

## 1

# Water and Wastewater Treatment: Historical Perspective of Activated Carbon Adsorption and its Integration with Biological Processes

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## 1.1

## Historical Appraisal of Activated Carbon

Activated carbon is broadly defined to include a wide range of amorphous carbon-based materials prepared in such a way that they exhibit a high degree of porosity and an extended surface area [1]. Moreover, all non-carbon impurities are removed and the surface is oxidized. Although today the term ‘activated carbon’ is taken for granted, a long time elapsed before it became generally adopted.

The use of activated carbon in its current form has only a short history. On the other hand, according to records, the use of carbon itself dates back to ancient times. The earliest known use of carbon in the form of wood chars (charcoal) by the Egyptians and Sumerians was in 3750 BC [2]. At that time, charcoal was used for various purposes such as reduction of ores in the manufacture of bronze, domestic smokeless fuel, and medicinal applications [3]. In Egyptian papyri dating from 1550 BC we find the first citation of the use of charcoal for the adsorption of odorous vapors – from putrefying wounds and the intestinal tract. The ancient Greeks used charcoal to ease the symptoms of food poisoning [4]. The beneficial effect was due to the adsorption of the toxins emitted by ingested bacteria, thereby reducing their toxic effects.

Hindu documents dating from 450 BC refer to the use of sand and charcoal filters for the purification of drinking water. Recent studies of the wrecks of Phoenician trading ships led to the discovery that drinking water was stored in charred wooden barrels in order to keep the water fresh [4]. In the time of Hippocrates (ca. 460–370 BC) and Pliny the Elder (AD 23–79) wood chars were employed for medicinal purposes [5]. In about 157 BC carbons of vegetable and animal origin were applied in the treatment of many diseases [2]. A Sanskrit text around 200 AD recommends the use of filtration of water through coal after storing it in copper vessels and exposing it to sunlight, providing probably one of the earliest documents describing the removal of compounds from water in order to disinfect it [6].

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In the fifteenth century, during the time of Columbus, sailors used to blacken the insides of wooden water barrels with fire, since they observed that the water would stay fresh much longer. It is likely that people at that time proceeded by intuition only, without having any insight into the mechanisms of the effect; these mechanisms were recognized beginning from the eighteenth century.

In the eighteenth century, carbons made from blood, wood, and animals were used for the purification of liquids. The specific adsorptive properties of charcoal (the forerunner of activated carbon) were first observed by Scheele in 1773 in the treatment of gases. Later, in 1786, Lowitz performed experiments on the decolorizing of solutions. He provided the first systematic account of the adsorptive power of charcoal in the liquid phase [7]. In those days, the sugar refining industry was looking for an effective means of decolorizing raw sugar syrups, but the wood charcoals then available were not particularly effective because of their limited porosity [4]. However, a few years later, in 1794, an English sugar refinery successfully used wood charcoal for decolorization. This application remained a secret until 1812 when the first patent appeared in England [2], although from 1805 wood charcoal was used in a large-scale sugar refining facility in France for decolorizing syrups, and by 1808 all sugar refineries in Europe were using charcoal as a decolorizer [4].

In 1811 it was shown that bone char had an even higher decolorizing ability for sugar syrups than wood char. Consequently, a switch took place from wood charcoal to bone char in the sugar industry. In 1817 Joseph de Cavaillon patented a method of regenerating used bone chars, but the method was not entirely successful. In 1822 Bussy demonstrated that the decolorizing abilities of carbons depended on the source material, the thermal processing, and the particle size of the finished product. His work constitutes the first example of producing an activated carbon by a combination of thermal and chemical processes. Later in the nineteenth century, systematic studies were carried out on the manufacture and regeneration of bone chars by Schatten in Germany and the application of charcoal air filters for removing vapors and gases in London sewers by Stenhouse [4].

In 1862, Lipscombe prepared a carbon material to purify potable water. This development paved the way for the commercial applications of activated carbon, first for potable water and then in the wastewater sector. In 1865 Hunter discovered the excellent gas adsorption properties of carbons derived from coconut shells. It is remarkable that the term 'adsorption' was first introduced by Kayser in 1881 to describe the uptake of gases by carbons [4].

