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Assessment of Sullage Chemical Properties Treated with Activated Carbon from Selected Agrowastes

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ABSTRACT

Sullage was treated with selected carbonized Agro-wastes (rice husk, corn cob and coconut husk). The Agro-wastes were first carbonized at 600^oC and chemically activated using phosphoric acid (H_3PO_4). They were then used as adsorbents for the removal of chemical compounds: pH, Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), nitrate (NO_3^-), phosphate (PO_4^-), chloride (Cl^-) and sulphate (SO_4^{2-}) from sullage using titration and spectrophotometer methods. Initial concentration of the chemical parameters of sullage samples showed COD to be 486.2±87 mg/l; BOD: 175.0±3.1 mg/l; pH: 7.33±0.1; Cl^-: 31.0±5.5 mg/l; NO_3^-: 28.6±24 ppm; PO_4^-: 7.08±3.2 ppm and SO_4^{2-}:347.08±67.1 ppm. After treatments, COD ranged from 122.2±10 - 190.5±25 mg/l; BOD: 44.0±3.5 – 68.6.0±9.0 mg/l; pH: 7.08±0.1 - 7.18±0.1; Cl^-: 17.28 - 21.13 mg/l; NO_3^-: 0.0 - 12.79 ppm and SO_4^{2-}: 117.7 - 251.7 ppm. There was significant difference ($p \leq 0.05$) between the untreated and the treated sullage samples. The efficiency of the individual adsorbents in adsorbing the chemical parameters was of this order: ricehusk>corncob>coconut husk. In combination it was, rice husk+corncob+coconuthusk > ricehusk+corncob > corncob+coconuthusk > ricehusk+coconuthusk. High percentage reduction observed of chemical properties revealed that activated carbons from rice husk, corn cob and coconut husk can be used singly or combined for the purification of sullage.

Introduction

Most of the freshwater bodies in Nigeria are daily taking in varieties of organic and inorganic pollutants from household sullage.[1] The daily amount generated is 36,493, 920 liters ($36494m^3$).[2] Organic compounds consist primarily of carbohydrates, proteins and fats which reflect the diet of the people.[3] Inorganic components consist of heavy metals, nitrogen, phosphorus, pH, sulphur, chlorides, alkalinity, toxic compounds etc. Inorganic compounds also include acidity caused by domestic discharge of ammonia from food processing waste, and chemical waste as industrial by-products, fertilizers, etc.[3] Other chemical properties of sullage include chemical Oxygen Demand, Biochemical Oxygen Demand.[4]

Department of Local Government, DLG 1998[5] identifies how domestic wastewater may harm the environment and is summarized as follows: by overloading the land application system with nutrients; by exceeding the hydraulic loading the land application system with water causing run off of polluted water to storm water drains, rivers, streams and other people's property; by altering the soil salinity, permeability, pH, soil electrical conductivity, soil sodality, soil cation exchange capacity, soil phosphorus sorption capacity, soil depressiveness; and by polluting the soil with chemical impurities which affect the properties of the soil to assimilate nutrients or water. Saeed *et al.*[6] conducted a study to evaluate the effect of household sullage (soap water) on growth of *Sesbania grandiflora L. S. grandiflora*. showed that higher concentration of detergents resulted in toxic effects such as poor growth and development of root nodules due to ionic toxicity or change of hydraulic properties of the soil.

Self-purification mechanism of land, air and water have been relied on the past to adsorb these pollutants[7] but the current discharge and concentrations exceed effective thresholds of the natural ecosystem.[8] Maton, Eziashi, Dodo & Olaku[9], recommended that reduction in the discharge of pollutants into the water bodies can be achieved by the removal of pollutants before discharging into the environment, creation of public awareness on the importance of water sanitation and good waste disposal method and enforcement of environmental laws.

Activated carbon based compound used for adsorption technique has numerous applications in removing pollutants from air or water such as in spill cleanup, groundwater remediation, drinking water filtration, air purification and volatile organic compounds capture from painting, dry cleaning, gasoline dispensing operations and other processes.[8] It is a carbon which has been processed to make it extremely porous and thus have a very large surface area available.[8] Its usefulness derives from its large micropore (and sometimes mesospore), volume and high surface area. The quest for economically safe activated carbon to be used as adsorbents has led to the exploration of biomass (rice husk, saw dust, oil palm shell etc.) for the removal of pollutants from wastewater. This research work determined the efficiency of using activated carbon from rice husk, corn cob and coconut husk in the treatment of sullage through the assessment

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of the sullage's chemical properties before and after treatment.

Material and Method

Collection of Samples

Sullage was fetched from male and female hostels of Federal University of Technology, Owerri, in a sterile 10 litre container. Agro-wastes were sourced from Relief Market, Owerri, Imo State, Nigeria.

