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ASSESSMENT OF SAWDUST ACTIVATED CARBON IN THE TREATMENT OF AQUACULTURE EFFLUENT

Nzubechi Augustine Oriaku*, Ogechi C. Ikpemo.
Department of Civil Engineering, Michael Okpara University of Agriculture, Umudike, Abia State, Nigeria.
ornzubechi@gmail.com, oriakunzube24@gmail.com,

ABSTRACT

This study is aimed at the assessment of sawdust activated carbon in the treatment of aquaculture effluent with various objectives which includes the determination of the SAC properties, effluent parameters, effect of operating conditions and application of relevant adsorption isotherms. The sawdust was first precarbonized at 500 C, then impregnated with potassium carbonate (activating agent) and activated at 450 C in a furnace. The surface morphology of the sawdust activated carbon (SAC) was determined using scanning electron microscope (SEM) which showed a very porous media. It was further characterized using BET surface area at 77K, and the resulting surface area was 831.83 ^{2}/g. The acquaculture effluent (AE) was found to be very polluted compared to the NESREA discharge standard, in COD, TSS, BOD, NITRATES and lead(II) ion. An optimization studies was done using batch treatment process at varying dosage, time and initial concentrations, and the optimum treatment dosage and time were found to be 2g/l at 60minutes which gave an overall 83.33% reduction of COD, 90.80% reduction of BOD, 73.24% removal of nitrate($_{3}$), 93.23% removal 0f TSS, and 99.35% removal of lead (Pb) ions. According to the ^{2} values, the kinetic studies showed that the adsorptionprocess was a pseudo second order reaction with a ^{2} value of 0.99. After the assessment, it was found that among other uses of sawdust, it is also a good precursor for the production of activated carbon, which is has been found in this study to be a good treatment media for the treatment of the polluted aquaculture effluent prior to its disposal into the environment.

1. INTRODUCTION

Aquaculture can be described as the production of aquatic organisms, both plant and animal under controlled or semi-controlled conditions. However, Adewumi(2015), defined aquaculture practice in Nigeria as the rearing of fish in an enclosed and fairly shallow body of water where all its life processes is being controlled. Typical wastewater from an aquaculture facility may include feaces and nutrients from excretion by aquatic animals, as well as uneaten feeds and chemicals such as drugs and cleanser residues. Aquaculture effluents contains dissolved and suspended solids that are basically oxygen demanding materials which makes the effluent to be high in biochemical oxygen demand(BOD) and nutrients like phosphorus (P) and nitrogen (N) which stems from fish excretion, feaces, and uneaten feed. Overtime, significant discharge of this untreated effluent into lakes, rivers, estuaries or any other receiving waters could cause adverse environmental impacts such as eutrophication.

In many instances however, secondary wastewater treatment such as trickling filters, oxidation ponds and aerated lagoons has been found inadequate in the treatment of certain waste water, hence the need to apply appropriate tertiary/advanced wastewater treatment methods, among which is adsorption. Common advanced wastewater treatment methods are ion exchange, membrane separation, electrolysis and adsorption. Among these methods, adsorption technology

has gained a wider application due to its inherent low cost, versatility and robustness. The other related technology are quite expensive, requiring a high level of technical know-how, steady energy supply, and chemicals along with specific equipment which may not be readily available, especially in developing countries.

Saw dust is a waste material from the timber industry, produced when timber is sawn into planks at saw mills. This process is a daily activity causing heaps of saw dust to be generated after each day. The need to convert this waste product into a useful by-product is the focus of the study. The size of sawdust particles depends on the kinds of wood from which the sawdust is obtained and also on the size of the saw teeth. About 10-13% of the total volume of the wood log is reduced to sawdust in milling operations and this sawdust generally depends largely on the average width of the saw kern and the thickness of the timber sawed. Sawdust has been used for several purposes, including sawdust briquettes, partial replacement of concrete, soil stabilization and also as an adsorbent.

