Carbon adsorption for rinse water purification in the precision cleaning of printed circuit boards can be optimized through basic guidelines.

A Guide to Activated Carbon for Semi-Aqueous Processing

BY REX L. BREUNSBACH

The role of activated carbon in semi-aqueous processing is to remove organic impurities from the water used to rinse parts after their exposure to an organic solvent wash. This is an alternative to separation of organic and other materials by membrane action (filtration or reverse osmosis).

In order to get optimal results from the use of activated carbon, the limits of adsorption and general characteristics of activated carbon must be understood. However, it must be emphasized that the application guidelines that follow are very basic, while adsorption mechanisms can be very complex.

What Is Activated Carbon?

Activated carbon, also called activated charcoal or filter carbon, is a nearly chemically pure amorphous form with a coal-like porous structure. It is produced by carbonizing coconut shells, peach pits, hardwood, sawdust, lignite, peat, bituminous coal, or petroleum coke. Subsequent activation treatment is accomplished with steam, chemical dehydration, or chemical reaction.

The type of activation applied determines the carbon's adsorption characteristics, especially the inner surface (adsorption) area, density, pore volume, pore size, and pore distribution. Active (inner) surface areas are 500 to 1800 square meters per gram of carbon.

Pore sizes are typically between 20 to 500 Angstroms, but larger sizes may be encountered. Pore sizes <20 A are called micropores; pores in sizes >1000 A are referred to as macropores, while sizes between those ranges are called mesopores or transitional pores.

The Way It Works

The basic mechanism is the selective adsorption of certain molecules to the inner surface of the carbon as a result of molecular attraction.

The capability of a certain activated carbon grade to adsorb, called the adsorptive capacity, depends not only on the inner surface area, but also on the molecule types that are to be adsorbed for optimal pore volume and pore sizes encountered. In order to be adsorbed, the molecules must be able to enter and wet the pores and there must be attraction between the molecules of the liquid and the carbon surface.

As a general rule, branched chain molecules with low or no polarity will be preferably adsorbed, while polar materials and those with straight chain structure will less likely be adsorbed. Larger molecules also will be more likely adsorbed than smaller ones.

Other factors such as surface tension, liquid temperature, contact time, and pH influence adsorption as well. Higher temperatures usually slow down adsorption. Low pH promotes adsorption of organic acids, while high pH promotes adsorption of organic bases.

Growth of microorganisms also plays a role in adsorption. Anaerobic conditions (idling period with no flow) will foster growth of anaerobic organisms. While presence of small amounts of these organisms may be considered beneficial, excessive growth can plug up pores and reduce the adsorptive capacity.

Solution flow through the carbon filter is another important factor, as a rapid flow will yield reduced adsorption. As a rule of thumb, the amount of carbon needed (gallons of volume) is calculated by multiplying the flow rate in gallons per minute (gpm) by a factor of 10. At a 3 gpm flow, for example, 30 gallons (4 cubic feet) of carbon is needed.

Selection Suggestions

Proper selection of carbon type and grade factors heavily in final results. Your carbon supplier is probably the best source of help in this process.

Activated carbon is available in granular or powder forms, the latter being lower in cost though messier to handle. Both types can be regenerated, but this may change pore sizes, distribution, and density among other properties. Commonly, usage of less than 400 pounds per day does not warrant regeneration.

Two grades of activated carbon are in general use: one for purification of gases, the other for purification of liquids. For gas purification, the grade with the highest pore volume in the micropore range is used, while liquid purification typically employs the grade with the highest pore volume in the transitional pore size range.

Optimum carbon type and grade selection will depend on what is being adsorbed. You'll want to specify a granular grade, rated for use with liquid, and also consider (from analysis) the composition of process water for selection of optimal surface area, pore size, and volume for specification refinement.

Performance Assessment

While continuous monitoring of water quality with regard to ionized impurities and performance of ion exchange beds is an easy task to perform with electrical resistivity or conductivity probes, carbon filter performance assessment is more complex.

