Chapter 16
Carbon-Based Polymers, Activated Carbons

16.1 Introduction

Carbon-based polymers and absorbents constitute a topic of huge scientific interest and great strategic importance in an interdisciplinary approach spanning applied physics, biology, pharmacy and medicine, mechanics, electronics, chemistry, and chemical engineering. Today carbon-based materials and adsorbents are widely used for energy storage and catalysis; for fabrication of electrodes and sensors operating in harsh environment; as hemoabsorbents of toxins and viruses in blood recovery; and as a biocompatible coating of medical implants, water purification, and wastewater treatment. Carbon is a surprisingly versatile element, able to hybridize in three different states, sp^1, sp^2, and sp^3**.** The changes in local bonding of carbon atoms account for the existence of extremely diverse allotropic phases, exhibiting a very broad range of physical and chemical properties. This element can crystallize as diamond (sp^3 hybridization) or graphite (sp^2 hybridization) and give rise to many noncrystalline phases (generally containing a mixture of sp^1, sp^2, and sp^3 hybridizations), such as fullerenes; carbon nanotubes; and disordered, nanostructured, and amorphous carbons. Strong tetrahedral δ bonds are responsible for the extreme physical properties of diamond, a wide gap semiconductor having the largest bulk modulus of any solid, the highest atom density, and the largest limiting electron and hole velocities of any semiconductor. Graphite, whose sheets (graphenes), featured by strong intralayer trigonal δ bonding, are held together by weak interlayer van der Waals' forces, is considered as an anisotropic metal.

16.2 Polymeric Carbon: Diamond

Natural diamonds are believed to have been formed millions of years ago when concentrations of pure carbon were subjected to great pressures and heat by the Earth's mantle (see Fig. 16.1). They are the hardest known material in nature. The majority of diamonds (nongem) are now man made. Most of the synthetic diamonds are no larger than a grain of common sand. The major use of synthetic diamonds is as industrial shaping and cutting agents to cut, grind, and bore (drill). By 1970, General Electric was manufacturing diamonds of gem quality and size through compressing pure carbon under extreme pressure and heat. It was found that addition of small amounts of boron to diamonds causes them to become semiconductors. Today, such doped diamonds are used to make transistors. While diamonds can be cut, shaping is done by trained cutters striking the rough diamond on one of its cleavage plates. These cleavage plates are called faces and represent sites of preferential cleavage and reflection of light. This balance between strength and flexibility, crystalline and amorphous regions is demonstrated to one extreme by diamonds that are very crystalline, resulting in a strong, inflexible, and brittle material [1].

O.V. Roussak and H.D. Gesser, *Applied Chemistry: A Textbook for Engineers and Technologists*,
DOI 10.1007/978-1-4614-4262-2_16,

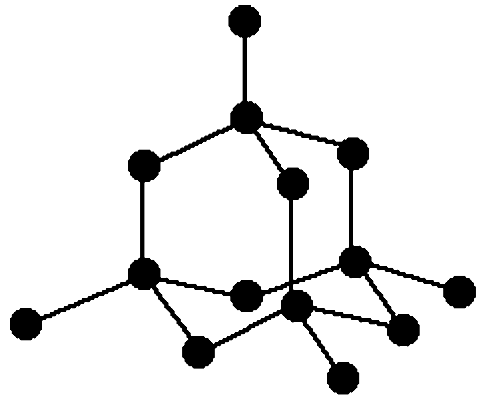

Fig. 16.1 Representation of diamond, where each carbon is at the center of a tetrahedron composed of four other carbon atoms

16.3 Polymeric Carbon: Graphite

While diamond is the hardest naturally occurring material, the most common form of crystalline carbon is the much softer and flexible graphite. Graphite occurs as sheets of hexagonally fused benzene rings (see Fig. 16.2) or "hexachicken wire." The bonds holding the fused hexagons together are traditional covalent bonds. The bonds holding the sheets together are weaker than the bonding within the sheets consisting of a weak overlapping of pi-electron orbitals. Thus, graphite exhibits many properties that are independent on the angle at which they are measured. They show some strength when measured along the sheet, but very little strength if the layers are allowed to slide past one another. This sliding allows the graphite its flexibility, much like the bending of bundles of proteins sliding past one another, allowing our hair flexibility. The fused hexagons are situated such that the atoms in each layer lie opposite to the centers of the six-membered rings in the next layer. This arrangement further weakens the overlapping of the pi electrons between layers such that the magnitude of layer-to-layer attraction is on the order of ordinary secondary van der Waals forces. The "slipperiness" of the layers accounts for graphite's ability to be a good lubricant.

The variance of property with angle of applied force, light, magnetism, and so on is called anisotropic behavior. Calcite is anisotropic in its crystal structure, resulting in a dependency of its interaction with light with the angle of incidence of the light.

