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Adsorption of Residual Pollutants in Pre-Treated Brewery Effluents onto Locally Produced Adsorbents: A Comparative Study

Raphael Terungwa Iwar[1, *], Kola Ogedengbe[2]

[1]Department of Agricultural and Environmental Engineering, College of Engineering, University of Agriculture, Makurdi, Nigeria
[2]Department of Agricultural and Environmental Engineering, Faculty of Technology, University of Ibadan, Ibadan, Nigeria

Email address

raphaeliwar@gmail.com (R. T. Iwar)
[*]Corresponding author

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Abstract

In the present study, activated carbons were produced from low-cost agricultural residues and characterized. These were; coconut shells carbon (CSC), palm kernel shells carbon (PKC) and a mixture of coconut and palm kernel shells carbon (CCPC). Batch equilibrium experiments were carried out in the laboratory using the adsorbents for the treatment of effluents from a brewery in Makurdi Metropolis, Nigeria. Carbon characteristics ranged between 6.58-7.03%, 0.57-0.65g/cm^3, 76.0-93.5%, 928.0-1126.0m^2/g, 23.0-33.7% and 192-303mg/g for ash content, bulk density, iodine sorption, specific surface area, carbon yield and methylene blue number respectively. In the batch equilibrium experiments, pollutants removal efficiency increased with increase in carbon dosage, contact time and pH. The highest removal efficiencies were observed at a dosage of 8g/100ml, contact time of 120minutes and pH of 8 for COD, TSS and TDS as well as for CSC, PKC and CCPC. Comparatively, CSC and CCPC performed better than PKC in terms of pollutants removal efficiency and carbon adsorption capacities in the order; CSC > CCPC > PKC.

Keywords

Activated Carbon, Removal Efficiency, Brewery Effluent, Coconut Shells, Palm Kernel Shells

1. Introduction

Most industrialist employ only primary and secondary treatments of their wastewater (physical and biological treatments), which do not efficiently remove all contaminants of concern in the wastewater. This is basically due to economic reasons, as every manufacturer is poised to maximizing profits. In other to meet stringent wastewater discharge standards, there is need for industrialists to employ tertiary and/or advanced treatments of their wastewater, especially for those whose wastewater constituents are difficult to remove by only physical and biological means.

Brewing industries are one of the major industrial users of water. These industries have one of the wastes most difficult to treat satisfactorily. The high organic content of brewery effluent classifies it as a very high-strength waste in terms of chemical oxygen demand, from 1000 mg/L to 4000 mg/L and biochemical oxygen demand of up to 1500 mg/L [1]. The treatment of brewery wastewater effluent is a costly task for the brewer in order to meet the government regulations and to practice environmentally friendly manufacturing. The untreated effluent discharge from these industries is coloured and highly intoxicating due to the presence of alcohol and can be toxic to aquatic life in receiving waters, hence the need for the treatment of brewery wastewater effluent before being discharged into water courses. However, the current problems in water and wastewater treatment stem from the increasing pollution of waters by organic compounds that are difficult to decompose biologically because these substances resist the self-purification capabilities of the rivers as well as

decomposition in conventional wastewater treatment plants. Consequently, conventional mechanical-biological purification no longer suffices and must be supplemented by an additional stage of processing. Among the physical-chemical processes that have proved useful for this, adsorption onto activated carbon is especially important because it is the dissolved, difficult-to-decompose organic substances in particular that can be selectively removed by activated carbon.

The use of activated carbons for the removal of toxic substances in wastewater has been reported to be efficient by various scholars [2, 3, 4]. In Nigeria and similar developing nations, activated carbon requirements are met by importation in enormous quantity at a very high cost, whereas vast quantity of agricultural residues, which can be used for its production to meet local demands and even for exportation, are generated annually.

The thrust of this study is to create wealth from waste, by converting these materials considered by-products into activated carbon adsorbents (a value added product and resource for other industries), which can be used for water and wastewater treatment, one of its most important fields in terms of consumption to remove hazardous organic compounds or those that impart odour or taste [3, 4] cleanup of off-gases containing volatile organic compounds, decolourization, solvent recovery and purification, treatment of industrial waste, surface and groundwater redemption, food processing, pharmaceutical and environmental remediation, amongst other uses.

