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REVIEW

A Review on Surface Chemistry of Activated Carbons

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ABSTRACT

Activated carbons derived from diverse carbonaceous bio-sources have been proving to be effective adsorbents in the removal of contaminants from air and water bodies. The present article reviews emphatically the activation of carbon materials by physical and chemical processes and formation of specific surface (acidic and basic) functional groups on the surface of activated carbons. These groups coupled with high surface area and porosities are endowing to the active carbons good sorption capacity towards potential pollutants through physisorption or chemisorptions or both. These aspects have been discussed. The increase in research interest in exploring the surface affinity of activated carbons in developing simple, economical and eco-friendly methodologies in the control of toxic ions has been discussed.

KEYWORDS

Activated Carbons, Surface chemistry, Toxic ions.

INTRODUCTION

Historical background of activated carbon: Activated carbon is defined to include a wide range of amorphous carbon based materials which have wide variety of properties and physical structures making them to be extensively used in variety of industrial and environmental applications [1]. The conversion of waste materials into more valuable active carbons in the advent of finding environmental friendly solutions, is one of the fascinating aspect pollution control research.

The activated carbons exhibit highly developed surface properties such as an extended surface area and high degree of porosity which are formed during the carbon activation process and show high adsorption capacities [2,3].

The wood chars were used for the reduction of ores in the manufacture of bronze by the Egyptians and Sumerians [4] even in 3750 BC. In Egyptian papyri dating from 1550 BC, the charcoal was used in medicinal applications [5] for the adsorption of odorous vapours-from putrefying wounds and the intestinal tract and the ancient Greeks used the charcoal to ease the symptoms of food poisoning [6]. The beneficial effect was due to the adsorption of the toxins emitted by ingested bacteria and thereby, reducing their toxic effects. Purification of water using charcoal and sand was practiced by Hindus and Egyptians even in 400 B.C. believing that such waters possesses antiseptic characteristics.

Lowitz in the year 1786 studied decolouration of solutions using charcoal and noticed strong sorption nature of the charcoal [7]. Many sugar factories in Europe started using charcoal for decolourization by the year 1808 itself [6]. In year 1811, it was shown that bone char had an even higher decolorizing ability for sugar syrups than wood char. Carbons prepared from coconut shell were used for purification of polluted gases by Hunter in year 1865. In fact, the term 'adsorption' is coined by Kayser in the year 1881 while studying the sorption of polluted gases [6].

Ostrejko [8] prepared active carbons in the year 1900 by loading the bio-material with metal chlorides before carbonization and also by selective oxidization with CO₂ at high temperatures. Using this gasification methodology, Fanto works, Austria prepared first industrial made active carbon with the trade name "Eponit" in the year 1911 which was used to decolorize the sugar and further, the capacity of Eponit was found to be increasing by heating it with zinc salt.

Initially, the powdered activated carbons were used mainly for decolorizing solutions in the chemical and food industries. But during the First World War, the coconut activated carbons were used in gas masks [9]. This new found utilization led to the manufacturing of granular activated carbon (GAC). Subsequently, these were used for water treatment, solvent recovery and air purification.

The production of active carbons is increasing worldwide, because of its application for the treatment of wastewater is picking up. Nowadays, activated carbon is very often utilized in the removal of various organic and inorganic species from surface water, groundwater and wastewater. The consumption of active carbon is the highest in the U.S. and Japan, which together consume two to four times more active carbons than European and other Asian countries. Materials derived from some high carbon content plants are being increasingly probed in exploring the potential uses of them in treating the industrial, agricultural and domestic wastes.

This article reviews the impact of changes in surface chemistry and formation of specific surface functional groups on the surface of activated carbons for the adsorption of contaminants.

Surface chemistry of activated carbons

Properties: Active carbons are not graphitic or graphitizable carbons but they are composed of highly disordered microstructures. Activated carbon is a family of micro porous materials and can be considered as a material of phenomenal surface area made up of millions of pores-rather like a molecular sponge. All non-carbon impurities are removed and the surface is oxidized. Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces [10]. Activated carbons have highly amorphous, micro crystalline and extensively developed internal pore structure. These materials are characterized by their extraordinary large specific surface areas, well-developed porosity and tenable surface-containing functional groups [11,12]. A variety of carbonaceous rich and low levels of inorganic content lignocelluloses agricultural and industrial waste products such as coal, wood, lignin and coconut shell, *etc.* are effective precursors for the production of activated carbons [13].

