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A Review on Activated Carbon: Preparation, Characterization and Applications

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Abstract: Charcoal has been used as an adsorbent material since ancient times. Its activation enhances the adsorbing capacity of the carbon and multiplies it manyfold. Activated carbon has proven to be very useful for removing impurities such as toxic metals, heavy metals, corrosive products, colour, odour, toxins, etc. from water, soil, air, the human body, and so on. The use of activated carbon is unlimited and dates back to very ancient times. It is cited in medicines and purification processes in Vedic and Egyptian literature. This paper describes in detail the various materials used by many researchers for the preparation of activated carbon. The other important parameters that are discussed in the paper are activating agents, activation temperature, carbonization, effect of various parameters, characterization techniques, etc. The paper also deals with the applications of activated carbon described by many authors in the purification and removal of impurities from various industrial effluents. The use of agro wastes as precursors for activated carbon material is mainly emphasized in the present study to make the material cheap and the purification process cost-effective.

Keywords: Activated carbon, Activation, Agro wastes, Adsorption, cost effective

I. INTRODUCTION

Activated carbon (AC) has been known for its useful properties and applications since ancient times. This traces back to 1500 BC, Egyptians used charcoal as an adsorbent for medicinal purposes and a purifying agent. Around 420 BC, Hippocrates dusted wounds with powdered charcoal to remove their odour. Ancient Hindu societies purified their water by filtering it through charcoal. In 1773, the Swedish chemist Karl Wilhelm Scheele was the first to observe the adsorption of gases on charcoal [1]. AC has always played a key role in chemistry. Recently, AC have been used to remove colour from pharmaceutical and food products, as air pollution control devices for industrial and automobile exhaust for chemical purification, as electrodes in batteries, etc. It has wide applications in body products, medicines, the purification of water, etc. All these applications make it an interesting subject to be studied in detail.

II. ACTIVATED CARBON

Activated Carbon (AC) is a solid, highly porous, dark, and tasteless carbonaceous material. Many scientists have defined AC [4], according to Marsh (1989) AC as "*a porous carbon material, usually chars, which have been subjected to reaction with gases during or after carbonization in order to increase porosity*". Norlia Baharun (1999) explained AC as: "*Activated carbon is an organic material that has an essentially graphitic structure*". Benaddi (2000) described AC on the basis of structure according to him "*the main features common to all of activated carbon are, graphite like planes which show varying degrees of disorientation and the resulting spaces between these planes which constitute porosity and the unit of condensed aromatic rings are referred to as basic structure units.*" Thus, the AC can generally be defined as a porous material obtained from the carbonization of organic materials from the liquid and gas phases [2]. Its pore volume typically ranges from 0.20 to 0.60 cm³/g and has been found to be as large as 1 cm³/g. Its surface area ranges typically from 800 to 1500 m²/g [3], but has been found to be in excess of 3000 m²/g.

III. SELECTION OF RAW MATERIALS FOR ACTIVATED CARBON

Organic materials rich in carbon can be used as raw materials for the manufacture of AC. Factors that are considered for the selection of the raw materials are listed below.

- 1) High carbon content
- 2) High density
- 3) An appreciable amount of volatile content
- 4) Low inorganic content
- 5) Potential extent of activation



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- *6)* Inexpensive material
- 7) Agro-waste materials
- 8) Low degradation upon storage

Materials rich in cellulose and lignin (fibres) are generally used for activation, keeping in mind that they should possess a low organic content but a relatively high volatile content that helps control the manufacturing process [4]. Agro wastes (raw materials) generally used for carbonization are: Gulmohar fruit shell [4], Karanj fruit shell [5], Almond tree pruning [6], Apricot stones [7], hazelnuts, Pistachios, walnuts [8] etc. They possess high density, high carbon content, hardness, and volatile content that are ideal for the manufacture of AC, as studied by many authors. The studies show the following order of suitability for the production of AC, coconut shells > peach stones > plum stones > hazelnut shells > walnut shells > cherry stones. Some of these raw materials include the fruit shells, the stones of fruits, woody materials, metal carbides, carbon blacks, scrap waste deposits from sewage, and polymer scraps. Different types of coal that already exist in a carbonaceous form with a developed pore structure can be used to create AC.

IV. METHODS OF ACTIVATION OF RAW MATERIALS

A. Physical Activation Method or Gas Activation Method

The physical activation method has been explained to be completed in two steps,

- Carbonization of the carbonaceous precursor in an inert atmosphere
- Activation of the resulting char in the presence of carbon gasification reactants such as carbon dioxide, steam, air, etc.

In this method, the pores are created by the reaction between the carbon atom and the oxidizing gas. During this reaction, the opening of the closed micropores takes place as a result of devolatilization. This is a major drawback of this method, as a large amount of internal carbon mass is eliminated to obtain a well-developed pore structure [9].

