

# Activated Carbon Adsorption for Wastewater Treatment

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## INTRODUCTION

This volume is a guide to the state of the art of activated carbon adsorption technology as applied to wastewater treatment. Current technology in this application is approximately 15- to 20-years-old. After a period of rapid changes, as real-life applications provided feedback, the technology has developed a body of knowledge and experience which serves as a basis for future applications. This book surveys this body of knowledge and is a detailed description of current technology.

A review of the Table of Contents will reveal the structure utilized. Subjects include: wastewater characterization; theoretical concepts of adsorption; physical and chemical properties of activated carbon and how they relate to adsorption phenomena; manufacture and regeneration of activated carbon; analyses and characterization parameters of activated carbon; adsorption test procedures; factors affecting adsorption phenomena; isotherm development, analysis, and interpretation; analytical technique; development of design parameters from lab test data; field evaluations; contacting processes and equipment; regeneration processes and equipment; integrated total process designs; economic analysis of activated carbon treatment; design of equipment components; common operating problems and solutions; and safety aspects. Design examples are utilized to demonstrate applications. Case histories are not presented, however, Appendix I includes case history reference sources for the interested reader.

Addition of powdered activation carbon to biological treatment systems is not addressed. This use of activated carbon is primarily an adjunct to biological treatment to enhance treatment effectiveness. It is not a utilization of activated carbon as a treatment process in its own right, which is the subject of this book. This exclusion is, of course, a matter of editorial discretion.

Each chapter is designed to be and can be read alone and understood. There is a minimum of interdependence on other chapters and therefore selected chapters can be addressed without the prerequisite of reading prior chapters. In using such an approach, there is naturally some duplication of material among different chapters. This is not necessarily undesirable as repetition begets learning. More important it allows the reader to approach a topic from several authors individual viewpoints, thus fostering a more balanced and complete comprehension.

The reader may detect some contradictions among authors. In a compiled book of this nature it is inevitable that some conflicts will be apparent. These conflicts have been minimized where possible, but some remain. These represent legitimate differences of opinion among experts. Certainly an author is allowed his interpretation or viewpoint when issues are unresolved or in areas of knowledge in which the final answers are not yet known. This exposes the reader to a variety of approaches rather than the dogma of a selected viewpoint.

A reading list is provided in Appendix I for those interested in sources of information regarding activated carbon adsorption technology. Appendix II is a list of U.S. suppliers of activated carbon.

It is a pleasure to acknowledge those who assisted in the preparation of this book, particularly the people at E. I. du Pont de Nemours and Co., Inc. under whose auspices I researched and coordinated this material. The authors are especially commended for the contribution of their expertise as well as their time and effort in preparing their chapters. Among those who provided secretarial support, I especially thank Mrs. Alberta Whitlow for her patience, diligence, and unflinching efforts in correspondence and manuscript preparation. Finally I am grateful for the interest, encouragement, and support from my family and especially my wife Jane.

Jerry R. Perrich

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# DEDICATION

To Jane, my wife.

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## Chapter 1

## WASTEWATER CHARACTERISTICS AND TREATMENT

D. L. Ford

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## EDITOR'S SUMMARY

A wastewater stream must be defined and characterized with respect to flows and composition before realistic treatment alternatives may be considered. Several common parameters and analytical techniques are discussed. Activated carbon adsorption is one of many potential wastewater treatment technologies and is suitable under certain conditions. Factors influencing the selection of activated carbon adsorption as a treatment alternative are discussed.

### I. CHARACTERISTICS OF WASTEWATER

The characterization of wastewaters, both quantitatively and qualitatively, is the logical beginning for evaluating process applicability. A brief discussion of this characterization program is described as follows.

#### A. Design Flows

The volume of wastewater to be treated, either municipal or industrial, is paramount in indicating not only the hydraulic design features of a treatment facility, but also the pounds of pollutants to be removed and the unit process size requirements based on kinetics of this removal. The development of design wastewater flows for municipalities and industries are discussed individually.

#### B. Municipal Wastewater Flow

The volumes of municipal wastewater flow have traditionally been projected on a per capita basis, making allowances for the maximum hourly quantity of connected flow as well as infiltration principally attributed to storm runoff. The guidelines for selecting the per capita flow and infiltration contribution should be sufficiently flexible to reflect local conditions (e.g., geographical location, economic structure, water use history of the area to be sewered, and the routing of the conveyance system with respect to location of storm runoff). The general guidelines established by the Texas State Health Department, for example, provide a residential per capita contribution of 80 to 200 gal/day. This suggests an infiltration allowance of 500 gal/day/in. diameter of pipe per mile, and indicates peak design flow to be 250% of average flow when considering primary mains and interceptors.