Aquaculture effluent has become large enough to have significant impacts on the environment, mainly in the form of eutrophication in rivers, as well as heavy metal pollution, which is supported by environmental activists and several scientists whose serious concern have been the pollution of limited fresh water. Since aquaculture effluent is being disposed indiscriminately, mainly because farmers see wastewater treatment as being an added expense that does not contribute to fish production. Hence, there is need to develop a viable, cost effective, and environmentally friendly means of treating fish farm effluent before its disposal.

In Nigeria, aquaculture industry products like fish and shrimps are gradually replacing animal meat as a source of dietary protein and business hub for teeming million youth with the most commonly grown fish being *Clarias Gariepinus* (catfish). Wastewater from catfish ponds usually has higher concentrations of nitrogen, phosphorus, organic matter and biochemical oxygen demand than natural surface waters in the vicinity. Boyd, Tucker and Robinson have reported that Concentrated Aquatic Animal Production (CAAP) facilities such as hatchery and fish ponds are major sources of oxygen demanding waste, which produces objectionable odor in the receiving adjacent streams where most fish farmers consider a convenient site for their effluent disposal.
Aquaculture effluent is constituted mainly of feaces, ammonia, uneaten food in the forms of dissolved compounds and suspended solids along with heavy metals been traced to be a product of the fish feeds which constitutes the bulk of aquaculture waste Uneaten food and fish feaces are usually in the form of suspended solids, which can make natural waters more turbid and eventually form organic deposits on the bottom of water bodies. The organic deposits in the effluent can reduce the oxygen content of the water through natural oxidation, which includes microbial respiration and aerobic decomposition. The feed and feacal wastes also contribute to biological oxygen demand (BOD) which is used as an index of pollution by dissolved organic substances or suspended particulate matter.

A number of physical, chemical and biological methods used in conventional wastewater treatment have been applied in aquaculture systems. Solids removal is accomplished by sedimentation, sand or mechanical filtration while biological processes such as submerged biofilters, trickling filters, rotating biological contactors, and fluidized bed reactors are employed for the oxidation of organic matter, nitrification, or denitrification.

These methods do help with phosphorus removal but are costly in terms of capital investment, energy consumption and maintenance requirements. Researchers have demonstrated that wetland systems can also remove significant amounts of suspended solids, organic matter, nitrogen, phosphorus, trace elements and microorganisms contained in wastewater. However, in urban areas with little or no space for wetlands, there is need for a more compact system like an adsorption batch or column treatment process which is equally effective in the removal of the various pollutants in aquaculture effluents, including heavy metals.

For the purpose of this research is to evaluate the feasibility of using sawdust activated carbon (SAC) in the treatment of aquaculture effluent.
For the purpose of this research, fishpond (catfish) effluent will be considered and the effluent will be obtained from a catfish farm within Umuahia. This study is focused on the production of activated carbon from sawdust to serve as an adsorbent in the treatment of aquaculture effluent towards safe disposal. The efficiency of the experimental batch treatment will be considered in terms of removal of organic and inorganic matter based on chemical oxygen demand (COD), biological oxygen demand(BOD), removal of total dissolved solids (TDS), total suspended solids (TSS), nitrates, phosphates along with removal of heavy metals.

2. Materials and Methods

2.1 Materials

In this research, three main categories of materials were adopted, these are the raw materials mainly in the form of the aquaculture effluents and sawdust; reagents and glassware; and the laboratory equipment. The following materials obtained are; Saw dust(800g)[*Ororo* tree], Distilled water, Effluent (10litres Catfish pond effluent). Equipment used for this research are; Furnace, Hot air oven, Shaker, Drying crucibles, Desiccator, UV visible spectrophotometer, Reflux condenser, Water bath, pH meter, absorption spectrophotometer (AAS) (Shimadzu Janay Spectrophotometer 6400), Autosorb-1, Quanta chrome equipment, SEM Balzers 050 evaporator. Chemical reagents for this research include the following: Sodium thiosulphate, Hydrochloric acid (HCl) Potassium carbonate, Methylene blue, Mercury (II) tetraoxosulphate (VI), Silver (I) tetraoxosulphate (VI) solution (Ag_4), Potassium heptaoxochromate (VI), Ferroin indicator.

