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PART I

INTRODUCTION

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History and Market Review

"For there is nothing good or bad but thinking makes it so"
Shakespeare Hamlet

1. INTRODUCTION

Purity is a subjective concept. Salt is desirable on a breakfast egg, but not in a glass of drinking water. Pesticides benefit a growing crop, and detergents help in the laundry, but both are considered contamination when discharged into waterways.

No substance—of itself and by itself—is an impurity. Conversely, any substance can become contamination, if on entering a system, it damages desirable properties and characteristics.

Some types of contamination can be converted into acceptable forms, for example, the bleaching of an unwanted color with chlorine. But in general purification is accomplished by providing effective means of separation. The separation may be accomplished by removing the desired constituent from the system, as when pure water is distilled from salt brines; and when white sugar is crystallized from a syrup. Alternatively, it may be the contamination that is removed, as when dissolved mercury salts are precipitated and separated from the solution by filtration; or when acid gases are removed from air by contact with an alkaline solution. Some separations are accomplished through the ability of fluid molecules to adhere to the surfaces of solids—a phenomena known as *adsorption*. For practicable use, certain requirements must be met. A large surface area is essential because only a very small weight of molecules adheres to each square meter of surface.

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The needed surface exists on porous solids known as *adsorbents*. Activated carbon adsorbents contain a myriad of micropores, the walls of which have surface areas that range from 400 to over 1800 square meters per gram in the various commercial brands. But more than large surface area is needed. To provide a means of separation, it is obvious that adsorption must be selective; certain species of molecules should be adsorbed in preference to others. To provide adequate purification, an adsorbent should be able to to take up and hold molecules of the substance to be removed without disturbing other constituents in the system. Moreover no single type of adsorbent surface will be appropriate for all diverse forms of contamination; therefore a variety of adsorbent surfaces should be available to handle the different needs.

It so happens that activated carbons can meet many of the diverse needs.[1,2,3] The adsorptive properties, which exist in primitive form in ordinary wood charcoal, can be developed in various forms by appropriate changes in manufacturing processes. Consequently, brands of commercial activated carbons made by dissimilar processes differ in adsorptive characteristics. Some excell for gas masks, others are superior for sugar refining, still others are best for water purification, and so on. Hence, we can quite properly consider that the term *activated carbon* comprises a family of adsorbents. That aspect extends and widens the potential utility because if one brand is tried and found wanting, possibilities remain that another brand will be suitable. Also to be considered is that the performance of carbon can often be guided into diverse channels by appropriate conditions. Consider the addition of activated carbon to a mixture of aniline and phenol in dilute aqueous solution: at pH 7, both will be equally adsorbed; at pH 10, aniline will be preferentially adsorbed; at pH 3, phenol will be preferentially adsorbed.

Features to be considered in the selection of carbon for use are considered in later chapters, but at this time we should mention the separate spheres of application of powdered and granular carbon. Powdered carbons are applied in a so-called *batch-contact* treatment: in this measured amounts of carbon and substance to be treated are mixed and subsequently separated by filtration. With granular carbons, the gas or liquid to be purified is passed continuously through a bed of carbon. For many years granular carbons (except bone char) were used primarily in vapor phase

systems because the early commercial brands lacked adsorptive characteristics needed for most liquid phase purifications. After World War II, new granular brands were developed having a broad spectrum of adsorptive powers, and today are in use in many liquid phase appplications. They have opened new markets that offer promise of important future growth.

The adsorptive properties of carbon were well known long before the terms *active* and *activated* had been coined. In early literature data on the adsorptive properties appear under many varied names: bone char, blood char, coconut char, and others. More recently the information appears under *decolorizing carbon* and also under individual commercial brand names.

2. HISTORY

Early history[1-2] The use of charcoal for purposes other than as a fuel and in metallurgy is very old; the use in medicine being mentioned in an Egyptian papyrus from 1550 B.C. In the time of Hippocrates wood chars were used to treat various ailments. Kehl in 1793 discussed the use of char for removal of odors from gangrenous ulcers.