Several studies have been conducted by various researchers on the use of agricultural waste biomass as low cost adsorbents and potential substitutes for conventional activated carbons, for the removal of pollutants in water and wastewater in Nigeria.

[2] studied the suitability of using activated carbon produced from locust bean pods in comparison with that from bone char, for the treatment of domestic wastewater. They asserted that, locust bean pod which is an agricultural waste is a suitable sorbent for the removal of Nitrates, Phosphates and chemical oxygen demand from domestic wastewater. However, they did not establish equilibrium and kinetic studies in the adsorption process.

In their studies, [5] compared the adsorption capacities of coconut and palm kernel shell activated carbons and found out that coconut shells activated carbon was a better adsorbent than palm kernel shells, with both acid and base activations. These findings suggest that the efficiency of activated carbons in removing pollutants in wastewater is a function of; type of feedstock, type of activating agent and nature of pollutant to be removed.

In a similar study, [6] found out that waste Nigerian Bamboo were good adsorbent for the removal of organic pollutants in wastewater if prepared and activated into granular activated carbons (GAC). They asserted that the experimental data fitted well into the Freundlich isotherms when compared with the Langmuir isotherm. A break through time of 1.5 hours was reported for the study. The effects of contact time on the removal of COD from the wastewater stream were also established.

[7], compared the adsorption efficiency of coconut shell-based granular activated carbon (Acid and barium chloride activation), with the adsorption efficiency of commercial carbon, (Calgon carbon F-300), with respect to organic matter from a beverage industrial wastewater. Freundlich adsorption isotherm was used to analyze the adsorption efficiencies of the two activated carbons. The studies indicated that the acid activated coconut shell carbon had higher adsorption for organic matter expressed as chemical oxygen demand (COD), than Calgon carbon (F – 300) at all carbon dosages used. In most of these studies, the efficiencies of removal and/or adsorption capacities of the locally produced carbons competed favourably with the conventional/commercial carbons which are made from non-renewable sources [7, 8, 9].

The performances of the activated carbons are reported to depend on a number of factors such as; contact time, carbonization and activation temperatures, activation agent (type of acid or base), carbon dosage (mass of carbon used per volume of wastewater), column height, pH of adsorbate solution and the characteristics of the produced GAC, which also depends on nature of the parent material from which the GAC was produced [6, 7, 10, 11].

In all of these findings in literature, there have been no efforts toward the production of GAC from a mixture of two or more agricultural waste materials. However, it is suspected that a proper combination of some selected agricultural materials for the production of GAC can greatly improve the efficiency of the GAC in removing pollutants in solution. These is because, most of the materials used to produce GAC do not poses all the required characteristics for efficient removal of pollutants in solution when used in isolation. Thus combining two or more agricultural waste materials together in known proportions, before carbonizing and activating them into GAC may greatly improve the overall characteristics of the GAC, thus improving their efficiencies and adsorptive capacities. The main objective of the present study is to produce and characterize acid based granular activated carbons (ABGACs) from palm kernel shells, coconut shells and a mixture of both. Other objectives are to comparatively study the effects of carbon dosage, contact time and initial effluent pH on the removal efficiencies of residual pollutants in brewery effluents by each of the produced ABGACs using laboratory-scale, batch equilibrium experiments.

2. Materials and Methods

2.1. Experimental Setup

The experimental work was divided into two major parts: (i) production of granular activated carbon using coconut shell, palm kernel shell and a mixture of both and characterization of the manufactured activated carbons, and (ii) treatment of brewery wastewater effluent in batch

equilibrium experiments, using the produced granular activated carbon (GAC) with a view to comparing them. Table 1 shows the list of materials and equipment used during the experiments.

2.2. Materials for Production of Activated Carbons

The coconut and palm kernel shells used for the production of activated carbons for the treatment of biologically pre-treated brewery effluents were gathered from a market and local palm oil processing company all in Makurdi, Benue State, respectively (Figure 1). Other materials used for the production process are, furnace (carbolite, model DH150) fitted with a thermocouple, electric oven, electric weighing balance, crucibles, activation agent (H_3PO_4), beakers, cylinders, pH meter, mortar, and pestle, sieves. The production process proceeded in two phases: Carbonization and Activation Phases [6, 8].