Carbonization and activation of absorbent

Grigis & El-Hendawy[10] procedure was adopted with slight modification. 750 g of each agro-waste were washed with deionized water, dried and crushed using locally made grinder. They were carbonized in electric muffle furnace at 600^oC for 45 min. The carbonized or pyrolyzed sample was sieved using 1.18 mm mesh size. Activation was achieved by mixing the carbonized sample with 20% H_3PO_4 solution at a ratio of 1:1 (Acid: Char) and stirring for 30 min. After that, the sample was then filtered, washed with deionized water until the washed off water gave a pH of 7.0. Washed activated carbon was then dried at 120^oC in an oven. The dried sample was stored in airtight container.

Adsorption column

An apparatus (burrete) of size 50 cm length and 1cm diameter (adsorption column), a tank and collection flask were provided for this experiment. An outlet was provided at the bottom of the tank. Experiment was conducted by placing the individual adsorbents in column apparatus separately at first and then in combination. From the tank waste water was allowed to pass in to the apparatus, collected water analyzed. Experiment was carried out at room temperature 30 ± 2^0 C.[11]

Characterization of Waste Water

Chemical Characterization [12]

pH

pH was determined by taking the pH readings for the before and after treatment of the effluent using pH meter. It was first calibrated with a buffer of 7.0.

Biochemical Oxygen Demand Analysis

The BOD_5 was determined using DO_2 meter. The DO_2 meter was calibrated using 5% sodium sulphate solution. The probe of the meter was inserted into the sample after which the meter switched on for about 10 minutes. The readings were recorded in mg/l. The sample was incubated in a 250 ml wrinkler's bottle for a period of 5days at 20^oC and the DO_2 of the fifth day recorded by inserting the probe again into the sample. The difference in the DO_2 (5) and DO_2 (1) was recorded and calculated as the BOD_5.

$$BOD_5\,(mg/L) = DO_2\,(5) - DO_2 \quad (1)$$

Chemical Oxygen Demand (COD)

Some 20 ml of the sample plus blank was measured into a 250 ml conical flask. After, 10ml of dichromate solution ($K_2Cr_2O_7$) (0.25N) and 30 ml of concentrated sulphuric acid were added. Samples were cooled in a cold waterbath. The mixture was diluted to about 150 ml and 3 drops of ferroin indicator added. The mixture was titrated with ferrous ammonium sulphate solution (O.5N $FeSO_4$. $7H_20$) until there was a change in colour. COD was calculated as:

$$\text{COD, (mg/litre)} = \frac{(B-A)(N)(8000)}{V} \quad (2)$$

Where

A = volume of ferrous ammonium sulphate solution used for sample titration in ml

B = volume of ferrous ammonium sulphate solution used for blank titration in ml

N = normality of the ferrous ammonium sulphate solution

V = sample volume in ml

Chloride (Argentometric Method)

Measured 50ml of the wastewater sample was transferred into conical flask and 3-4 drops of indicator 5% potassium chromate indicator added. The solution in the conical flask was titrated with 0.02N silver nitrate to a reddish brown end point using a micro burette. A blank titration was done as above using deionized water.

$$Chloride, (mg/l) = \frac{(V_1 - V_2)xNx35.5x1000}{Volume\ of\ sample} \quad (3)$$

Where

V_1 = volume of sample titer

V_2 = volume of blank titer

N = normality of silver nitrate used (0.02N)

Phosphate (Ascorbic Acid Method)

Ammonium heptamolybdate (6.0 g) was weighed and dissolved in 150 ml distilled water in 250ml conical flask. Ascorbic acid (2.6 g) was dissolved in 50 ml of distilled water in 1litre volumetric flask to give 0.0007 M. Then, 0.4 g of disodium ETDA was dissolved in the 0.0007M ascorbic acid and O.14 g of potassium antimony tartrate weighed and dissolved in 20 ml distilled water (0.0000086 M). Stock of concentrated sulfuric acid (1M) was prepared by dissolving 10ml of the stock in 50ml distilled water.

Phosphate stock (1000 mg/L PO_4^{3-}) was prepared by weighing accurately 1.532 g of potassium phosphate trihydrate in 250 ml of distilled water. Thereafter, 0.5, 1.5, 2.0, and 2.5 ppm PO_4 were prepared by proper dilution with distilled water for calibration curve.

Measured volume (12.4 ml) of the ammonium molybdate solution was transferred into a 50 ml volumetric flask and then 10 ml sulfuric acid was added, swirled, and 2.3 ml of antimony potassium tartrate added. The mixture was swirled properly to mix and the mixture made up to the mark with distilled water.

Spectrophotometric determination was carried out by adding 0.4 ml molybdate reagent to 20ml of standard or sample in a test tube and swirled to mix. Also, 0.4 ml of L-ascorbic acid was added and swirled. The light absorption of the solution was measured at 820 nm wavelength using spectrophotometer.