Activated carbon was first produced on an industrial scale at the beginning of the twentieth century, and major developments then took place in Europe. However, at the beginning of the twentieth century activated carbon was only available in the form of powdered activated carbon (PAC). The Swedish chemist von Ostrejko obtained two patents, in 1900 and 1901, covering the basic concepts of chemical and thermal (or physical) activation of carbon, with metal chlorides and with carbon dioxide and steam, respectively [7]. In 1909, a plant named 'Chemische Werke' was built to manufacture, for the first time on a commercial scale, the powdered activated carbon Eponit<sup>®</sup> from wood, adopting von Ostrejko's gasification approach [8]. Other activated carbons known as Norit<sup>®</sup> and Purit<sup>®</sup>

were produced in this plant by the activation of peat with steam. The NORIT company, a manufacturer in Holland, first appeared in about 1911 and became widely known in the sugar industry [5]. The powdered activated carbons were used at that time mainly for decolorizing solutions in the chemical and food industries.

On an industrial scale, the process of chemical activation of sawdust with zinc chloride was carried out for the first time in an Austrian plant at Ausing in 1914, and also in the dye plant of Bayer in 1915 [9]. This type of activation involved pyrolytical heating of the carbonaceous material in the presence of dehydrating chemicals such as zinc chloride or phosphoric acid [10].

In parallel to the developments in Europe, in the United States the first activated carbon was produced from black ash, a waste product of soda production, after it was accidentally discovered that the ash was effective in decolorizing liquids [5]. The first commercial production of activated carbon in the United States took place in 1913 [11]. Activated carbon in the form of PAC was used for the first time in 1928 by Chicago meat packers for taste and odor control [12].

The use of poisonous gases in the First World War paved the way for the development and large-scale production of granular activated carbon (GAC). These carbons were used in gas masks for the adsorption of poisonous gases. Subsequently, they were used for water treatment, solvent recovery, and air purification. After the First World War, considerable progress was made in Europe in the manufacture of activated carbons using new raw carbonaceous materials such as coconut and almond shells. The treatment with zinc chloride yielded activated carbons with high mechanical strength and high adsorptive capacities for gases and vapors. Later, in 1935–1940, pelletized carbons were produced from sawdust by zinc chloride activation for the recovery of volatile solvents and the removal of benzene from town gas. Nowadays, the zinc chloride process of chemical activation has been largely superseded by the use of phosphoric acid [4].

## 1.2

### General Use of Activated Carbon

Nowadays, activated carbon finds wide application in many areas, but especially in the environmental field. Aside from environmental pollution control, activated carbon is mainly used in industry in various liquid and gas phase adsorptions [1]. Among liquid phase applications one can list food processing, preparation of alcoholic beverages, decolorization of oils and fats, product purification in sugar refining, purification of chemicals (acids, amines, glycerin, glycol, etc.), enzyme purification, decaffeination of coffee, gold recovery, refining of liquid fuels, purification in electroplating operations, purification in the clothing, textile, personal care, cosmetics, and pharmaceutical industries, and applications in the chemical and petrochemical industries. Gas phase applications include recovery of organic solvents, removal of sulfur-containing toxic components from exhaust gases and recovery of sulfur, biogas purification, use in gas masks, among others. Activated

carbon is also used in medical and veterinary applications, soil improvement, removal of pesticide residues, and nuclear and vacuum technologies.

### 1.3

#### **Application of Activated Carbon in Environmental Pollution**

Although the use of carbon-based materials dates back to ancient times, the use of activated carbon in its current form began in the second half of the twentieth century as a consequence of the rising awareness of environmental pollution. Today, activated carbon is very often utilized in the removal of various organic and inorganic species from surface water, groundwater, and wastewater.

#### 1.3.1

##### **Activated Carbon in Drinking Water Treatment**

Adsorption by activated carbon is employed today in drinking water treatment for various purposes. An overview of historical development shows that the first application of activated carbon in the form of GAC was in the year 1910 in Reading, England for the purpose of dechlorination of chlorinated water [12]. In the 1930s and 1940s, in particular in Europe, water works used high chlorine doses for the disinfection of water following the growing pollution of surface waters. Often, GAC filtration was used for dechlorination purposes. However, the dechlorination in these filters cannot be regarded as an adsorptive process since the removal of chlorine depends on a catalytic reaction taking place on the carbon surface. However, the use of GAC for dechlorination purposes was abandoned a long time ago because of the formation of additional haloforms and other chlorine compounds within filters [7].