#### C. Industrial Wastewater Flow

The design flows for industrial complexes, as graphically illustrated in Figure 1 generally consisting of the following:

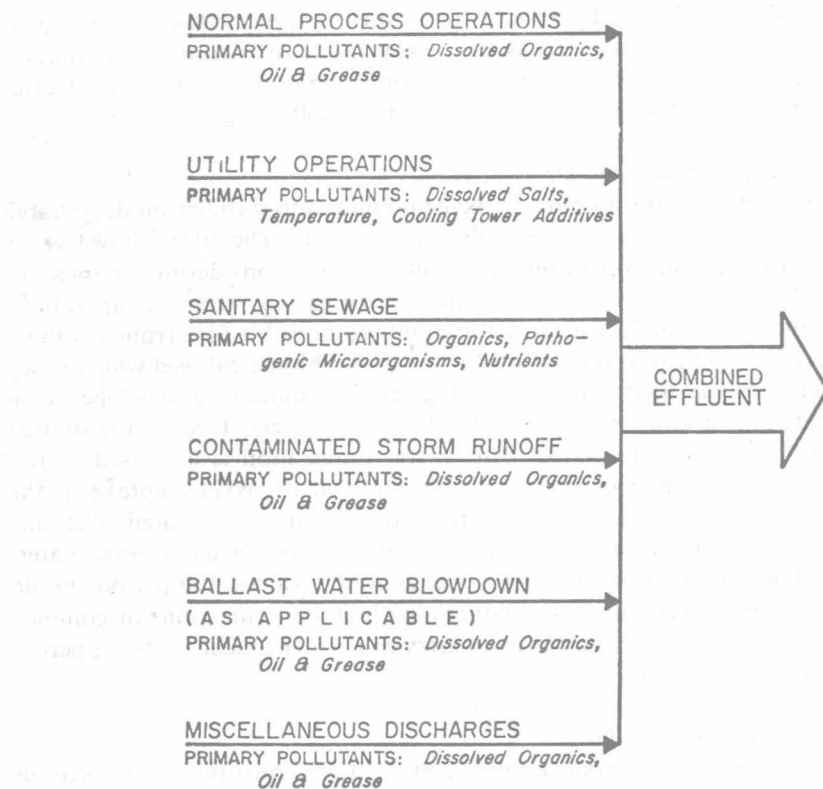


FIGURE 1. General sources of wastewaters.

1. Base process flows resulting from normal production operations
2. Sanitary sewage
3. Contaminated storm runoff
4. Other sources (extraordinary dumps, tank draining, ballast discharge, spills, etc.)

The base flow and sanitary contribution can be measured in open channels or closed conduits using a variety of methods such as automatic metering devices, weirs, or less sophisticated equipment. Care should be taken to insure flows are measured during workday and weekend operations, different work shifts, and during sufficiently long periods of time to reflect the statistical reliability.

Within the last decade, contaminated storm runoff has become an area of increasing concern within industrial complexes. Storm flow is intermittent and unpredictable in nature, and little data has been collected to typify its characteristics. The level of flow and degree of contamination varies with time during the course of the storm, as well as with each individual area within an installation, as each has its own geometric characteristics which influence patterns of surface runoff.

#### D. Composition of Municipal and Industrial Wastewaters

Municipal or domestic effluents contain soluble, suspended, and colloidal organic materials, primarily of human origin, which exert a deleterious impact on receiving bodies of water unless properly treated. Most of these constituents are biodegradable and sorptive, inferring that biological systems and activated carbon are applicable methods for treatment. Conversely, industrial wastewaters contain a myriad of organic

and inorganic pollutants, the removal of which depends on many factors. For this reason, biological and/or activated carbon systems must first be evaluated in terms of process efficiency before being selected as the proper mode of treatment. A comprehensive wastewater characterization analysis is the first step in this evaluation. A brief review of the analytical parameters is thus discussed in the following narrative.