Nitrate, (NO_3^-) determination

To each of standard nitrate solution of different concentrations were added 10 ml of H_2SO_4 and swirled. The beakers containing the solutions were allowed to reach thermal equilibrium inside a cold water bath prior to heating. To each of the beakers was added 0.5 ml of brucine-sulphahilic acid reagent, swirled properly to mix and placed in 100^oC water bath for 25 minutes. After heating, the beakers were removed from the hot water bath, immersed in a cold water bath and allowed to reach thermal equilibrium of $20\text{-}25^oC$. The mixtures in the beakers were hence analyzed with spectrophotometer and the absorbance read at 410 nm wavelength. The samples and blank were treated in the same manner.

Sulphate (Turbidometric Method)

Deionized water (100 ml) was poured into a clean and acid-washed beaker with 50 ml of buffer solution, and then transferred into a clean, 125 ml Erlenmeyer flask containing a clean magnetic stirring bar. Some 10 ml of deionized water, 6 ml of buffer reagent, and 10 ml of the standard solution were added into the flask. It was swirled gently to ensure mixing. About 0.1 g - 0.2 g of $BaCl_2$ was added to the flask which was immediately placed on the magnetic stirrer and stirred for 58 to 62 seconds. After a minute of stirring, the solution was allowed to stand undisturbed for 2 minutes. The absorbance was measured by a spectrophotometer at the wavelength of 420 nm.

Efficiency of Percentage Removal

The efficiency of percentage removal was done according to Ahmad *et al.*[13] as:

$$Removal\ efficiency = \frac{C_o - C_k}{C_o} X100 \quad (4)$$

Where

C_o = Initial concentration

C_k = concentration after treatment

Statistical Analysis

Analysis of Variance (ANOVA) and Student t-Test at 0.05 (95%) significant level were used to analyze data generated.

Results and Discussion

Chemical properties of untreated sullage.

From Table 1, apart from pH, phosphate, chloride and nitrate, other physicochemical parameters of hostel sullage of Federal University of technology, Owerri (FUTO) were above the Environmental Protection Agency[14] and World Health Organisation[15] permissible limits. COD mean values ranged from 342.2±71 mg/l - 482.2±87 mg/l while BOD mean values ranged from 1.23.2±25 mg/l - 175.0±31 mg/l. pH mean values ranged from 7.01±0.1 - 7.33±0.1. Phosphate had a range of 4.25±1.3 mg/l - 8.77±1.7 mg/l; Chloride, 21.9±4.4 mg/l - 31.0±5.5 mg/l; Nitrate, 7.74±9.5 ppm - 28.6±24 ppm and sulphate, 289.51±21.7 ppm -347.08±67.1 ppm. The high values of the parameters can distort the homeostatic balance of the receiving water. This correlates the work of Adebayo *et al.*[16] on Ota Mmirri river at FUTO station who suggested that the high values of some physicochemical properties might be from the wastewater discharged from FUTO hostels. The chemical properties of sullage samples from male and female hostels varied within the groups. This might be as a result of the quality of the water used by students, personal habits and lifestyles, the type of cosmetic used and laundry activities in the hostels[16]. Onyekuru *et al.*[17] reported that hand pump wells near septic tank units, which are a major source of water in FUTO hostels do not impair the quality of water supply in aquifer system as anticipated. The values of the physicochemical parameters of hostel sullage may be traced to the contaminants from shampoos, hair dyes, toothpastes and cleaning chemicals. Since more compounds are chemically oxidized than biologically oxidized[18], COD values were higher than the BOD values. Sulphate contamination might be from laundry detergents and soaps[19].

Chemical parameters of sullage treated with absorbents

From Table 2, there was a reduction in the physicochemical properties of the sullage after the treatment with activated carbon forms of the agrowastes. Apart from BOD, their levels were below the EPA and WHO discharge limit, thus does not pose any threat to environment. COD ranged from 122.4 to 190.5 mg/l; BOD, 44 – 68 mg/l; pH, 7.07 – 7.17; PO4-, 3.61 – 6.63 ppm; Cl-, 17.28 – 21.13 mg/l; NO3-, 0 – 12.79 ppm and sulphate, 117.7 – 251.7 ppm. Egdon *et al.*20 reported that chemically activated carbon from maize cob produced at 250oC reduced the physicochemical parameters like pH, COD, BOD and nitrate in salon wastewater which was attributed to the unique features like large surface area, high degree of surface reactivity and favorable pore size.