As with diamond, graphite's discovery and initial usage are lost in antiquity. It was long confused with other minerals such as molybdenite MoS_2. At one time, it was known as plumbago (like lead), crayon noir, silver lead, black lead, and carbon minerals. Werner in 1789 first named it graphite, meaning (in Greek) "to write."

The Acheson process for graphite production begins by heating a mixture of charcoal, or coke, and sand. Silica is believed to be reduced to silicon that combines with carbon forming silicon carbide, which subsequently dissociates into carbon and silicon. The silicon vaporizes, and the carbon condenses forming graphite. Graphite is also produced using other techniques.

Today, graphite is mixed with clay to form the "lead" in pencils. Graphite conducts electricity and is not easily burned, so many industrial electrical contact points (electrodes) are made of graphite. Graphite is a good conductor of heat and is chemically inert, even at high temperature. Thus, many crucibles for melting metals are graphite-lined. Graphite has good stability to even strong acids; thus, it is employed to coat acid tanks. It is also effective at slowing down neutrons, and thus, composite bricks and rods (often called carbon rods) are used in some nuclear generators to regulate the progress of the nuclear reaction (see Chap. 7). Its slipperiness allows its use as a lubricant for clocks, door locks, and handheld tools and skies. Graphite is also the major starting material for the synthesis of

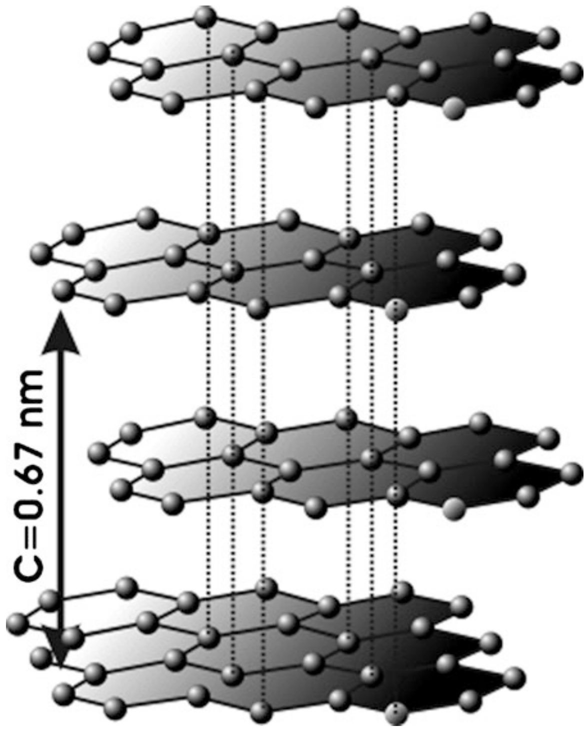

Fig. 16.2 Representation of graphite emphasizing the layered and sheet nature of graphite

synthetic diamonds. Graphite is sometimes used as a component of industrial coatings. Dry cells and some types of alkali-storage batteries also employed graphite. Graphite fibers are used for the reinforcement of certain composites [1].

16.4 Graphene

The first material in this new class is graphene, a single atomic layer of carbon (see Figs. 16.3 and 16.4). This new material has a number of unique properties, which makes it interesting for both fundamental studies and future applications. Graphene is a single layer of carbon packed in a hexagonal (honeycomb) lattice, with a carbon-carbon distance of 0.142 nm. It is the first truly two-dimensional crystalline material, and it is representative of a whole class of 2D materials including, for example, single layers of Boron-Nitride (BN) and Molybdenum-disulphide (MoS_2), which have both been produced after 2004 [2]. The electronic properties of this 2D-material lead to, for instance, an unusual quantum Hall effect. It is a transparent conductor that is one atom thin. It also gives rise to analogies with particle physics, including an exotic type of tunneling which was predicted by the Swedish physicist Oscar Klein [2]. Graphene is practically transparent. In the optical region, it absorbs only 2.3 % of the light. In contrast to low-temperature 2D systems based on semiconductors, graphene maintains its 2D properties at room temperature. Graphene also has several other interesting properties, which it shares with carbon nanotubes. It is substantially stronger than steel and very stretchable.