- 1) Advantages
- Avoids the incorporation of impurities from the activating agent.
- The process is not corrosive.
- Washing stage is not required.
- Cheaper

2) Disadvantages

- The activated carbon are obtained in two steps.
- Higher temperatures of activation (800–1000 °C)
- Poorer control of the porosity

B. Chemical activation method

It is explained as a single-step process for the preparation of AC. Here, the carbonization of an organic precursor is done in the presence of chemical agents. Activating agents such as KOH [10], NaOH [11], Li₂CO₃, Na₂CO₃, K₂CO₃ [12], Rb₂CO₃, Cs₂CO₃ [13], ZnCl₂ [14], H₃PO₄ [15], CH₃COOH [16] etc. are employed for activation. These agents facilitate dehydration that promotes pyrolytic decomposition, thus inhibiting the formation of tar, and enhancing the yield of carbon. Activation temperatures are low in chemical activation compared to the physical activation process [9].

1) Advantages

- Shorter activation time
- Lower temperature of pyrolysis (600 °C to 800 °C)
- Better control of textural properties
- High yield
- High surface area of the AC
- Well-developed microporosity
- Narrow micropore size distributions
- Reduction of the mineral matter content



2) Disadvantages

- Corrosiveness of the process
- Requires a washing stage.
- Inorganic impurities
- More expensive [16]

V. TEMPERATURES FOR CARBONIZATION

During carbonization [17], most of the non-carbon elements, hydrogen, and oxygen are first removed in gaseous form by pyrolytic decomposition of the starting materials, and the carbon atoms get organized into crystallographic formations known as elementary graphite crystallites. The temperature plays an important role during the carbonization of the activated precursor; hence, proper care should be taken during carbonization. Carbonization temperature up to 400 $^{\circ}$ C there is water removal, cellulose evaporation, and lignin evaporation, while for carbon refining processes occur at a temperature of 500 – 800 $^{\circ}$ C [18].

VI. TEST FOR QUALITY STANDARDS FOR ACTIVATED CARBON

A. Moisture Content

It is undesirable as it lowers the calorific value of the AC. It is defined as the loss in weight of the AC after heating to around 105 - 110 °C for 1 hr in a hot air oven [19 - 21].

Percentage of moisture =
$$\begin{array}{c} \text{Loss in weight} \\ \hline \text{Weight of AC} \end{array} X 100$$

B. Volatile Matter Content

Volatile matter is the presence of combustible and non-combustible gases present in the AC such as sulphur, CO_2 , nitrogen, methane etc. It is determined as the loss in weight of the moisture free AC when heated at 950 °C in a muffle furnace for about 7 mins [19 - 21].

Percentage of volatile matter = $\frac{\text{Loss in weight}}{\text{Weight of moisture free AC}} X 100$

C. Ash Content

Ash is an undesirable matter in the AC and it is a mixture of minerals which if present can lead to clogging of the pores on the surface of AC thus reducing its adsorption capacity and also the mechanical strength of AC. Weight of the moisture free AC when heated at 800 $^{\circ}$ C in a muffle furnace for about 4 hours to become ash [19 - 21].

D. Fixed Carbon

It's the most desirable content in the AC and is determined by deducting the sum of moisture, volatile matter and ash content from 100 [19 - 21].

Percentage of fixed carbon = 100 - % (moisture + volatile matter + ash) content

E. pH of the Activated Carbon

pH of the AC can be defined as the pH of a suspension of AC in distilled water. The presence of acidic functional group such as carboxyl, phenolic and others on the surface of AC may cause the acidic property of AC [21] and affects the adsorptive capacity of the AC.



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VII. CHARACTERIZATION OF ACTIVATED CARBON

The various techniques employed to characterize the surface of the AC are summarised as follows:

A. X-ray Photoelectron Spectroscopy (XPS)

Use of X-ray photoelectron spectroscopy to identify the functional states of carbon that exist on the surfaces of various AC. It also helps in determining the relative percentages of carbon, oxygen, and trace elements that comprise the surfaces of the AC [22].

B. Scanning Electron Microscope (SEM)

The surface structure and development of porosity of AC can be determined by Scanning Electron microscopy [23 - 25]. The SEM micrographs of AC can help in identifying the cavities, pores, and rough surfaces on the AC samples. With its help, the influence of the activating agent on the topographical characteristics of the carbon surface can be determined.

C. X-Ray Diffraction Spectrometer (XRD)

The crystalline structure of the AC was determined by XRD. [25, 26]

D. IR Spectroscopy

IR Spectroscopy provides qualitative information on characteristic functional groups on the surface of AC [25].

E. Fourier Transform Infrared Analysis (FTIR Spectrometer)

FTIR Spectrometer was employed to determine the presence of surface functional groups in AC [24 - 28].

