

Production and Characterization of Activated Carbon Produced from a Suitable Industrial Sludge

Laxman Attri¹, Kuldeep Singh²

¹Assistant Professor, Department of Mechanical Engineering, Rattan Institute of Technology and Management, Haryana, India

²Research Scholar, Department of Mechanical Engineering, Rattan Institute of Technology and Management, Haryana, India

ABSTRACT

The purpose of this study is the preparation of activated carbon using a suitable industrial sludge. The characterization of two types of industrial sludge's showed that paper sludge was a better option over rice husk. The activated carbon produced from pyrolysis of paper mill sludge was chemically activated with various activating agents like zinc chloride, potassium hydroxide and potassium chloride. A systematic investigation of the effect of impregnation ratio, activation temperature and activation time on the properties of the activated carbon was done. The chemically activated carbons were characterized by measuring iodine and yield percentage. The activated carbon prepared from paper mill sludge in this study had maximum iodine of 764.80 mg/g with ZnCl₂ as the activating agent. The FT-IR analysis depicts the presence of a variety of functional groups. The XRD analysis reveals that the produced activated carbon has low content of inorganic constituents compared with the precursor.

INTRODUCTION

Activated carbon, also widely known as activated charcoal or activated coal is a form of carbon which has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions.^[1] The word active is sometimes used in place of activated. Due to such high degree of micro porosity, just 1 gram of activated carbon has a surface area in excess of 500 m² (about one tenth the size of an American football field), as typically determined by nitrogen gas adsorption. Sufficient activation for useful applications may come solely from the high surface area, though further chemical treatment generally enhances the adsorbing properties of the material. Activated carbon is most commonly derived from charcoal.

Preparation of Activated Carbon:

Activated carbon is nothing but carbon produced from carbonaceous source materials like nutshells, peat, wood, coir, lignite, coal and petroleum pitch.

LITERATURE REVIEW

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.^[5] These materials are characterized by their extraordinary large specific surface areas, well-developed porosity and tunable surface-containing functional groups^[6]. For these reasons, activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater.^[7] The surface oxygen functional groups can be easily introduced to the carbon by different activation methods including dry and wet oxidizing agents.

Dry oxidation methods involve the reaction with hot oxidizing gas such as steam and CO₂ at temperatures above 700 C.^[8] Wet oxidation methods involve the reaction between the carbon surface and solutions of oxidizing agents such as phosphoric acid H₃PO₄, nitric acid HNO₃, hydrogen peroxide H₂O₂, zinc chloride ZnCl₂, potassium permanganate KMnO₄, ammonium per sulphate (NH₄)₂SO₈, potassium hydroxide KOH, etc. From the above oxidizing agents, phosphoric acid and zinc chloride are usually used for the activation of lignocellulose materials, which have not been carbonized before.^[9] On the other hand, potassium hydroxide is usually used to activate coal or char's precursors. It has been reported that zinc chloride produces activated carbon with higher specific area than that produced by using phosphoric acid.^[10] However,

phosphoric acid activation is widely preferred over zinc chloride because $ZnCl_2$ has bad environmental impact and the activated carbon produced when using it cannot be used in the food and pharmaceutical industries. ^[11] Activated carbon usually increases the cost of the treatment process.

Classification:

Activated carbons are complex products which are difficult to classify on the basis of their behavior, surface characteristics and preparation methods. However, some broad classification is made for general purpose based on their physical characteristics.

Powdered activated carbon(PAC)

A micrograph of activated charcoal under bright field illumination on a light microscope. Notice the fractal-like shape of the particles hinting at their enormous surface area. Each particle in this image, despite being only around 0.1 mm wide, has a surface area of several square meters. This image of activated charcoal in water is at a scale of 6.236 pixels/ μm , the entire image covers a region of approximately 1.1 by 0.7mm. Traditionally, active carbons are made in particulate form as powders or fine granules less than 1.0 mm in size with an average diameter between .

Granular activated carbon(GAC)

Granular activated carbon has a relatively larger particle size compared to powdered activated carbon and consequently, presents a smaller external surface. Diffusion of the adsorbate is thus an important factor. These carbons are therefore preferred for all adsorption of gases and vapors as their rate of diffusion are faster. Granulated carbons are used for water treatment, deodorization and separation of components of flow system. GAC can be either in the granular form or extruded. GAC is designated by sizes such as 8×20, 20×40, or 8×30 for liquid phase applications and 4×6, 4×8 or 4×10 for vapor phase applications. A 20×40 carbon is made of particles that will pass through a U.S. Standard Mesh Size No. 20 sieve (0.84 mm) (generally specified as 85% passing) but be retained on a U.S. Standard Mesh Size No. 40 sieve (0.42 mm) (generally specified as 95% retained). AWWA (1992) B604 uses the 50-mesh sieve (0.297mm) as the minimum GAC size. The most popular aqueous phase carbons are the 12×40 and 8×30 sizes because they have a good balance of size, surface area, and head loss characteristics.

