FILTRATION

By Henry Nowicki, Ph.D., Mick Greenbank, Ph.D. and Homer Yute, M.S., PACS

Evaluating Activated Carbons

ASTM, AWWA and EPA Standard Methods and New Test Methods for AC

Table 1. ASTM Activated Carbon Test Methods

Method Number	Test Method Description
D 2652-94	Terminology Relating to AC.
D 2854-96	Apparent density of AC.
D 2862-92	Particle size distribution of GAC.
D 2866-94	Total ash content of AC.
D 2867-95	Moisture in AC.
D 3379-75	Tensile Strength and Young's modulus for high-modulus single-filament materials.
D 3466-76	Ignition temperature of GAC.
D 3467-94	Carbon tetrachloride activity of AC.
D 3802-79	Ball-Pan hardness of AC.
D 5919-96	Adsorptive capacity of AC by a microisotherm technique, Determination of.
D 3860-89a	Adsorptive capacity of carbon by isotherm technique, Determination of.
D 3922-89	Operating performance of GAC for removal of soluble pollutants from water, Estimating the.
D 2865-96	Apparent density of AC.
D 3803-89	Nuclear-grade AC.
D 3838-80	pH of AC.
D 3860-89	Practice for determination of adsorptive capacity of AC by aqueous phase isotherm technique.
D 3922-89	Practice for estimating the operating performance of GAC for removal of soluble pollutants from water.
D 4069-95	Impregnated AC used to remove gaseous radio-iodines from gas streams.
D 4607-94	Iodine number of AC, Determination of.
D 5029-89	Water solubles in AC.
D 5158-93	Particle size of powdered AC, Determination of.
D 5159-91	Dusting attrition of GAC.
D 5160-95	Gas-phase adsorption testing of AC.
D 5228-92	Determination of butane working capacity of AC.
D 5742-95	Determination of butane activity of AC.
D 5832-95	Volatile matter content of AC samples.
D 5919-96	Practice for determination of adsorptive capacity of AC by a microisotherm technique for adsorbates at ppb concentrations.

Source: ASTM

AWWA Standard for Powdered AC

B 600-96	Phenol Value
B 600-96	Tannin Value

Source: AWWA

The American Society for Testing Methods (ASTM) and American Water Works Association (AWWA) have standard test methods for commerce between buyers and sellers of activated carbons (AC). These methods have been discussed in other articles[1,2] and are listed in Table 1 by title and method number. The methods are used for purchasing and monitoring after the AC is installed. New challenges are emerging in the industry that require new methods and product developments. This article discusses additional test methods for the AC industry.

Blending GAC Lots

All ACs are not equal. It is common practice to blend different lots of AC with different standard method iodine number values to meet the customer specifications. Often in drinking water applications, used granular activated carbon (GAC) from drinking water adsorbers is furnace reactivated to provide a resource for additional use cycles. Only dedicated furnaces for regeneration of used GAC from water filters can be used when the GAC will be returned to drinking water filtration. Since each cycle of the reactivation process loses abut 10 percent of the original GAC volume, unused make-up GAC must be added to the reactivated lot to get the original volume to fill the customers adsorber.

New test methods are needed to detect blended lots of GAC. The ASTM iodine number is an average of the blended lot, thus 50:50 blend of 1,000 and 700 iodine numbers yields a 850 iodine number. This heterogeneous lot (1,000:700) meets the 850 client specification but will have significantly different performance compared to a homogenous 850. The authors are developing new methods to detect heterogeneous GAC (granule-to-granule for intergranule heterogeneity and intragranule, outside granule versus inside granule heterogeneity). Currently, the AC industry does not have test methods for heterogeneity determination; presently, average and index values are provided.

GAC granules from blended lots can be separated based on apparent specific gravity test methods. Once the granules are separated they can be tested with standard AWWA, ASTM or heat-of-immersion test methods under development, commonly referred to as the AC Tester, which is the method PACS and ASTM's method development group committee D-19 are developing. The external portion of the separated granules can be removed by using a gem polisher. Once the exterior is worn off, the dust can be separated by using standard U.S. sieves. The gem polisher also can be used to remove coatings from other media. Again, the separated portions, exterior and interior granule parts, can be tested with standard AWWA or ASTM methods or the AC Tester.

The AC Tester results should be used by a professional who has knowledge about the specific adsorption systems being studied. The decision when the carbon is exhausted and needs replaced is a site-specific decision process. It depends on the toxicity of the adsorbates, competition of adsorbates and possibly moisture effects on the adsorption capacity.

AC Tester—Remaining AC Service Life

Two common questions concerning GAC installations are "How good is this GAC?" and "How long can I use this GAC before it needs to be changed?" The first question partially is answered using ASTM methods listed in Table 1, before purchase and installation. The second question is more difficult because there are no standard methods for testing used GAC to estimate remaining service. The authors have been working on a method to answer this important question and they have come up with a simple and low-cost test device called the AC Tester. This method is based on the concept that as the adsorption space in GAC fills, less heat is given off when adsorptive materials challenge it. In an initial space distribution in a bed of GAC, 40 percent of the volume is inside the GAC granules. Thus the AC Tester determines the heat rise of the unused GAC to compare with an equal volume of the used GAC. Rationing the temperature rises gives an estimate of the adsorption volume used (i.e., 4° C rise for unused and 1° C rise for used indicates only 50 percent of the original adsorption space is occupied.) Thus this example suggests adsorption space still remains in this used GAC, but the operators need to proceed with caution.

