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Effect of Preparation Conditions on the Characteristics of Activated Carbons Produced In Laboratory

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Abstract: **The central theme of this investigation is to evaluate the feasibility of using coconut as a precursor material for the production of activated carbons using chemical activation processes. The chemical activation process was accomplished by impregnating the raw materials with different dehydrating agents (KOH, $ZnCl2_{)}$ in different ratios and concentrations, prior to heat treatment. Characterization of activated carbon was done using scanning electron microscope Activation temperature of 600^{o}C and holding time of 4 h, proved to be the optimum conditions for preparing activated carbon. Coconut shell treated with 10 wt% KOH displayed activated carbon of larger pore size (0.28nm) than those treated with 10 wt% $ZnCl_2$ (0.14nm).**

Keywords: **Activated carbon; Holding time, Chemical activation; Activation temperature, Pore size.**

1. INTRODUCTION

Activated carbon is a microcrystalline form of carbon with very high porosity and surface area. It is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions. (Odesola and Daramola, 2009). Activated Carbon is distinguished from elemental carbon by the removal of all non-carbon impurities and the oxidation of the carbon surface. (Benaddi, 2000). The main features common to all 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 built of condensed aromatic rings are referred to as Basic Structure Units (BSU). Activated Carbon contain physical characteristic such as internal surface area and pore volume. The large surface area results in a high capacity for absorbing chemicals from gases or liquids. The adsorptive property stems from the extensive internal pore structure that develops during the activation process. Activated carbons with highly developed surface area are widely used in a variety of industries for applications which include separation/purification of liquids and gases, removal of toxic substances, as catalysts and catalyst support (Moon and Shim, 2006; Fuente et al., 2001). With the development of technology, the applications of activated carbons keep expanding, with newer applications such as super-capacitors, electrodes, gas storage, and so on (Yuan and Zhang, 2006; Oda and Nakagawa, 2003; Biloé et al., 2002). Activated carbons can be prepared from a variety of precursors with a high carbon content and low levels of inorganic compounds. Various carbonaceous materials such as coal, lignite, nutshells, wood, and peat are used in the production of commercial activated carbon. However, abundance and availability of agricultural by-products make them good sources of raw materials for activated carbons.

The raw material for an activated carbon plays a major part in determining the ability of the final product to adsorb certain molecular species. Activated carbons produced from coconut shells exhibit a predominance of micropores, while coal based carbons have a wider range of transitional pores. The development of an extensive macropore structure is found when either peat or wood is used as the raw material. (Odesola and Daramola 2009). Guo and Lua (2003) mentioned that the characteristics of activated carbon depend on the physical and chemical properties of the raw materials as well as activation method used. Physical properties of AC, such as ash content and moisture content can affect the use of a granular AC and render them either suitable or unsuitable for specific applications.

2. PRODUCTION OF ACTIVATED CARBON

Two basic methods are available for the production of activated carbon; physical and chemical activation. The challenge is to produce activated carbon with the desired pore size distribution and surface chemistry from low cost carbons (Benaddi et la 2000). Physical activation includes carbonization of the precursor in an inert atmosphere and activation of the resulting char by an activation agent such as CO_2, steam or air. The carbonization (pyrolysis) process is usually conducted in the absence of air. During the carbonization step, most of the non-carbon elements, mainly volatiles are removed, leaving a char with high carbon content and slight porosity. Chemical activation is a single-step process including the impregnation of the carbonaceous material with dehydrating agent prior to activation. The significance of the impregnation is to enhance the pore structure of precursor and hence increases its surface area. The chemical agents used in the chemical process are normally alkali and alkaline earth metal containing substances and some acids such as KOH, K_2CO_3, NaOH, Na $_2CO_3$, $ZnCl_2$, $MgCl_2$ and H_3PO_4. Although, phosphoric acid is shown to be the most environmentally sound chemical for the activation processes, most studies have used zinc chloride due to its effective activating capability (Park, and Jung 2003). Chemical activation of carbonaceous materials has been the subject of considerable interest in the past because activated carbon, with a well-developed porosity, can be produced by this process in a single operation. Also, it has been found that the effect of some chemicals on the carbon precursor yields more char and less tar than the untreated sample (Ahmadpour and Do,1996). If chemical activation is essentially considered as a reaction between the solid precursor and the chemical, it is clear that concentration, temperature and activation time determine the extent of the reaction. However, such reaction cannot proceed to completion because it would mean the destruction of the precursor (Molina-Sabio, and Rodr´ıguez-Reinoso, 2004). The extent of the chemical activation can significantly alter the characteristics of the carbons produced.