The earliest date at which adsorptive powers were definitely recognized was 1773 when Scheele[1] described experiments with gases. In 1785, Lowitz[4] called attention to decolorizing effects of charcoal on solutions. A few years later, wood char was employed to purify cane sugar, and in 1808 was applied to the then infant beet-sugar industry. Figuers[1] discovery in 1811 of greater decolorizing power of bone char led to its almost immediate adoption by the sugar refiners. At first, pulverized bone char was applied on a single use and discard basis, but limited supplies made regeneration necessary. A method of regenerating granular bone char was developed and that process is still in general use in refining cane sugar.

During the 19th century, many studies were made to develop decolorizing carbons from other source materials. Bussy[5], in 1822, heated blood with potash and produced a carbon with with 20 to 50 times the decolorizing power of bone char. Blood char so produced was used for years in many laboratory studies. Hunter[6], in 1865, reported the the gas-adsorbing power of cocoa-

nut char. Stenhouse,[7] in 1865, prepared a decolorizing char by heating a mixture of flour, tar, and magnesium carbonate. Winser and Swindells[8] heated paper mill waste with phosphates. Many of the disclosures are similar to processes now in industrial use, and it is natural to inquire why they were not then developed on a commercial scale.

One answer is found in the manufacturing problems that are involved. Although activated carbon can be prepared with relative ease in the laboratory, the industrial production is attended by engineering difficulties. The corrosive action of many activation conditions requires special structural materials not then available. Moreover, successful industrial production depends on the skill of controlling manufacturing conditions within narrow limits, and suitable instrumentation for such control is relatively recent. The development was also retarded by the absence of apparent needs for a more adsorptive carbon than bone char. However, it is of interest to note that some early suggestions have today become important markets for activated carbon. Thus, Lipscombe[9] in 1862 prepared a carbon to purify potable water; and Stenhouse in 1854 described the forerunner of the modern gas mask.

Recent History

Work by Ostrejko[10] patented in 1900 and 1901 led to the development of modern commercial activated carbons. In one process, metallic chlorides were incorporated with carbonaceous materials before carbonization; another basic patent described selective oxidation with carbon dioxide at elevated temperatures.

Early interest in the development of activated carbon was stimulated by a belief that a large potential market existed in the cane sugar industry. Some early studies seemed to indicate that powdered activated carbon could be applied directly to the raw cane juice and thereby eliminate the need for an intermediate state in which raw sugar is first produced, which then must be redissolved and recrystallized. However, this hope was not realized; factory operation disclosed that the intermediate stage of raw sugar was still necessary.

Powdered activated carbon was found to be an effective substitute for bone char in established operations but required the installation of new specialized equipment. Inasmuch as the existing

equipment for bone char represented a large capital investment, very few refiners were induced to make the change.

Sales opportunities were found for the purification of other products that require decolorization but the total extent of such markets fell far short of fulfilling the hopes of those that had pioneered in the development of what were then known as decolorizing carbons.

Such was the situation prevailing in 1915 when an event which was to shock the world brought fame to active carbon. Following the initial stage of rapid offensive operations, World War I had become a defensive action, with the opposing armies landlocked in trenches. Seeking to break the impasse, the Germans, on April 22, 1915, released chlorine from cylinders on a four-mile front near Ypres and a breeze carried the yellowish-green vapor over no-man's land into the Allied trenches. Against this weapon, the Allied soldiers had no protection; the agonized men choking and gasping could only flee, leaving a breach in the line. Had the Germans conducted the gas attack over a wider front and provided effective reserves to follow through the breach, they might have won a decisive victory. But the German soldiers had no protective armour against the gas and could not follow through. Soon after, another attack was made with similar results. The German command turned a study of other methods of gas warfare and the interval enabled the allies to provide protection in the form of gas masks containing activated carbon. Had it not been for that development, history might have followed a different course. Although powdered activated carbon had been manufactured for some years the powdered form was not suitable for gas masks. Consequently granular grades had to be developed. The rapid progress in developing effective methods of production is a brilliant episode in the annals of industrial chemistry.

3. MARKETS

The publicity given activated carbon in World War I stimulated a search for new peacetime markets, and the years since have witnessed steady growth.

Vapor and Gas Phase Applications

At the conclusion of the war, research found uses for granular carbon in various industrial operations. Processes were developed to recover gasoline from natural gas, and to extract benzene from manufactured gas.

