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Preparation of Activated Carbon from Neem (*Azadirachta indica*) Husk by Chemical Activation with H_3PO_4, KOH and $ZnCl_2$

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ABSTRACT

A study of the effect of the preparation conditions on the yield and quantity of activated neem (Azadirachta Indica) husk was made using $ZnCl_2$, H_3PO_4 and KOH as activating agents. The raw neem husk was washed, dried and carbonized at 400^0C and the corresponding yield was 63.6%. The optimum conditions for the activated carbon production was evaluated based on the determination of various adsorption parameters of methylene blue. Using methylene blue as the adsorbate with a maximum wavelength of 660nm, it was found that the optimum activation temperature for activating $ZnCl_2$, H_3PO_4 and KOH were 400^0C, 500^0C and 350^0C respectively while the optimum concentration and time were 1minute and 10minutes respectively for the 3 activating agents used. The effect of particle size, shaking time, adsorbent dosage and adsorbent regeneration were also studied. Adsorption studies were carried out using carbonized neem carbon, activated neem carbon and commercial adsorbent from coconut shell on methylene blue.

Keyword: Adsorption, Carbonization, Activation, neem husk, dye

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INTRODUCTION

Neem tree (Azadirachta Indica) is small of about 10m high with pink to reddish and rough bark tissues. The leaves are evergreen and asymmetric at the base. The margins of the leaves are dentate and glabrous. The fruit are berry-like usually green but yellow at maturity. The plant is wide spread in the tropical countries while in Nigeria it is common in the northern part of the country. It is usually cultivated for its wood, shade and medicinal value such as anti-malarial and anti-inflammatory [1].

Due to the high increase of environmental pollution as a result of 'unfavorable alteration of our surrounding' created by man due to rapid industrialization and urbanization which has resulted in the deterioration of air quality, water purity and soil quality, some of this pollutants end up in rivers destroying key ecological elements. In the past, some of the conventional techniques employed include; coagulation and flocculation [2], reverse osmosis [3], chemical oxidation, membrane separation process, electrochemical, and aerobic and anaerobic microbial degradation. All of these methods suffer from one or more limitations (efficiency, cost-effectiveness, availability, management etc.) except 'adsorption' [4]. Commercial adsorbents that have been used in large-scale adsorptive separation and purification processes include activated carbon, zeolites, activated alumina, silica gel, and polymeric adsorbents. The estimated world wide sales of these sorbents are as follows: Activated carbon: $1 billion, Zeolite: $1.07 billion, Activated alumina: $63 million, Silica gel: $71 million and Polymeric adsorbents: $50 million [5]. This is expensive but some agricultural by-products such as coconut shell, rice husk, corn cob, groundnut shell, etc have been discovered to be good sources of activated carbon which makes them useful to a host of applications.

In a largely agrarian country such as Nigeria, there are so many agricultural by-products lying waste, littering and polluting the environment. The need to research on these wastes to convert them into agents of environmental control in order to reduce waste and conserve cost is the key to the present research work. Presently, National Research Institute for Chemical Technology (NARICT) Bassawa, Zaria is researching on the neem tree (Azadirachta indica). Neem husk is a waste product from the processes employed to utilize neem's beneficial properties. It is this neem husk that this research work investigates. Principally adsorbent properties of activated carbon produced from neem husk will be investigated.

MATERIALS AND METHODS

2.1 Materials

Sample (Neem husk) was collected from the National Research Institute for Chemical Technology (NARICT) Basawa, Zaria.

2.2. Carbonization

The method of [7] was used to determine the minimum temperature for carbonization of the neem husk. 2g of the neem husk sample was weighed at a time into a clean crucible of known weight and heated in a muffle furnace at temperature ranging from 200 to 500^{o}C for 10minutes.

2.2.3 Characterization of the carbonized carbon

The following characteristics of the carbonized neem carbon were determined:
Elemental analysis, Moisture and dry matter content, Ash content and Bulk density,

2.3 Activation of carbonized carbon sample

Before activating the sample, the following parameters were determined: Optimum activation temperature, concentration, time, particle size, shaking time and adsorbent dosage,

RESULT AND DISCUSSION

Table 1 Physicochemical parameters of activated carbon from neem (*Azadirachta indica*) husk:

Parameters	Raw sample
Moisture (%)	6.31
Dry matter (%)	93.69
Ash Content (%)	3.60
Bulk density(g/cm^3)	0.95
Yield (%)	63.60
Particle size (µm)	850
pH measurement	
(i) Raw	6.76
(ii) Carbonized	7.70

3.1 Some physicochemical changes occurring during the carbonization process

Fig 1 shows variation in weight loss of neem husk against temperature (°C). The graph is characterized by a dehydration stage, observed at temperature between 0 - 300°C, this process removed moisture present in the material, while a steep slope was observed between 300°C and 350°C, indicating total dehydration. High yield of char carbon were obtained at lower temperatures of carbonization. As it is seen in figure 1, there is a progressive weight loss upon increasing the temperature of the carbonization. Weight loss at higher temperature is greater than that of the weight loss at lower temperature. Probably during carbonization at higher temperature, chemisorbed water is released associated with cleavage of the hydrogen bonds. This implies that a correlation could exist between volatile matters or groups and the weight loss during carbonization at various temperatures. Conversion of char carbon to CO or CO_2 in presence of oxygen, thus decreasing the weight of carbon at equilibrium