2.2 Methods

2.2.1 **Preparation of activated carbon**

The preparation of the sawdust activated carbon started with the collection of the sawdust from a local sawmill at Owerri, Imo state and it was then sieved, dried, carbonized, washed, dried, impregnated, activated and then finally washed with a weak acid to adjust the pH accordingly.

Sawdust (SD) sample of 2kg weight was collected from a local sawmill at Owerri, Imo state, Nigeria.
The sawdust was oven dried at 100 C overnight for 17hours.
The sample was sieved with a 2 mm mesh size sieve and the less than 2 mm samples were stored in an airtight container to avoid absorption of moisture.

The carbonized sample was washed, using 0.1 M HCl to remove surface ash which was followed by hot water wash and further washing with distilled water to remove residual acid.
The sample was then further dried in the oven overnight at 100 C.
The activated sample was washed with ice-cold water, and excess water was drained off.
Washing was continued until the pH of sample solution was in the range of 6 -8,
The washed sawdust activated carbon was then dried in the oven at a temperature of 105 c and stored in an airtight container to prevent moisture uptake.

2.2.2 Characterization of the sawdust activated carbon

The produced activated carbon was further characterized based on the carbon yield, pH, solubility in water, moisture content, ash content, methylene blue number, bulk density, surface morphology and surface area, in order to fully understand the effectiveness of the activation process and also for comparison with previous researches on activated carbon.

2.2.2.1 Carbon yield

The total yields were determined after sample processing in terms of raw material mass. The dried weight, W, of each pre-treated sample was determined using weighing balance and the carbon yield calculated as: $Y_{CH} = \frac{W_S}{W_0} X\ 100$ (1)

Where, is carbon yield (%), is dried weight of AC prepared and is pre-treated sample used in the carbonization and activation processes.

2.2.2.2 *pH*

The standard test method for determination of activated carbon pH ASTMD3838-80 was used. 1.0 g of SAC was weighed and transferred into a beaker. One Hundred milliliters (100ml) of distilled water was measured and added and stirred for 1 hr. The samples were allowed to stabilize before the pH was measured. The pH was determined using a pH meter.

2.2.2.3 Solubility in Water

For water solubility (S), 0.5 g SAC samples were added to 100 ml of distilled water in 250 ml flask and shaken at 200 rpm in a shaker for 2 hrs at ambient temperature. The mixtures were filtered through pre-weighed filter papers. The filter papers containing the residual carbons were dried in the oven for 12 hrs at 105 C. After cooling to ambient temperature, in a desiccator, the filter papers together with residual carbons were weighed. The percentage solubility in water was calculated as the weight ratio of unrecovered carbon to the original sample.

$$S(\%) = \left| \frac{\text{Loss in Weight on dissolution}}{\text{Weight of original carbon}} \right| \text{x100} \qquad (2)$$

2.2.2.4 Moisture Content

Thermal drying method was used in the determination of moisture content of the SAC (Atef 2016). 1g of the powdered SAC was taken in a previously weighed crucible. The crucible was placed in an electric hot air oven maintained at about 110 C. After one hour the crucible was taken out, cooled in a desiccator and weighed again. The loss in weight of the powder reported on percentage basis gives moisture content in the sample as:

$$\left| \frac{\text{Loss in Weight on drying}}{\text{Initial Sample}} \right|$$

Moisture content (100%) x100 (3)

2.2.2.5 Ash Content

The ash content of the SAC was determined using the standards ASTM procedure D2866-94. This method involves heating the sample at 650 C for several hours until constant weight has been achieved. The ash is then weighed and the ash content of the SAC is calculated as:

$$\text{Total ash (\%)} = \frac{D-B}{C-B}x \quad 100 \qquad (4)$$

Where, B is the weight of the crucible (g), C is the weight of crucible plus original carbon sample (g) and D is weight of crucible plus ash containing sample (g).