Granular carbons are employed to purify various industrial gases. Sulfur compounds are removed from hydrogen and from acetylene; pyridine is removed from ammonia. Carbon dioxide for dry ice and carbonated beverages is deodorized.

A large market developed for recovery of vapors of organic solvents used in diverse industrial operations: as processing mediums in the manufacture of plastics and explosives; and as agents to apply a product to its intended use as in painting and printing. Many such solvents are volatile and the escape of the vapors into work rooms creates hazards to health, and from fire and explosion. Moreover, the amount of vapor thus vaporized constitutes an appreciable cost and recovery becomes important for economic reasons.

The need to protect the environment makes it necessary to end the discharge of alien vapors and gases to the atmosphere. Much research is now devoted to the removal of sulfur dioxide from stack gases.

The gas mask principle is employed to provide freshness to air in work rooms and living quarters. Purity of indoor air can be maintained by continuous recirculation through a bed of activated carbon. The incorporation of such use of carbon into an air conditioning system avoids the cost of ventilation with outdoor air during hot summer days. In areas where smog prevails outdoor air should be passed through carbon before being admitted to living quarters. Individuals living in smog areas can then go indoors to get a breath of fresh air. In submarines, activated carbon preserves the freshness of air that is reused very many times.

Liquid Phase Applications

The publicity given to activated carbon in World War I renewed the search for additional liquid phase markets. Although the prewar endeavor had fallen far short of the hopes of the entrepre-

neurs, it resulted in commercial production of carbons having great adsorptive power, and the knowledge and skills acquired were put to use. In addition, experience brought an awareness of the practical value of certain purification properties that had received very little prior attention. As we reflect back on the early use of powdered carbon we realize the dominant objective was color removal, and the term then generally used, *decolorizing carbon,* mirrors the thinking. However, those who used carbon for decolorizing found other benefits of practical value such as: improved flavor of food products; longer shelf life of packaged goods. In some applications, the use of carbon provides benefits to the operation of various stages of the manufacturing process. The presence of traces of adsorbable impurities can create difficulties listed in Table 1–1. Pretreatment of the process liquor with a suitable adsorbent will often avoid those difficulties.

TABLE 1:1

SOME OPERATIONAL DIFFICULTIES WHICH CAN OFTEN BE CORRECTED BY ACTIVATED CARBON

Slow and/or difficult filtration
Retarded and incomplete crystallization
Emulsion formation
Foaming during concentration and/or distillation

The liquid phase applications are reviewed in Chapter 7. Purification of most liquid systems by carbon is accomplished by adsorbing the contamination, the desired purified product remaining in the liquid. A different procedure is employed when a valuable adsorbable substance is present in minute concentrations. Then the desired substance is removed by selective adsorption and subsequently extracted in a purified and more concentrated condition. The operation known as desorption* is effected by specific changes in the environment; thus many vapors can be desorbed by an elevation of temperature. The desorption of substances taken up from liquids is more involved, and the details are examined in Chapter 5. The general features are illustrated by production of penicillin during World War II. In that process, activated carbon was added to an inoculated broth containing about 30 ppm penicillin. After removal from the broth, elution of the carbon yielded a relatively con-

*The overall process is known as adsorption-desorption.

centrated penicillin solution. At the time, adsorption-desorption provided the only immediately available method for large scale production of penicillin. Since then, experiences with adsorption-desorption in liquid systems have provided many disappointments, and the hopes once held for large markets remain unfulfilled. In contrast adsorption-desorption processes have been very successful in many vapor phase applications

When increasing amounts of carbon are added to a system, additional molecules become bonded to the carbon surface. Finally, practically all the adsorbable molecules are anchored at the surface and are immobilized. This feature is utilized to prevent the migration of color bodies in plastic solids as applied in the manufacture of white-sidewall tires. These are formed by placing a layer of white rubber over a carcass of dark rubber. Color molecules in the carcass rubber can migrate and stain the white-sidewall. This staining is prevented by incorporating activated carbon in the carcass rubber. Then, color molecules will migrate only until they reach a particle of carbon, whereupon they are adsorbed and anchored.

The *advanced treatment* (see chapter 8) of domestic and industrial waste waters is a promising future market for activated carbon. Aspects of that market recall the search for ways and means to purify potable water supplies. During the early decades of this century, an acute problem was the presence of objectionable tastes and odors in municipal water supplies. Several factors were responsible. One was the discharge of much greater quantities of domestic and industrial wastes into waterways. Another was the use of chlorine for disinfection.

