



Treatment of Organic Pollutants in Wastewater Using Activated Carbon Generated from *Oryza Sativa* Husk as Adsorbent

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Abstract-The effectiveness of locally produced activated carbon from *Oryza sativa* husk was investigated and analyzed by carbonizing the material and activated with phosphoric acid and treated with sodium bicarbonate in order to open up the pores of the char for proper adsorption. The samples were carbonized at different temperatures viz 300^oc, 500^oc and 600^oc respectively. Following the basic pretreatment procedure such as sorting, washing, drying and sample activation, the adsorption capacity of the activated carbon was tested in naphthalene and acenaphthene using Gas Chromatography by subjecting the samples to standard testing conditions. The adsorbent was also used to adsorb different concentration of naphthalene and acenaphthene (50-150mg/L) in wastewater sourced from a nearby industry. The removal efficiency of *Oryza sativa* husk ranges from 63.85-80.56%, and the outcome of the adsorption capacity of the experimented sample was fitted for the Freundlich adsorption isotherms and the values of 1/n ranges from 0.2390-1.1498 and it indicates that tested adsorbent is good for adsorption of polycyclic aromatic hydrocarbons (PAHs) from wastewater. The research also analyzed and compared the adsorption capacity of the activated carbon in 2 rings and 3 rings PAHs and the result shows that the adsorbent is more effective in removing pollutants in the 3 rings PAHs than in the 2 rings PAHs.

Keywords- Activated Carbon, Organic Pollutants, Wastewater, Adsorbents, PAHs, Adsorption Isotherms

I. INTRODUCTION

The quest for the removal and treatment of polyaromatic hydrocarbons from wastewater and other organic materials has become source of concern to the relative stakeholders for several years now. It was also discovered in recently that environmental pollution related to organic materials has gone beyond what was obtainable and needs urgent attention and perfect solution. This initiative of wastewater treatment is to recycle water which will serve as a means to replenish the existing water bodies, mainly in the area where there is shortage of water supply to meet the demand of fast-growing population in some parts of the world (ATSDR, 2007). Industrial development over the years has brought about increase in the level of contaminants otherwise known as pollutants and has impacted the wastewater hence need for their treatment and these include dyes, heavy metals and some

organic materials and these are varied from the common inorganic pollutants such as NO₂, SO₂ and CO researchers were familiar with in the past. (Domeno and Nerin, 2003).

It is important to know that polyaromatic hydrocarbons (PAHs) became interesting subject in the research arena across the globe because of the carcinogenic and mutagenic characteristics (ATSDR; 2007) although the number of the organic PAHs pollutants being released into the environment are slightly lower compared to those inorganic counterparts (Brenner, 2002).

As part of efforts geared toward the treatment of contamination caused by PAHs, USEPA (2004) recommended the use of activated carbon produced by extraction, preparation, characterization and carbonization of agricultural wastes. Polyaromatic hydrocarbons may be released or dispersed into the environment through incomplete combustion of fossil fuels such as petroleum, coal and other industrial wastes like wood, paper and rubber (tyre) that contain carbon and hydrogen (Domeno and Nerin, 2003). Likewise, some imbalance in the molecular structure, slow rate of photochemical decomposition and biodegradation also contribute immensely to the growth of PAHs in the environment (Liu et al, 2000). However, PAHs are not known to static in nature but are rather dynamic and can be subjected to redistribution and transformation (Simonich and Hites, 1995). They can also be formed by both natural and anthropogenic process (Fernandes, 2001).

Despite their formation through pyrolysis or incomplete combustion of organic materials that contain carbon and hydrogen, at higher temperatures, the pyrolysis of organic compounds generates molecular fragment and radicals which combined to produce PAHs (Fetzer, 2000). The composition of the resulting products of the pyrosynthesis is dependent on the nature of the wastewater, the temperature and the residence time in the hot region. The increase in the proportion of higher molecular weight PAHs is considered to be an indication of contamination of mainly pyrolytic origin (Kanally and Harayama, 2000).

Anthropogenic sources include combustion of fossil fuels, coal gasification and liquefaction processes, waste incineration and production of coke, carbon black, coal tar pitch, asphalt and petroleum cracking (Domeno, 2003). PAHs are usually solids at room temperature and have very low volatility (Manoli and Samara, 2000). Judging by their aromatic

character, the PAHs absorb ultraviolet light and give characteristic fluorescence spectra (Mastral et al, 2004).