F. Single-point BET Surface Area Analysis

To study the effect of different activation methods on the surface area of AC samples, a single-point BET surface area analysis is done. This analysis technique helps in determining the specific surface area and porosities of the samples before and after activation. [28]

G. FESEM Analysis

FESEM micrographs provide information on the structural changes in raw materials for analysis during the activation process. [25]

VIII. EFFECTS OF VARIOUS VARIABLES ON THE ADSORPTION PROCESS

The significance of various parameters such as contact time, dye volume, adsorbent dosage and pH plays an important role on the adsorption of dye and are described as under:

A. Effect of Contact Time

Contact time refers to the duration of time the adsorbent is in contact with the dye solution. Adsorption of dye on adsorbent surface is fast initially leading to the formation of monolayer. The adsorption continues but slows down gradually till equilibrium is reached. This is due to a large number of vacant surface sites at the initial stages available for adsorption but after a interval of time, the vacant surface sites are difficult to be occupied by the adsorbate due to the repulsive forces acting between the adsorbate molecules on the AC and in the solution [26]. However, after a legging period the percentage colour removal and adsorption of dye on the AC increases. Adsorption process, can alternatively be explained as a physical phenomenon where the adsorbate molecules reach the boundary of the AC and get diffused into the adsorbent layer from where they penetrate and reach into the interior of the adsorbent. Since, this is a lengthy process hence it requires extended contact time between adsorbent and adsorbate [27].

B. Effect of Dye Volume

The adsorption of dye on AC decreases with an increase in the dye volume. This is due to the competition among the adsorbate molecules (dye) for getting adsorbed on the AC with increasing volume. The concentration of the dye in the solution generally increases with the increase in volume of dye thus lowering its percentage removal from the solution. However, qe which is the maximum adsorption capacity of AC increases with an increase in the volume of the dye. This is due to the increase in the number of available binding sites when the dye concentration is increased [28].



C. Effect of Adsorbent Dosage

The quantity of AC in grams required for the removal of adsorbate molecules (dye) from its aqueous solution is called adsorbent dosage. The determination of dosage of AC plays an important role as it not only determines the efficiency of AC for removal of adsorbate but is also a measure of prediction of the cost of AC per ml of solution to be treated. The efficiency of removal of adsorbate increases significantly as adsorbent dosage is increased. This is due to the increase in the surface area available for adsorption along with the availability of more active binding sites on the adsorbent surface. However, the maximum adsorption capacity (qe) shows a reverse tendency i.e, with the increase in adsorbent dosage, the adsorption capacity decreases. This is due to the decrease in the total adsorption surface area available to adsorbate molecules for getting adsorbed and is a result of overlapping or clustering of adsorbent gets reduced, which further results in a decrease in qe value [29-32].

D. Effect of pH of the Dye Solution

pH is an important parameter which affects the physical phenomenon of adsorption on the surface of adsorbent and dye in the solution [33, 34]. It controls the charge on the adsorbent surface and the degree of ionization of dye in the solution. It not only affects the dissociation of functional groups on the active sites of the adsorbent molecules but also in the solution of the dye. The effect of pH is due to the H^+ and OH ions that affects the adsorption of other ions on the surface of the adsorbent. These ions also affect the adsorption of dye molecules on the surface of the adsorbent.

VIII. APPLICATIONS

AC finds a variety of applications in day-to-day life of great importance, such as: It is used in the removal of gasoline vapours from automobile exhaust, and in the removal of heavy metal ions (mercury, lead, cadmium, molybdenum, etc.) from drinking water. In the removal of organic impurities from plating bath solutions in metal finishing. In the removal of overdosage of the drugs or poison by oral ingestion of AC.

Used as the stationary phase in low-pressure chromatographic separation of carbohydrates (mono-, di-trisaccharides) where ethanol solutions are in the mobile phase. It is used for the measurement of radon concentrations in the air. It is largely employed in water filtration systems to remove colour.

Filters charged with activated charcoal are employed to purify compressed air. It is also used in gas purification to remove hydrocarbons and oil vapours, as well as different odours, from the air. AC is commonly used as a purifier to purify organic solutions from undesirable organic impurities. AC saturated with sulphur or iodine is extensively used to remove mercury from coal-fired power stations, furnaces, and wells containing natural gases [35 - 51]. Thus, AC is used in the removal of almost all the impurities, such as odour, colour, heavy and toxic metals, and complex organic compounds, from the solutions or water without disturbing the ecological balance of nature.

IX. CONCLUSION

The present study reveals that AC can be used in purification processes very effectively. The major concern is the high cost involved in its production, which renders the purification processes very expensive and almost unaffordable for common people. The authors suggest using agro-waste, waste generated from ornamental trees and plants, domestic waste, etc. as precursors for activation. The other areas that should be addressed are the minimization of energy consumption by optimizing the temperature and the minimal use of chemicals employed for activation. This can be achieved by adopting green activation techniques. The present study will definitely be a great help to the researchers and students working in this field in their better learning and understanding of AC.

X. FUTURE SCOPE

Activated Carbon has a wide range of applications in almost all industries. Academic research on low-cost precursors, yield of AC, adsorption efficiency, regeneration of AC, and green activation techniques are the areas where attention is required.

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