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The Influence of Activating Agents on the Properties of Activated Carbon

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ABSTRACT
The issues of water pollution have continually poised a great challenge to aquatic environment and have altered natural ecosystems. This has informed the need for various treatment technologies to be put in place. However, the sad reality is that most of these technologies are expensive and have cannot be afforded by local residents that need them. Adsorption can be described as the selective uptake of ions. Most commercial adsorbents are expensive; consequently, there is need for adsorbents to be sourced locally by producing activated carbon using locally available materials. The performance of these locally sourced activated carbons is influenced by numerous factors which include pH, temperature, initial ion concentration, adsorbent dosage, contact time as well as type of activating agent used. In this work, the influence of five activating agents on the properties of activated carbon was investigated. Proximate analysis was used to check the percentage compositions for various properties (density, volatile matter, organic carbon, ash content & moisture content). Additionally, scanning electron microscope was used to investigate the surface morphological changes due to the use of these activating agents. The activating agents used in this work were Potassium hydroxide, sodium hydroxide activated, sodium carbonate, potassium hydroxide and zinc chloride. It was observed that unactivated carbon samples and potassium hydroxide activated carbon yielded the highest and least values for ash content and volatile matter respectively. Potassium hydroxide activated carbon yielded maximum organic carbon content and moisture content of 93.1% and 7.93% respectively. However, the highest value for density (5.71) was observed in sodium carbonate activated carbon.

Keywords: Pollution, adsorption, activating agents, proximate analysis, scanning electron microscope.

I. INTRODUCTION

What is Water Pollution?

Water Pollution occurs when harmful compounds are introduced into water bodies thereby changing the characteristics of the water body. According to Hasan et al (2019), although Bangladesh has plenty water sources, they are continuously polluted resulting in many deaths as a result of water borne diseases. The pollution of water bodies with contaminants especially fecal matter and subsequent consumption of such polluted water has lead to many diarrheal deaths across the globe. According to W.H.O (2017), diarrhea is the 2nd leading cause of death in children less than five years old and is responsible for about 525,000 deaths globally every year. According to UNICEF (2018), about 1300 children under the age of five die of as a result of diarrhea despite the use of simple treatment methods especially in sub-saharan Africa. In addition , nutrition, proper hygiene, hand washing and water treatment are associated with childhood diarrhea (Archarya et al, 2017).

Water pollution can be defined as the pollution of water bodies as a result of pollutants being discharged without appropriate treatment. It can also be said to be the contamination of water sources as a result of human activities. Water pollution has continually given rise to health concerns around the world. Chaudhry and Malik (2017), opined that pollutants are substances that cause undesirable effects when present in the environment. Sources of pollution include sewage discharge, industrial effluents and agricultural runoff (Dwivedi, 2017). Sarkar et al (2019), observed that water bodies in Bangladesh have been polluted because industries in this region discharge waste into these water bodies without proper pretreatment. Water pollution is a global problem in modern times as it affects the safety of aquatic ecosystems and restricts the development of humans, society and economy (Xiang et al, 2016).

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When compared with conditions in the past, polluting compounds have become increasingly complex. The uncontrolled discharge of these complex compounds into drinking water sources has contributed immensely to water pollution (Brian et al, 2018). Researchers in different parts of the world have reported health problems associated with prolonged use and consumption of polluted river water. They include: dysentery, diarrhea, gastric/duo dermal ulcers etc (Purnamitta, 2012). Dipak (2017), opined that river pollution is a serious and emerging problem in developing countries occasioned by rapid industrialization and resulting in an increase in the amount of effluent being disposed to natural water bodies. It was observed by Shadella et al (2018), that conventional wastewater treatment also serve as a major source of pollution by releasing effluents containing pharmaceuticals into water bodies. Viegas et al (2021), opined that agriculture is a major contributor to surface and groundwater pollution in Europe and that water treatment methods that are cheap should be advocated for.