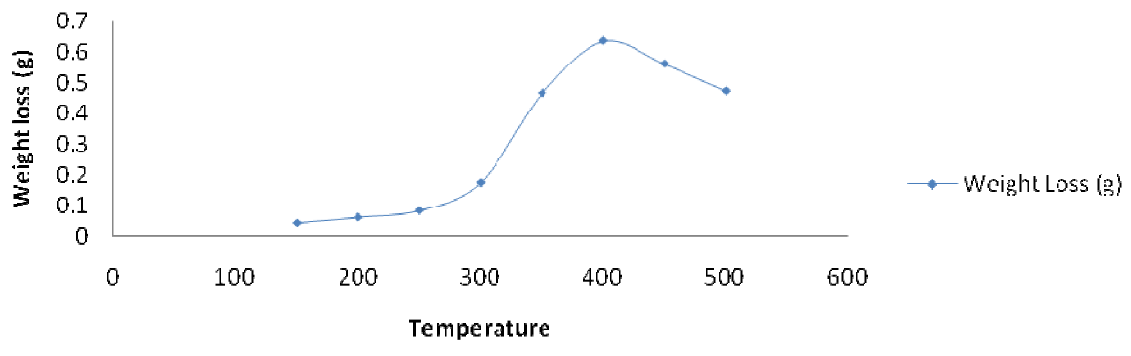


Figure 1 Determination of minimum carbonization temperature

3.2 The **optimum activation temperature, time and concentration for KOH, H_3PO_4, $ZnCl_2$**

The optimum activation temperature for the three activating agents after activation were determine using methylene blue as the adsorbate and found to be at 350°C for KOH, 500°C for H_3PO_4 and 400°C for $ZnCl_2$. The optimum activation concentration and time were found to be the same for the three activating agents as in 1M and 10 minutes respectively.

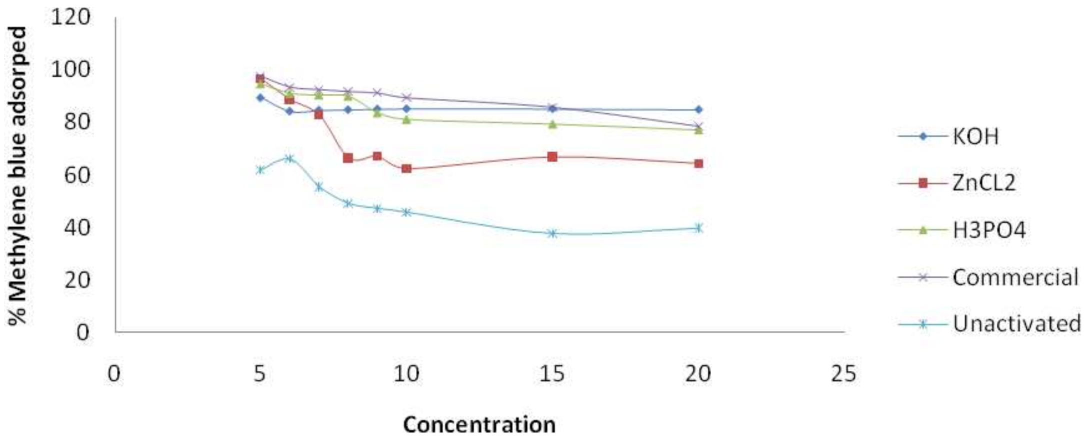
3.3 Adsorption of methylene blue on activated neem carbon, carbonized neem carbon and commercial activated carbon

The adsorption studies show that the activation was actually successful when compared to the carbonized neem husk that was not activated. At initial concentration of 5ppm, it is observed that the neem carbon activated with $ZnCl_2$ adsorbed 96.69% of the methylene blue and these competed very well with the commercial activated carbon from coconut which has 97.29% while KOH and H_3PO_4 had lower values but as the methylene blue concentration increases the percent methylene blue adsorption tend to decreases.

Figure 2 Adsorption of methylene blue on neem and commercial activated carbon

CONCLUSION

The present study shows that: Neem (*Azadirachta indica*) husk could be developed into activated carbon having very high efficiency for methylene blue. The chemistry of carbonization is immensely complex, it involves too many chemical reactions, some occurring simultaneously.



According [7], it will not be possible to know in detail the chemistry of all the pyrolytic reactions of carbonization. However, an attempt has been made in this work to study the minimum and optimum carbonization temperature of neem husk between the ranges of 150-500°C. The optimized conditions (Activation concentration, temperature and time, shaking time and adsorbent dosage) were all studied. For economic reasons and to avoid further pollution of the environment with the used adsorbent. Regeneration study was carried out on both the prepared adsorbent and commercial adsorbent and was found out that the adsorbent could be actually be regenerated.

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