2.2.1. Carbonization

Coconut and palm kernel shells were dried in the sun for 10 hours to remove moisture content. After drying the shells were crushed to pieces of 10-15mm. Shells were then separated from other materials such as fibres and sand, cleaned and prepared to be placed in the furnace. 5000g of the coconut shell was weighed, put on a crucible and then put into the furnace for carbonization. The furnace was switched on, and the temperature adjusted until it reached 600°C and was maintained for a period of 2 hours with limited supply of oxygen. After 2 hours the carbonized samples were collected from the furnace after quenching with cold water and transferred to the oven for further drying at 110°C for a period of 30 minutes. After this, the samples were ready for acid activation (Figure 2). The process was repeated for palm kernel shells as well as for a mixture of palm kernel and coconut shells, which comprised of 2500g of coconut shell and 2500g of palm kernel shells.

2.2.2. Activation

The carbonized coconut shells were weighed and mixed with 0.1M phosphoric acid in a beaker for the purpose of activation and stirred into a paste. The resulting moist paste upon mixing the char with phosphoric acid (H_3PO_4) (30% by weight) was again charged into the furnace and heated at a temperature of 800°C, for a period of 1hour. Cold water was used to quench the activated carbon produced, and this further dried for 60 minutes at 110°C, till a constant weight of activated carbon was obtained. After the chemical activation, the activated carbon was rinsed thoroughly. Washing was used to remove the remaining phosphoric acid and ash in the carbon to a pH of 6-7. This was accomplished by washing with distilled water. The activated carbon was then drained and spread on a tray at room temperature. The activated carbon was dried in an oven at a temperature of 110°C for 3 h. Weight of activated carbon produced was taken and yield calculated. The activated carbon was then crushed using a mortar and pestle and sieved with an American standard mesh 200 to obtain granular activated carbon particles of 1.185 to 2.5 mm. The activation process was repeated for the palm kernel shells and the mixture of both. The activated carbons were thus ready for the characterization process (Figure 3).

2.3. Characterization of Activated Carbon

The produced activated carbons were each subjected to characterization. The parameters determined were, bulk density, specific surface area, ash content, carbon yield, methylene blue value and iodine sorption, all experiments were done in triplicates and the mean values were obtained and reported for each of the produced activated carbons.

All carbon characteristics were determined experimentally according to the methods described by [12] and [13].

2.4. Treatment of Brewery Effluents with Granular Activated Carbons

A fresh sample of biologically pre-treated brewery effluent was collected from the effluent outfall point in the month of August by grab method. Samples were stored in a cooler containing ice block at 4°C and immediately taken to the laboratory. In the laboratory, samples were tested to determine the initial concentrations of the following criteria, residual pollutants: COD, Phosphates, Total Dissolved Solids (TDS), Total Suspended Solids (TSS) and Ammonia. The samples were tested in replicates based on the standard methods for the examination of water and wastewater [15] and values were recorded as means. Samples were then fixed and stored in a refrigerator at 4°C for use during the batch equilibrium experiments.

The tested samples were then subjected to batch experimental studies (treatments), using each of the produced activated carbons. Adsorption capacities and pollutant removal efficiencies were determined for each GAC produced. The effects of carbon dosage, pH, and contact time on the removal efficiencies of the residual pollutants for each GAC were also evaluated and compared according to the method described by [7, 6] and [10] respectively.

2.5. Statistical Analysis

A summary index was used to determine the mean values of results obtained from various parameters. One-way ANOVA was used to test for significant differences in characteristics of the prepared activated carbons. The results were investigated by using the least significant difference at a 95% confidence level using SPSS 20 [15].

Table 1. *List of Instruments and Materials used in Experimental Analysis.*

S/N	Instrument/Material	Description	Quantity
1	Furnace	Manufactured by Carbolite in 2011, Model S302AU	1
2	Electric Oven	Manufacture by Newlife in 2012, Model NL-9023A	1