The preparation of activate carbons involves mainly two steps: physical activation and chemical activation [14]. Activated carbons with tailored properties for the adsorption of particular compounds may be manufactured by controlling the activation process conditions [15]. The final pore structure on the surface of activated carbon is strongly influenced by the nature of the raw material used, the nature of the activating agent and the conditions of the carbonization and activation process [16]. Due to their high adsorption capacity, activated carbons are used in a myriad of commercial applications as adsorbents for the removal of gaseous and liquid pollutants as well as in many other applications.

Significance of activation: Activated carbons, being porous comprising of micro holes and tunnels depending upon the nature and method of preparation of the activate carbon, offer an ideal large surface area for adsorption (holding) of impurities and thereby, purging the solutions. For increasing the sorption affinity towards the toxic ions, the prevailing holes in the active carbons can be filled with suitable materials and thus the surface area acts as a substrate for the doped or loaded materials.

The nature of applications of porous carbon depends on specific surface area, pore structure and nature of surface chemical functional groups [17,18]. Through the activation process, all of the volatile compounds are removed, as layer after layer of carbon atoms are peeled off, enlarging the internal pores and leaving behind a carbon skeleton. By decreasing the number of carbon atoms, the internal surface area of the material is increased.

The surface chemical functional groups which are derived from activation process are found to be responsible for the variety in physico-chemical and catalytic properties of the adsorbents [3,19,20]. So, many researchers focussed on how to modify as well as to characterize the surface functional groups of carbon materials in order to improve or extend their practical applications [19,21,22]. Radovic *et al.* [23] reviewed the carbon materials as adsorbents in aqueous solution and pointed out that the control of chemical and physical conditions might be harnessed to produce carbon surfaces suitable for particular adsorption applications.

Physical structure: The activated carbon adsorption properties are attributed to its physical structure. The process of activation comprises of carbonization of raw material and the subsequently activating it at elevated temperatures ranging from 800 and 1100 °C in the presence of oxidizing gases like carbon-dioxide or steam [24,25]. During carbonization (physical activation) process, most of the non-carbon elements such as nitrogen, oxygen and hydrogen are eliminated as volatile gaseous species by the pyrolytic decomposition of raw material. The residual carbon atoms are comprised to ordered aromatic planes. These graphitic planes form crystallites similar to graphite. However, the angular orientations of the planes in the activated carbon are random to each other, whereas in the graphite they are well ordered. This irregular arrangement of the aromatic sheets leaves free interstices. These give rise to pores which make activated carbons excellent adsorbents. This pore structure in carbonized material is further developed and enhanced with the randomly distributed pores of various sizes

and shapes during the activation process [26]. This leads to an extended and extremely high surface area for the activated carbon. The large surface area of activated carbon relative to the size of the actual carbon particle makes it easy to remove large amounts of impurities in a relatively small enclosed space.

Chemical structure: In addition to porous structure, activated carbon surface has a chemical structure. The adsorption properties are strongly influenced by the chemical structure of the activated carbon surface. Carbon matrix is invariably associated with appreciable amounts of hetero atoms, *i.e.* atoms present in the carbon structure that are not carbon, such as oxygen, hydrogen and small amounts of nitrogen, that exist in the form of functional groups and/or atoms chemically bonded to the structure giving rise to carbon-oxygen, carbon-hydrogen and carbon-nitrogen surface compounds and the presence of these surface compounds modifies the surface characteristics of the carbon. In the carbon matrix, oxygen is the predominant heteroatom present in the form of functional groups, including carboxyl, carbonyl, phenols, lactones, quinones and other groups bound to the edges of the graphite-like layers [10,27]. The nature and concentration of these groups may be modified by various post-activation treatment methods [22], the most important is oxidation.

Chemical activation is a single step process in which carbonization and activation are carried out simultaneously. In chemical activation (oxidation), the precursor is mixed with chemical activating agent and then fired to high temperatures [28,29]. The surface oxygen functional groups of activated carbon are mainly created by two major oxidation methods, namely dry and wet. The former is a method involving reactions with oxidizing gases such as steam, oxygen, carbon dioxide and air at high temperatures ($> 700\text{ }^{\circ}\text{C}$) [30], while wet oxidation involves reactions between activated carbon surfaces

and oxidizing solutions such as aqueous nitric acid, sulphuric acid and orthophosphoric acid, hydrogen peroxide, zinc chloride, potassium permanganate, potassium thiocyanate [31-38] at low or reflux temperatures (about $100\text{ }^{\circ}\text{C}$).