The thermal and electrical conductivity is very high, and it can be used as a flexible conductor. Its thermal conductivity is much higher than that of silver. Graphene has a number of properties which makes it interesting for several different applications. It is an ultimately thin, mechanically very strong, transparent, and flexible conductor. Its conductivity can be modified over a large range either by

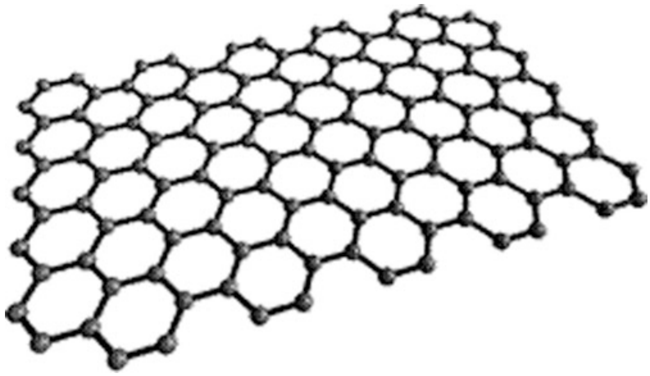

Fig. 16.3 Structure of graphene

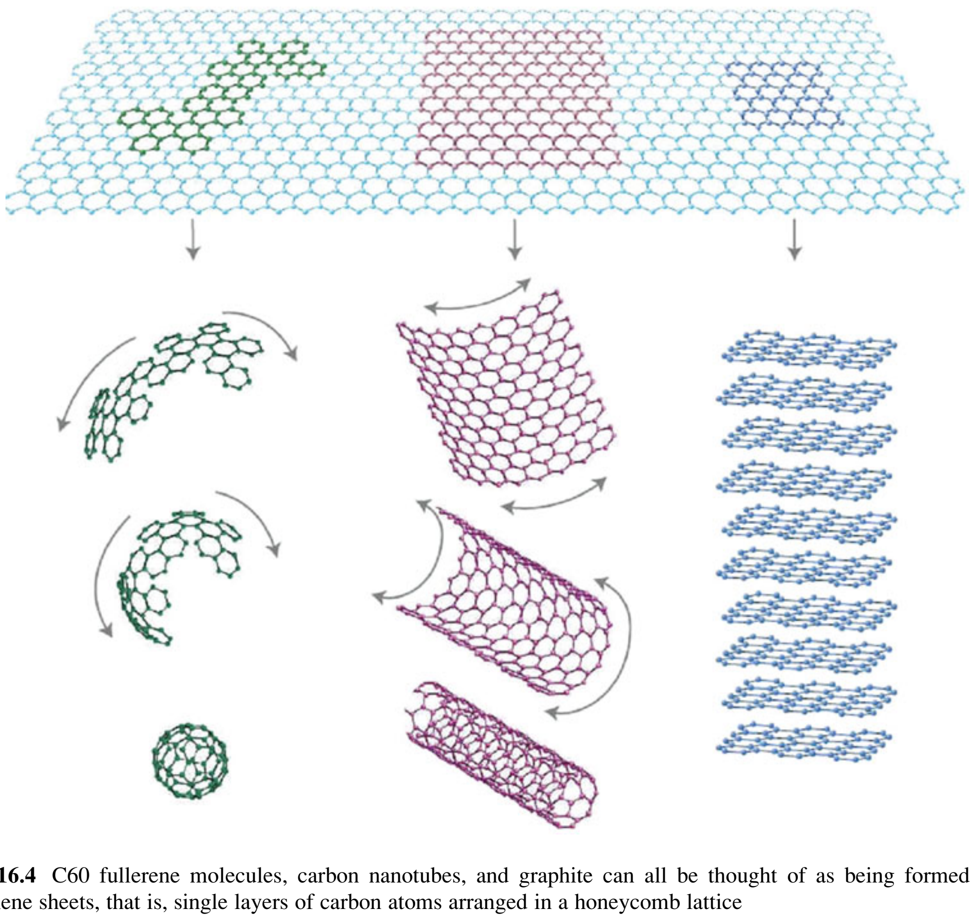

Fig. 16.4 C60 fullerene molecules, carbon nanotubes, and graphite can all be thought of as being formed from graphene sheets, that is, single layers of carbon atoms arranged in a honeycomb lattice

chemical doping or by an electric field. The mobility of graphene is very high, which makes the material very interesting for electronic high-frequency applications. Recently it has become possible to fabricate large sheets of graphene. Using near-industrial methods, sheets with a width of 70 cm have been produced. Since graphene is a transparent conductor, it can be used in applications, such as touch screens, light panels, and solar cells, where it can replace the rather fragile and expensive indium tin oxide (ITO). Flexible electronics and gas sensors are other potential applications. The quantum Hall effect in graphene could also possibly contribute to an even more accurate resistance standard in metrology. New types of composite materials based on graphene with great strength and low weight could also become interesting for use in satellites and aircraft [2].

The Nobel Prize in Physics 2010 honors two scientists who have made the decisive contributions to this development. They are *Andre K. Geim* and *Konstantin S. Novoselov*, both at the University of Manchester, UK. They have succeeded in producing, isolating, identifying, and characterizing graphene [2].