NASA has used very expensive instrumentation to continuously measure total organic carbon in water recycling. Currently, however, this concept appears impractical for common industrial processes. Yet, if carbon filters are not monitored, total saturation occurs and their performance will drop suddenly. Hence, periodic "organic passage" checks are necessary.

The frequency required for these checks will become apparent when the user is "tuned in" on a given process scenario. Changes in materials and process conditions may require re-establishing the required test frequency.

Tests commonly employed for this purpose include total organic carbon (TOC), chemical oxygen demand (COD), and biological oxygen demand (BOD). The latter requires several weeks, because of the inherent incubation time needed. Therefore, BOD tests are typically used for initial characterization only.

Figure 1 displays typical performance of a two-filter carbon filtration system. CODs were measured and plotted as a function of how much organic material was adsorbed. Measurements were taken from the outset of the first and second carbon filter. As illustrated, first carbon COD levels climb slowly until 7600 ml was adsorbed, then they climb rapidly. Output of the second carbon stays well under the 50 ppm level.

For best carbon filter performance, the following is recommended:

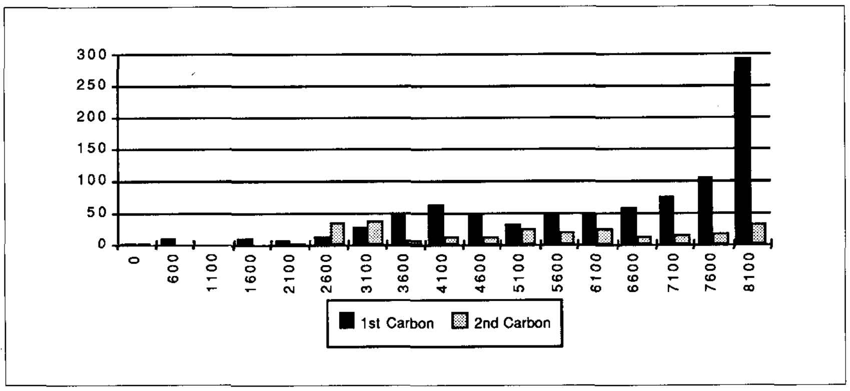
- Measure COD or TOC periodically to catch "break-through''(sudden drop in adsorption at exhaustion point). Inexpensive COD test kits are available for such detection. More accurate COD and TOC tests can be performed by local commercial water analysis laboratories. These tests should be done initially during a pilot run to determine if carbon filtration is effective for the given process scenario.
- Employ two carbon filters and run routine COD tests. Take samples after the first filter. When the first filter breaks through, effective carbon action is assured by the second. Filters are then rotated: After breakthrough in the first filter, the back-up filter is changed to the number one position and the second filter is replaced with a new carbon filter.

Prolonging Filter Life

High amounts of adsorbable organics will reduce filter life significantly. In order to reduce the expense of new filter media, labor required to change filters, and process "down time," the following should be observed:

- Minimize drag-out losses by mechanically removing organics before rinsing.
- Perform efficient phase separation to remove as much as possible of the organic solvent and other organic materials. The aqueous phase may be separated from the organic solvent phase through centrifugal gravity, followed by decanting.

Figure 1
Checking Carbon Filter Performance



- Avoid process compounds and temporary soldermasks containing very fine particles of silica, used as thixotropic agents. These particles are in the submicron range, and masks also have a high sodium content that will reduce ion exchange media life. Nanofiltration is recommended for such process scenarios.
- Minimize organic materials introduced by soldering and peripheral assembly operations.
- Provide adequate mechanical filtration before solution entry into carbon to avoid "carbon plugging" by particles. As fine carbon particles could be dragged from the carbon filter in the ion exchange beds, the installation of a fine mechanical filter will be helpful.
- Avoid use of hard water or water with high solids content.
- Avoid prolonged anaerobic conditions (non-moving liquid) in carbon.