Activated carbon is an end product of carbonization and activation of carbonaceous materials (Bowen et al, 1989). This process requires large surface area and provides carbon with the strength to absorb gases and vapors from dissolved substances in liquids. It can be prepared from agricultural wastes like rice husk, coconut shaft, banana peels, palm kernel shell, cassava peels, yam peels etc.

The adsorptive property of activated carbon makes it a suitable material for a wide range of applications which include its industrial use in solution purification, solvent reclamation and research laboratories as decolourising agents and catalyst support.

Activated carbon exists in two different forms viz; powdered activated carbon (PAC) and granular activated carbon (GAC), (Schweitzer, P.A., 2005). Aksu and Yener, (2001) explained that adsorption occurs whenever molecules diffuse from bulk of the fluid to the surface of the solid adsorbent forming a distinct adsorbed phase and this was performed using activated carbon as the adsorbent. Adsorption process is applicable for the removal of trace components from the liquid phase and may be used in recovering the component or to rid an industrial effluent of poisonous substances (Potgieter, 1991).

Adsorbents are available as irregular granules, extruded pellets and formed spheres (Strelko, V., et al, 2002). Meanwhile, during adsorption experiment, some isotherms are applied and they include; Langmuir isotherm, BET isotherm and Freundlich isotherm. These adsorption isotherms show the relationship between the adsorption capacities and the concentrations of PAHs (Ayranci, E., 2005). Some of the notable PAHs recently classified as priority pollutants include naphthalene (Np), acenaphthalene (Acy), acenaphthene (Ace), fluorene (Fl), pyrene (Pyr), phenanthrene (Ph) and anthracene (Ant).

It was also discovered that PAHs are ubiquitous in nature that is, they can be found in water, soil and air (Binet, P., et al, 2000). They emanate from both natural and anthropogenic processes (Fetzer, J.C., 2000). The other sources may come from combustion of refuse and wood and can as well be from spillage of raw and refined petroleum (Domeno, C., and Nerin, C., 2003). They are also likely to be found in smoked and fried foods like fishes and other agricultural products (Svendsen, T.C., et al, 2006). PAHs toxicity and their effects on human health and living environment was examined and listed as mutagenic, carcinogenic and tetragenic (Luch, 2005).

II. METHODOLOGY

A. Reagents and Apparatus

Some of the reagents and apparatus used include distilled water, PAHs (naphthalene 2 rings; 100g, mw), (acenaphthene 3 rings; 100g, mw), Merck, 128:18g, commercial activated carbon (5kg), phosphoric acid (H_3PO_4 , 8% Conc.), sodium hydrogen carbonate ($NaHCO_3$) and acetone (BDH Chemicals

Ltd, mw 58.08g, 0.789-0.791 $g^{0}C$); beakers, measuring cylinders, magnetic stirrer, weighing balance, mortar and pestle, Vectra furnace, standard flask (100ml), funnel, filter papers and spatula.

B. Source of Raw Material, Sample Preparation and Pretreatment

The raw material (*Oryza sativa* husk) used for the experiment was sourced from a milling center at Arada local market in Ogbomoso, Oyo state, Nigeria.

After collecting the needed quantity of the raw material, it was transported to the laboratory for sorting and preservation. It was washed with distilled water and sun dried to reduce the moisture content. Dried sample was further grinded for better size reduction and increased surface area and was then packed and stored for carbonization.

C. Carbonization

1 kg of the sample was measured into four crucibles using weighing balance and were carbonized to char at different temperatures viz $300^{\circ}C$, $400^{\circ}C$, $500^{\circ}C$ and $600^{\circ}C$ respectively in the Vectra furnace (Model 184A, Italy) for 2 hours using the thermostat for time control and the pores were opened for adsorption process.

D. Activation

Acid activation method (Phosphoric acid) was used for this research work. The samples were sieved with 10 BSS mesh size (over screen) and 100 BSS mesh size (under screen) and carbonized at different temperatures and the activated adsorbents were weighed and kept in labelled beakers and soaked with excess phosphoric acid for 3 hours and then charged into the furnace at $200^{\circ}C$ for 24 hours to dry. The dried samples were removed at the end of the set time and cooled for 2 hours. Later washed with bicarbonate solution and distilled water and tested on P^{H} for confirmation of acidity level.

E. Preparation of Adsorbates

After preparing all the glass wares needed for the experiment, 30ml of acetone was measured into 1dm³ standard flask and 50mg of the PAH sample was added and mixed together and allowed to dissolve. Then, 700ml of distilled water was added and thoroughly shaken to ensure the dissolution of the adsorbates. The flask was covered with filter paper and well tightened to prevent evaporation. The procedure was also repeated for the 100mg and 150mg of PAH respectively.