A new form of molecular carbon is the so-called fullerenes (see Fig. 16.4). The most common, called C60, contains 60 carbon atoms and looks like a football (soccer ball) made up from 20 hexagons and 12 pentagons which allow the surface to form a sphere. The discoverer of fullerenes was awarded the Nobel Prize in Chemistry in 1996 [2].

A related quasi-one-dimensional form of carbon, carbon nanotubes, has been known for several decades, and the single-walled nanotubes, since 1993 [1, 2]. These can be formed from graphene sheets which are rolled up to form tubes, and their ends are half spherical in the same way as the fullerenes. The electronic and mechanical properties of metallic single-walled nanotubes have many similarities with graphene.

Thus, the difficulty was not to fabricate the graphene structures, but to isolate sufficiently large individual sheets in order to identify and characterize the graphene and to verify its unique two-dimensional (2D) properties. This is what Geim, Novoselov, and their collaborators succeeded in doing.

16.4.1 Density of Graphene

The unit hexagonal cell of graphene contains two carbon atoms and has an area of 0.052 nm^2. We can thus calculate its density as being 0.77 mg/m^2.

A hypothetical hammock measuring 1 m^2 made from graphene would thus weigh 0.77 mg.

16.4.2 Optical Transparency of Graphene

Graphene is almost transparent; it absorbs only 2.3% of the light intensity, independent of the wavelength in the optical domain. Thus, suspended graphene does not have any color.

16.4.3 Strength of Graphene

Graphene has a breaking strength of 42 N/m^2. Steel has a breaking strength in the range of 250–1,200 MPa $= 0.25-1.2 \times 10^9$ N/m^2. For a hypothetical steel film of the same thickness as graphene (which can be taken to be 3.35 Å $= 3.35 \times 10^{-10}$ m, i.e., the layer thickness in graphite), this would give a 2D breaking strength of 0.084–0.40 N/m^2. Thus, graphene is more than 100 times stronger than the strongest steel.

In our 1-m^2 hammock tied between two trees, you could place a weight of approximately 4 kg before it would break. It should thus be possible to make an almost invisible hammock out of graphene that could hold a cat without breaking. The hammock would weigh less than one mg, corresponding to the weight of one of the cat’s whiskers.

16.4.4 Electrical Conductivity of Graphene

The mobility is theoretically limited to $\mu = 200{,}000\ \text{cm}^2\ \text{V}^{-1}\ \text{s}^{-1}$ by acoustic phonons at a carrier density of $n = 10^{12}\ \text{cm}^{-2}$. The 2D sheet resistivity, also called the resistance per square, is then 31 Ω.

Our fictional hammock measuring 1 m^2 would thus have a resistance of 31 Ω.

Using the layer thickness, we get a bulk conductivity of $0.96 \times 10^6\ \Omega^{-1}\ \text{cm}^{-1}$ for graphene. This is somewhat higher than the conductivity of copper which is $0.60 \times 10^6\ \Omega^{-1}\ \text{cm}^{-1}$.

16.4.5 Thermal Conductivity of Graphene

The thermal conductivity of graphene is dominated by phonons and has been measured to be approximately 5,000 $\text{W m}^{-1}\ \text{K}^{-1}$. Copper at room temperature has a thermal conductivity of 401 $\text{W m}^{-1}\ \text{K}^{-1}$. Thus, graphene conducts heat ten times better than copper.

16.5 Activated Carbons

With its immense capacity for adsorption from gas and liquid phases, activated carbon is a unique material. It occupies a special place in terms of producing a clean environment involving water purification and water treatment; decolorizing; chemical, nuclear, and pharmaceutical processing; food processing; air and gas purification; solvent vapor recovery; and motor vehicle emission canisters. In these roles, it exhibits a remarkable efficiency, as the world production capacity is estimated to be around 400,000 tonnes per year (excluding China and other Eastern countries, for which figures are not accurately known), with perhaps two million tons being in continuous use. This is equivalent of 200 mg per person of the world population to be compared with the world use of fossil fuels of 2 tonnes per person of the world population [3].

The largest producer of activated carbons is the USA (accounting for more than 40 wt.% of the total production capacity), followed by Europe, Japan, and Pacific Rim countries. The largest producer in the USA is Calgon Carbon Corporation, with plants in several locations. Its annual capacity represents around 42% of the total production capacity of the country; the main activation process is thermal, and the precursors used are bituminous coal, coconut shell, and charcoal. The second largest producer is Norit Americas, Inc., with around 23% of total capacity. Thermal activation is carried out with lignite and bituminous coal, and chemical activation (with phosphoric acid) is carried out with wood and peat. The third producer is Westvaco Corporation, with around 12% sawdust being the main precursor for both thermal and chemical (with phosphoric acid) activation. Regeneration capacity of spent activated carbon has increased considerably in the last few years, this being estimated to be over 50, 000 tonnes per year now.