2.2.2.6 Bulk Density

In accordance to standard ASTM methods, an empty dry graduated cylinder was weighed. A sample of dry activated carbon SAC was packed into the cylinder and reweighed as:

$$\text{Density (g cm}^{-3}\text{)} = \frac{\text{Weight of dry activated carbon}}{\text{Volume of dry material}} \qquad (5)$$

2.2.3 Adsorbate preparation and characterization

A 10 liter effluent sample was collected from a local fish farm in Umuahia, Abia state. A 10 litre sample container (jerry can) was filled to the brim with the effluent in order to expel entrapped air. The can was corked and remained sealed until the commencement of the analysis. Physicochemical analyses including COD, BOD, nitrates, phosphates, pH and metal ion concentration of the effluent sample were determined following the standard method of water and wastewater purification.

2.2.3.1 Physical tests

The temperature and pH of the samples were obtained using the thermometer and multi-parameter photometer respectively. The total suspended solids (TSS) was determined using membrane filters of 0.45 µm that were dried at 103 C for one hour after which they were weighed and placed in a desiccator until they were ready for use. Sample was collected in sterile container and mixed thoroughly by inverting the bottles several times to obtain a uniform mix. 100 mL of sample was poured into the membrane filter assembly holding the previously weighed membrane filter and attached to a suction pump and then filtered. The filter paper was then dried at 103°C and re-weighed. Total suspended solids (TSS) in mg/L was then determined by subtracting the initial weight of the filter paper from its final weight.

2.2.3.2 Determination of COD

The chemical oxygen demand test was carried out by placing 50ml of the effluent sample in a 500 ml refluxing flask and glass boiling beads was added to serve as anti-bumping aid followed by the addition of 1g of mercury (II) tetraoxosulphate (VI) crystal. 5 ml ofconcentrated tetraoxosulphate (VI) acid/silver (I) tetraoxosulphate (VI) solution (Ag_4) was added, and mix until the $_4$ was dissolved in the solution. Accurately measured 25 ml of 0.25N potassium heptaoxochromate (VI) was added and mixed; while mixing, an additional 70 ml of concentrated $_{2\,4}$- Ag_4 solution was added. After thorough mixing, the flask was attached to the reflux

condenser; the mixture was gently heated in a water bath, and reflux for 2 h. The apparatus was then cooled to room temperature (28) after the refluxing period. Washing down of the interior of the condenser and flask twice, with approximately 25 ml portions of distilled water was carried out. The flask was removed from the condenser and diluted to a final volume of approximately 350 ml with distilled water and, further cooling was done with running tap water. 2 to 3 drops of ferroin indicator was added and stirred with a magnetic stirring bar. The resulting mixture was rapidly titrated with 0.1 N iron ((II) ammonium tetraoxosulphate (VI), to the first red-brown endpoint. The same procedure was repeated for other samples.

2.3 Data Analysis

The kinetics of adsorption was analyzed using two kinetic models, namely; pseudo first order and pseudo second order kinetic models. Other data were fully analyzed using different mathematical tools which include; graphs, charts and tables to illustrate the treatment process.

3 RESULTS AND DISCUSSIONS

3.1 Aquaculture Pond Effluent Characteristics

The result of the physicochemical analyses of the effluent sample is presented in Table 1. Concentrations of the parameters including pH, phosphate, and some of the heavy metals (cadmium, zinc, iron, copper) were below the National Environmental Standards and Regulations Enforcement Agency (NESREA) effluent discharge limits; While others, like chemical oxygen demand(COD), total suspended solids(TSS), lead (Pb), nitrate ($_3$) and biological oxygen demand ($_5$) values were far beyond the limits with very wide margins.

Table 1: Physicochemical Characteristics of Aquaculture Effluent

PHYSICOCHEMICAL PARAMETERS	EFFLUENT CONCENTRATION	NESREA LIMIT
pH	6.3	6-9
COD(mg/l)	600	60
Nitrates(mg/l)	19.88	10
Phosphates(mg/l)	3.48	3.5
TSS(mg/l)	787.5	35
$_5$(mg/l)	120	20
Lead (mg/l)	1.54	0.05
Zinc (mg/l)	0.0021	2.0
Cadmium (mg/l)	0.0012	0.01
Copper (mg/l)	0.001	0.5
Iron (mg/l)	0.0239	0.5

Comparing the results with the findings of Boyd (2020) on the pollution of aquaculture effluent, it is confirmed that the effluent ought to be treated before final disposal into the environment.