II. ADSORPTION

This is the process by which a solid hold molecules of a gas or liquid or solutes as a thin film. This treatment process Is effected by the use of adsorbents. Wolowiec et al (2019), opined that heavy metal contamination is a very important environmental issue requiring appropriate steps to mitigate; a viable treatment method is adsorption. Adsorption is a viable means of removing pharmaceuticals from water (Haro et al 2017). Among several chemical and physical methods of wastewater treatment, the adsorption onto activated carbon has been found to be superior to other techniques because of its capacity to adsorb a broad range of different types of adsorbates efficiently and the simplicity of its design (Rajappa et al,. 2014). According to Adeolu et al. (2016), adsorption technology is being used extensively for the removal of heavy metals from aqueous solutions due to the fact that it is a cleaner, more efficient and cheaper method. As an economic and efficient method, adsorption technique has been widely applied to remove heavy metals from wastewater (El-Bartouti and Ahmed, 2014). According to Orodu et al (2014), though adsorption technique has demonstrated significant potential in the laboratory, its industrial application remains a challenge due to the following reasons:

i. Versatility and robustness;
ii. Ease of regeneration; and
iii. Time constraints etc.

Adsorption is the physical or chemical process in which a substance is accumulated at an interface between two phases. This phase may be solid-liquid, liquid-liquid, gas-liquid or gas-solid. Adsorption refers to the disappearance of solutes from solution with a presumption of adsorption on a solid phase. Adsorption may result from physical or chemical interactions with the surface. Abdel-Raouf & Abdul-Raheim (2017), opined that adsorption is one of the best methods for water treatment because it is cheap, versatile and ecofriendly. Adsorption is a phenomenon marked by increase in the density of a fluid near the surface. In the case of gas adsorption, this happens when molecules of the gas get to the vicinity of the surface and undergo an interaction with it, temporarily departing from the gas phase. Molecules in this new condensed phase formed at the surface remain for a period of time and return to the gas phase. Adsorption is a surface phenomenon governed by the unique propertied of bulk materials that exists only at the surface due to bonding deficiencies. Adsorption process generally affected by factors which include pH, temperature, adsorbent dose, contact time etc (Brahmaiah, 2015)

2.1 Factors affecting adsorption

Numerous factors affect the performance of the adsorption process. These factors may sometimes influence the chemical reactions between the adsorbent and the adsorbate. In a study carried out by Iftekhar et al (2018), to understand the factors affecting the adsorption of Lanthanum using different adsorbents; it was observed that pH, contact time, adsorbent dosage and temperature were some of the factors affecting the performance of the adsorption process. Various activated carbons were prepared and characterized from tobacco stem using various chemical activating agents (KOH, K_2CO_3 & $ZnCl_2$). The effect of impregnation ratio and activating agent type was investigated; from the various characterizations carried out using scanning electronmicroscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), x-ray photoelectron spectro-scopy (XPS), and thermogravimetry (TG), $ZnCl_2$ was observed to act as a better activating agent because the activated carbon obtained from it was more porous than the other two(Chen et al, 2017). In a study on the efficient adsorption of sulfamethazine onto modified activated carbon by Liu et al (2017), it was observed from this study that adsorption process was influenced by a change in pH and temperature. In a study on the adsorption of antibiotics using biochar prepared at various temperatures, it was observed that various factors

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such as biochar dosage, pH, initial antibiotic concentration, contact time and temperature affect the adsorption of antibiotic adsorption. Batch experiment studies was carried out investigate the effects of effluent pH, adsorbent dosage and contact time on dissolved solids adsorption; from this study, it was observed that optimum adsorption occurred at a pH between 4-6, adsorbent dosage of 30mg/l and that an increase in contact time increased the adsorption process (Ani et al, 2019). Eletta et al (2020) studied the effect of factors like temperature, solute pH, adsorbent dosage, agitation time and initial concentration on the production of precursors for the production of adsorbents using cocoa pod husk. From the study, it could be understood that these parameters affect the performance of adsorbents prepared from cocoa pod husk greatly. Treated eggplant peels were applied as low cost adsorbents for the elimination of Pb^{2+} from water; the results of this experiment showed that optimum adsorption was achieved at contact time of 110mins, adsorbent dose of 0.01g/l, initial Pb^{2+} concentration of 70ppm, pH of 4 and temperature of 25^0C(Darvanjooghi et al, 2018). Okoya et al (2020) opined that Nigeria being the largest producer of rice in Africa is bound to generate huge quantities of rice-related waste such as rice husk, in addition; due to an increase in the production of rice, there is a consequent increase in the use of pesticides which can accidently be introduced into water bodies. Therefore a scenario that utilizes these waste produce from rice farming and production for the treatment of pesticides contaminated water will be applauded. However, it was also observed that for the optimum adsorption treatment, certain factors like adsorbent dosage, initial ion concentration and contact time can never be neglected. Tiadi et al (2017) studied the adsorption behavior of an industrial waste for the removal of chromium from aqueous solution; in this study, various parameters were varied, they include contact time, adsorbent dosage, adsorbate concentration, pH; the results of this study revealed that optimum chromium adsorption was observed at pH of 2, adsorbent dose of 20g/l. Ukanwa et al (2019) opined that the activating agents play a major role in the production of activated carbon depending on the type of biomass used and the working temperature. Due to the cost of some commercial adsorbents, there has recently been a lot of interest from indigenous researchers to produce activated carbon/adsorbents from locally available materials. Guo et al (2020) studied the adsorption of CO_2 using porous activated derived from waste sugar bagasse using activating agents such as CO_2, H_3PO_4, NaOH. From the results obtained from this experiment, it was observed that chemically activated carbon showed better promise that physically activated carbon. In addition, NaOH activated carbon showed larger surface area than those activated with other activating agents. In a bid to develop an appropriate treatment for produced water, a study on the effects of salts, acids and bases ($ZnCl_2$, H_3PO_4, NaOH) on the structure three activated carbon and their performance for the removal of oils and greases was carried out; from the results of the characterization, H_3PO_4 proved to be the most efficient because it had the largest surface area and yielded the most adsorption.