F. Adsorption Process

Electric weighing balance was used to measure 1g of adsorbent and mixed with 50mg/l of the prepared adsorbate solution. The mixture was then placed on a magnetic stirrer set at 150rpm and allowed to operate for 30 minutes. It was then removed and allowed to settle for about 45 minutes. The resultant supernatant solution was filtered into an already washed and labelled sample bottled using filter paper. The same steps were repeated for 100mg/l and 150mg/l of the selected adsorbate respectively.

G. Gas Chromatography (GC) Analysis

The experimental analysis was performed using a PERIKINELMER AUTO-SYSTEM gas chromatography equipped with flame ionization detector (FID). A 30ms-fused capillary column with internal diameter 0.25um and 0.25/um film thickness were used and the peak area was analyzed with a 3RI model and 203 peak sample chromatography data system. The column temperature was 60°C for 2mn to 320°C set at 10°C/mn. Nitrogen was used as carrier gas with a flowrate of 35psi, hydrogen and air flowrates were 22psi and 28psi respectively. Injector port and detector temperature were 250°C and 320°C respectively.

1-2 ml of sample was injected after checking the calibration standard to ascertain column performance, peak height and resolution and the limits of detection. Compounds were identified mainly by their retention times and the abundance of quantification of analyzed samples with respect to authentic PAH standard.

III. RESULTS AND DISCUSSION

Following the analysis of samples and presentation of values, it is observed that in Table 1, there is a corresponding reduction in the weight of the samples after carbonization over a period of 2 hours. The samples reduced accordingly with

increase in activation temperature and it is obvious in the percentage burn-off. Also, from the Table 2, the result of the weight reduction showed that increase in temperature has a reduction effect on the carbonization of samples. Similarly, Table 3 shows the adsorption capacity of rice husk carbonized at 300°C for naphthalene and the removal efficiency (RE %) increased with increase in concentration. At initial concentration of 50 mg/l, the removal efficiency is 63.854% and at 100 mg/l, it is 66.337% while at 150 mg/l, it rose to 67.165%. The values of the log Ce and log qe have similar effect.

Table 4 shows adsorption capacity of rice husk carbonated at 500°C for Naphthalene in waste water. The clear observation from the experimental analysis is, increase in concentration from 50-150mg/L is directly proportional to the removal efficiencies (RE) i.e 66.040-67.537%. Table 5 has similar effect and it shows minimal increment in the removal efficiency.

Also, from Table 6 to 8, the data obtained for the Acenaphthene (3 rings) in waste water treatment shows that the adsorption efficiencies are higher when compared to that of Naphthalene and therefore confirm that rice husk activated carbon is comparatively strong in removing pollutants from PAHs. Table 9 shows the relationship between 1/n and kf, the lower the values of Freundlich intensity (1/n) the higher the values of Freundlich (kf).

TABLE I. CARBONIZATION PROCESS OF THE ORYZA SATIVA HUSK FOR ACTIVATED CARBON

Agricultural waste	Activation Temperature (°C)	Sample code	Carbonization period (hour)	Initial weight (g)	Final weight (g)	Percentage Burn-off (%)
Rice husk	300	RH3	2	1000	550	45
	500	RH5	2	1000	400	60
	600	RH6	2	1000	200	80

TABLE II. TABLE SHOWING THE ACTIVATION OF SAMPLES AND THE PERCENTAGE REDUCTION

Activated carbon	Initial weight (g)	Final weight (g)	Percentage reduction in weight (%)
RH300	160.0	139.3	12.94
RH500	323.0	292.3	9.50
RH600	330	287.6	12.85

TABLE III. ADSORPTION CAPACITY OF ORYZA SATIVA (RICE) HUSK CARBONIZED AT 300°C FOR NAPHTHALENE IN WASTE WATER

Group ID (°C)	Initial Conc. (Co) mg/L	Final Conc. (Ce) mg/L	Concentration adsorbed (qe) mg/g	Log Ce	Log qe	Removal Efficiency (RE) (%)
RH300	50	18.07281	1596.36	1.257	3.203	63.854
RH300	100	33.66312	3316.84	1.527	3.520	66.337
RH300	150	49.25342	5037.33	1.692	3.702	67.165