Extruded activated carbon(EAC)

Extruded activated carbon combines powdered activated carbon with a binder, which are fused together and extruded into a cylindrical shaped activated carbon block with diameters from 0.8 to 130mm. These are mainly used for gas phase applications because of their low pressure drop, high mechanical strength and low dust content.

Polymer coated carbon:

This is a process by which a porous carbon can be coated with a biocompatible polymer to give a smooth and permeable coat without blocking the pores. The resulting carbon is useful for hemoperfusion. Hemoperfusion is a treatment technique in which large volumes of the patient's blood are passed over an adsorbent substance in order to remove toxic substances from the blood.

Other: Activated carbon is also available in special forms such as cloths and fibers. The "carbon cloth" for instance is used in personnel protection for the military.

Applications of activated carbon:

Activated carbon is used in gas purification, gold purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks and respirators, filters in compressed air and many other applications.

Recently Activated Carbon filters have gained popularity among recreational users of Cannabis, and other smoking herbs for their use in effectively filtering out "Tar" from the smoke. They are becoming quick competition for Vaporizers as they are only a fraction of the cost and achieve nearly the same thing.

PRECURSOR SELECTION:

Precursor (raw material) selection for the production of activated carbon was obviously the first step of the project. Conventionally, activated carbon is produced from carbonaceous source material such as wood, peat, coal, and wastes of vegetable origin (e.g. nutshells, fruit stones).

Today, one promising approach for the production of cheap and efficient activated carbon is the reuse of waste sludge, such as bio solids produced at municipal or industrial waste water treatment facilities. The usage of waste sludge is

especially important due to its mass production and resulting occupation of valuable landfill space.

CHNS elemental analyzer:

CHNS elemental analyzers provide a means for the rapid determination of carbon, hydrogen, nitrogen and Sulphur inorganic matrices and other types of materials. They are capable of handling a wide variety of sample types, including solids, liquids, volatile and viscous samples, in the fields of pharmaceuticals, polymers, chemicals, environment, food and energy.

Proximate Analysis:

The proximate analysis of a substance is a simple means of determining the distribution of products obtained when the coal sample is heated under specified conditions. As defined by ASTM D 121, proximate analysis separates the products into four groups: (1) moisture, (2) volatile matter, consisting of gases and vapors driven off during pyrolysis, (3) fixed carbon, the nonvolatile fraction of coal, and (4) ash, the inorganic residue remaining after combustion. Proximate analysis is the most often used analysis for characterizing a material in connection with their utilization.

Volatile matter:

The sample was measured and placed in a closed crucible. It was then heated up to 925 C for exactly 7.5 min in a furnace. The crucible was then cooled in a desiccator and weighed

Ash content:

Sample was measured and taken in a crucible. It was then heated to 750 C for 1.5 hr. During this test the crucible was left open. After the required heating, the crucible was cooled in a desiccator and then weighed.

Moisture content:

Sample was measured and taken in a petri dish. It was spread nicely on the dish. It was then heated at 105 C for 1.5 hr. the petri-dish was left open during the heating process. After heating petri-dish was removed, cooled in desiccator and then weighed.

CHARACTERIZATION OF ACTIVATED CARBON PRODUCED

Iodine value of the activated carbon:

To gain further knowledge of the porous structure of activated carbon, iodine adsorption from liquid phase was adopted by other researchers^[53] in the characterization of sludge-based activated carbons. The adsorption of aqueous I₂ is considered a simple and quick test for evaluating the surface area of activated carbons associated with pores larger than 1 nm.^[54] The iodine value, defined as the amount of iodine adsorbed per gram of activated carbon at an equilibrium concentration of 0.02 N, was measured according to the procedure established by the American Society for Testing and Materials (ASTM 2006). The mean values of data for each experiment were presented. Standard deviation was calculated from triplicate samples.

Characterization of activated carbon produced:

The iodine value, defined as the amount of iodine adsorbed per gram of activated carbon at an equilibrium concentration of 0.02 N, was measured according to the procedure established by the American Society for Testing and Materials (ASTM 2006). The mean values of data for each experiment were presented. Standard deviation was calculated from triplicate samples.

RESULT AND DISCUSSION

Result of elemental analysis and proximate analysis of the sludge's:

From the CHNS analysis and proximate analysis it was clearly seen that there was not much difference in the fixed carbon content. Hence both are viable options for activated carbon production. But the mass per unit volume of paper sludge being much higher than that of rice huts, it would be more economical to choose paper sludge as the precursor of activated carbon production. Hence paper sludge is fixed a sour precursor in the further production and analysis of activated carbon.

Effect of chemical activating agents on activated carbon product used

In chemical activation, different activating agents are expected to significantly affect the extent of activation.^[55] In the present study, activating with ZnCl₂, KOH and KCl were evaluated and compared. Table 3 shows the effect of different activating agents on the iodine value and yield of activated carbon from sludge in N₂ atmosphere at 600 C for 1 h. The

concentration of various chemical activating agents was fixed at 1 N and the impregnation time for these agents was maintained as 20 h.