The unique adsorption properties of activated carbons can be significantly influenced mainly by these carbon-oxygen surface functional groups. These groups which are mainly present on the outer surface or edge of the basal plane contribute toward the chemical nature of the carbon. As these outer sites constitute the majority of the adsorption surface, the concentration of oxygen on the surface has a great impact on the adsorption capabilities of the carbon [20,39-41]. The activation process (oxidation) usually involves cleaning out of tars-clogging, forming of new pores and finally, enlarging the surface areas. Due to increase in their adsorptive properties such as high surface area, adequate pore volume and variety of pore size distributions, chemical activation process is preferred [42,43].

Nitrogen functional groups can be introduced on the surface of active carbons by treating the carbons with nitrogen containing reagents such as nitric acid, ammonia, amines, *etc.* [3,44-52]. Sorption nature of the active carbons is due to the nature of surface functional groups present on the inter-surface area and the surface heterogeneity [53]. The nature and abundance of the functional groups depend upon the starting material and/or the treating methods of activation [54,55]. The functional groups and delocalized electrons of the carbon structure, impart either acidic or basic nature [56].

Acidic surfaces: The acidic nature of the activated carbons may be attributed to the presence of oxygen possessing functional groups such as carboxylic, lactone, carbonyl, phenol, pyrone, quinone, chromone and ether groups [57-63,172] are as shown in Fig. 1. The $-\text{COOH}$ groups may also exist in the

