ACTIVATED CARBON:

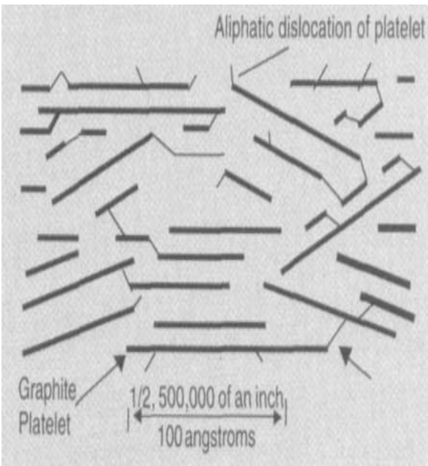
Portable Analytical Instrument Measures Life of Activated Carbon

By Henry G. Nowicki, Ph.D, and Homer Yute

Summary: A simple testing device has been designed to determine quality of activated carbon (AC), the best available technology to purify waterandaircontaminated with an assortment of organic compounds. However, AC is a manufactured carbonaceous material with open space, or pores, that fills with adsorbates as it removes organics from water. A common concern for AC users is how much longer they can use carbon before breakthrough—which indicates a contaminant in the influent has shown up in the effluent. The AC must then be replaced to maintain the specified effluent quality. The device described here is intended to help users determine the status of filtration systems.

The power of activated carbon is its tremendous surface area, which is more than any other known material: one gram of AC has a surface area of a football field. Much of this surface area is due to a microscopic labyrinth of slits, canals, worm-holes and other terms used to describe this porous structure. A common term used to describe these narrow passageways is "pore." Eventually, AC filters lose their ability to adsorb or-ganics and with no more available space in the AC to accommodate additional adsorbates, breakthrough occurs. Organics in the aqueous phase pass through the AC into the effluent, instead of being trapped and removed from the water. A device has been developed that's both a thermometer and a reservoir to hold a hydrocarbon solvent. This simple device takes advantage of the fact that heat is given off when AC takes up organics into its pores, called an exothermic phenomena or exotherm. An exotherm is directly proportional to the available space in the AC. The volume of this adsorption space decreases with use of the AC adsorber. Simply measuring the heat rise with the thermometer after immersing the AC sample into the solvent provides a quick estimation of the remaining adsorption system life. A "control" group of unused AC is used to estimate the original adsorption space, by measuring the heat of immersion in this simple device. Typically, a used AC adsorber which gave no temperature rise when a sample of spent AC was put into the AC tester would need to be changed, because it is exhausted. Many uses for the AC tester have been reported to the authors for the last six years.

Figure 2. Molecular Structure of Coal-Based Activated Carbon



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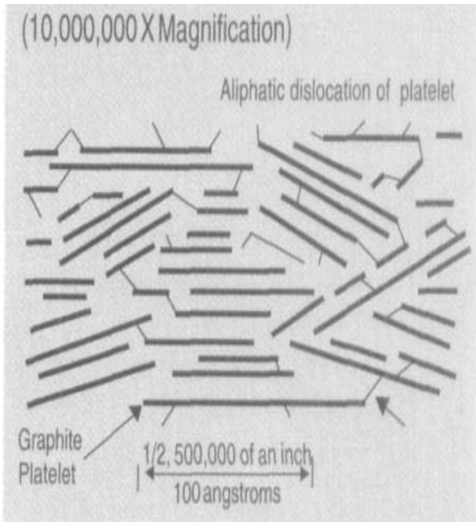
History and operation

During the first International Activated Carbon Conference (IACC) in Pittsburgh, Pa., in 1993, Dr. Milton Manes—a professor emeritus at Kent State University with 30 years of AC experience and over 100 published works—suggested the measurement of adsorption heat might be a good way to determine the quality of an AC specimen.[1] Professional Analytical "Consulting Service (PACS) became interested in Manes hypothesis, seeing this idea as a solution to several clients' problems. PACS's clients desired a quick and easy way to estimate the remaining adsorption service life of working AC systems. PACS subsequently

Figure 3. MolecularStructure of Coconut-Based Activated Carbon



spent the next five years developing and commercializing this method.