S/N	Instrument/Material	Description	Quantity
3	Weighing Balance	25 Kg Capacity, Manufactured by Mettle Telode, Model AB204	1
4	Crucibles	250 ml capacity	8
5	Measuring Beakers	250 ml Capacity	4
6	Measuring Cylinder	30ml Capacity, Superior BS 604 Made in West Germany	6
7	Sieves	American Standard Mesh 200	1
8	Mortar and Pestle	Locally Made and laboratory-Based	1
9	pH Meter	pH Meter Model H196107, Manufactured by Hanna Instruments	1
10	Activating Agent	Phosphoric Acid supplied by Allied Laboratories, Makurdi	4 Litres
11	Palm Kernel and Coconut Shells	Locally Sourced	10 Kilograms each
12	Spectrophotometer	Portable Data logging type, Model DR 2010, manufactured by HACH	1
13	Erlenmeyer Flasks	250 ml capacity	8
14	Stoppered Flask	50 ml capacity	4
15	Electromechanical Shakers	Manufactured by Seedboro equipment Company, Model 2512/C	8
16	Stopwatch	Digital stopwatch model N009221, manufactured in India	1
17	WhatmanFilter Papers	Grade No 1, Manufactured by Whatman, Model 1001-110, Pack of 100	1
18	Thermometer	ELE Thermometer, Model HPC7H15, manufactured in 2010	1
19	Deionized water	Supplied by Allied laboratories, Makurdi	2 Litres
20	TDS Meter	Manufactured by HACH, Model CO 150.	1

Figure 1. *Precursors used to produce granular activated carbons (GAC).*

Figure 2. *Carbonized products.*

Figure 3. *Experimentally Produced Granular Activated Carbons (GAC).*

3. Results and Discussion

3.1. Production and Characterization of Experimental Activated Carbons

The mean characteristics of the activated carbons are presented in Table 2. The ash content (%), bulk density (g/cm^3), carbon yield (%), iodine sorption (%), methylene blue number (mg/g) and specific surface area (m^2/g), ranged between 6.58-7.03, 0.57-0.65, 23.0-33.7, 76.0-93.5, 192.0-308.0 and 928.0-1126.0 respectively.

The CSC had the highest ash content, iodine sorption and specific surface area and was closely followed by CCPC, while the PKC had the lowest values of these parameters. This may be due to the low carbon content of CSC as compared to PKC and CCPC, which indicates low output of carbon per given mass of precursor material. The CCPC had the highest methylene blue number and was closely followed by CSC, while PKC had the lowest methylene blue number.

These may be due to the molecular attractions that exist differently in each of the activated carbons, which mean that CCPC has the highest affinity for methylene blue as against CSC and PKC. CCPC also had the highest carbon yield, followed by PKC, while CSC had the lowest value in carbon yield. The PKC however, had the highest bulk density and CPC had the lowest, these shows that PKC and CCPC will be less subjective to attrition during usage as adsorbents as compared to the CSC. There was significant difference in means of all the carbon characteristics between the three carbons at p = 0.05 level of significance.

3.2. Characteristics of Residual Pollutants Before Treatment with Activated Carbons

The pollutants characteristics determined were, COD, TSS, TDS, PO_3^-, and NH_4^+. The results of the residual pollutants characteristics before treatment with granular activated carbons (GACs) are presented in Table 3. From Table 3, the mean values for COD, TSS, TDS, PO_4^{-3} and ammonia were, 318.00, 69.00, 185.50, 7.00 and 3.80 mg/l respectively. The effluent mean pH value was 6.80, while ambient and wastewater temperatures were respectively, 31.0 and 35.0°C. Considering the FEPA discharge limits, these values are too high especially COD, TSS and TDS for acceptable disposal into the surface water bodies.

3.3. Batch Equilibrium Experiments

The results of the batch equilibrium experiments conducted for each experimental carbon in comparison with CSC are presented in the following sub-sections. These include the effects of carbon dosage, wastewater pH and contact time on the removal efficiencies of the identified residual pollutants.

3.3.1. Effects of Carbon Dosage on Removal Efficiency of Residual Pollutants

This was done for COD, TSS and TDS. Carbon dosages used were 0-8g/100ml (0-80g/l) of the wastewater. (Figures 4-6). CSC exhibited the highest removal efficiencies of 78.46, 100, 93.53%, for COD, TSS and TDS, respectively at the highest dosage of 8g/100ml (80g/l), except for TSS which recorded a 100% removal efficiency even at a dosage of 6g/100ml (60g/l).

CCPC exhibited a close removal efficiency of pollutants to that of the CSC, with COD, TSS and TDS, having values of 72.5, 100 and 88.14%, respectively at the maximum dosage of 8g/100ml. PKC, however, had the lowest removal percentages of 67.79, 73.62 and 67.65%, for COD, TSS and TDS, respectively at the same dosage of 8g/100ml.