III. MATERIALS AND METHODS

3.1 Material Collection

The adsorbent to be used in this research work were obtained from carbon rods in spent batteries. These spent batteries were sourced from rural communities in Enugu, Anambra, Kogi and Benue states. After collection, these batteries were air dried and subsequently broken or cut longitudinally. This will enable the whole length carbon rods to be extracted without any part of the rod remaining inside the batteries. These carbon rods were again air dried and scrubbed to further purify the carbon rods by removing impurities that may stick to the surface of the carbon rods. Thereafter the carbon rods were crushed and stored into an air tight container ready for activation.

3.2 Preparation Of Activating Agents

For the purpose of this experiment, five different activating agents was used to activate the samples in a two-step activation process. These include Sodium hydroxide(NaOH), Sodium Carbonate(Na_2CO_3), Potassium hydroxide(KOH), Potassium Carbonate(K_2CO_3) and Zinc Chloride($ZnCl_2$). 1 molar solution of each of these activating agents was prepared by following standard methods.

The activation of granular carbon will be done in two steps

1. Thermal activation: The granular carbon rods was carbonized in muffle furnace at 600^oC for 6hours. The furnace was allowed to cool and the carbonized granular carbon rods was further cooled using desiccators. The dessicators was used to ensure that granular carbon does not trap moisture from the air during the cooling process.
2. Chemical activation: Five different beakers will be labeled SHAC, SCAC, PHAC, PCAC and ZCAC. 250grams of the carbonized granular carbon rods were weighed out five times and placed into these five different

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beakers. Five different activated carbon will be prepared by pouring the already prepared 1 molar solution of the various activated carbons in the correspondingly labeled beakers and stirring till a uniform consistency is achieved.

3. After the 5 different mixtures have been formed, the mixtures were poured into 5 stainless flat plates that that have been previously labeled with SHAC, SCAC, PHAC, PCAC and ZCAC. These flat plates were placed in the oven for 3hours at 250^{o}C. The dried granular carbon already activated will be washed with distilled water until the pH of each sample was neutral (7) and dried some more
4. Five different containers was labeled SHAC, SCAC, PHAC, PCAC and ZCAC and the activated carbon samples was poured into air tight containers bearing the corresponding labels

3.3 Characterization of the different activated carbon samples

The characterization of the five different types of activated carbon was done using the following

Proximate Analysis: This analysis will be carried out to determine the amount of the following on the 5 different activated carbon samples and the un-activated sample. This analysis was to find out the following about the samples: % ash content, % moisture content, % volatile matter, % organic matter and density. It is important that these parameters of the activated carbon be looked at because they influence the behavior of the activated carbon samples during the adsorption process.