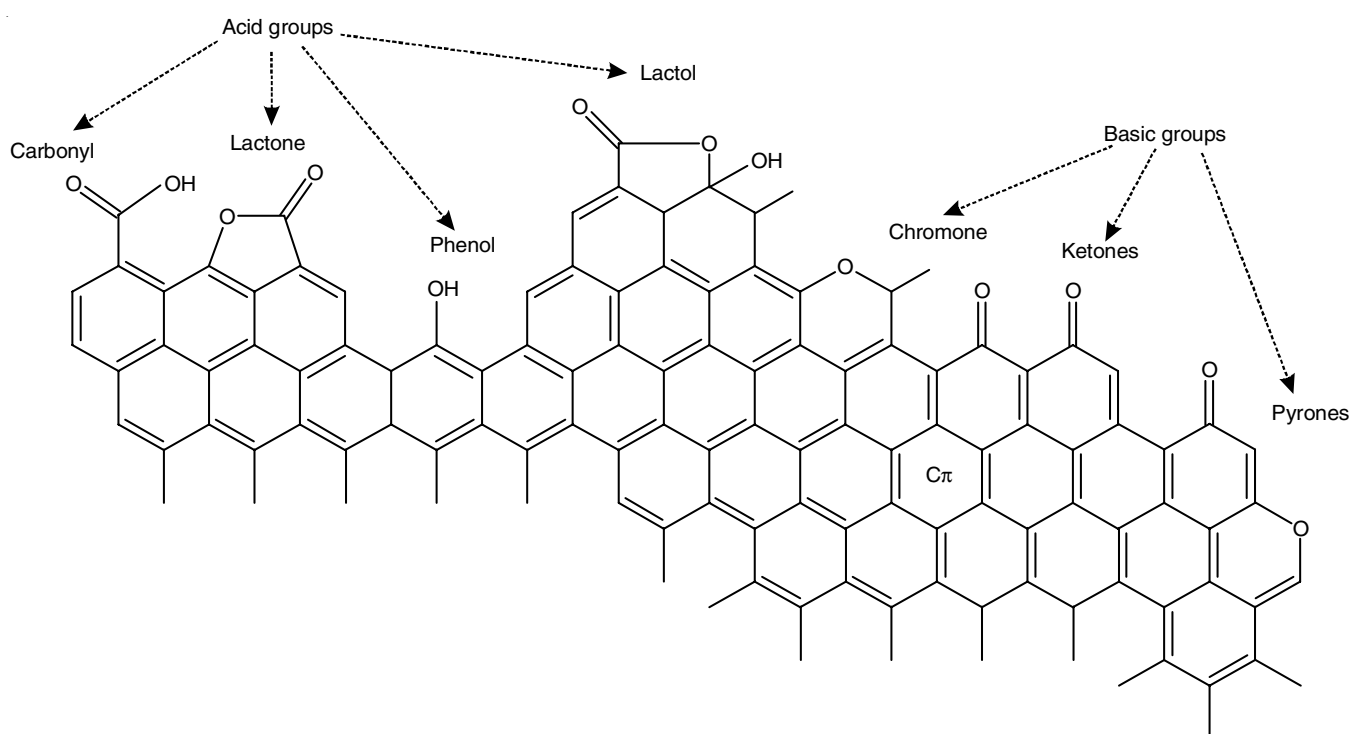


Fig. 1. Acidic and basic oxygen surface functional groups on a carbon basal plane

form of its anhydride [10,39,64-66]. These oxygen functional groups can be classified into three classes according to their chemical properties, *i.e.*, acidic, basic, neutral.

With low temperature oxidation processes, strong acidic groups (*e.g.*, carboxylic) while at elevated temperatures weak acid groups (*e.g.*, phenolic), will be generated on the surface [32-34,64,66]. The oxidation process can be carried either in gas phase or in liquid phase [18,39,57,67-70]. The gaseous phase oxidation increase mainly the concentration of hydroxyl and carbonyl surface groups while liquid phase oxidation done by using nitric acid or an admixture of nitric acid and sulphuric acid incorporates mainly $-\text{COOH}$, phenol and $-\text{OH}$ groups even at low temperatures compared to the gas phase oxidation [18,34].

The treatment with sodium hydroxide replaces H^+ of surface acid groups by Na^+ and thus, the acidity of activated carbon is decreased. Shim *et al.* [21] used the pitch-based activated carbon fibers treated with nitric acid and sodium hydroxide. Coal-based activated carbons are modified by chemical treatment with nitric acid and thermal treatment under nitrogen flow [71].

Basic surfaces: The basic nature of the active carbon is attributed to the nitrogen containing functional groups and/or to the affinity of π -cloud prevailing on the aromatic rings of the sorbents towards the positively charged adsorbates [63,72-75]. Various nitrogen containing functional groups such as amine, amide, imide, pyrrolic and pyridinic groups, *etc.*, are as shown in Fig. 2 [46-51,76-79,172].

The delocalized π -electrons of grapheme layers [58,63,72] could act as Lewis bases [80] and in fact, the contribution of basal planes to basicity has been reported by some researchers [80-82]. Leon *et al.* [72] studied the surface basicity of two series carbons and demonstrated that oxygen-free carbon sites can absorb protons from solution. These sites are located in π -electron rich regions on the basal plane of carbon crystallites. So, some basic sites are Lewis type associated with the carbon

structure itself [64]. Nitrogen functional groups generally provide basic property, which can enhance the interaction between carbon surface and acid molecules such as dipole-dipole, H-bonding, covalent bonding, *etc.* [83]. It was also proposed that certain oxygen containing surface functional groups (Fig. 1) such as chromene [60-62], ketone [84] and pyrone [85] might contribute to the carbon basicity.

Basic surfaces with heat treatment: The decomposition of oxygen-functional groups at the higher temperatures (800-1000 °C) has been found to be causing the increase of basicity to the adsorbent [18,57,82,86]. At low temperatures, strong acidic functional groups like $-\text{COOH}$, anhydrides and lactones are found to be decomposed while at higher temperatures weak acidic groups such as carbonyl, phenol and quinone are decomposed [87,88] (Fig. 3).

At elevated temperatures, large amounts of CO_2 are released causing the loss of major functional groups while loss of CO is minimal resulting pyrone and chromene type structures [87,89]. The treatment of carbons with temperature under hydrogen or inert atmosphere (nitrogen or helium) can increase carbon hydrophobicity by removing hydrophilic surface functionalities particularly various acidic groups [90-92]. H_2 treatment at 900 °C was found to produce highly stable and basic carbons [91].

Thus the basic nature for the thermally treated samples are due to the oxygen-free Lewis basic sites on the graphene layers and also from the few basic oxygen containing groups (pyrone and chromene) remaining in the carbon surface [87,89].