Table 2. *Characteristic of the Experimental Activated Carbons.*

S/No	Characteristics	CSC	PKC	CCPC
1	Ash Content (%)	$7.03^b \pm 0.23$	$6.58^a \pm 0.07$	$6.82^a \pm 0.18$
2	Bulk Density (kg/m^3)	$0.57^a \pm 0.02$	$0.65^a \pm 0.06$	$0.60^a \pm 0.02$
3	Carbon Yield (%)	$23.00^a \pm 4.36$	$31.70^b \pm 1.47$	$33.70^b \pm 1.53$
4	Iodine Sorption (%)	$93.50^b \pm 5.63$	$76.00^a \pm 2.00$	$88.00^b \pm 2.00$
5	Methylene Blue Value (mg/g)	$223.00^b \pm 2.89$	$192.00^a \pm 8.74$	$308.00^c \pm 14.42$
6	Specific Surface Area (m^2/g)	$1126.00^c \pm 4.04$	$928.00^a \pm 2.51$	$1027.00^b \pm 37.15$

CSC is Coconut Shell Granular Activated Carbon, PKC is Palm Kernel Shell Granular Activated Carbon, CCPC is Combined Coconut and Palm Kernel Shell Activated Carbon. Values are means and standard deviations of triplicate experiments. Means in the same row with same superscript are not significantly different at P=0.05.

Table 3. *Brewery wastewater Characteristics before Treatment with GACs.*

S/No	Characteristics	Replicate 1	Replicate 2	Mean
1	COD (mg/l)	315.00	320.00	318.00
2	TSS (mg/l)	68.50	69.50	69.00
3	TDS (mg/l)	185.00	186.00	185.50
4	Phosphates (mg/l)	6.90	7.10	7.00
5	Ammonium ion (mg/l)	3.80	3.80	3.80
6	pH	6.80	6.80	6.80

Ambient Temperature = 31.0°C and Wastewater Temperature =35.0°C

This can be attributed to the pore structure and specific surface area of the various carbons, as those with higher specific surface areas tend to adsorb more pollutant than those with small specific surface areas [11]. The poor removal of pollutants by adsorption is usually as a result of their low initial concentrations in the wastewater before treatment with the carbons. The pollutants with higher concentrations tend to diffuse faster into the available adsorption site in the carbons as compared to those with low initial concentrations [16].

The general trend shows that the pollutant removal efficiency increased with increase in carbon dosage for each of the experimental carbons used. Among all the carbons studied, only CSC reduced COD concentration below the FEPA discharge limits, however TSS and TDS were both reduced to acceptable FEPA limits [17] by all the experimental carbons at a dosage of 8g/100ml. There was significant difference between treatments (experimental carbons) and (dosage) at p=0.05, for the removal of COD, TSS, and TDS.

3.3.2. Effects of Contact Time on Removal Efficiency of Pollutants

The effects of contact time between the wastewater and the experimental carbons on the removal efficiencies of COD, TSS and TDS were studied for all the three activated carbons (Figures 7-9). Contact times of 0-120 minutes, at successive intervals of 30 minutes were used in this study. From the tables, CSC exhibited the highest removal efficiencies of 78.56, 100 and 93.5%, after a contact time of 120 minutes for COD, TSS and TDS, respectively. The removal efficiencies of CCPC were close to those of CSC at the same contact time with values of 71.82, 100.0 and 88.25%, respectively. PKC, however showed the least removal efficiencies for all pollutants studied, with values for COD, TSS and TDS after a contact time of 120 minutes being, 66.48, 73.19 and 69.37%, respectively.

The general trend for each experimental carbon and pollutant studied shows that the removal efficiencies increased with contact time, throughout the period of the experiment. This is in agreement with the findings of [8-10] who reported a similar trend for the adsorption of pollutants in industrial effluents.