Consumption of activated carbon is estimated to be around 200, 000 tonnes per year (2005) in the USA, but the market is negatively affected by imports from the Asia-Pacific region. More than 80% of that amount was used in liquid-phase applications. Sweetener decolorization used to be the main end-use of activated carbon, but it has been displaced by water treatment as a consequence of the increasing severity of government legislation on pollution control. Potable water is now the largest end-use market for activated carbon, around 26% of total consumption, 47% of which is powdered and 53% granular.

In the USA, the price of granular activated carbon ranged from $ 1.65 US to $ 9.90 US per kg, when most powdered activated carbon was priced in the range of $ 0.80 US to $ 2.00 US per kg. The largest difference in price is for carbons used in water treatment, with granular carbons being up to three times more expensive.

However, in such applications as gas and air purification, the costs of granular and powdered activated carbon are similar [3].

Usually untreated coal-based carbons contain up to 20 wt.% ash content, such as Si, Al, Fe, Mn, Ni, Cu, Sn, and Pb (according to the ASTM 2866–70 ash content test). Coconut shell carbons have less ash content about 1–3 wt.%. Also disarmament and partial substitution of military equipment and reorganization of some chemical production can release significant amounts of the used gas—proof carbons and chemical absorbents base on activated carbons. As a rule, these materials contain up to 20% nickel or iron chlorides or copper sulfate, and most of these components are water soluble. But some applications need very pure activated carbons, and to comply with this need, many manufacturers remove most of the ash components by washing the carbon with water or acids such as hydrochloric or nitric acid [4]. In some cases, a more exhaustive washing requires the use of hydrochloric and hydrofluoric acids to eliminate the aluminosilicate components of the ash. The washing must be particularly thorough when the carbon is to be used in pharmaceutical and medical preparations, in food industries, or in catalysis (when the carbon is used either as a hemoabsorbent or as a catalyst).

The main mechanism by which activated carbon removes impurities is one of physical adsorption, this being a reversible process. Consequently, one can expect that desorption of the impurities will render the carbon surface available again for adsorption. Regeneration of spent activated carbon is not only important from the point of view of restoring the adsorption capacity of carbon but also because in many cases, the recovery of the adsorbed species is important. If the adsorption is of chemical type (chemisorption), the formation of a bond between the carbon and the adsorbate makes the process nonreversible, and even if desorption is possible, the desorbed species will be different from those originally adsorbed. Additionally, adsorption (especially in liquid phase) is often accompanied by precipitation of species which cannot be removed by simple desorption.

Activated carbon is porosity (space) enclosed by carbon atoms. Porosity has the size of molecules and is probably slit shaped. Porosity within a porous solid is space which is accessible to molecules from the gas/vapor and liquid phases. Micropores have entrance dimensions less 2.0 nm. Mesopores have dimensions between 2 and 50 nm. Micropores have entrance dimensions higher than 50 nm. It is porosity within activated carbon which imparts their dominant characteristics of adsorption.

The *Langmuir equation* or *Langmuir isotherm* or *Langmuir adsorption equation* or *Hill-Langmuir equation* relates the coverage or adsorption of molecules on a solid surface to gas pressure or concentration of a medium above the solid surface at a fixed temperature (see the Langmuir equation and isotherm in Appendix C, p. 333).

Dubinin and his coworkers—Kaganer, Radushkevich—discovered adsorption using potential theory originally formulated by Polanyi. They developed adsorption methods for pore volume and specific surface (the methods of Dubinin and of Kaganer). Dubinin and his coworkers advance arguments favoring the view of the adsorption space may be expressed as a Gaussian function of the corresponding adsorption potential. Dubinin's method makes it possible to calculate the micropore volume from the low pressure part of isotherm, the region where the adsorption is still much below the plateau value. It also offers the possibility of using different adsorbates as molecular probes. Dubinin has plotted the results for adsorption isotherms of nitrogen, saturated hydrocarbons, benzene, and cyclohexane and has found equation for calculation of the micropore volume W_o (see the Dubinin equation in Appendix C, p. 333). Dubinin's treatment has been modified by Kaganer to yield a method for the calculation of specific surface from the isotherm [5, 6].