For assessing the nature of functional groups, one of the parameter widely used is pH_{ZPC} and it is the pH at which the surface charge on the active carbon is “zero”. If the pH of the contacting solution is less than pH_{ZPC} , then the surface of the activated carbon acquires positive charge and it is attributed to the protonation of basic groups like pyrones or chromenes or electron rich regions of graphene layers. The negative charge for the active carbon surface is imparted, if the pH of the

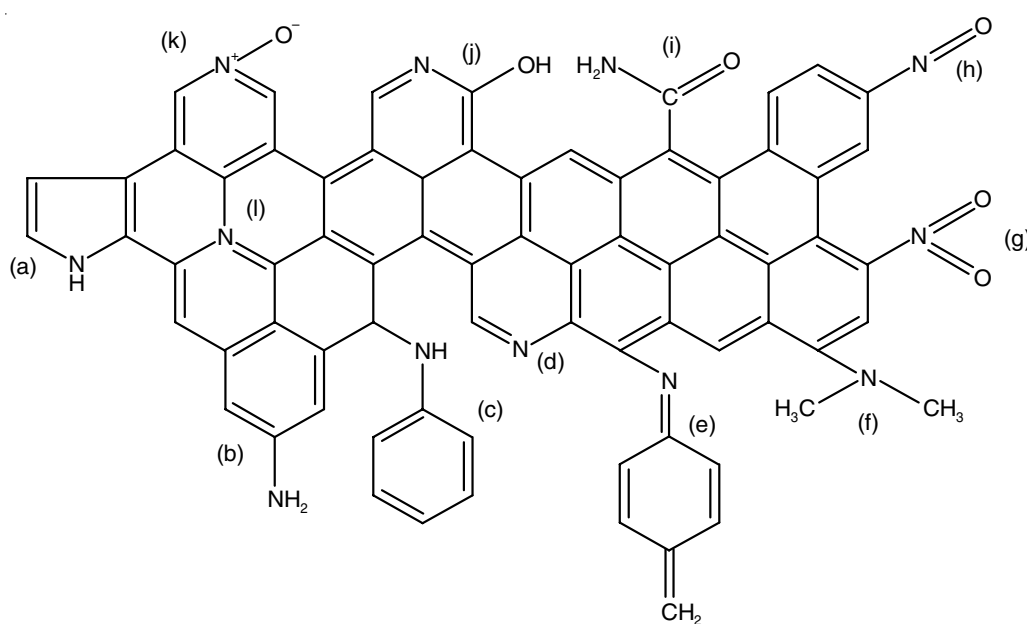


Fig. 2. Nitrogen surface functional groups: (a) pyrrole, (b) primary amine, (c) secondary amine, (d) pyridine, (e) imine, (f) tertiary amine, (g) nitro, (h) nitroso, (i) amide, (j) pyridone, (k) pyridine-N-oxide, (l) quaternary nitrogen