Proximate Analysis of the different carbon samples:

i. Percentage moisture content: 6 moisture dishes were weighed and labeled. 10grams of each activated carbon samples and 10grams of unactivated carbon samples were weighed and put into the dishes with corresponding labels and weighed again, the weight of the samples plus the moisture dishes were noted. These five different containers were placed in an oven for five hours at a temperature of 100^{o}C. These dishes were removed from the oven, covered, placed in a dessicator allowed to cool and weighed. This process was repeated till a constant weight is achieved. Percentage moisture was calculated using the following relationship.(See equation 3.1)

$$\%\mathbf{M.R} = \frac{\mathbf{100(B-F)}}{\mathbf{(B-G)}} \quad \textbf{.....(3.1)}$$

Where G is weight of each empty crucible before use;

B is weight of crucible + activated carbon before heating; and

F is weight of crucible + activated carbon after heating.

The process will also be repeated for the un-activated carbon sample

ii. Percentage Ash content. A silica crucible of known weight was ignited in a muffle furnace for 90minutes. The crucible was placed in a dessicator and cooled to room temperature and weighed again. 10grams of a sample of activated carbon was dried in hot air oven at 150®C for three hours and was put in the crucible. The crucible was placed back in the muffle furnace at 750®C for 90minutes. The crucible was removed from the furnace and allowed to cool to room temperature and reweighed. The ash content can be calculated using the following relationship. (See equation 3.2)

$$\mathbf{A} = \frac{\mathbf{100(F-G)}}{\mathbf{(B-G)}} \quad \textbf{.....(3.2)}$$

Where G is the weight of the empty silica crucible;

B is the weight of crucible plus activated carbon sample before heating; and

F is the weight of crucible plus activated carbon sample after heating.

iii. Percentage Volatile Matter: A crucible covered with its lid was weighed. A known 10grams of the activated carbon sample was put into the crucible, covered and placed in the muffle furnace at 920®C for 7 minutes and allowed to cool in a dessicator and reweighed. The percentage volatile matter was calculated from the following relationship. (See equation 3.3)

$$\mathbf{VM} = \frac{\mathbf{[100(B-F)-M(B-G)]}}{\mathbf{[(B-G)(100-M)]}} \quad \textbf{.....(3.3)}$$

Where G is the weight of empty crucible plus lid;

B is the weight of crucible and lid plus activated carbon sample before heating; and

F is the weight of crucible plus lid plus activated carbon sample after heating.

This process will be repeated for each of the activated carbon samples and un-activated carbon samples

iv. Density: This can be defined as the volume of a full recipient that contains a determined mass of activated carbon. A known mass of the activated carbon sample was weighed out

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and put in a graduated cylinder. The cylinder was tapered on a bench till the sample of the activated carbon stopped decreasing in volume. Density may be calculated from the following relationship (see equation 3.4)

$$\textbf{density} = \frac{\textbf{mass}}{\textbf{volume}} \text{.....(3.4)}$$

This process was repeated for the other activated carbon samples and unactivated carbon sample

v. The percentage organic Carbon was calculated from the following formula

$$\%\mathbf{OC} = \mathbf{100} - (\%\mathbf{MR} + \%\mathbf{ash} + \%\mathbf{VM}) \text{.....(3.5)}$$

Scanning Electron Microscope: The scanning electron microscope was used to check the activation process by the different activating agent. The un-activated carbon samples was also scanned so that difference in changes were observed for the different samples. The scanning electron microscope produced images by detecting secondary electrons which were emitted from the surface of due to excitation by the primary electron beam. The electron beam was scanned across the surface of the sample activated with Potassium Hydroxide, Sodium Hydroxide, Potassium Carbonate, Sodium Carbonate Zinc Chloride and also samples of unactivated carbon; in a "raste" pattern with electrons building up an image by mapping the detected signals with beam positions. The samples scanned (characterized) were preserved by dehydration. This was done by removing water from the sample and replacing with organic solvents

IV. RESULTS AND DISCUSSIONS

4.1 Characterization of the adsorbents

The characterization of the adsorbents was carried out using the following methods

i. Proximate Analysis; and
ii. Scanning Electron microscope.