Regeneration of activated carbons is based on principle that carbon is a stable material that can withstand changes in temperature and it is resistant to acidic and basic media. The more common regeneration process is by passing a flow of superheated steam through the carbon bed at a temperature lower than the one used during activation. As the temperature increases during regeneration, there is a desorption of the more volatile adsorbed compounds; above 400°C, there is also decomposition of adsorbed organic material, thus leaving some less-organized carbon, which is eliminated by reaction with steam at around 600°C. Of course, although the less-organized carbon is more reactive, a portion of the activated carbon is also gasified, and regeneration usually means a loss of 8–15 wt.% of the original activated carbon. For this reason, when regeneration is carried out under these conditions, it is also called reactivations. It is important to note that the off-gases must be properly handled, passing them through a post-burner and a scrubber, where they are destroyed. If regeneration is properly carried out, the bulk density and the surface area of the resulting carbon will be similar to that of the original carbon. If the surface area is larger after regeneration, this means that a proper reactivation has taken place, with the subsequent reduction in bulk density.

Additional regeneration procedures include desorption by hot inert gases, desorption under vacuum, and, more important, use of conventional liquid solvents. The main problem for the latter is the slow desorption and the difficult solvent regeneration, and the last few years have seen the development of regeneration with supercritical solvents, mainly carbon dioxide and water. Complete recovery of components and environmental safety of the corresponding industrial operations require also development of low-waste or waste-free extraction procedures and the corresponding process apparatus.

One of directions of low-waste extraction is multiple treatment of a material with azeotropic nitric acid (13.4 wt.%) and water or hydrochloric acid (20.9 wt.%) and water in the Soxhlet laboratory apparatus invented in 1879 by Franz von Soxhlet and shown in the Fig. 16.5. The acids are boiled, and its vapor of azeotropic acid travels upward through the extraction tube into the condenser tube. The cool water flowing around the outside of the condenser tube condenses the vapor, which then drips into the thimble, containing the material. Because the mineral alkaline impurities are soluble in azeotropic acids, they move into the condensed acids as they accumulated in the thimble. The solution, now containing the acids and dissolved impurities, build up in the thimble. Once the liquid reaches the level of the bypass arm, it is siphoned back into the flask. This continuous condensation, buildup, and siphoning is known as the reflux event. The advantage of the Soxhlet is that once the acids and dissolved mineral impurities are brought into solution and siphoned back into the flask, they stay in the flask—so that the sample in the extraction thimble is continuously reexposed to fresh, heated extraction agent—thus greatly increasing the extraction rate.

Demineralization ratio **d** is calculated from the content of the mineral components (wt. %) before and after extraction treatment according to the formula: $\mathbf{d} = (\mathbf{a} - \mathbf{b}) \times \mathbf{100/a}$. The purification coefficient **K** is calculated from the formula $\mathbf{K} = \mathbf{a/b}$, where **a** and **b** denote the contents of mineral components in the sample before and after extraction treatment, respectively. The azeotropic mixture of hydrochloric acid and water more effectively removes mineral impurities from commercial carbons than azeotropic nitric acid does due to the difference in the concentration of the corresponding acids (the concentration of nitric acid is reduced due to oxidation of the carbon sorbent) and to different nature of the acid anions. It has been experimentally found that demineralization ratio for most English carbons was higher than 41.5% and only in the case of NORIT RBXC carbon was it 6.5%, indicating profound preliminary demineralization of this sorbent. A single treatment with acid and water significantly decreases the total content of mineral components (Mn; Fe; Ni; Cu; Pb) and increases the exchange capacity of the resins too. For example, in case of VP-1AP anion-exchange resin, very large decontamination coefficients for nickel (K = 180,000) and iron (K = 1, 078) have been observed [7].

It is well known that the Soxhlet extractor underwent numerous improvements, and the most cardinal change was installation of a valve connecting extraction in the bottom section of the extractor, which allows operation both in mode of batch washing and in the of ideal displacement and vapor treatment (Figs. 16.6 and 16.7).

But there was lack of industrial application of the Soxhlet apparatus with large volume and capacity because glass extractors with a charge volume of up to 5 Liters were produced only in the United States. In 1984 Prof. Anatoliy I. Loskutov and Oleg V. Roussak designed and proposed industrial Soxhlet apparatus that allowed sampling of the solvent, charging and discharging of the