3.2 Activated Carbon Characteristics

The characteristics of the sawdust activated carbon (SAC) are presented in Table 2.

Table 2: Result of Characterization of Sawdust Activated Carbon (SAC)

Parameters	Values
Carbon yield (%)	31.25
Ash content (%)	4.92
Solubility in $_2$ (%)	12.0
Moisture content (%)	3.54
Ph	8.07
Bulk density (g/ ^{3})	0.27
Methylene blue number	61.10
Surface area (^{2}/g)	831.83
Pore volume (^{3}/g)	0.34
Surface morphology	Figure I

3.2.1 Carbon yield

The yield of activated carbons was calculated from sample weight after activation relative to its initial weight according to equation 18. Table 2 shows the percentage yield of SAC to be 31.1%, prepared at temperature of 450, at time 10 min, and impregnation ratio of 1:1 with potassium carbonate.

3.2.2 Ash content

The inorganic constituents in the sawdust activated carbon were found to be 4.9% when the carbon was washed; this is lower when compared to the 6.5% and 6.6% ash content obtained by Oriaku Nzubechi, from acid activated sawdust carbon. Ash content can lead to increased hydrophobicity and can have catalytic effects, causing restructuring during regeneration of used activated carbon. The inorganic material contained in activated carbon measured as ash content should generally be in the range of 2 and 10%. High ash content raw materials contain high levels of impurities that lead to blockage of pores during activation process, thereby reducing the surface area of the activated carbon, it also affect the efficiency of reactivation of spent carbon. Therefore, the lower the ash content, the better the activated carbon can be used as an adsorbent.

3.2.3 Solubility in water

The water solubility of the sawdust activated carbon was found to be 12%. Any degree of solubility in either water or acid is an indication of presence of impurities. Pure carbon does not dissolve in either water or acid, since carbon is very unreactive due to lack of electron donating or accepting species in its structure such as lone pair of electrons. A lower solubility of 8.4% was produced from phosphoric acid activated sawdust. The high value of water soluble matter expressed in this carbonate activated sawdust suggests that a large amount of carbonate salts could have been incorporated into the carbon structure.

3.2.4 Moisture content

The moisture content obtained for the activated carbon in this study is 3.54% which is better than the 5.6% moisture content from the study of NH_4Cl-activated sawdust. A much higher percent moisture of 10.35 and 12.57 %, for prepared activated carbon and that of commercial activated carbon respectively, while on the other hand, lower percentage moisture of 4.8 % was observed. The recommended AC storage moisture content is <3%. So the produced sawdust activated carbon in this study has its moisture content within the recommended limit. If the moisture content of AC is high, fungi and other micro-organisms degrade the carbon, utilizing it in their metabolic processes. The micro-organisms can also multiply within the AC, blocking the pore structure, thereby reducing the adsorptive capacity of the carbon.

3.2.5 pH

The pH result obtained is shown in Table 3.2. A pH of 9.03 was reported which is quite basic and not suitable for most adsorption as it has been reported that for water treatment, carbon pH 6-8 is acceptable. Thus the activated carbon to be applied in the adsorption process was further neutralized with 0.1 M HCL and washed to a pH of 8.07, which is more within the acceptable range, which can be reduced with further washing . A similar result was obtained a pH range of 6.3, 6.8, 7.6 and 7.6 - 8.7, for physically activated carbon and chemically activated carbon respectively. The initial high pH is as a result of the basicity of the activating agent.

3.2.6 Bulk density

The value of bulk density of prepared sawdust activated carbon as shown in Table 4.1 is 0.27g/ ^{3}, bulk density for a good adsorbent should not be less than 0.25g/ ^{3}. Activated carbon was prepared from palm kernel shell (PKS), Coconut shell (CS) and sawdust (SD) and the result of the study indicated that the activated carbon from sawdust (SD) had the lowest bulk density of 0.142 g/ ^{3} and the highest was from the palm kernel shell (PKS) at 0.722g/ ^{3}.