### 1. Biochemical Oxygen Demand (BOD)

The BOD is an estimate of the amount of oxygen required to stabilize biodegradable organic materials by a heterogeneous microbial population. The BOD, however, is subject to many variables and constraints, particularly when considering complex industrial wastes. These include time of incubation, nitrification effects, temperature, pH, and seed acclimation. The factor most commonly responsible for erroneous BOD results, particularly for industrial wastewaters is the use of biological seed which is not properly acclimated to the wastewater. A biological seed should be developed in a continuous or batch laboratory reactor (preferably the former), feeding the diluted wastewater to the initial microbial seed. The waste composition is increased to full strength over a period of time; once the organic removal or oxygen uptake in the reactor reaches the maximum level, the seed can be considered as acclimated. The time required to obtain this acclimation depends on the nature of the seed and wastewater. For domestic wastewaters or combined industrial-domestic wastes, the period should be less than 1 week. However, for wastes containing high concentrations of complex organic compounds such as those present in refinery or petrochemical wastes, a period of several weeks may be required.

### 2. Chemical Oxygen Demand (COD)

The COD is a measure of the oxygen equivalent of those constituents in a sample which are susceptible to permanganate or dichromate oxidation in an acid solution. Although it is independent of many of the variables which affect the BOD test, there are still factors which influence the COD value of the sample in question.

Generally, one would expect the ultimate BOD of a wastewater to approach its COD value. There are several factors, however, which prevent a consistent  $BOD_{ult}/COD$  ratio of unity. These include:

1. Many organic compounds are dichromate or permanganate oxidizable but are resistant to biochemical oxidation.
2. The BOD results may be affected by lack of seed acclimation, giving erroneously low readings.
3. Certain inorganic substances such as sulfides, sulfites, thiosulfates, nitrates, and ferrous iron are oxidized by dichromate, creating an inorganic COD.
4. Chlorides interfere with the COD analysis (but provisions have been made to eliminate this interference).

### 3. Total Organic Carbon (TOC)

Although TOC is a parameter that has been applied in the field for many years, the advent of the carbon analyzer has provided a rapid and simple method for determining organic carbon levels in aqueous samples, enhancing the popularity of TOC as a fundamental measure of pollution. The organic carbon determination is free of the many variables inherent in COD or BOD analyses, with more reliable and reproducible data being the net result. As the analysis time using the carbon analyzer is only several minutes, the efficacy of using this parameter is apparent particularly when a TOC-COD or TOC-BOD correlation can be established.

#### 4. Total Oxygen Demand (TOD)

Another analyzer has been developed to measure the amount of oxygen required to combust the impurities in an aqueous sample. This measurement is achieved by providing a continuous analysis of the oxygen concentration present in a nitrogen carrier gas. The oxidizable constituents in the liquid are converted to their stable oxides in a platinum catalyzed combustion chamber. This disturbs the oxygen equilibrium at a platinum surface which is restored by the oxygen in the carrier gas stream. This depletion is detected by a silver-lead fuel cell and is recorded as a negative peak related to the oxygen demand of the sample.

#### 5. Oil and Grease

One of the more important parameters applied in characterizing many industrial wastes such as refinery and petrochemical wastewaters is the oil and grease measurement. This is particularly true since oils have both a recovery value and create problems in treatment unit processes. Therefore, oil separation and recovery facilities are required for all oily wastewater streams.

Extraction techniques using various organic solvents, such as n-hexane, petroleum ether, chloroform, and trichloro-trifluoro-ethane are used to evaluate the oil and grease content of wastewaters. The method outlined by the U.S. Environmental Protection Agency (EPA) measures hexane extractable matter from wastewaters but excludes hydrocarbons that volatilize at temperatures below 80°C. Additionally, not all emulsifying oils are measured using these extraction techniques. However, a modified procedure provides for the release of water-soluble oils by saturating the acidified sample with salt followed by isolation on the filter in the accepted manner.

#### 6. Phenolic Compounds

Phenols and related compounds are generally prevalent in refinery and petrochemical wastewaters and are of particular significance as they are potentially toxic to marine life, create an oxygen demand in receiving waters, and impart a taste to drinking water with even minute concentrations of their chlorinated derivatives. Primary sources of phenolics are in wastewaters from benzene refining plants, oil refineries, coke plants, chemical operations, and plants which are processing phenols to plastics.

Phenols, or the hydroxy derivatives of benzene, are measured using the distillation approach or by other miscellaneous colorimetric, spectroscopic, or chromatographic techniques.

