problems occur. Also, a representative sample is important for laboratory testing because decisions are based on a relatively small amount (one to 100 grams are typical test sizes). A sample received by a laboratory may be a small part of several tons, and testing laboratories need to ensure representative samples are used. This is best ac-
complished by asking AC users to send samples from the top, middle and bottom of the column. A representative sample is acquired by coning or riffling the entire field sample; the concept is that every received particle has an equal chance to be a part of the tested sample. Coning is done by pouring the sample into a cone, followed by taking a random sample size for a specific test method. Riffling is done with a mechanical device shaped like an upside-down letter Y. The sample is slowly poured through the top and a splitter separates the sample into two equal halves; repetition is used until a sample size is obtained for the specific test method. If a representative sample is not used, biased results can occur. For example, with a quart of granular activated carbon (GAC) in a bottle, the fine dust will work its way to the bottom of the container. If the sample is taken from the top, there is no representative dust, which is small and may be more activated or burned out. Water plants remove dust by backwashing before they start operations, but AC can be used to destroy strong oxidants, as AC is a reducing agent. In this oxidation-reduction role, heat is generated and fine dust can block air flow through the bed and possibly explode—it is a safety problem. Dust from unused GAC is not toxic because the particles are too big to penetrate deep into lungs. Activated carbon with contaminants need to be handled as toxic.

**Activated carbon physical forms**

Activated carbons come in different forms: powder, granular, pellets, cloth and carbon block. Granular activated carbon is the dominant form used for drinking water applications. Historically, coal-based GAC was used more than coconut-shell-based, but with the US Department of Commerce customs tariff on coal steam-activated carbons from China, the relative price of coconut-shell-based carbon is now competitive with coal-based and has gained market share.

Coconut-shell-based GAC is somewhat harder and has an advantage for reactivation yield. Reactivated cost is about half of virgin or unused GAC. Coconut-shell may have more micropore volume than coal-based GAC. Micropores are needed for small molecules, like trihalomethanes, methyl tertiary butyl ether (MTBE), geosmin and methyl isoborneol (MIB) physical adsorption. Mother Nature provides coconut-shell breathing tubes, which are large vessels within the plant to transport gases and liquids. These are useful in finished AC to transport contaminated water to the particles’ interior active adsorption sites. Since water diffusion inside carbon granules is the slowest step in physical adsorption, coconut-shell breathing tubes provide the major water-flow path to the interior. Coal-based carbons can be manufactured to create high-flow pathways to the interior using a process called agglomeration.

Powdered activated carbon is used for predictable episodic taste and odor (T&O) problems. Water plant personnel can add powdered AC before settling tanks to remove T&O compounds, then remove the settled powder before distribution of the finished water. These smaller GAC particles provide faster kinetics for T&O removal because the particle surface-area-to-volume ratio is lower with powder than granular. Thus, a molecule in bulk water can travel much faster to the center of powder than granular. Powder, however, does not reduce T&O compounds to non-detect levels like a column of GAC can do. But general users do not incur the capital cost of adsorbers to hold and process GAC. Powder is used in batch processes where a given volume of water is dosed with a known mass of powder. Granular is a continuous process; thus, powder provides a point on an isotherm, leaving a known amount in water and a known loading on powdered activated carbon. GAC columns can reduce contaminants to non-detect levels in the effluent because the column is designed to maintain the mass transfer zone (MTZ) in the column. A segregated GAC column has a distribution of particle sizes, smaller on top and bigger on bottom. It maintains its mass-transfer-zone shape and location after backwashing, with proper slow settling after backwashing to maintain MTZ column location. Backwashing reverses water-flow direction to suspend packed AC particles. This loosens and removes dirt and large particulate matter from the influent and controls head pressure on the GAC bed. Carbon blocks are made by combining a thermoplastic with small GAC particles and heated to melt the plastic and glue the particles together. Carbon blocks are tested by breaking them down with physical force. They have little dust and are conveniently used as drop-in filter replacements for refrigerator and POU devices for water purification. AC pellets do have a place in water treatment, but not very often. Pellets provide the least resistance to water flow through the bed and have the smallest amount of dust. Thus, water treatment applications with no suspended solids and no need for backwashing could use pellets. You cannot backwash pellets and maintain column MTZ shape, size and location because pellets are all equivalent and randomly settle after backwashing.

**Mass transfer zone**

A GAC column working in an established equilibrium with influent contaminated water has three distinct GAC column zones:

- **Zone 1.** The first zone at the column face receives influent with contaminants. Carbon becomes saturated with contaminants and the water will be the same as influent. This zone is no longer removing contaminants and will get longer as more water passes through the bed.
- **Zone 2.** The second zone in the column is a gradient, ranging from small contaminant reduction to complete influent removal. This is the mass transfer zone where contaminants are removed from the water to the carbon. MTZ needs to be maintained in the column to provide contaminant-free pure water.
- **Zone 3.** The third zone at the effluent column side is clean.
carbon and water. The size and shape of mature MTZ remains the same as it moves through the column. There is no detectable contaminant appearing in the effluent until the MTZ migrates out of GAC columns. The contaminant then becomes detectable in effluent and rises to influent level with MTZ shape.