Effect of concentration of $ZnCl_2$ solution used for producing activated carbons

The yields and iodine values of activated carbons prepared from paper sludge in N_2 atmosphere at $600^\circ C$ for 1 h are presented in figure 2. The impregnation process was carried on with various concentrations of $ZnCl_2$ solution but identical solid sludge by dry weight of 6.2 g for a fixed impregnation time of 20 h prior to activation.

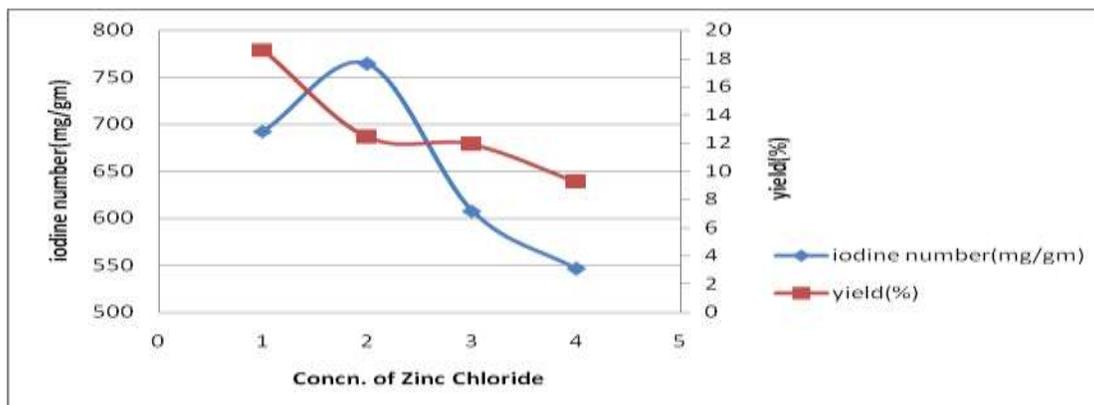


Figure 2: Effect of Concentration Of Activating Agent On Yield Percent And Iodine Value Of Activated Carbon.

CONCLUSIONS

The results of this study show that it is feasible to prepare activated carbons with relatively high surface areas and pore volumes from paper sludge by direct chemical activation. An activation with $ZnCl_2$ produced activated carbons with better developed porosities than with KOH or KCl.

The iodine value of the activated carbon product increased with concentration of $ZnCl_2$ solution (up to 2 N). As the impregnation time increased, the iodine value rose steeply, reaching a maximum value of 764.8mg/gm after 20 h. The iodine value also increased with activation temperature up to $600^\circ C$, beyond which it gradually decreased, presumably due to excessive carbonization.

While prolonging the activation time, the iodine value of $ZnCl_2$ -activated carbon increased and then reached its maximum at 1 h. A longer activation time could induce negative effect on the carbon structure, and, thus, decrease the iodine value.

REFERENCES

- [1]. "Properties of Activated Carbon", CPL Caron Link, accessed 2008-05-02
- [2]. <http://en.mimi.hu/astronomy/granule.html>
- [3]. Value Added Products from Gasification – Activated Carbon, By Shoba jhadhav, The Combustion, Gasification and Propulsion Laboratory (CGPL) at the Indian Institute of Science (IISc)
- [4]. Michael M, Brittain M, NagaiJ, etal. Phase II study of activated charcoal to prevent irinotecan-induced diarrhea, J Clin Oncol. 22 (21), 4410–7 (2004).
- [5]. Mattson J. S. and Mark H. B., activated carbon, New York: Dekker, (1971).
- [6]. Baker F.S., Miller C.E., Repic A. J. and Tolles E.D., Activated carbon. Kirk-Othmer Encyclopedia of Chemical Technology, 4, 1015-1037 (1992).
- [7]. El-Hendawy A. A., Influence of HNO_3 oxidation on the structured and adsorptive properties of corncob activated carbon. Carbon, 41, 713-722 (2003).
- [8]. Smisek M. and Cerney S., Active carbon: manufacture, properties and applications, Elsevier: Amsterdam (1970).
- [9]. Puziy A. M., poddubnaya O. I., Matinez-Alonso A., Suarez-Garcia F. and Tascon J. M. D., Characterization of synthetic carbon activated with phosphoric acid, applied surface science 200, 196-202 (2002).



- [10]. Thomas, J. M. and Thomas, W. J., Principles and practice of heterogeneous catalysis, VCH Verlagsgesellschaft mbH, Weinheim; Federal Republic of Germany, 267-275 (1997). Srinivasakannan C. and AbuBaker M.Z., Production of activated carbon from rubber wood sawdust. Biomass Bioenergy in press.9, 234-235 (2006)
- [11]. Rengarag S., Seung-Hyeon Moon, Sivabalan S., Arabindoo B. and Murugesan V., Agricultural solid waste for the removal of organics: Adsorption of phenol from water and wastewater by palm seed coat activated carbon. Waste Management, 22, 543-548 (2002).