#### 7. Acidity and Alkalinity

The acidity of a wastewater, or its capacity to donate protons, is important because a neutral or near-neutral water is required before biological treatment can be deemed effective. Many regulatory authorities have criteria which establish strict pH limits to final discharges. Acidity is attributable to the un-ionized portions of weak ionizing acids, hydrolizing salts, and free mineral acids. The latter is probably the most significant as it is difficult to predict neutralization requirements when mineral alkalinity, or the ability of a wastewater to accept protons, is significant in the same general sense as acidity and can affect both biological systems (as a buffer) or carbon systems (possibly affecting the rate phenomenon). The impact, if any, of acidity or alkalinity should therefore be assessed before process design is finalized.

#### 8. Suspended Solids

Organic and inorganic solid matter held in suspension in the wastewater is usually measured via a filtration and drying technique and described as Total Suspended Solids

(TSS). These solids are normally removed from a wastewater by settling or filtration in a pretreatment step prior to the major organic removal treatment process.

### 9. Dissolved Solids

The dissolved solids can have a pronounced deleterious effect on many unit processes included in the waste treatment system. This is true for both biological and carbon systems. Although municipal effluents do not present a problem in this regard, industrial wastes with total dissolved solids (TDS) levels of 3000 mg/l and higher should be tested with respect to evaluating this impact on process performance.

### 10. Ammonia Nitrogen and Sulfur Compounds

Ammonia nitrogen is present in municipal wastewater in the range of 15 to 40 mg/l, although industrial streams often contain exceedingly high concentrations. Biological systems are sensitive to ammonia nitrogen, although carbon systems can tolerate rather high concentrations. Little ammonia removal occurs using activated carbon, although its presence has not been reported to adversely affect the removal of other constituents.

Municipal wastewaters contain sulfates, the exact concentration varying to some extent depending on the carrier water. Industrial effluents cover the whole spectrum of sulfur compound concentrations, depending on the source. The important thing to recognize is that the biochemical reduction of sulfates to sulfides is the precursor to many process operational problems, either in an anaerobic biological system or in a fixed-bed granular carbon column. Evaluating the sulfur content of a wastewater is then an integral part of the wastewater characterization program.

### 11. Heavy Metals

The influence of heavy metals on biological and activated carbon unit processes has been the subject of many investigations particularly in light of the "priority pollutant" emphasis by the regulatory authorities pursuant to PL 92-500 and the settlement agreement between the EPA and the Natural Resource Defense Council (NRDC).

Several techniques for heavy metal analysis are given in *Standard Methods* although atomic adsorption flame photometry is an effective and rapid method for determining small quantities of metals. This method is based on the measurement of a light adsorbed at a given wavelength by the unexcited atoms of the element being analyzed.

Heavy metals in municipal wastewaters are normally attributed to the carrier water, while those in industrial effluents can be traced to instrumentation leaks, process catalysts, corrosion products, and many other factors.

## E. Summary

It is apparent that the volume and characteristics of municipal and industrial wastewaters vary markedly, and can affect the concept and design of efficiencies such as activated carbon systems in different ways. In summary, BOD, by definition, is an expression of the impurities in oxygen equivalents which can be degraded biochemically. These constituents may or may not be removed by activated carbon. Therefore, the removal of BOD or other parameters such as COD, TOC, or TOD by activated carbon should be realistically assessed before committing to a process concept. This is particularly true of industrial wastes. The first step in any water pollution treatment program is thus defining the characteristics of the wastewaters in question, using the aforementioned and other parameters, establishing the applicability of a unit process such as activated carbon, then predicting performance levels, effluent quality, and formulating process design criteria.

## II. CAPABILITY OF ACTIVATED CARBON SYSTEMS TO COMPLY WITH EFFLUENT GUIDELINE REQUIREMENTS

The applicability of the activated carbon process in treating municipal and industrial wastewaters is contingent on many factors, including the amenability of the dissolved constituents to sorption, the presence of other substances which enhance or impede the sorption process, the soundness of engineering, and proper operation and maintenance of the system.

It is recognized that carbon technology is changing continuously. It is imperative, therefore, that in evaluating carbon applicability, developing design bases, predicting effluent quality, and finalizing management decisions in terms of constructing carbon facilities with a corresponding commitment of capital, one must carefully predicate these judgments on the known process capabilities and limitations of activated carbon, particularly when pursuing Best Available Technology (BAT) or "priority pollutant" effluent requirements.