Table 1.Chemical properties of untreated sullage

Parameter	Sample			EPA limits	WHO limits
	M	**F**	**MF**		
COD (mg/l)	486.2±87.0	342.2±71.0	395.5±64.0	250	200
BOD (mg/l)	175.0±31.0	123.2±25.0	142.4±23.0	50	40
pH	7.33±0.1	7.01±0.1	7.27±0.1	6-9	6.5-8.5
PO_4^-(mg/l)	7.68±3.2	4.25±1.3	8.77±1.7	10	10
Cl^- (mg/l)	31.0±5.5	24.9±3.9	21.9±4.4	250	200
NO_3^-(mg/l)	28.6±24	7.74±9.5	13.83±11.8	50	45
SO_4^{2-}(mg/l)	347.08±67.1	319.58±24.6	289.51±21.7	200	200

All values were expressed as Mean±SEM (Standard Error of Mean).

M - sullage from male hostel; F - sullage from female hostel; MF – combined sullage from male and female hostels. EPA Limits= Permissible limits.

Table 2.chemical properties of sullage treated with adsorbents

Parameters	Rh	Cc	Ch Adsorbents	Rh +Ch	Rh+ Cc	Cc +Ch	Rh+Cc+ Ch	EPA limits	WHO limits
COD(mg/l)	158.7±21	190.5±25	188.1±15	170.1±19	127.8±30	187.7±31	122.4±10*	250	200
BOD(mg/l)	57.1±76*	68.6±9.1	67.7±5.2	46.0±11*	61.3±6.7	67.6±11.3	44.0±3.5*	50	40
pH	7.15±0.1	7.18±0.1	7.18±0.1	7.12±0.1	7.13±0.1	7.13±0.1	7.08±0.1*	6-9	6.5-8.5
CL^- (mg/l)	20.5±5.5	17.3±3.3	20.3±4.0	18.5±3.1	20.9±4.6	17.8±2.4	21.1±4.4	250	200
PO_4^-(ppm)	3.61±0.4*	5.61±1.6	5.34±1.9	4.88±2.9	6.60±0.7	6.30±1.1	3.89±05	10	10
NO_3^-(ppm)	8.8±10.7	12.5±12.7	4.1±3.2	12.8±14	0±0.7	11.1±13.3	0±4.3	50	45
SO_4^{2}(ppm)	147.8±7.5*	175.3±23.3	251.7±22.3	163.2±1.7*	178.7±9.7	183.9±31.2	117.7±25.3*	200	200

All values were expressed as Mean±SEM (Standard Error of Mean). MF= combined untreated sullage from male and female hostels. Rh=rice husk; Cc=corn cob; Ch=coconut husk; Rh+Ch= rice husk+coconuthusk; Rh+Cc=rice husk+corncob; Cc+Ch=corncob+coconuthusk; Rh+Cc+Ch=rice husk +corn cob +coconut husk. LSD *, shows the value with significant different compared with MF at p≤0.05.

Efficiency of the adsorbents in the removal of Chemical Properties from sullage

From fig 1, nitrate was better adsorbed by the activated carbon followed by phosphate and sulphate, then chlorine. The efficiency of removal was in this order: Rice husk + corn cob +coconut husk > rice husk + Corn cob > rice husk > rice husk + coconut husk > corn cob > coconut husk > corn cob+ coconut husk and rice husk + coconut husk. For their individual strengths, activated carbon performed in this order: rice husk > corn cob > coconut husk. In combination, rice husk + corn cob + coconut husk > rice husk + corn cob > corn cob + coconut husk > rice husk + coconut husk. A combination of rice husk, corn cob and coconut husk best adsorbed the four inorganic compounds. Zahid[21] reported that chemically activated rice husk had high efficiency in removing inorganic compounds in wastewater.

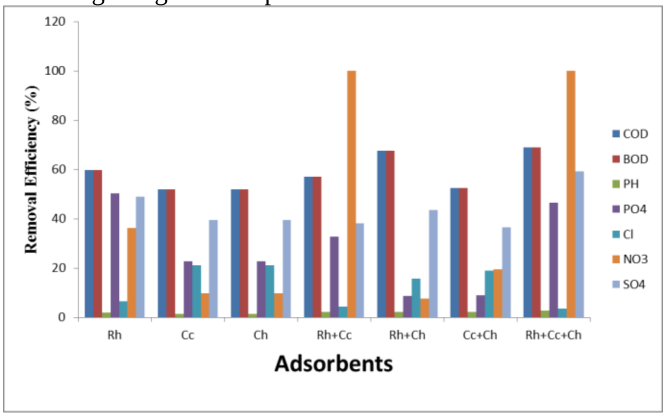

Fig 1. Efficiency of the adsorbents in the removal chemicals properties from sullage

4. Conclusion

The results obtained from this study revealed that activated carbon obtained from rice husk, corn cob and coconut husk have the potential to effectively treat the chemical properties of sullage from domestic sources. The consortium efficiency of other activated carbon forms of agrowastes should be investigated. Other factors which may affect the efficiency of the activated agrowastes in effluent treatment should be critically studied.

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