A schematic of the Model 1 AC tester is shown in Figure 1. This solvent container has graduated volume marks and is filled to the 30-milliliter (ml) line with mineral oil. The thermometer has a range of 10-to-30”C, with 0.5 degree-calibrated lines, was compared to a primary standard thermometer calibrated at the National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards (NBS). The indications of this thermometer are traceable to N1ST. The development of a Model 2 version based on hand-held computer technology will eventually be introduced.[2]

The AC tester

As mentioned earlier, when AC adsorbs material from the aqueous or gas-phase, heat is given off. This heat is a result of the Van der waals forces between the AC surface and the adsorbates concentrated in the pores, resulting in a decreased capacity from the original AC.The remaining capacity is the main interest of most users of AC.

Johannes D. Van der Waals, 1837-1923, was the first person to describe the weak attractive force between atoms or non-polar molecules caused by an instantaneous dipole movement (attraction) of one atom or molecule inducing a similar, temporary dipole movement in adjacent atoms or molecules. Activated carbon adsorption is viewed as advanced liquification, one example being when non-polar butane gas molecules are passed through AC, the atoms in the AC create and instantaneous dipole (plus and minus parts in the butane molecule due to dislocation of electrons in the original non-polar butane molecule) which results in attraction between each other through electrostatic interactions. Concentrations of butane molecules in AC pores result in liquification, i.e., separated molecules in the gas become close neighbors as liquid in the AC pores.

The heat phenomenon used in this tester was developed using hydrocarbons as the solvent to immerse the AC specimen into. Hydrocarbons used include mineral oil, diesel fuel and hexane. Hydrocarbons were selected as the immersion solvent because they don'thave strong competitive displacement of existing adsorbates on the AC surface, i.e., hydrocarbons don't replace existing adsorbates; they only fill remaining free space in the service AC. The AC tester was designed to help clients evaluate virgin as well as partially used or spent AC samples.

The development

Prior work in our laboratory over the last five years has shown the AC tester to be a useful tool. It can be used as a portable analytical instrument or in conventional laboratory environments. When testing granular AC, the amount of adsorption heat does not depend on either the particle size or the amount of water in the AC. Different particle sizes and water contents of the same commercial AC have given the same temperature rise at our lab.[3]

By mixing varying amounts of spent AC with virgin AC, a series of control mixtures was obtained, varying from 5 percent (spent) to 100 percent (exhausted).[4] The temperature rise of these synthetic control samples in the tester was directly proportional to the remaining adsorption capacity of the immersed sample.

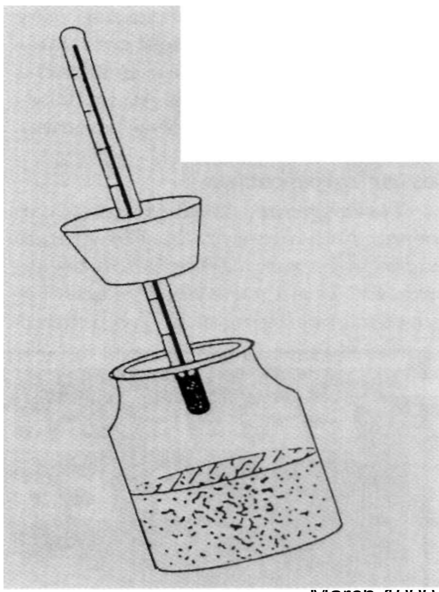
Temperature rise in the tester depends on the porous structure of the AC. Typically, coconut shell AC will give a higher rise than bituminous AC, because there is more surface area for adsorption. In Figures 2 and 3, we
show theCalgon Carbon model for the porous nature of coal and coconut shell-based AC.

Activated carbon can be manufactured from a wide variety of high-car-bon-percenased raw materials. Common materials with a high percent of carbon used are coal, coconut shells, wood and nutshel is. I empera ture rises in the tester are typically 2-to-4:C in 1-to-5 minutes for virgin AC samples; partially spent AC samples give less temperature rise. Carbon that's completely exhausted does not give any temperature rise when evaluated in the tester.