Source Reduction

While older process scenarios had relatively few restrictions with regard to quantities of soils and other foreign matter to be cleaned, increased awareness of waste management now demands minimization or elimination of contaminant sources.

Effectively addressing these new demands may require a restructuring of the processes used. The cost of these efforts often is quickly amortized by the savings realized from reduced overall process costs.

Following are some basic tips for reducing organic materials in process water during the manufacture of printed circuit boards:

- Solder flux: Avoid high-solids fluxes. Use the optimal amount. Use spray fluxers instead of wave types. Use fluxes that do not impair phase separation, as high amounts of surfactants will cause emulsions to form and increase separation time. Overall, optimize flux selection with the separation technology employed.
- Decanting: Perform effective decanting before process water enters the filtration system. Evaluate the separation characteristics of the semi-aqueous system to be used; select one that separates fast. (The presently available semi-aqueous systems vary considerably from brand to brand in this regard.) Allow enough time for phase separation, and minimize turbulence in the decanter.

- Temporary (dissolvable) soldermasks: Avoid if at all possible, or confine screening to those portions that must be masked. Alternatives include peelable masks (for small process volume and assemblies that require only minimal masking) or boards with buried vias. Design boards without need for masking.
- When baskets are employed, choose mesh size as coarse as possible. Fine mesh will retain organic solvent and yield high organic solvent drag-out levels. If possible, avoid using the same baskets for water rinsing and solvent cleaning.
- Wave oil or solder shield fluid: Use materials that are non-water-soluble and easy to separate by decanting or using dry wave without shield.
- In-line lead cutting: Use component stabilizers that are easy to separate. Avoid excessive build-up or use “solder-cut-solder’’ process.
- Permanent soldermask: Avoid use of undercured soldermask, as mask constituents can be extracted by wash. Evaluate soldermask type/brand for retention of the cleaning medium. Porous masks, masks with rough surfaces, and those having a high surface energy level will retain by absorption and adsorb more wash medium — hence, drag-out will be higher.

Design for Process and Environment (DFPE)

This new trend in design philosophy addresses an optimal balance between performance of the manufactured product, producibility, and considerations for minimizing the process’s environmental impact. A product‘s design can dictate potential processes, which in turn influence environmental impact as well as overall manufacturing Costs.

For example, cleanability must be designed into a printed wiring assembly, providing free-flowing wash and rinse through spacings between component and board surfaces.

A cleaning process should be chosen to yield the required degree of cleanliness without damage to the assembly, at an acceptable cost and with minimal waste. In order to increase semi-aqueous process efficiency, design and packaging engineers should consider the following:

- Avoid geometries that may entrap cleaning media, causing excessive drag-out. These include open components, crevices such as unfavorable stand-off dimensions (<5 mil) that hold solvent by capillary action, certain connector types, as well as insulated wire and cable.
- Specify materials that are compatible with the semi-aqueous process used. Certain kinds of materials tend to adsorb semi-aqueous cleaners and are harder to rinse: others are dissolved or attacked by semi-aqueous agents.
- Specify contamination controls to eliminate handling and storage soils.
- Specify that received boards shall meet ionic cleanliness standards, e.g. MIL-C-28809, and shall be free of organic contaminants when tested, e.g. IPC 2.3.38 of Test Manual TM-650.

Advice for Advantage

Many factors affect the performance and efficiency of carbon adsorption systems in semi-aqueous processing. With the aforementioned considerations effectively addressed, the organic impurities in the rinse wall will be significantly reduced and carbon filter life optimized.

About the Author

Rex Breunsbach, founder and president of Electronic Controls Design (Milwaukie, OR), has 25 years experience in the design and manufacture of electronic instrumentation for medical and industrial processes. Recent work focuses on batch aqueous and semi-aqueous closed-loop cleaning systems for electronic assemblies and precision metal parts.