1. **Proximate Analysis**: This was carried out to determine amount of moisture, ash, volatile matter and organic matter. The density of each sample was also calculated from the proximate analysis. The results of the proximate analysis are shown in the Table 4.1

Table 4.1: proximate analysis result for all types of activated carbon

Type of adsorbent	% Ash	Volatile matter	Organic carbon	Density	%moisture
SHAC	3.64	1.60	89.33	5.43	7.70
SCAC	3.62	1.60	89.07	5.71	7.89
PHAC	2.99	0.95	93.1	2.96	7.93
PCAC	4.82	1.60	88.22	5.36	5.27
ZCAC	5.350	1.69	88.44	4.52	5.18
Unactivated	11.25	6.38	81.02	1.350	4.73

Where; SHAC connotes Sodium Hydroxide Activated Carbon

SCAC connotes Sodium Carbonate Activated Carbon

PHAC connotes Potassium Hydroxide Activated Carbon

PCAC connotes Potassium Carbonate Activated Carbon

ZCAC connotes Zinc Chloride Activated carbon

From the results obtained from the proximate analysis carried out on the different samples SHAC, SCAC, PHAC, PCAC, ZCAC and unactivated samples; it was observed that PHAC contained a very high amount of organic carbon (93.1%) while unactivated sample contained a relatively low amount of organic carbon (81.02%). Unactivated carbon samples was also observed to contain the most amount of ash and volatile matter. It also contained the least amount of moisture and was the least dense of all the samples. PHAC on the other hand, contained the highest amount of organic carbon and highest amount of moisture; it also contained the least amount of volatile matter and the least amount of ash and was observed to be the densest sample.

Scanning Electron Microscope: This showed the surface morphological changes that the different activating agents had on the carbon samples. The results from this analysis is pictorial and is presented below for the different samples of activated carbon and the inactivated carbon

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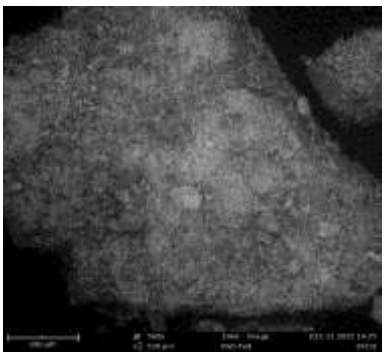
Fig 1: $ZnCl_2$

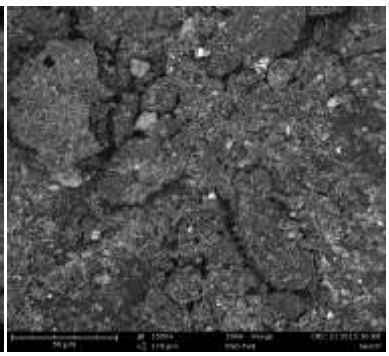
Fig 2: NaOH

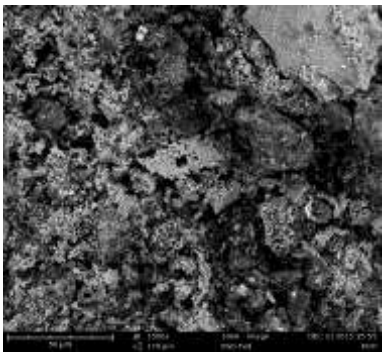
Fig 3: KOH

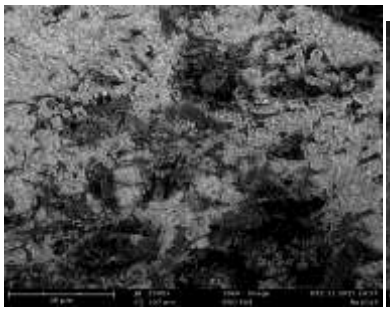
Fig 4: Na_2CO_3

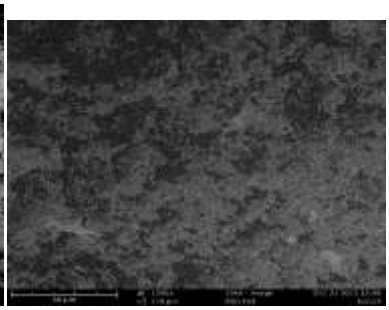
Fig5: K_2CO_3

V. CONCLUSIONS

From the results obtained from this experiment, it can be observed that the activating agent used has a great influence on the properties of the activated carbon used. These properties include density, percentage ash content, moisture content volatile matter and organic carbon. Additionally, from the images obtained from the Scanning electron microscope, the type of activating agent used affect the structure of the subsequent activated carbon. Further research should be carried out on the other factors affecting the performance of adsorbents used for adsorption such as adsorbate pH, activating temperature, particle size of adsorbent and adsorbent dosage.

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