3.2.7 Surface area and pore volume

A high surface area of 831.8 ^{2}/g and pore volume of 0.34 ^{3}/g was gotten obtained the sawdust activated carbon using BET analysis, which signifies a microporous carbon and an enormous improvement to the pure washed sawdust with BET surface area of 1.21 ^{2}/g. Attia also produced sawdust carbon activated with phosphoric acid and reported a BET surface area of 831 ^{2}/g and pore volume of 0.39 ^{3}/g.

3.2.8 Surface morphology

The SEM image of the sawdust activated carbon is as shown in Figure 1. The figure shows that the activation process produced extensive external surface areas with oval shaped pores which was accomplished through the evaporation of the chemical reagent during activation process, hereby leaving empty pores. The pores are of different shapes and are irregular in their distribution. Raw sawdust is usually characterized by a highly oriented structure in the form of filaments filled with materials, conferring an anisotropic character to the sawdust, which was eliminated by treatment with potassium carbonate. Firstly, it clears the filament, thereby eliminating sawdust anisotropy and leaving empty channels. Then it further reacts with the sawdust component leaving a honey comb structure as seen in Figure 1.

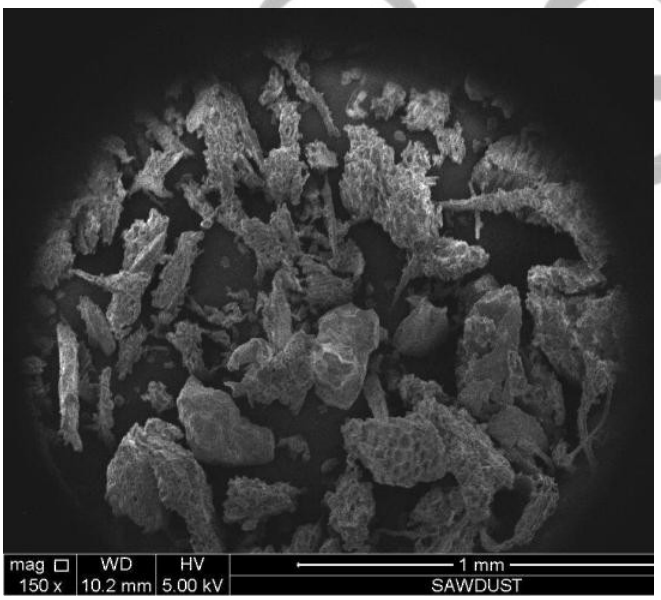

Figure 1: SEM image of sawdust activated carbon at 150x magnification

3.2.9 Batch Adsorption

The batch adsorption was carried out with varying adsorbent dosage and contact time at ambient room temperature. The effect of adsorbent dosage was studied at 2g/l, 4g/l, 6g/l, 8g/l and 10g/l, while the effect of contact time was observed at 20min, 40min, 60min and 80min. The overall treatment process showed a maximum removal efficiency of 99.17% (5mg/l), 96.61% (26.67mg/l), 99.35% (0.01mg/l) and 87.53% (2.48mg/l) for COD, TSS, lead and nitrate respectively as shown in Figure 2.