The use of activated carbon in water treatment for removal of substances responsible for taste and odor dates back to the late 1920s [11]. The undesirable taste and odor in drinking water was mainly attributed to the presence of chlorophenols formed in water as a result of the chlorination of phenols at the disinfection stage [7].

PAC was used for the control of taste and odor in drinking water for the first time in the USA in 1929–1931 [7]. The first GAC filters were installed in Germany in 1929 and in the USA in 1930 for taste and odor removal. By 1932 about 400 water treatment works in the USA were adding PAC to their water to improve taste and odor, and this number increased to 1200 by 1943. The first major GAC filter for public water supply was installed in the USA at the Hopewell, VA, water treatment plant in 1961 [12]. By 1970 the number of waterworks which added PAC to their units or used GAC adsorbers was estimated at 10000 worldwide [7]. In later years, PAC adsorption for water treatment was also integrated with Dissolved Air Flotation (DAF), in which PAC served as an adsorbent for various pollutants and was subsequently floated to the surface by DAF [13].

When activated carbon was used in granular or powdered form in the early 1960s in water treatment, the main aim was the removal of taste and odor. In Europe, where surface waters were heavily polluted, early breakthroughs of

odor-causing species were observed in GAC filters, necessitating frequent regenerations. Intensive investigations beginning in the early 1960s revealed that pretreatment of water with ozone was an effective solution to this problem since it extended the GAC bed life. The well-known Mülheim process was developed as a result of these efforts [7]. Details of this process can be found in Chapter 8.

Currently, problems in drinking water treatment extend beyond the scope of taste and odor control. Much attention is being paid to the regulation and control of numerous organic and inorganic compounds in water. Concerns about the presence of Synthetic Organic Compounds (SOC) arose in 1960s. Beginning in the 1970s it was recognized that disinfection of water with chlorine gas or chlorine-containing compounds led to the generation of organic compounds, collectively termed Disinfection By-Products (DBPs), which were suspected of having adverse effects on health [7]. In this regard, Natural Organic Matter (NOM) constitutes the key group of organics acting as precursors for DBP formation. It was also shown that pretreatment of water with ozone led to inorganic hazardous by-products such as bromates. For many decades, adsorption onto activated carbon has appeared to be one of the most reliable methods of NOM and DBP control. This type of treatment is usually conducted in GAC filters. These are usually placed after sand filtration and before disinfection, but, depending on the characteristics of the water and the object of the treatment, GAC filters may also be positioned at other locations within the treatment train.

The presence of synthetic organic contaminants in surface and groundwaters is largely attributed to the discharge of municipal and industrial wastewaters into receiving waters in treated or untreated form. The increased use of fertilizers and pesticides in agriculture is another factor contributing to pollution. Further, discharges into surface waters from non-point sources such as urban runoff also add to pollution.

Raw waters taken from surface and groundwater supplies contain many organic compounds such as phenols, pesticides, herbicides, aliphatic and aromatic hydrocarbons and their chlorinated counterparts, dyes, surfactants, organic sulfur compounds, ethers, amines, nitro compounds, and newly emerging substances such as Endocrine Disrupting Compounds (EDCs). More than 800 specific organic and inorganic chemicals have been identified in various drinking waters, and many more are suspected to be present [1]. Therefore, concerns are frequently expressed about the presence of these compounds, which can be present at levels as low as  $\text{ng L}^{-1}$  or  $\mu\text{g L}^{-1}$ . Because of their proven or suspected health and environmental effects, great efforts are made to control and/or remove them, and one of the major methods of doing this is by adsorption onto activated carbon.