It's important to equilibrate— bring into balance or counterbalance—the temperature of the AC tester and test sample before use. If the AC sample is exceptionally warm or cold, this will change the solvent temperature when the sample is added to the tester. In typical field operations, a level teaspoon full of

AC is placed in the tester when a weighing balance is not available.

It's best to weigh 2-to-2.5 grams of the sample to the nearest 0.1 -gram. It's also best to avoid any changes in solvent tempera ture for any reason besides true adsorption heat and it's important to have a tester as a control to determine where



ambient temperature may be quickly changing. The amount of adsorption heat correlates with the degree the carbon is spent,; a common term used in the AC industry to indicate the amount of exhaustion.

Tester applications

Testing your activated carbon can answer such questions as: How much longer can I use my carbonbefore breakthrough? What's the relative adsorption efficiency rating of AC from different vendors? Is the AC being equally used indifferentparts of the adsorber? Is the AC received today as good as last year's? Where's the mass transfer zone (MTZ), located in operating activated carbon beds? Screening AC samples with this tester also reduces extensive testing. For example, if you have a large AC shipment which needs over a 100 iodine number determinations (which can take several hours each), using the AC tester (which takes minutes) can reduce the number of iodine number determinations

An AC client recently needed to obtain information about the amount of base and acid on virgin and service carbon samples.' AC can be impregnated with acid or base to control emissions such as hydrogen sulfide or ammonia. The strategy is to convert a toxic gas to its salt and water on the AC surface. The samples and appropriate controls were then immersed in water instead of a hydrocarbon solvent in the AC tester (acids and bases generate heat when dissolved into water). The base virgin AC, without any acid or base added to AC, doesn't produce any heatin the tester. Immersion heats were directly proportional to the remaining acid and base on the samples. The carbon itself doesn't produce any heat

when immersed in water because water has negligible adsorption capacity. This modification to the tester helped solve the client's problem. The MTZ could be defined in the working AC adsorption systems.

Industry associations and trade organizations also have a need for an AC tester. The American Society for Testing and Materials (ASTM) has a group of laboratory tests used by the AC industry. The ASTM methods are designed for clean AC, i.e., newly manufactured virgin AC or spent AC that has been regenerated back to new AC. The tester is designed for use with new or partially used AC.

The test results need to be evaluated and used by a professional understanding the workings and prior history of the AC adsorption system under evaluation. The AC tester is a new tool in the toolbox of the water professional. The tester is at minimal cost, and can be used many times by a technician; the cost for the tester is the equivalent to what some labs charge for a single iodine number.

Conclusion

The development of this AC testing device helps users answer the common question: How much longer can 1 use my activated carbon before its ad

Service Time Calculations

The calculation for relating the heat of immersion to the estimated remaining service time (ERST) of the activated carbon can be expressed as:

ERST= Temperatureise from rise

Temperatuerise of Virgins

A typical set of heat of immersion numbers is a four degree rise from the virgin or unused sample of carbon and a two degree rise from the service or partially used sample. Rationing these two heat of immersion temperature rises in the formula above gives 0.5. Thus it is logical that the service activated carbon sample has about half its original porous volume filled; i.e. it has an estimated remaining service time of about the same amount of time and conditions (volume and concentration of historical contaminants) up until the sample was obtained, to bring the heat of immersion for the service sample to 0° C**.**

It's good practice to report the ERST as plus or minus 10 percent to indicate to the user of your calculated ERST that it is not a precise number. Information provided by the AC tester is designed for use by an expert who knows the specific activated carbon application. Determining the relative heats of immersion can provide valuable information about when the carbon needs to be replaced.

sorptive qualities are exhausted? Benefits of the device include a low cost and use in the field by a technician that does not require a lot of knowledge about AC. However, it's recommended some training courses on activated carbon adsorption be taken by all technicians. The knowledge gained is not only a benefit to an AC consumer but would also be useful in an ongoing schedule of professional career development.

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About the authors

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