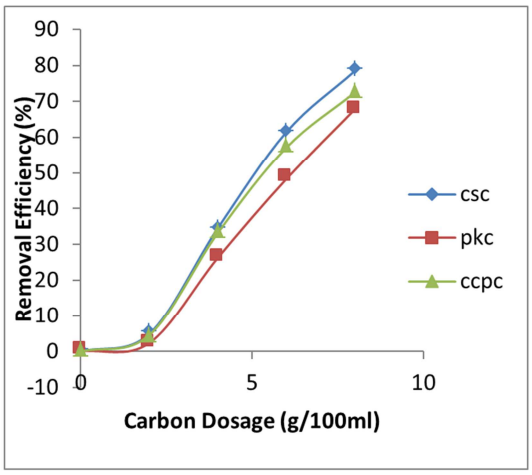

Figure 4. *Effect of carbon dosage on COD removal efficiency.*

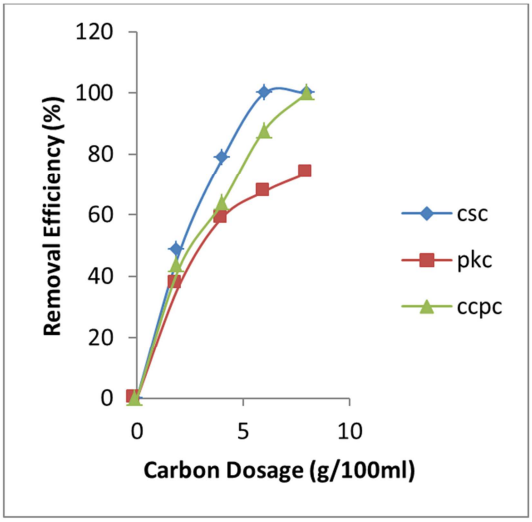

Figure 5. *Effect of carbon dosage on TSS removal efficiency.*

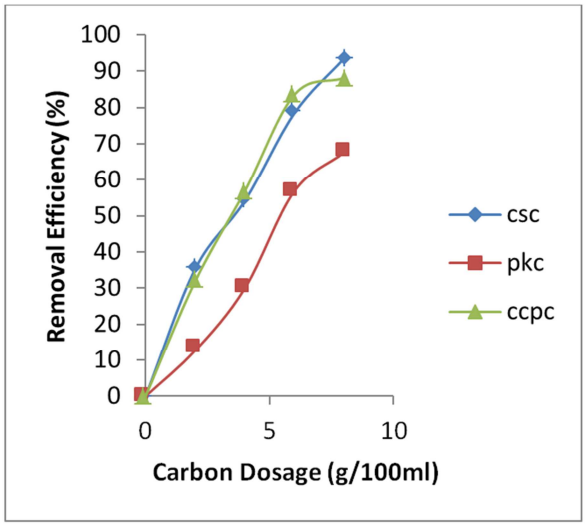

Figure 6. *Effect of carbon dosage on TDS removal efficiency.*

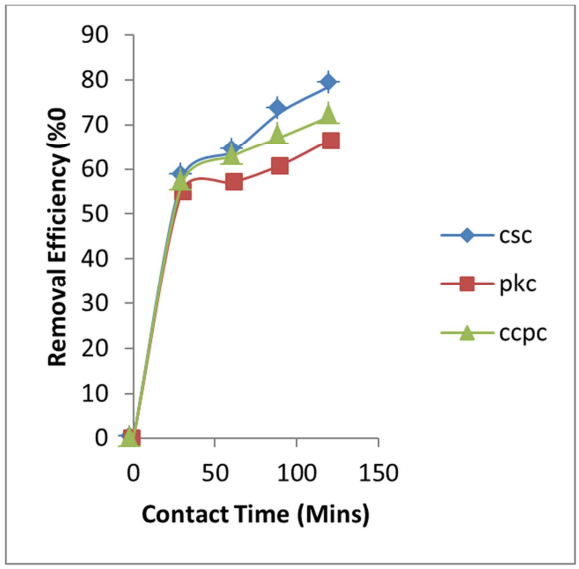

Figure 7. *Effects of Contact Time on COD Removal Efficiency.*

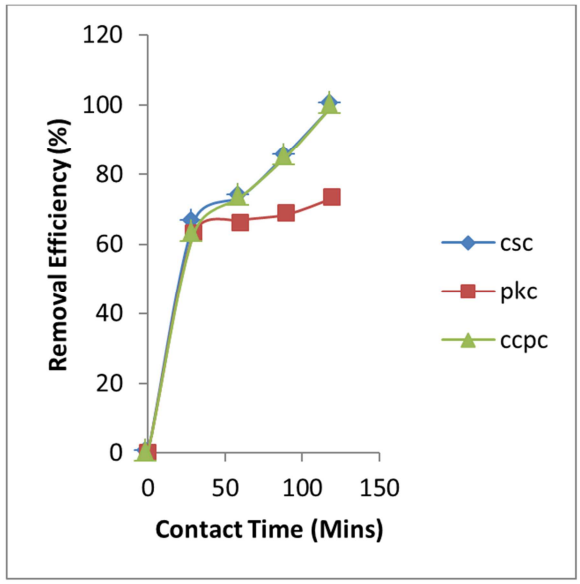

Figure 8. *Effects of Contact Time on TSS Removal Efficiency.*

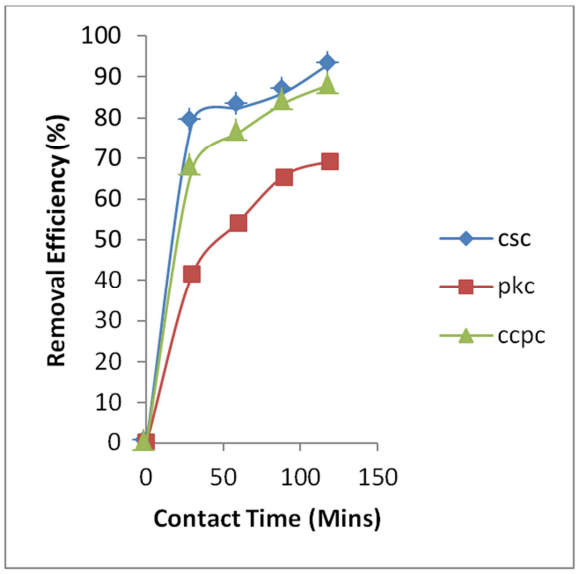

Figure 9. *Effects of Contact Time on TDS Removal Efficiency.*

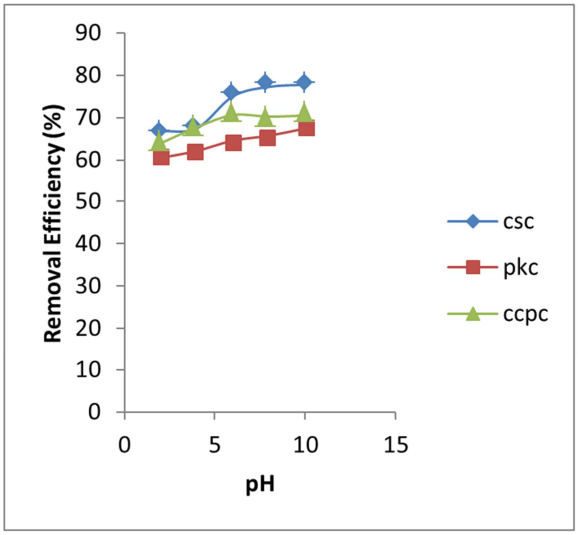

Figure 10. *Effects of pH on COD Removal Efficiency.*

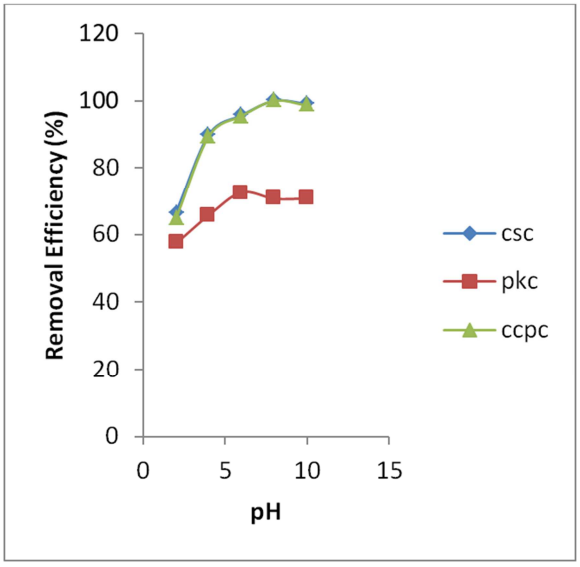

Figure 11. *Effects of pH on TSS Removal Efficiency.*

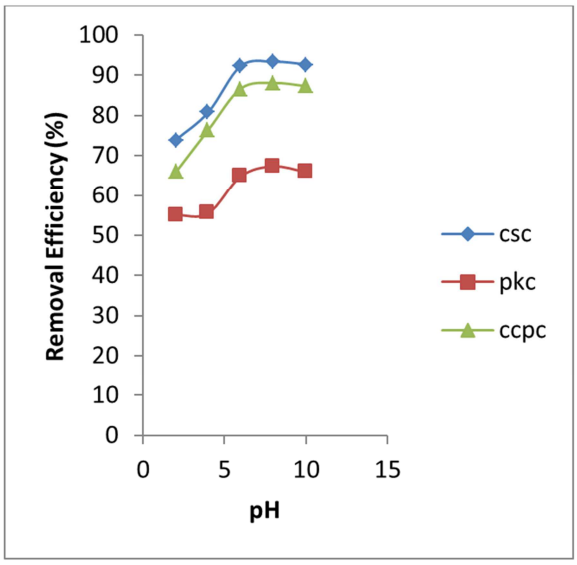

Figure 12. *Effects of pH on TDS Removal Efficiency.*

Among the three carbons studied, only CSC reduced COD concentrations below the acceptable FEPA limits of 80mg/l after a contact time of 120 minutes. TSS and TDS were however, reduced below the FEPA set limits by each of the experimental carbons after a contact period of 120 minutes. Statistical analysis showed that the mean removal efficiencies of all the pollutants were significantly different between treatments at $p = 0.05$.

3.3.3. Effects of pH on the Removal Efficiency of Pollutants