### 1.3.2

#### Activated Carbon in Wastewater Treatment

The groups of organics that are generally amenable to adsorption onto activated carbon include pesticides, herbicides, aromatic solvents, polynuclear aromatics, chlorinated aromatics, phenolics, chlorinated solvents, high-molecular-weight (HMW) aliphatic

acids and aromatic acids, HMW amines and aromatic amines, fuels, esters, ethers, alcohols, surfactants, and soluble organic dyes. Compounds having low molecular weight (LMW) and high polarity, such as LMW amines, nitrosamines, glycols, and certain ethers, are not amenable to adsorption [11].

Many compounds falling into these categories are encountered in the effluents of various industries and to some extent also in municipal wastewaters and drinking water supplies. Activated carbon has gained importance especially since the mid 1960s as an adsorptive material in the treatment of municipal and industrial wastewaters.

#### 1.3.2.1

##### **Municipal Wastewater Treatment**

The first full-scale advanced (tertiary) wastewater treatment plant incorporating GAC was put into operation in 1965 in South Lake Tahoe, California. The use of GAC beds as a unit process became common in the tertiary treatment train [11]. The purpose in employing GAC was to reuse the effluent of municipal wastewater treatment plants for purposes such as industrial cooling water, irrigation of parks, and so on.

Physicochemical treatment options involving PAC adsorption were also tested in lieu of biological treatment. The idea was that primary settling was followed by coagulation and PAC adsorption, settling and perhaps filtration. However, secondary treatment could not be replaced with a merely physicochemical process because of cost [11].

Today, GAC filtration or PAC-assisted membrane operation are mainly conducted as a tertiary treatment step to remove dissolved and refractory organic matter from secondary sewage effluent. The main goal remains to be the reuse of effluent for various purposes.

#### 1.3.2.2

##### **Industrial Wastewater Treatment**

Activated carbon adsorption is most commonly applied in industrial wastewater treatment to meet stringent regulations for discharge into receiving waters. In industrial wastewater treatment, activated carbon adsorption can be utilized as a separate unit process. It may be placed after various physicochemical treatment steps such as coagulation/clarification, filtration, and dissolved air flotation. Another alternative is to use activated carbon adsorption prior to biological treatment to remove compounds which might be toxic to a biological system. However, the most widely adopted procedure is to place activated carbon adsorption as a tertiary or advanced treatment step subsequent to biological treatment for removal of refractory organics. To some extent this procedure may also be effective in the removal of inorganics.

Nowadays, activated carbon finds wide application in the treatment of wastewaters generated from industries such as food, textile, chemical, pharmaceutical, pesticides and herbicides production, coke plant, munitions factories, petroleum refineries and storage installations, organic pigments and dyes, mineral processing plants, insecticides, pesticides, resins, detergents, explosives, and dyestuffs. It is also employed in the treatment of sanitary and hazardous landfill leachates.

## 1.3.3

**Applications of Activated Carbon in Other Environmental Media**

## 1.3.3.1

**Remediation of Contaminated Groundwater and Soil**

Groundwaters are significantly polluted with organic and inorganic substances as a result of industrial spills, accidents, discharges, and so on. Activated carbon adsorption is often employed in remediation of groundwaters for drinking purposes. In groundwater remediation, activated carbon may either directly adsorb contaminants or remove them after their transfer into the gas phase by air sparging or stripping.

Today, activated carbon is also applied in the remediation of contaminated soils. Remediation of soils contaminated with petroleum hydrocarbon and other substances involves the use of thermal desorption methods. The resulting off-gases containing Volatile Organic Compounds (VOCs) are usually treated with PAC or GAC. In contaminated soils, PAC may also be used as a soil additive to immobilize organic contaminants.

## 1.3.3.2

**Treatment of Flue Gases**

Activated carbon also finds application in the purification of flue gases such as those emerging from incinerators, and in the removal of gases such as radon, hydrogen sulfide, and other sulfur compounds from gas streams [2].

## 1.3.3.3

**Water Preparation for Industrial Purposes**

Activated carbon is utilized in industrial facilities for the production of the water required for various plant items such as steam generators, heat exchangers, cooling towers, and also in the production of ultrapure water.