3. EXPERIMENTAL

The present work focuses on the evaluation of the potential use of an inexpensive material which is available at large quantities (coconut shell) as precursor material for the production of highly effective activated carbons using chemical activation processes. Different types of activated carbons produced were investigated. Two dehydrating agents were used to treat the coconut shells prior to pyrolysis to further improve the porous properties of the activated carbon. The dehydrating agents (chemical activators) were used in different concentrations to assess the effect of dehydrating agent/coconut ratio on the characteristics of the activated carbons.

3.1 Procedure:

200 grams of the grinded coconut shell sample were dried in an electric oven at 110^0C for 48 hours in order to remove all moisture present in the sample. This was impregnated with two dehydrating agents at ratio 1, 1.5 and 3 (KOH: Sample). A typical carbonization run began by charging 20 grams of impregnated sample in the reactor and heating it up to the carbonization temperature in flowing stream of nitrogen (Wan Daud and Wan Ali 2004) .The temperature of the reactor was increased at the rate of 10^0C/min until it reached the final carbonization temperature.Since most literatures reported that optimum activation temperature for most biomass materials generally fall between 400^0C and 500^0C,(Srinivassakannan and Zailani AbuBakar 2004),450^0C was chosen as the final carbonization temperature.

Since most literatures reported that optimum activation temperature for most biomass materials generally fall between 400^0C and 500^0C,(Srinivassakannan and Zailani AbuBakar 2004)The carbonized samples were subjected to this temperature for 1hour and 3 hours. The samples were cooled down under nitrogen flow and were washed sequentially with warm water and finally with cold water to remove residual chemicals .The samples were dried at 110^0C in an electric oven.

3.2 Characterisation:

3.2. 1Surface area and pore size distribution:

Analysis of the surface physical properties of the activated carbon includes determination of the total surface area, total pore volume and pore size distribution. Gas sorption analysis can be considered as one of the most important techniques

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used to determine the physical characteristics of the activated carbon. The adsorption of nitrogen gas at liquid nitrogen temperature (77 K) is routinely used to characterise the porous texture of the activated carbons. Surface area, total pore volume and pore size distribution of the activated carbons were determined from N_2 adsorption/desorption isotherms.

3.2.2. Scanning electron microscopy (SEM):

Texture and surface properties of the adsorbents play an essential role in determining their performance and the final application of carbon materials will depend on their characteristics.

Electron microscopy is widely used technique to probe the surface topography of activated carbon. Scanning Electron Microscope, was used to characterise surface properties of the activated carbons.

4. RESULTS AND DISCUSSIONS

In this study, a coconut shell was used as precursor materials in an attempt to produce activated carbons with high surface area, well-developed porous structure and match the requirements of industry. Chemical activation process was adopted. The effects chemical activators and various activating conditions on the structure and texture properties of the resulting activated carbons were investigated.

4.1 Activated carbons characteristics:

Electron microscopy is commonly used for the characterization of the topography of activated carbons. It provides useful information relating to the porous nature of carbon's exterior and adsorbent porosity. Scanning electron microscopy was conducted to study the surface topography of activated carbons. SEM image of coconut shell based chemically treated activated carbon (ACS) which clearly shows its porous structure and complete opening of cell pores on the surface. As can be seen from the micrograph, there is a formation of pores which may be due to release of some volatiles and activating agent from the sample. The external surface of ACS was full of cavities which are due to the removal of dehydrating agents thereby leaving the space which was previously occupied by the activating agent. In this way it provides a larger surface area. The examination of the activated carbon impregnated with KOH (Fig.1) shows the presence of a few micropores of size (0.28nm).as against that impregnated with $ZnCl_2$ porosity to 0.14nm (fig 2). In both samples a regular microporous and homogeneous surface are obtained by activation at for 4 hours at 600^{o}C.