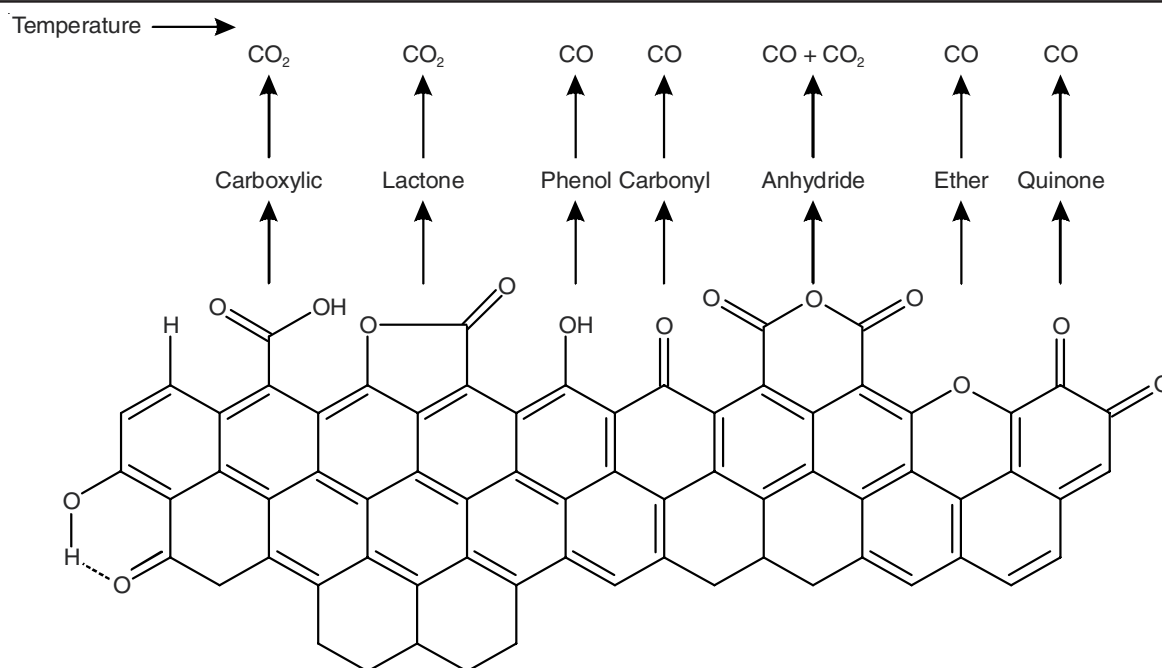


Fig. 3. Decomposition of oxygen containing groups on carbon surface [Ref. 172]

contacting solution is more than pH_{ZPC} [93] and it is due to the dissociation of functional groups like $-\text{OH}$, $-\text{COOH}$.

Removal of contaminants from water: Due to the increasingly stricter air and water legislation, the interest for the preparation of activated carbons with certain surface functional groups has been arisen, for the removal of particular substances with a variety of chemical properties. The term activated carbon defines a group of materials with highly developed internal surface area and porosity and hence a large capacity for adsorbing chemicals from gases and liquids. The major toxic impurities present in water are inorganic, heavy metals (arsenic, lead and mercury), ionic (fluoride and cyanide), organic (phenol and tri chloro ethylene) and microbial contaminants. Activated carbons are efficiently used in several pollution control processes due to their high adsorption capacity. Activated carbons are being extensively used as adsorbent in a variety of industrial and environmental applications [1,94,95]. These active carbons are found to be effective in removing the toxic organic substances and metal ions of environmental or economic concern from air, gases, potable water and wastewaters [96] besides their emphatic use in the removal of a large number of contaminants from a liquid or gas stream during their passage through an activated carbon bed in the process of purification or recovery of the chemical constituents.

The activated carbons also find use as catalysts or catalyst supports [97-99]. The important properties such as surface area, pore volume and pore size distribution are strongly associated to the adsorption capacity [3]. The large surface area and high pore volume are rendering these active carbons as good catalysts and further, large total surface area supports the accessibility of active sites relating to the catalytic activity [100].

Course of action in removal of contaminants: Adsorption is one of the most effective and economic techniques. The nature of functional groups present on the surface of adsorbent, has profound say in effecting the adsorption process

[93]. Adsorption is the formation of a gaseous or liquid layer by molecules in a fluid phase on the surface of a solid either by molecular attraction of the vander Waals type or by chemical bond formation between the adsorbate to the functional groups presents on the surface of the adsorbent. The former is said to be the physisorption and latter is chemisorptions.

The molecules of the contaminants present in the water are adsorbed onto the heterogeneous surfaces of the activated carbon by reversible physical attraction or by irreversible chemical bond formation. Physical attractions do not alter the adsorbate molecular structure while chemical adsorption results in changing the adsorbate molecular structure. Activated carbon adsorption proceeds through three basic steps after adsorbates diffuse to the active site.

- Substances adsorb to the exterior of the carbon surface.
- Substances move into the carbon adsorption pore with the highest adsorption potential energy.
- Substances adsorb to the interior graphitic platelets of the carbon.

Fig. 4 depicts the adsorption process showing transfer of adsorbate molecules through the bulk gas phase to the surface of the solid and diffusion onto the internal surfaces of the pores in the solid adsorbent [173]. Activated carbons have different kinds of pore systems varying in size and shape; the pore size ranges from a nanometer to thousands of nanometers.

Depending on the preparation methods used, the pore sizes of the activated carbon can be categorized by their sizes [14] usually into three groups (i) macro pores having average diameter more than 50 nm, (ii) meso pores with diameter 2-50 nm and (iii) micro pores having average diameter less than 2 nm. These are further divided into super micro pores (0.7-2.0 nm) and ultra micro pores of diameter less than 0.7 nm. The heterogeneity of activated carbons is due to the presence of these pores and in fact, the micro pores and mesopores contribute to the internal surface to a great extent and thereby, the total pore volume.