Carbon adsorbers are designed to maintain the MTZ in the working column. It is a good idea for carbon users to have multiple columns in series to obtain full GAC utilization before replacement. The first column is run until the MTZ breaks through column one and enters back-up column two; column one is run until influent and effluent are the same. At this time, the user has maximized gallons treated per pound of exhausted GAC in column one. This exhausted GAC can still be functioning as a filter and trapping millimeter- and micron-sized particles from the influent, which can be periodically removed to decrease head pressure and water-level rise by backwashing the trapped material out of the column. (The words bed and column are often used interchangeably; typically bed is for small POU devices and column for large GAC commercial operations.) Adsorption and absorption are often used interchangeably in technical literature, but they are not the same. Adsorption occurs when contaminant(s) enter the GAC granule and are accumulated on carbon surfaces and there is no increase in granule size, no swelling. Absorption occurs when contaminants infiltrate the sorbent material homogeneously and the sorbent swells.

**ASTM test methods**

International ASTM testing standards on activated carbon need to be used for apparent density, moisture, particle sizing, hardness number and other standard tests. AC users can and should acquire the latest ASTM carbon methods. These methods are committee-validated by manufacturers, users and consultants and are most used between sellers’ and purchasers’ specifications. There are also many other non-ASTM test methods that drive the AC industry. Instrumental test methods for GAC characterization include GAED for full characterization and low-cost isotherms, ICP for metals detection and quantification and GC/MS for identifying organic contaminants on carbons.

**Apparent density**

Apparent density (AD; bulk density or packed density) is determined on a granular AC (minimum 90 percent being larger than 30 mesh or 30 holes per square inch of wire mesh screen). Mass per milliliter or density is determined by slowly delivering GAC (0.75 to 1.0 mL/s) into an appropriately sized graduated cylinder, using a funnel with an outside diameter that just fits into the graduated cylinder. This provides uniform bed filling to accurately read the filled cylinder volume without shaking. Getting 12 particles or more across the bed diameter avoids cylinder-wall effect (which simply means poor particle packing) and low biased density result; i.e., observable void volume spaces next to the cylinder wall. To remove these void spaces, increase the cylinder diameter size to get more than 12 particles lying on the AC bed face. Apparent density numbers provide users with the packed density of GAC. Related to mechanical strength, this information is needed for designing GAC vessels, provide backwashing and for ordering purposes when purchasing material for refill. If AC is going to fines in the process, using a denser AC may solve mechanical strength problems.

**GAC moisture level**

Granular activated carbon does not like water, which is one reason it effectively removes contaminants from water. The typical purchasing specification for the amount of water on unused GAC is less than two percent weight/weight. Moisture level can be determined by comparing received and dry apparent densities for unused, non-contaminated carbons through drying the AD material three hours at 150°C (302°F). Water-percent calculation is received AD minus dry AD, to provide mass of water, divided by received AD times 100; this provides percent moisture. If GAC comes in contact with VOCs, they will be adsorbed. Some weakly adsorbed volatile organic compounds (VOCs) could shed off GAC with water at 150°C, thus providing a false high-water reading and missing VOCs. The definitive moisture test method is called Dean-stark, based on a xylene Soxhlet extraction of the GAC sample and collection of distilled sample water into a graduated Dean-stark tube attached to the distillation column.

**Proper wetting of activated carbons**

The most common user complaint about GAC not working is related to improper wetting before use. Clients can purchase fully wetted, ready-to-use GAC. Dry GAC requires water soaking and backwashing several times to remove dust before use and ensure proper wetting; i.e., complete removal of entrapped air and filling micropores with water. Micropores concentrate ambient air two to three times above ambient pressure, making air removal difficult.

To get maximum GAC performance, contaminants in bulk water must be transported to the deepest particle adsorption sites. Proper wetting takes 72 hours before putting GAC into service. It is absolutely critical that GAC micropores or adsorption spaces are filled with water and original entrapped air-filled adsorption spaces are not accessible to aqueous contaminants. Be patient.

**Particle sizing**

Particle sizing is used to determine the nominal particle size (NPS) of small and large ends of the sample size distribution. GAC nominal particle sizes on both ends exclude not more than five percent of particle size distribution mass per side. Determining the screen size for large-end nominal particle size allows selection of hardness test sieve. The selected sieve has half the mesh size opening of large-end nominal particle size. It is necessary to know the distribution of GAC particle sizes in order to provide proper contact time in liquid packed beds and column applications. Smaller particle sizes fit closer together, providing increased resistance to flow and more contact time. Flow must not be too slow, in order to provide practical filter use and avoid excessive head pressure and water backup. Changes in particle sizing distribution can affect the rate of adsorption, MTZ size and shape in a GAC bed and can change pressure drop across the bed. Particle sizing data can provide mean particle diameter (MPD), effective size (ES) and uniformity coefficient (UC) values that are needed in municipal water treatment applications where gravity flow is important.