The effect of pH on adsorption of pollutants in pre-treated brewery effluents by locally produced adsorbents was studied within a pH range of 2 – 10 covering both the acidic and the alkaline ranges. Figures 10, 11, and 12 show the results obtained. From the Tables, it was observed that the general trend indicated that removal efficiencies were higher in the alkaline (higher) pH range than in the acidic (lower) range, for all pollutants studied and for all experimental carbons used. CSC exhibited better removal efficiencies at a pH of 8 for all parameters and was closely followed by CCPC at the same pH, while PKC exhibited the lowest removal efficiencies at all the pH ranges considered. This finding is in line with those of [18] who noted that adsorption of lead on modified palm kernel shell activated carbon was highly dependent on the pH of the source water. Statistics show that there was significant difference in means between treatments (activated carbons) and (pH) for all the pollutants studied.

4. Conclusion

Three different granular Activated carbons (GACs) were produced and characterized. These were; CSC (Coconut based), PKC (Palm kernel based) and CCPC (coconut and palm kernel based). From the characteristics determined, it was observed that CSC had the highest ash content, specific surface area and iodine sorption and was closely followed by CCPC, while PKC had the lowest values of the mentioned

characteristics. CCPC had the highest methylene blue number, followed by CSC and then PKC. As for bulk density, PKC had the highest value and was followed closely by CCPC, then CSC. Carbon yield was higher in CCPC, followed by PKC, then CSC. There was significant difference in mean characteristics between the three carbons considered at $p = 0.05$.

The experimentally produced carbons were used to remove the residual pollutants, through the use of batch equilibrium/ adsorption experiments. The effects of carbon dosage, contact time and pH on the removal efficiencies of the pollutants were investigated.

Results show that adsorption increased with increasing carbon dosage for all the activated carbons and pollutants studied. The highest removal efficiencies were recorded at a dosage of 8g/100ml for all the carbons and pollutants. In all cases, the COD, TSS and TDS were reduced to acceptable discharge limits at the highest dosage of 8g/100ml.

Removal efficiencies also increased with increase in contact time and were highest at a contact time of 120 minutes for all the carbons used and pollutants treated.

The experimental results also revealed that, best removal efficiencies were more favoured in the alkaline pH ranges than in the acidic pH ranges. The highest removal efficiencies were recorded at a pH of 8 for all the carbons and pollutants. There was significant difference in means between the carbons and within all treatments levels, for all pollutants treated at $p = 0.05$.

Thus, it could be concluded that, CSC and CCPC are better adsorbents for the removal of COD, TSS and TDS in terms of adsorption capacity and intensity, while PKC is not suitable for complete or acceptable removal of the studied pollutants in pre-treated brewery effluents.

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