Intensive research demonstrated that the use of activated carbon was essential to furnish a completely palatable water. Today, the use of activated carbon to purify municipal water supplies extends throughout the entire world. The development encountered many difficulties, and much of the rapid progress can be traced to a spirit of cooperation that prevails within the water works industry.

Most new specialized applications of carbon are initiated by personnel working within the industry because they are in a position to know what needs to be done to improve the operation. However, the progress of a new application can often be expedited by consultation with the carbon supplier.

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Elementary Aspects of Adsorption

1. INTRODUCTION

Atoms and molecules are held together in a solid by cohesive forces that range from strong valence bonds to the relatively weak Van Der Waals forces of attraction. Molecules in the interior of a solid are completely surrounded, consequently their attractive forces are satisfied on all sides. The attractive forces do not cease abruptly at the surface. Instead they extend outward and can capture wandering fluid molecules—a phenomenon that is known as *adsorption*. The terms *adsorbent* and *adsorbate* describe the solid and its captured molecules.

But the forces radiating from the solid surface do not act alone they join with attractive forces radiating from the fluid molecules, and the combined attraction weaves an adsorption affinity. It follows that substances having great affinity will be adsorbed in preference to those with less affinity *if* other forces do not intervene. Other properties such as condensibility of a vapor or solubility of a solute may sometimes augment a certain adsorption and diminish another.

Adsorption is viewed as a surface phenomenon, and it is well to understand the full significance of that concept. Subconsciously, we think of a surface as the exterior of a material substance. However, a surface is really a boundary, because where the mass

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of one body ends, the mass of another begins. Consider a solid immersed in a liquid: there, the surface of the solid faces a corresponding surface of the liquid; and the region enclosed by those two surfaces is an *interface*. It is within that interfacial region that adsorption occurs.

In view of this, it is perhaps unfortunate that we generally refer to adsorption as ocurring on a surface, but it would be pedantic to change the custom. However, we can and should keep in mind that the solid surface is just a part of an interfacial region, and realize that the masses on both sides of the interface participate. The adsorption-affinity which pulls molecules to the interface can be assisted or opposed by forces on the other side of the interface. To take a single example: the dissolving power of the solvent will pull molecules away from the surface and will have a stronger pull for those that are more soluble. Our knowledge of the interplay of all the varied forces operating at the interface is dim and indistinct. However experience has taught us ways and means whereby we can often improve the effectivness of an adsorption process.

2. CHARACTERISTICS OF ADSORPTION FROM GAS/VAPOR PHASE[1,2]

Molecules remain in the gas/vapor state because of the energy of separation and translation contained in the moving molecules. In order to adhere to the interface, that energy must be displaced to bring the molecules to a state of rest. When activated carbon is present in the system, we can visualize that the adsorption-affinity squeezes away the energy, which is released in the form of *heat of adsorption*.

If an insufficient amount of carbon is present, some gas/vapor molecules will not be adsorbed but will remain in the gas/vapor side of the interface. These can be adsorbed by adding more carbon; or alternatively by lowering the temperature of the system. When the temperature is lowered, energy is removed from the moving molecules, and less attractive force is needed to displace the residual energy from each molecule. The force thus saved is available to bring more gas/vapor molecues to a state of rest.

Many features of our present knowledge of adsorption of gas

and vapor are outlined in an investigation of de Saussure[3] published in 1814. He found that porous substances such as charcoal will take up many gases but in different amounts, and that the more condensible gases are adsorbed in greater quantities. Many subsequent studies have confirmed that when adsorptions are conducted at an identical temperature, e.g., 20°C, a correlation exists between the adsorbability and the boiling point, also to the critical temperature. Or as we may otherwise express it, vapors that readily condense to a liquid state will be more readily adsorbed than gases that liquefy only if and when cooled to a low temperature. Such interdependence is to be expected inasmuch as molecules which can more readily slow down to the comparative rest or the liquid state should by the same token be more easily captured by a solid surface.