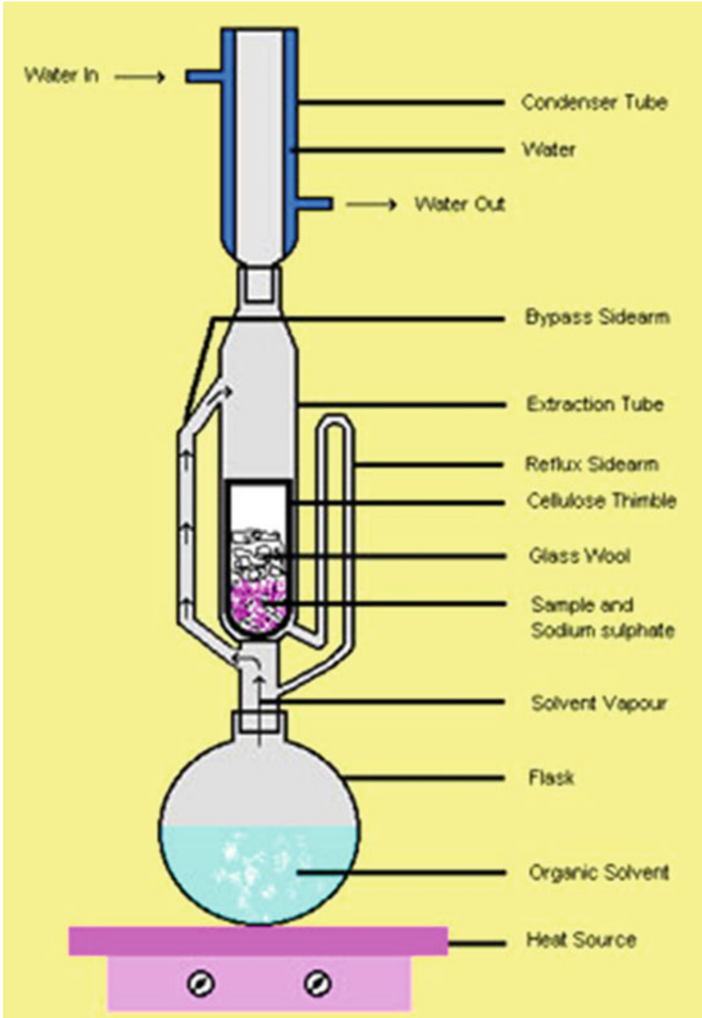

Fig. 16.5 Laboratory Soxhlet extractor

carbon material without complete dismantling of the setup, and stirring and treatment of the whole material with any solvent at its boiling temperature in the following four modes: vapor treatment, batch washing, ideal displacement, and heating (Fig. 16.8). An improved industrial Soxhlet apparatus has been tested for regeneration of granular activated carbons AG-3 and AG-5 as well as powdered activated carbons SKT-6A that are widely used in a solvent recuperation and wastewater treatment. The reagents of regenerated activated carbons were 20% aqueous solutions of ammonium hydroxide, potassium hydroxide, sodium hydroxide, distilled water, and azeotropic hydrochloric acid in various sequences of treatment: acid-water, alkali-water, and alkali-water-acid-water. It helped to solve problems of regeneration of powdered activated carbons (SKT-6A carbon) as well as regenerated calcium-containing activated carbons, which significantly increase safety, economical efficiency of regeneration, and reduce volume of liquid wastes [8, 9].

Extraction of activated carbons by a multiple treatment with a mixture of azeotropic hydrochloric acid and distilled water and large-scale production of high-purity activated carbon absorbents have been successfully applied to wastewater treatment and water purification and pharmaceutical and medical preparations.