TABLE IV. ADSORPTION CAPACITY OF ORYZA SATIVA (RICE) HUSK CARBONIZED AT 500°C FOR NAPHTHALENE IN WASTE WATER

Group ID (°C)	Initial Conc. (Co) mg/L	Final Conc. (Ce) mg/L	Concentration adsorbed (qe) mg/g	Log Ce	Log qe	Removal Efficiency (RE) (%)
RH500	50	16.98004	1650.99	1.229	3.218	66.040
RH500	100	32.83687	3358.16	1.516	3.526	67.163
RH500	150	48.69369	5065.32	1.687	3.706	67.537

TABLE V. ADSORPTION CAPACITY OF ORYZA SATIVA (RICE) HUSK CARBONIZED AT 600°C FOR NAPHTHALENE IN WASTE WATER

Group ID (°C)	Initial Conc. (Co) mg/L	Final Conc. (Ce) mg/L	Concentration adsorbed (qe) mg/g	Log Ce	Log qe	Removal Efficiency (RE) (%)
RH600	50	14.38250	1780.88	1.158	3.250	71.235
RH600	100	28.65936	3567.03	1.457	3.552	71.340
RH600	150	42.93593	5353.20	1.632	3.728	71.376

TABLE VI. ADSORPTION CAPACITY OF ORYZA SATIVA (RICE) HUSK CARBONIZED AT 300°C FOR ACENAPHTHENE IN WASTE WATER

Group ID (°C)	Initial Conc. (Co) mg/L	Final Conc. (Ce) mg/L	Concentration adsorbed (qe) mg/g	Log Ce	Log qe	Removal Efficiency (RE) (%)
RH300	50	13.47562	1826.22	1.130	3.261	73.048
RH300	100	28.20883	3589.55	1.450	3.555	71.791
RH300	150	42.91204	5352.89	1.633	3.729	71.372

TABLE VII. ADSORPTION CAPACITY OF ORYZA SATIVA (RICE) HUSK CARBONIZED AT 500°C FOR ACENAPHTHENE IN WASTE WATER

Group ID (°C)	Initial Conc. (Co) mg/L	Final Conc. (Ce) mg/L	Concentration adsorbed (qe) mg/g	Log Ce	Log qe	Removal Efficiency (RE) (%)
RH500	50	10.79773	1960.11	1.033	3.292	78.405
RH500	100	25.62269	3718.86	1.408	3.570	74.377
RH500	150	40.44764	4477.62	1.606	3.738	73.035

TABLE VIII. ADSORPTION CAPACITY OF ORYZA SATIVA (RICE) HUSK CARBONIZED AT 600°C FOR ACENAPHTHENE IN WASTE WATER

Group ID (°C)	Initial Conc. (Co) mg/L	Final Conc. (Ce) mg/L	Concentration adsorbed (qe) mg/g	Log Ce	Log qe	Removal Efficiency (RE) (%)
RH600	50	9.71828	2014.08	0.899	3.304	80.563
RH600	100	24.31926	3784.04	1.386	3.578	75.680
RH600	150	38.92024	5553.98	1.590	3.744	74.053

TABLE IX. EXPERIMENTAL DATA SHOWING 1/N VERSUS KF

Activated carbon	1/n	KF
RH300 (2rings)	1.1498	57.478
RH500 (2rings)	1.0643	81.395
RH600 (2rings)	0.2390	107.647
RH300 (3rings)	0.9273	163.080
RH500 (3rings)	0.7735	308.958
RH600 (3rings)	0.7251	383.620

IV. CONCLUSION

The treatment of organic pollutants in wastewater using activated carbon produced from oryza sativa husk as adsorbent was experimented. Based on the analyzed data and results from the research, it was observed that locally made activated carbon when produced under standard conditions, can be effective for adsorption of polycyclic aromatic hydrocarbons.

Gas chromatography analysis done on the rice husk activated carbon at 600°C for both acenaphthene and naphthalene almost equals the required standard for commercial activated carbon in terms of adsorption capacity. It is concluded that the finer the particle sizes, the better the adsorption rate or affinity of organic pollutants as demonstrated with oryza sativa husk.

Also, assessing the outcome of the adsorption experiment from the 2rings and 3rings PAHs, there is a clear indication that increase in the temperature of carbonization process has a higher effect on the removal efficiency of the adsorbent.

Conclusively, the utilization of agricultural wastes as raw materials required for adsorption and treatment of pollutants from wastewater will go a long way in proffering solution to the environmental pollution issue and help to minimize waste management problem.

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