**Particle sizing test parameters**

Seven wire-mesh sieves (height two inches and diameter eight inches) are stacked onto a receiving pan in order of increasing size opening, bottom to top. A volume with a known weight of GAC is added to the top of the sieve stack, depending on the AD. The stack is then shaken with a mechanical device for 10 minutes and weights are determined on each sieve and receiver pan. The sum must add up to the starting amount. Software compliant with ASTM is used to interpret and archive the data.

**Hardness, abrasion numbers**

The most frequently used test method to determine the resistance of AC to particle-size degradation is called ball-pan hardness number. This test method is not designed to predict
degredation in real-world applications using real-world process conditions, but has long been used as a physical characteristic of GAC mechanical strength. The highest hardness number is 100 and most specifications call for 97 to 98. After eliminating sub-nominal particle sizes, material is weighed, placed in a special hardness pan with stainless steel balls and vigorously rotated for 30 minutes; particles retained on the hardness test sieve are determined using a specific calculation method. The following equation is used to calculate hardness number:

\[ H = 100 \frac{B}{A} \]

\( H \) = Ball-pan hardness number  
\( B \) = Weight of sample retained on hardness test sieve  
\( A \) = Weight of sample loaded onto hardness pan before shaking

The hardness number is used to compare different GAC samples and is a good relative measure of physical degradation characteristics. It is also used to determine if the process application is degrading GAC as time goes on; hardness numbers are determined at different process times and compared.

An abrasion test is similar to hardness number, but more steel balls are used and it is more aggressive. One example of differences in test results involves two samples with a 98 hardness number. After the abrasion test, results are 96 and 71. This tells us that the 96 abrasion number sample is harder than 71. This carbon would probably be selected for a gold-recovery application because hardness is a critical trait for this AC application. Any AC degraded material leaving the adsorber that contains gold because hardness is a critical trait for this AC application. Any AC degraded material leaving the adsorber that contains gold is simply money lost.

Quality assurance/quality control program established program

Characteristic testing results for samples need to be obtained from a competent, professionally staffed laboratory with a quality assurance/quality control program (QA/QC) that includes training new employees; a structured problem solving approach; blind and known control samples with client sample runs; duplicates; blanks; the ability to develop new test methods; the capability of answering activated carbon questions and being state-of-the-art, with continuous improvement objectives. Addressing safety issues is a part of a good QA/QC program.

Conclusion

Activated carbon users need to apply routine and modern, advanced laboratory testing to maximize the effectiveness of products to improve drinking water quality. Field and lab AC sampling is important to ensure users get representative tested samples and relevant information. It is too easy to overlook the importance of sampling. Service laboratories need to integrate business into consulting, sampling and R&D—growing quality relationships will keep clients coming back for more. Activated carbon users have a responsibility to educate themselves on this complex and evolving subject to improve performance and lower operating costs.

References
2. ASTM Stock Numbers ACTCARBON, ASTM Standards on activated carbon. service@astm.org.

About the authors

Corresponding author Henry Nowicki, PhD, MBA, is President and Senior Scientist for Professional Analytical & Consulting Services, Inc. (PACS). He is a member of the WC&P Technical Review Committee, on the editorial review board of International Filtration News and chairperson for the bi-annual International Activated Carbon Conference and Activated Carbon School. Nowicki presents the Activated Carbon Adsorption: Principles, Practices and Opportunities short course. He can be reached at Henry@pacsllabs.com or (724) 457-6576. Wayne Schuliger, PE is an activated carbon expert with 45 years of highly varied experience. He presents the Activated Carbon Adsorption: Principles, Practices and Opportunities short course. Schuliger can be reached at Wayne@pacsllabs.com. George Novicki, BS, directs the PACS laboratory operations and provides consulting. He can be reached at George@pacsllabs.com. Chris Brunning provides laboratory technical services and is the site webmaster. He can be reached at webmaster@pacsllabs.com. Gary Pittman is a PACS Senior Lab Technician. Barbara Sherman, BS Manager of Operations. She directs the International Activated Carbon Conference and PACS monthly and on-site short course programs. Sherman can be reached at Barb@pacsllabs.com.

About the company

Professional Analytical & Consulting Services, Inc. (PACS) provides a one-stop shop for activated carbon laboratory testing, consulting, expert witness services in science subjects and training to grow the size and quality of the activated carbon industry. PACS sponsors the International Activated Carbon Conference and Activated Carbon School.

Call for papers and vendors for the 32nd International Activated Carbon Conference (IACC-32) September 25-26 at the Sheraton Hotel near Pittsburgh, PA. It is never too late to participate. Short courses are provided before and after IACC-32 as well as at a client’s time and place. A registration form is available at www.pacsllabs.com for the Conference and Activated Carbon School Courses.