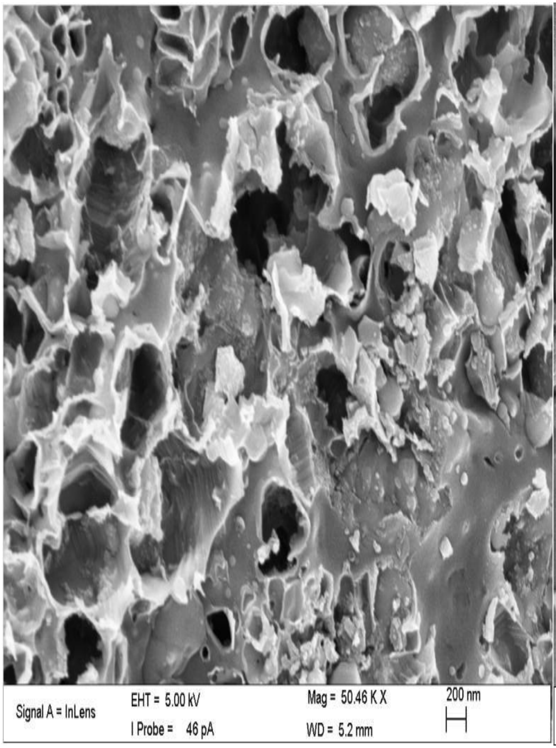

Fig 1 KOH AC

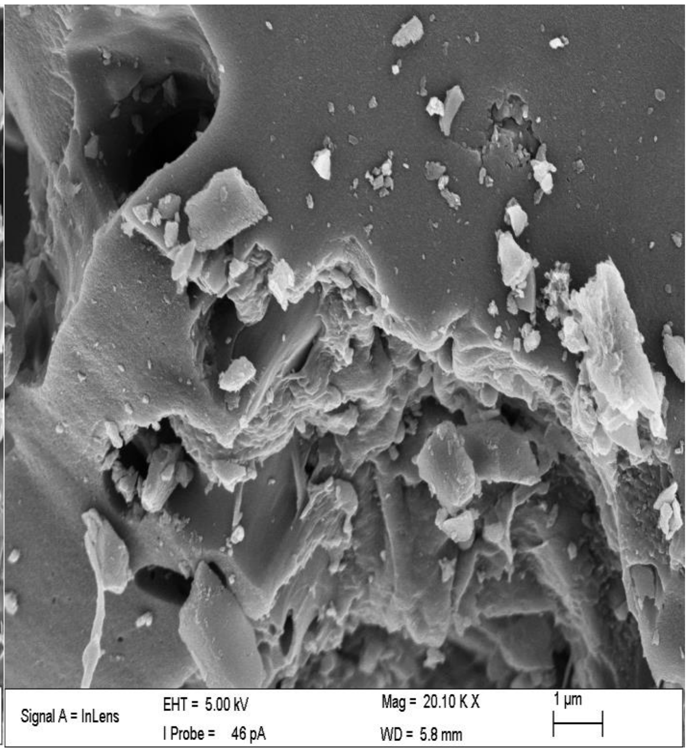

Fig 2 $ZnCl_2$ AC

5. CONCLUSION

The focus of this research was to investigate the effects of dehydrating agents and preparation conditions on the physical characteristics of the activated carbon produced from coconut shell as a precursor material. Chemical activation process was employed for the production processes. Electron microscopy was used for the characterization of the topography of activated carbons. On the basis of present studies, many conclusions can be drawn:

• Activating temperature and Holding time had significant role in imparting physical and chemical characteristics of the activated carbon.

•Activating temperature of 600^oC and holding time for 4 hours proved to be the optimum charring conditions for producing activated carbon as indicated by the superior physical characteristics.

• Regardless of the dehydrating agents used, approximately, all samples produced from coconut shell are essentially microporous.

• Scanning of the surface topography using electron microscope indicates microporosity of the activated carbons produced.

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