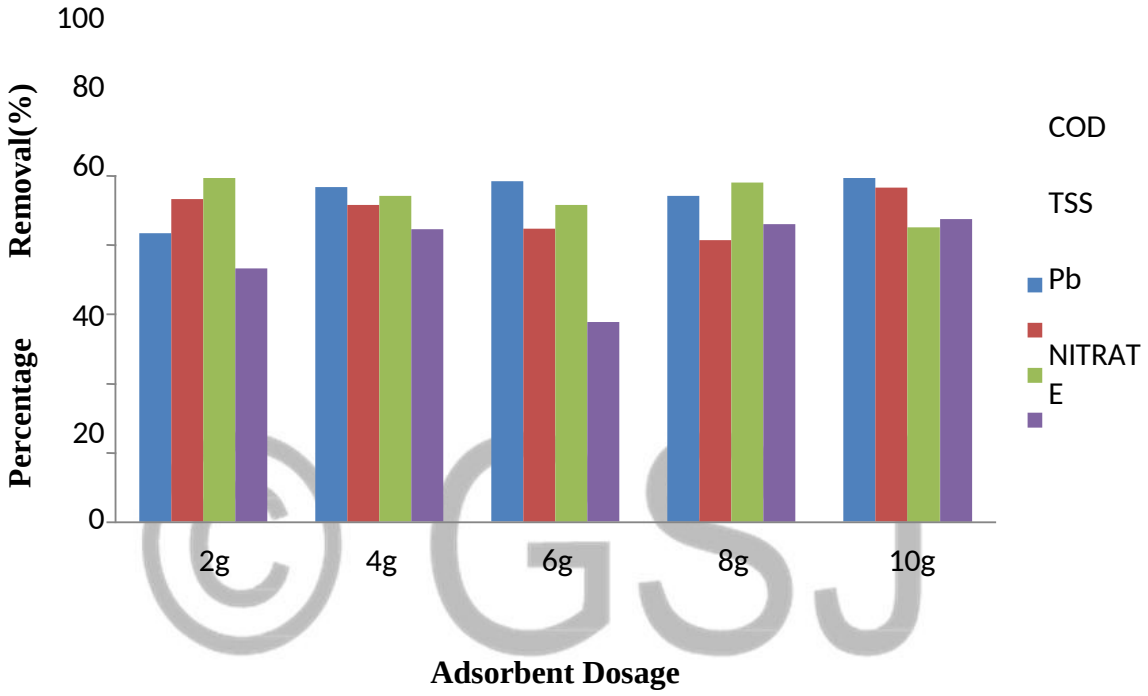

Figure 2: Optimum Percentage Removal at Varying Dosage

4 CONCLUSION

Activated carbon was prepared from sawdust by chemical activation with potassium carbonate in the 1:1 w/w ratio, at an activation temperature of 450^{o}C. Different parameters like pH, moisture content, bulk density, methylene blue adsorption, iodine number, surface morphology and BET surface area were determined and the results obtained indicated that the prepared sawdust activated carbons has good adsorptive properties with as much as 831 ^{2}/g for the surface area. After the optimization study, an equilibrium time of 60min was recorded and an adsorbent dosage of 2g per liter of the effluent. The physicochemical tests on the raw effluent showed that the raw aquaculture effluent is polluted and should be treated before disposal according to NESREA standard. At the end of experimental batch treatment process, the physicochemical parameters of the fishpond effluent had been reduced to meet the NESREA standards for effluent disposal. So, it can be concluded that “potassium carbonate sawdust activated carbon” is a good adsorbent for the treatment of aquaculture effluent. After the various assessment of the use of sawdust activated carbon in the treatment of aquaculture effluent, it can be concluded that; Aquaculture effluent contains pollutants that are beyond the NESREA discharge limit, hence, should be treated prior to disposal. The use of sawdust activated carbon in the treatment of

aquaculture effluent, it can be concluded that, while potassium carbonate is a good activating agent, producing a large surface area(831.8m^2/g), SAC is also a good adsorbent for the treatment of aquaculture effluent with a success of 66.67%. The bulk density (0.27 g/cm^3), moisture content (3.54%), pore volume (0.34 cm^3/g), surface morphology and BET Surface area (831 g/cm^3) properties of the SAC showed it to be a good adsorbent. SAC is an effective medium in the treatment of aquaculture effluent prior to disposal with a COD, BOD, Nitrate, Suspended solids, Pb ion and phosphate percentage removal of 83.3, 92.3,73.2, 93.2, 99.3 and 60% respectively. Optimum operating conditions are concluded to be a contact time of 60minutes at an adsorbent dosage of 2g at ambient room temperature. I recommend that further research on the prepared activated carbons should be carried out, such as reduction of the precursor to activating agent ratio, in order to minimize cost of production, the use of a continuous test, and also the regeneration of the spent carbon.

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