## 1.3.4

**Integration of Activated Carbon Adsorption with Biological Processes in Wastewater and Water Treatment**

Nowadays, adsorption and biological processes for the control of various pollutants generally take place in separate unit, whereas the combination of adsorption and biological processes in the same reactor is relatively less common and more complicated. The main purpose of this book is to provide an insight into this system of integrated application, whose usefulness has been clearly recognized since the 1970s, in both wastewater and water treatment.

## 1.3.4.1

**Wastewater Treatment**1.3.4.1.1 **Combined Suspended-Growth Processes**

**The Powdered Activated Carbon Treatment (PACT) process** The PACT process is essentially a modification of the activated sludge process by the addition of PAC. The application of concurrent adsorption and biodegradation in the same

suspended-growth reactor is an effective alternative for the removal of biodegradable and nonbiodegradable compounds. The PACT system has also been adopted for anaerobic treatment.

**Integration of PAC adsorption with membrane processes** In recent years PAC has been integrated into Membrane Bioreactors (MBR) to bring about a positive effect on contaminant removal and to prevent membrane biofouling.

#### 1.3.4.1.2 Combined Attached-Growth Processes

**The Biological Activated Carbon (BAC) Process** While PACT is a modification of a suspended-growth process, BAC is essentially a biofilm process that is based on the establishment of biological activity in a GAC adsorber by gradual attachment of microorganisms and development of a biofilm.

Since 1970s, both PAC- and GAC-based biological processes have been applied in the treatment of industrial wastewaters such as organic chemicals, petrochemicals, refineries, textiles/dyes, in joint treatment of municipal and industrial wastewaters for discharge or reuse purposes, and in the treatment of sanitary and hazardous landfill leachates. Detailed discussion of such applications is presented in Chapters 3–7 of this book.

#### 1.3.4.2

##### **Water Treatment**

Since the 1970s, a gradual development has taken place in the direction of integrating adsorptive and biological processes in the treatment of surface water or groundwaters. In this regard, BAC filtration is a well-known unit process that combines the merits of adsorption and biological removal in the same reactor. While the majority of GAC adsorption applications target the removal of natural and/or anthropogenic organic compounds, BAC filtration is also suited to some extent for the elimination of inorganics such as ammonia, perchlorate, and bromate. The characteristics of this unit process is extensively addressed throughout Chapters 8–11.

#### 1.3.5

##### **Improved Control of Pollutants through Integrated Adsorption and Biological Treatment**

Water and wastewaters are multicomponent mixtures. As such, it is impossible to measure the presence and removal of a large number of compounds present in treatment or remediation systems. Therefore, in the case of organic compounds, monitoring is commonly carried out by the use of sum (collective) parameters such as TOC, DOC and UV absorbance, and the BOD and COD parameters, in water and wastewater treatment, respectively (Table 1.1).

Over the years, more specific parameters have been developed. In the characterization of waters and wastewaters one of the widely used parameters is

**Table 1.1** Reduction in parameters or pollutant groups achieved by adsorption, biological removal, or integrated means.