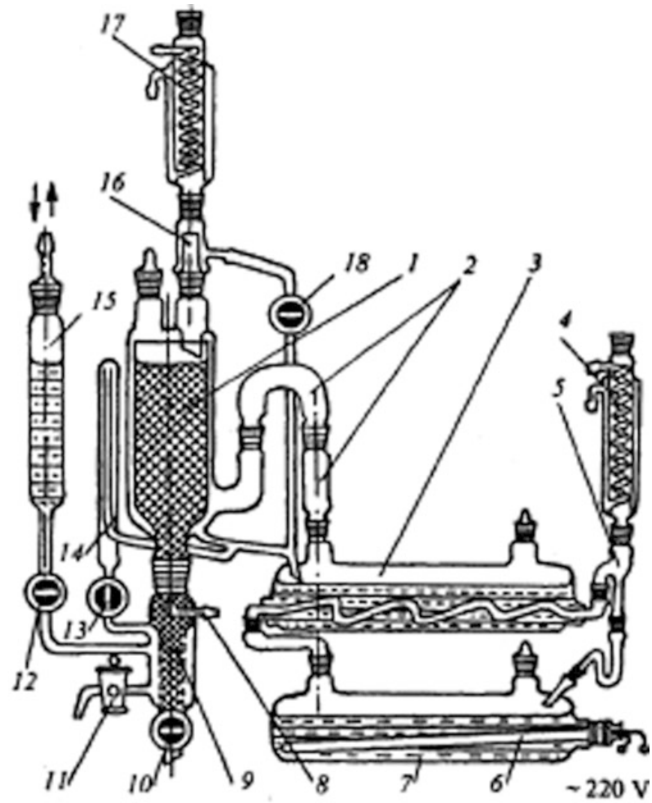

Fig. 16.6 Improved vertical Soxhlet extractor

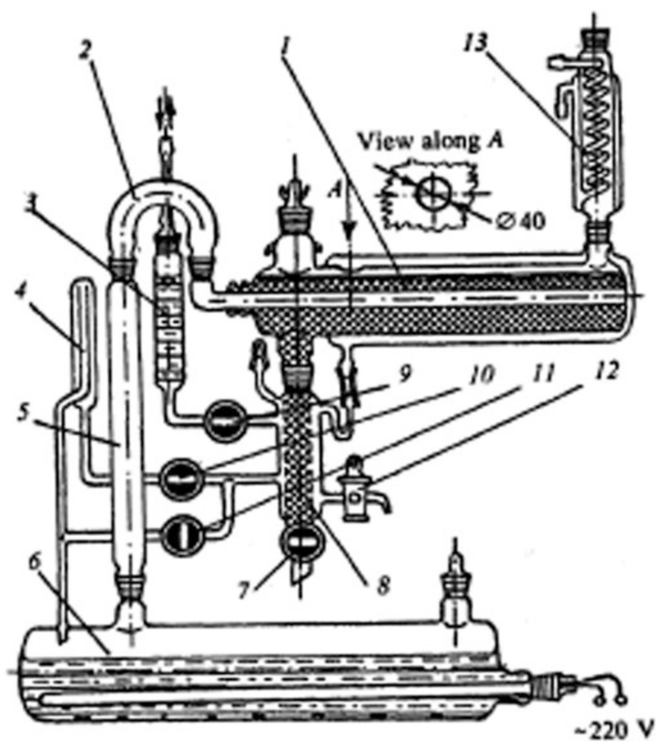

Fig. 16.7 Improved horizontal Soxhlet extractor

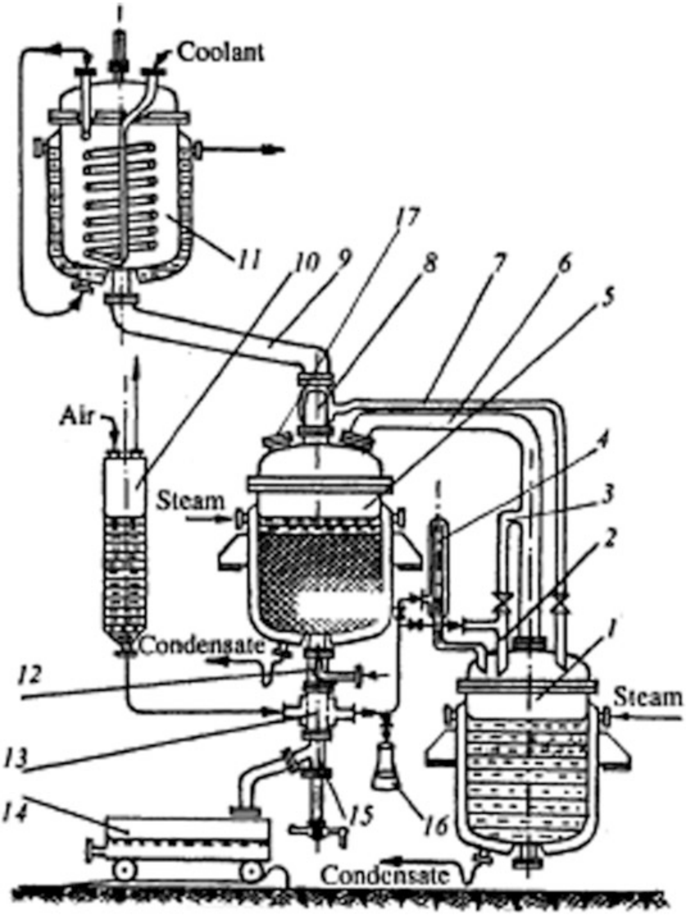

Fig. 16.8 Improved industrial Soxhlet extractor

It also can be used for the low-waste, environmentally friendly, clean and safe production of especially pure materials; ion-exchange resins of nuclear cleanliness; low-waste extraction of components from the solid materials (for instance, the extraction of platinum and palladium from the spent catalysts); and deactivation of materials and equipment of radiochemical enterprises [10, 11]. The industrial Soxhlet extractor and evaporator were made from steel covered with acid-resistant Ftoroplast coating sheets. The charge volume of the industrial Soxhlet apparatus could be significantly increased up to 16 cubic meters using stainless steel apparatus with acid-resistant ftoroplast coating sheets.

Exercises

1. What is activated carbon?
2. What is the size and shape of porosity?
3. What are activated carbons used for?
4. What is activation?
5. What are activated carbons made for?
6. Can any natural materials be used to make activated carbon?

7. Are all activated carbons very similar to each other?
8. What are adsorption isotherms?
9. Is there a common structure in activated carbons?
10. Is shape and size of porosity all important for activated carbons?

Further Reading

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