EPA Toxicity Characteristic Leaching Procedure

The EPA toxicity characteristic leaching procedure (TCLP) is a third-generation regulatory test method to determine if a waste material has potential to contaminate groundwater. This method consists of leaching used GAC with a simulated landfill aqueous acetate solution. The water extracted GAC adsorbates are analyzed for a target list of organic and inorganic toxic compounds. The 10 highest concentrations of organics and metals in the extracts in addition to the target list are qualitatively and quantitatively determined. Experience using the TCLP on used AC has shown the adsorbed organics have a negligible recovery using TCLP, but metals often are recovered. These recoveries are reasonable because the carbon adsorbed were accumulated from water passing through carbon. The high energy binding sites in the carbon hold on to adsorbates strongly and are not detached by buffered acetate solutions. California has a more aggressive extraction method based on using citric acid to evaluate a materials potential to leach and contaminate the ground water.

The authors have reported on liquid cocktails that can recover organics from used GAC.[3,4] These extracting liquids were developed for a new commercial regeneration process, "Liquid Regeneration of Commercial Used GAC," and found to be very efficient.[4] A version of this kicker/chase solvent for used GAC adsorbate recovery and analysis is under development as a stream monitor.[5]

Presently, there needs to be a test method capable of detecting 10-9 to 10-12 molar organics in water. The pharmaceutical food and specialty chemical industry have the potential of adding these concentrations to public waters. Some of these compounds are suspected to have endocrine disrupter and other deleterious biological effects.[6] GAC can enrich water organics 10,000–100,000 fold, which is the basis of this new stream monitor method. WQP

Learn More!
For more information related to this article, go to www.waterinfocenter.com/lm.cfm/wq060204

Acknowledgement

The authors thank Professor Emeritus Dr. Milton Manes for regularly sharing his thoughts about new opportunities in the AC industry. He is the originator of the kicker/chaser concept and many other innovations presently used in the AC industry. Manes has provided a short course for 12 years titled, "AC Adsorption: Principles and Applications," for PACS and the American Chemical Society.

References

1 Nowicki, H.G., B.S. Nowicki and H. Yute. "ASTM and AWWA Test Methods in Evaluating Activated Carbon," *Water Conditioning & Purification*, 2001.
2 PACS. www.pacslabs.com.
3 Nowicki, H.G. "Solvent Regeneration of Used GAC," International Activated Carbon Conference-2 (IACC-2), Pittsburgh, Pa., 1994.
4 Ben Franklin Technology Grant, State of Pennsylvania. "New Liquid Regeneration of Spent Activated Carbons," 96W.CE00272R-1 and R-2, 1997 and 1999.
5 Greenbank, Mick. "Detective Work and Future New Test Methods for the Activated Carbon Industry," IACC-10, Pittsburgh, Pa., September 2002.
6 Chemical Engineering News, 2001.

Glossary of AC Terms

- **AC Tester—**A device designed to determine the remaining service life in partially used and unused carbons such as powder, GAC, pellets and carbon cloth.
- **Activated Carbon—**Technology for removing organics from vapor and aqueous phase streams.
- **GAC Heterogeneity—**Granule-to-granule GAC heterogeneity (interheterogeneity) and exterior/interior granule heterogeneity (intraheterogeneity). Example of interheterogeneity: mixing 1,000 and 700 iodine numbered GAC lots.
- **Toxicity Characteristic Leaching Procedure (TCLP)—**The U.S. EPA-approved method to evaluate a material's potential to contaminate the ground water. For example, testing used ACs to determine the extractable adsorbates and identify and quantify a list of target compounds and the major compounds leached.
- **Heat of Adsorption—**Heat given off when an adsorbate molecule is attracted to a sorbent surface. For example, heat given off when GAC is immersed in mineral oil in the AC Tester.
- **Chemisorption Heat—**Heat given off during exothermic chemical reactions.
- **Gem Polisher—**A device used for removing the exterior layer of GAC granule off of the core. After polishing, the exterior and interior portions can be separated using standard sieving techniques, and the portions can be tested using standard methods used for buying and selling carbons.
- **ASTM Standard Methods—**American Society for Testing and Material Standards. (See Table 1 or visit www.astm.org.)
- **AWWA Standard Methods—**American Water Works Association. (See Table 1 or visit www.awwa.org.)
- **Unused GAC—**Older terms used in the carbon industry are new, virgin or first-time used. Used GAC is the term recommended by ASTM for the older term spent AC.

* *Additional AC terms are available at www.pacslabs.com*

About the Authors

Henry G. Nowicki, Ph.D. and MBA, directs the PACS Laboratory testing and consulting services and new business developments at PACS. He has obtained three patents and published more than 100 articles about environmental issues and AC adsorption and has been an expert witness in more than 30 legal cases. Dr. Nowicki may be reached at hnpacs@aol.com; www.pacslabs.com.

Mick Greenbank, Ph.D., is a surface chemist with 23 years of varied experiences in AC and holds seven patents. He directs new test methods development and application and provides special projects, consulting and training for PACS. Dr. Greenbank teaches "Selecting the Best Activated Carbon for the Application," a PACS shortcourse. He may be reached at mickpacs@aol.com.

Homer Yute is a mathematics and computer programming expert who has developed seven software programs for the AC industry.

All authors may be reached at PACS, Inc., 409 Meade Dr., Coraopolis, PA 15108; 724-457-6576.

For more information on this subject, write in 1014 on the reader service card.