| Parameters/pollutant groups           | Importance in water (W), groundwater (GW) or wastewater (WW) treatment | Activated Carbon Adsorption | Biological Removal or Transformation | Removal in Integrated Adsorption and Biological Treatment |
|---------------------------------------|------------------------------------------------------------------------|-----------------------------|--------------------------------------|-----------------------------------------------------------|
| <b>Organic pollutants</b>             |                                                                        |                             |                                      |                                                           |
| <i>Oxygen demand parameters</i>       |                                                                        |                             |                                      |                                                           |
| BOD                                   | WW                                                                     | F to G                      | G to E                               | E                                                         |
| COD                                   | WW                                                                     | G                           | L to G                               | G to E                                                    |
| <i>Organic carbon parameters</i>      |                                                                        |                             |                                      |                                                           |
| TOC                                   | W, WW, GW                                                              | G                           | L to G                               | G to E                                                    |
| DOC                                   | W, GW, WW                                                              | G                           | L to G                               | G to E                                                    |
| VOC <sup>c</sup>                      | W, GW, WW                                                              | L to G                      | L to G                               | G to E                                                    |
| BDOC                                  | W, GW                                                                  | F to G                      | G to E                               | E                                                         |
| AOC                                   | W, GW                                                                  | F to G                      | E                                    | E                                                         |
| <i>Other organic parameters</i>       |                                                                        |                             |                                      |                                                           |
| AOX                                   | W, GW, WW                                                              | G to E                      | L to F                               | G to E                                                    |
| UV <sub>254</sub>                     | W, GW, WW                                                              | G                           | L                                    | G to E                                                    |
| <b>Organic Pollutant Groups</b>       |                                                                        |                             |                                      |                                                           |
| THMs                                  | W, GW                                                                  | G                           | P to L                               | G                                                         |
| HAAs                                  | W, GW                                                                  | G                           | G                                    | G to E                                                    |
| Pesticides                            | W, GW                                                                  | F to E                      | L to F                               | G                                                         |
| Pharmaceuticals                       | W, WW                                                                  | F to E                      | F to E                               | G to E                                                    |
| Endocrine Disrupting Compounds (EDCs) | W, WW                                                                  | G                           | P to G                               | G to E                                                    |
| Chlorinated hydrocarbons              | GW, WW                                                                 | F to E                      | L to G                               | G to E                                                    |
| <b>Inorganic Pollutants</b>           |                                                                        |                             |                                      |                                                           |
| Bromate                               | W                                                                      | L to F <sup>a</sup>         | F to G <sup>d</sup>                  | G <sup>d</sup>                                            |
| Perchlorate                           | GW                                                                     | N                           | G                                    | G to E                                                    |
| Ammonia                               | W, WW                                                                  | N                           | G                                    | G to E                                                    |
| Nitrate                               | GW, WW                                                                 | N                           | G                                    | G to E                                                    |
| Heavy Metals                          | W, GW, WW                                                              | P to G <sup>b</sup>         | F <sup>c</sup>                       | G <sup>c</sup>                                            |

E: excellent, G: good, F: fair, L: low, P: poor, N: none

<sup>a</sup>reduction at surface<sup>b</sup>dependent on type and conditions<sup>c</sup>possible transformation and/or biosorption<sup>d</sup>uncertain<sup>e</sup>Volatile Organic Carbon: Volatile Organic Compounds are measured based on the surrogate 'carbon.' They can also be measured as individual compounds.

referred to as Adsorbable Organic Xenobiotics (AOX), and represents halogenated organics that have a high affinity towards activated carbon. This parameter is most often used to indicate the chlorinated organic compounds (AOCl).

There are also definitions that are specific to water treatment. In this context, one can list the Disinfection By-Products (DBPs). Specific groups among DBPs are referred to by terms such as Trihalomethanes (THMs) and Haloacetic Acids (HAAs). The respective formation potentials of these groups are abbreviated as THMFP and HAAFP.

In addition to these, in recent decades, a large number of new compounds have been detected in water and wastewater media. Since in raw and finished waters these compounds, referred to as micropollutants, are present at  $\mu\text{g L}^{-1}$  or  $\text{ng L}^{-1}$  levels, the sum parameters mentioned above prove to be useless in their monitoring. Due to this fact, efforts are made to monitor them individually by advanced analytical techniques. Within this context, one can list various pharmaceuticals and EDCs that have received a great deal of attention in the last decades.

Various pollutants found in water and wastewater systems are amenable to either adsorption or biological degradation or transformation. Still, a number of them can be removed by both adsorptive and biological means. Combination of activated carbon adsorption and biological processes in the same unit often offers a synergism, in that a higher removal is achieved than expected from adsorption or biodegradation alone. For many pollutants that are considered to be slowly biodegradable or even nonbiodegradable, integration of adsorption with biological removal may provide the opportunity for biological degradation. This integrated approach can also enable the effective elimination of micropollutants at trace levels.

Various organic and inorganic pollutants are encountered in surface waters, groundwaters, and wastewaters. Table 1.1 provides a brief overview of the relative reduction achieved in parameters or specific groups by means of adsorption, biological activity or integration of both. However, the evaluation presented here is rather general, relative, and qualitative. Comprehensive discussion of the elimination mechanisms, the synergism in integrated adsorption and biological removal, the laboratory-, pilot- and full-scale studies, and the modeling of integrated adsorption and biological removal in wastewater and water treatment is presented throughout Chapters 3–11.

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