Early investigators described adsorption as surface condensation; and today it is generally accepted that the forces that bind molecules or a liquid (van der Waals) are a large factor in physical* adsorption at solid surfaces. A strong attraction is contributed by the solid surfaces-this is demonstrated by the fact that vapors will condense on a solid surface under conditions that are inadequate to cause normal condensation to a liquid. A familiar illustration is the way in which dust particles enable a supersaturated vapor to condense to a liquid; or the way dew forms on solid surfaces when the humidity is not sufficient to precipitate as a fog. In presenting the contribution of solid surfaces we must not lose sight of the fact that the cooperation of vapor molecules is essential, and a greater measure of assistance can be furnished by molecules that more readily condense to a liquid.

Gases having a low boiling point and critical temperature are not appreciably adsorbed at 0°C. However, they can be adsorbed in large quantities at a sufficiently low temperature. By appropriate adjustment of the temperature it is often possible to separate such gases by adsorption. The proper temperature depends on the gases to be separated. Yagi[4] points out that carbon dioxide is effectively separated from air between −90°C and −110°C; at higher temperatures carbon dioxide is incompletely adsorbed, and at lower temperature adsorption of nitrogen and oxygen occurs.

*The roles of physical and chemical forces in adsorption phenomena are reviewed in Chapter 13.

Although condensibility is a major factor, the amount of gas or vapor adsorbed is not always in exact correlation with the boiling point or critical temperature. Deviations appear in Table 2:1, and Bancroft[5] and Mantell[6] call attention ot others. The deviations suggest that we lack full knowledge of all forces that are involved. In many studies, the adsorption of different vapors are measured at identical temperatures, a condition that augments the influence of the condensibility factor. This can be illustrated by a comparison of methane (boiling point, −160°C) and methanol (boiling point, 64°C). If both are adsorbed at 25°C it is apparent that the adsorption of methanol will be far greater because of the relative ease or condensation at the temperature.

TABLE 2:1

ADSORPTION OF GASES AT 0° C ON CHARCOAL*

Gas	Boiling point	Critical temperature	Gas adsorbed cc/g charcoal	
			Wood, 10 cm pressure	Coconut, 76 cm pressure
	° C	° C		
Helium	−268	−267	—	2
Hydrogen	−253	−241	0.3	4
Nitrogen	−196	−149	1.5	15
Carbon monoxide	−190	−136	—	21
Oxygen	−183	−119	2	18
Argon	−186	−117	—	12
Carbon dioxide	− 78	+131	20	—
Ammonia	− 33.5	+130	50	—

* From A. B. Lamb, R. E. Wilson, and N. K. Chaney, *Ind. Eng. Chem.* 11:420 (1919), by permission of the American Chemical Society.

Various ways have been searched to equalize the condensibility factor. One approach is to measure the adsorption of vapors at equal ratios of partial pressure to saturation pressure.

Pearce[7] sought to equalize the influence of condensibility by conducting the adsorption of each vapor at a temperature having the same definite ratio to its boiling point. Pearce found a relation to exist between molecular structure and the amount adsorbed, but the relation depends on the pressure at which the adsorption is measured.

At low pressures the adsorption increases with molecular size in an homologous series. The adsorption also increases when an

atom of oxygen. chlorine, or nitrogen is introduced into an organic chemical molecule-an effect that has been interpreted as indicating a direct attraction to carbon for such substituent atoms.

The foregoing behavior occurs only at low pressures. Increasing the pressure promotes the adsorption of smaller molecules. and eventually reverses the order found at low pressures. The reversal has been ascribed to a crowding effect that appears sooner for large molecules which have greater covering power and take more room.

In evaluating the relative adsorbability of vapors, the interpretation of data can depend on whether the amount adsorbed is calculated as mols or grams. Thus carbon N adsorbed approximately the same number of millimoles of chloroform as of carbon tetrachloride, and on that basis, both vapors would be viewed as equally adsorbable. But on a gram basis the carbon tetrachloride would be judged more adsorbable because of a greater molecular weight.[8]

Mixtures of Gases and Vapors

From a mixture of two gases or vapors, the one more strongly adsorbed from the pure state will usually be preferentially adsorbed from a mixture; but each may be less adsorbed from a mixture than when present separately at the same partial pressure in a pure state. In most industrial applications, the adsorptions of some components are so small as to be negligible, and the adsorbable component can be viewed as though it were present alone. An example is the adsorption of organic vapors from air.