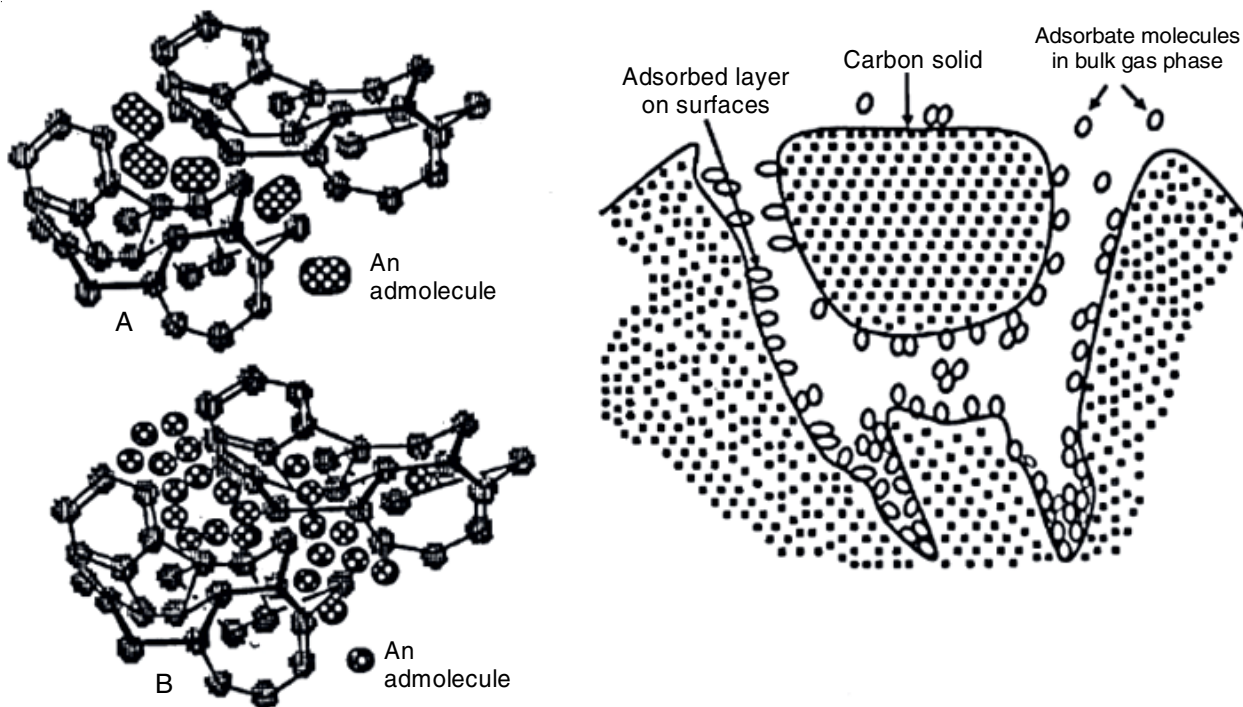


Fig. 4. Adsorption process in activated carbons: Transfer of adsorbate molecules to adsorbent

TABLE-1
LIST OF A FEW ACTIVATED CARBONS IN REMOVAL OF POLLUTANTS FROM WASTE WATERS

Pollutant	Activated carbons	Ref.	Pollutant	Activated carbons	Ref.
Arsenic	<i>Peltophorum pterocarpum</i>	[104]	Manganese	Coconut shells	[114]
	Jute stick	[105]		<i>Ziziphus spinachristi</i> seeds	[134]
	Coconut husk	[106]		Coconut shells	[114]
Cadmium	Palmyra palm fruit seed	[107]	Iron	Date pits	[127]
	Olive stones	[108]		Acanthaceae	[135]
	Date stems	[109]	Cobalt	Date pits	[127]
	Wood apple shell	[110]		<i>Thespesia populnea</i> bark	[136]
Copper	Wood of derris indica	[111]	Molybdenum	Coir pith	[137]
	Pigeon pea husks	[112]	Vanadium	Coconut coir pith	[138]
	Olive stones	[108]			
	Agricultural materials	[113]			
Chromium	Coconut shells	[114]	Dyes	Activated carbons	Ref.
	Cucumis melo peel	[115]	Acid blue 25	Pecan nut shells	[122]
	Wood apple shell	[116]	Azure B	Rice husk	[139]
	Gingelly oil cake	[117]	Methylene blue	Rice husk	[140]
	Peanut shell	[118]		Palm kernel shell	[141]
<i>Ricinus communis</i> seed shell	[119]	Coconut Shell		[142]	
Lead	Palm shell	[120]	Rice husk	[143]	
	Pine cone	[121]	Eriochrome black T	Mosambi peel	[144]
	Pecan nut shells	[122]	Aniline blue	Rice husk	[145]
	<i>Mangostana garcinia</i> shell	[123]	Rhodamine-B	Rice husk	[146]
	Maize tassel	[124]	Orange G and Safranin O	Tamarind seed	[147]
Mercury	Olive stones	[108]	Phenol	Babul sawdust	[148]
	Coconut shell	[125]		Rice husk	[149]
	Maize cob	[126]	4-Chloro-2-methoxyphenol	Date-pit	[150]
	Date pits	[127]	2,4-Dichlorophenol	Oil palm shell	[151]
	Walnut shell	[128]	Nitrate and nitrite	Agricultural waste	[152]
	Olive stones	[129]	Nitrate	Olive stones	[153]
	Palm shell	[130]	Fluoride ion	Pistachio, walnut and almond shells	[154]
Nickel	Wood apple shell	[131]		Coconut coir pith	[155]
	Olive stones	[108]	Activated coconut shell	[156]	
	Maize cob	[126]	Rice straw	[157]	
	Coconut shells	[114]	Cashew nut shell	[158]	
Zinc	Date pits	[127]	<i>Typha angustata</i>	[159]	
	Van apple pulp	[132]	<i>Vitex negundo</i>	[160]	
	Rice Husk	[133]			