Some of the significant factors include the following:

1. Adsorption theory is rigorous for single solutes, but becomes less definitive when applied to wastewaters containing multiple components with varying molecular weights and chemical characteristics.
2. Batch isotherm studies are not necessarily indicative of continuous flow carbon treatment systems.
3. Many classes of organic compounds are not amenable to carbon adsorption — particularly oxygenated organics — and show up as residual BOD, COD, or TOC in carbon column effluents. This limits the overall process efficiency of activated carbon when treating many industrial wastewaters.
4. As effluent quality requirements become more stringent under the 1984 BAT guidelines, activated carbon will be considered as a candidate process, primarily through the selective treatment of individual process streams or as an effluent polishing process. It is imperative, however, that its limitations be understood when considering its application in terms of BAT quality requirements.

## III. THEORY AND PROPERTIES OF ACTIVATED CARBON ADSORPTION

### A. Theoretical Concepts

Molecules are held together by cohesive forces ranging from strong valence bonds to the weaker Van Der Waals forces of attraction. These attractive forces are satisfied in the solid phase interior molecules, having the ability to capture certain fluid molecules as they contact the surface. Van Der Waals forces are the bases for the adsorption of wastewater constituents onto carbon which has been activated to maximize this interphase accumulation of liquid constituents at the surface of interphase of the solid phase.

The rate at which substances are removed from the liquid phase (adsorbate) to the solid phase (adsorbent) is of paramount importance when evaluating the efficacy of activated carbon as a wastewater treatment process. Unfortunately, the task of quantifying the many forces acting at the solid-liquid interface is a formidable one. Developing a mathematical expression which describes the dynamic phenomenon occurring in a continuous-flow/fixed-bed reactor has been difficult because of multivariable influences. The overall adsorption rate represents the combined effects of diffusion



through a laminar layer of fluid surrounding the constituent, surface diffusion, and adsorption on the internal pore surfaces. Most mathematical solutions for equations which describe concentration/time profiles are limited to the special case in which only one of these phenomena controls the overall rate of adsorption.

One expression for the continuous-flow regime assumes the diffusion of the constituent through the liquid phase and through the pores of the carbon (which are rate-limiting), then combining these resistances in an overall mass coefficient term. Using this rationale:

$$q \frac{dc}{dM} = \frac{k_2 r}{x} (C_s - C) \quad (1)$$

where:  $q$  = flow rate,  $C_s$  = concentration of the adsorbate,  $D$  = adsorbent bed depth,  $C$  = equilibrium adsorbate concentration, and  $K_2 r$  = overall mass transfer coefficient.

A more convenient expression of Equation 1 is in terms of the adsorbate rate with respect to the weight of the carbon in the columns:

$$q dC/dM = k_2 \frac{r}{x} (C_s - C) \quad (2)$$

where:  $M$  = weight of the carbon in the column, and  $X$  = packed density of the carbon in the column.

Another proposed model predicts four successively decreasing adsorption rates would be observed as the adsorption proceeds to equilibrium. The initial rate would be limited by the rate of adsorbate transfer across the film layer, film diffusion, or, if sufficient turbulence existed, control would be exerted by the combined rate of external surface adsorption and macropore filling. After the external surface adsorption capacity was exhausted, there would exist three secondary adsorption rates controlled, respectively, by the filling of the macropore (an effective radius of 5000 to 20,000 Å), the transitional pore (20 to 100 Å), and the micropore (10 to 20 Å effective radius). This model is illustrated in Figure 2. It is inherent in this model that the intraparticle transport occurs as a series of adsorption/desorption steps, each linear with respect to time, and their summation resulting in a time/linear function.

The development of adsorbate removal kinetics on a batch basis can be used to approximate carbon effectiveness and predict organic residual levels. The adsorption isotherm is used for this purpose and is defined as a functional expression for the variation of adsorption with concentration of adsorbate in bulk solution at a constant temperature. The isotherm is expressed in terms of removal of an impurity — such as BOD, COD, and color — per unit weight of carbon as a function of the equilibrium impurity remaining in solution. Linear plots as shown in Figure 3 can be expressed in terms of the empirical Freundlich equation. This expression relates the amount of impurity in the adsorbed phase to that in solution:

$$X_m = KC^{1/n} \quad (3)$$

where:  $X$  = amount of impurity adsorbed,  $M$  = weight of carbon,  $C$  = equilibrium concentration of impurity in solution, and  $K, n$  = constants.

The Freundlich isotherm is valid within the context of a batch test for pure substances and some dilute wastewaters. As shown in Figure 3 its application is limited in certain cases when a significant portion of the organic impurities are not amenable to sorption, resulting in a constant residual, regardless of the carbon dosage.

The constants “ $n$ ” and “ $K$ ” can be used to define both the nature of the carbon