Effects of High Pressure

A large increase in pressure has little effect on the adsorption of the more adsorbable vapors, many of which approach saturated adsorption at normal pressures. The less adsorbable gases, however, often show a marked increase at high pressures. The relative amounts adsorbed from mixtures can vary with changes in pressure, Lorenz and Wiedbrauk,[9] in a study of mixtures of ethylene and carbon dioxide, found the adsorption of ethylene to be greater at low pressures; whereas this was reversed at high pressures. Richardson and Woodhouse[10] observed that at 2870 millimeters

pressure, carbon dioxide and nitrous oxide are adsorbed in almost equal volumes; whereas at 72 millimeters pressure, two-thirds of the adsorbed gas is nitrous oxide.

3. CHARACTERISTICS OF ADSORPTION FROM THE LIQUID PHASE

The manifold forces acting at the solid liquid interface provide a complex phenomena that is formidable to analyze. The algerbraic sum of all the forces (Table 2:2) is measured by the quantity of substance adsorbed by a given weight of carbon. We lack quantitative knowledge of *separate strengths* of the individual factors, namely: the solvent the brand of carbon and the solute to be adsorbed. However, experience has developed guide lines that aid in the selection or appropriate operating conditions.

TABLE 2:2

SOME FACTORS INFLUENCING ADSORPTION AT CARBON/LIQUID INTERFACE

- *a)* Attraction of carbon for solute
- *b)* Attraction óf carbon for solvent
- *c)* Solubilizing power of solvent for solute
- *d)* Association
- *e)* Ionization
- *f)* Effect of solvent on orientation at interface
- *g)* Competition for interface in presence of multiple solutes
- *h)* Interactions of multiple solutes
- *i)* Coadsorption
- *j)* Molecular size of molecules in the system
- *k)* Pore size distribution in carbon
- *l)* Surface area of carbon
- *m)* Concentration of constituents.

Adsorbable Solutes

Inorganic compounds reveal a wide range of adsorbability: At one extreme are dissociated salts such as potassium chloride and sodium sulfate which (for all practical purposes) are viewed as non-adsorbable by carbon. At another extreme we find iodine which is one of the most adsorbable substances known. Between those extremes are all degrees of adsorbability. The adsorption of some substances is accompanied by chemical changes.

The published data, on adsorption of organic compounds from solution, provide ample evidence that the architecture of a molecule is an important factor in adsorption phenomena. Some reported trends are listed in Tables 2:3.

TABLE 2:3

INFLUENCE OF MOLECULAR ARCHITECTURE ON ADSORBABILITY

a) Aromatic compounds are in general more adsorbable than aliphatic compounds of similar molecular size.

b) Branched chains are usually more adsorbable than straight chains.

c) Influence of substituent group is modified by position occupied, *e.g.*, ortho, meta, para.

d) Stereoisomers show inconsistent pattern; thus fumaric acid (*trans*) is more adsorbable than maleic (*cis*), but the *trans* form of hydrobenzoin is less adsorbed than the *cis* form.

e) Optical isomers, *dextro* and *levo*, appear to be equally adsorbed.

In comparing the data of different investigations, one should be aware that quantitative data (i.e., precise quantities of substance adsorbed) are reproducible only when the studies are conducted under identical operating conditions. A different temperature or concentration, or the use of a different brand of carbon can alter the amount adsorbed; and on occasion may even reverse the apparent relative adsorbability of some compounds.

Much published data are based on studies of adsorption from solutions containing only a single solute. Such data can seldom be directly related to industrial processes in which the impurities to be removed are generally a mixture of substances of unknown identity. However, studies using a single solute are essential in research seeking to trace the influence of the separate factors in Table 2:2. On this, our information is far from complete. As of now, we see only the probable direction and the possible strength of each influence, the latter only in a very qualitative way. Let us examine portions of what we now know.

Solubility

An increase in solubility reflects a greater affinity between the solvent and the solute, and acts to oppose the attraction to the carbon. Consequently, any change that increases solubility may be accompanied by reduced adsorbability. Thus, polar groups (characterized by an affinity for water) usually diminish adsorption from aqueous solutions. Conversely, the greater adsorption of