In recent years, there is a growing research interest in the production of activated carbons and surface modified activated carbons from renewable and inexpensive precursors which are mainly industrial, plant materials and agricultural by-products, particularly used as water-purifying agents in wastewater treatment techniques [101,102].

In adsorption technique, active carbons are used as most effective adsorbents for the removal of a variety of pollutants such as heavy metals, fluorides, dyes, phenols and other organic and inorganic toxic compounds from aqueous solutions. The activated carbon with high surface area, micro porosity and wide variety of surface functional groups show good adsorption capacity in the removal of pollutants from wastewaters through physiosorption or chemisorptions. The presence of functional groups on carbon surface such as carboxylic, hydroxyl, carbonyl, ether, quinone, lactone and anhydride indicates the occurrence of many types of pollutant-carbon interactions [103].

The naturally occurring, low-cost and more effective activated carbons derived from plant and wood based agricultural biomaterials are investigated by various researchers. Some of the examples are listed in Table-1.

Besides the above pollutants, activated carbon has also been successfully used for the removal of detergents [161,162], pesticides [163,164], humic substances [165,166], chlorinated hydrocarbons [167,168] and many other chemicals and organisms [169-171].

Conclusion

In this study, the impact of changes in surface chemistry and formation of specific surface functional groups on the surface of activated carbons for the adsorption of contaminants is reviewed. A variety of carbonaceous rich materials such as coal, wood, lignin and coconut shell, *etc.* are used for the production of activated carbons. During physical activation process in the absence of air, most of the non-carbon elements such as nitrogen, oxygen and hydrogen are eliminated as volatile gaseous species by the pyrolytic decomposition of the raw material. In chemical activation process, the oxygen containing surface functional groups are produced by treating with oxidizing gases such as steam, oxygen, carbon dioxide and air at high temperatures ($> 700\text{ }^{\circ}\text{C}$) (dry oxidation) or by reacting with oxidizing solutions such as aqueous nitric, sulphuric and orthophosphoric acids, hydrogen peroxide, zinc chloride, potassium permanganate, potassium thiocyanate at low temperatures (about $100\text{ }^{\circ}\text{C}$) (wet oxidation) and the nitrogen containing surface functional groups are produced by treating with nitric acid, ammonia, amines, *etc.*

The acidic nature of the activated carbons is due to the presence of oxygen possessing functional groups such as carboxylic, lactone, carbonyl, phenol, pyrone, quinone, chromene and ether groups. The basic nature of the activated carbons is due to the presence of various nitrogen containing functional groups such as amine, amide, imide, pyrrolic and pyridinic groups, *etc.* It is also observed that the basicity of the activated carbon is increased due to the functional groups such as pyrone and chromene type which are formed by the decomposition of oxygen-functional groups at the higher temperatures.

The activated carbons are used as most effective adsorbents in adsorption technique. Thus we conclude that the activated carbons with high surface area and porosity show good adsorption capacity in the removal of pollutants from wastewaters through physiosorption or chemisorptions.

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