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Utilization of Processed Activated Carbon from Coconut Shell in Wastewater Treatment

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Abstract

Activated carbons were prepared from a variety of carbonaceous raw materials such as coal, coconut shells, wood and lignite. The present research work is emphasized on the investigation of carbonization process of coconut shell. Firstly, physico-chemical properties of coconut shell and distillery wastewater were determined. The effects of carbonization temperature and time on the properties of prepared coconut shell activated carbon were investigated. The nature of prepared coconut shell activated carbon was analyzed by X-ray Diffraction (XRD) method. The characteristics of prepared coconut shell activated carbons such as moisture content, ash content, volatile matter content, bulk density and fixed carbon content were also determined. Then, the prepared coconut shell activated carbon and commercial activated carbon were utilized for treatment of wastewater from a distillery plant. The characteristics of distillery wastewater before and after treatment with prepared coconut shell activated carbon as well as commercial activated carbon were determined. The effects of important parameters such as temperature, initial pH, activated carbon (adsorbent) dose and contact time on the removal of total chemical oxygen demand (COD) in distillery wastewater were also investigated.

Key words: Carbonization, adsorbent, activated carbon, XRD method

Introduction

In most of the coastal cities and towns *Cocos-nucifera* commonly known as coconut trees. In some places the green nuts are dried and the fibres are removed for various purposes like rope making, floor mats or rubberized coir mattresses, etc. However, the shells are not much used other than for use as fuel. Coconut shells are made up of "stone cells" and are hard, porous, impregnated with lignin, tannin and a little oil. It is felt that

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the shells can be used for producing of an adsorbent for treating distillery spent wash using adsorption processes (Patil, *et al*, 2007).

Activated carbon (AC) is black, amorphous solid containing major portion of fixed carbon content and other materials such as ash, water vapour and volatile matter in small percentage (http://www. benefits of activated carbon.com). Carbonization is generally accomplished by heating the source material usually in the absence or limited amount of air to a temperature sufficiently high to dry and volatilise substances in the carbonaceous material. Coconut shell contains about 65-75% volatile matter and moisture which are removed largely during the carbonization process (Gimba and Muyiwa, 2008).

Industrial wastes may be defined briefly as the wastewaters that resulted from industrial processing operations. Pollution of water and soil by organic and inorganic chemicals are of serious environmental concern. As the number of process industries increased, there was a corresponding increase in the discharge of pollutional wastewaters contaminated with acids or alkalies, chemicals, floating and suspended solids, silt, oils and grease, odours, colours, and putrescible organic matter (http://www. industrial wastewater.com). The aim of this research work was to obtain the effective treatment of industrial effluent with activated carbon as adsorbent that synthesized by carbonization of coconut shells.

Materials and Methods

Materials

In this research work, wastewater was collected from Royal distillery plant, No.270, Mahawgani Street, Shwe Pyi Thar Township, Yangon Region. Coconut Shells were collected from Kamayut Ywa-ma Monastery, Hlaing Township, Yangon Region. 95% Sulphuric acid, sodium hydroxide, potassium dichromate, ferrous ammonium sulphate (FAS), ferrion indicator, hydrochloric acid, silver sulphate and mercury (II) sulphate (analar grade, British Drug House Co. Ltd., England) were used.

Methods

Preliminary Preparation and Characterization of Coconut Shell

The coconut shell was washed with water to remove dust and fiber and then sun dried at 32-35 ºC for 24 hours. The dried coconut shell was cut into small pieces (5 mm size) and stored. The physico-chemical characteristics such as moisture content, ash content, volatile matter

content, bulk density and fixed carbon content of coconut shell were also determined.

Preparation of Coconut Shell Activated Carbons

For uniform carbonization, (10) g of cleaned and dried coconut shell (5mm size) was soaked in (10) mL 95% sulphuric acid for 5 minutes. The treated coconut shell was placed in the porcelain crucible and covered. It was carbonized in the Electric Muffle Furnace (LEF-105S), at 300ºC for 120 minutes. Then, the carbonized carbons were rinsed with distilled water to obtain neutral condition. Rinsed activated carbons were dried in an oven at 105ºC for 10 minutes. The dried coconut shell activated carbons were stored in desiccator at room temperature for further studies.

Effects of Carbonization Temperature and Time on the Quality of Coconut Shell Activated Carbons

The coconut shell activated carbons were prepared according to the above procedure using the various temperature ranging from 300ºC to 600ºC for 120 minutes and various times ranging from 60 minutes to 150 minutes at 400ºC. The appearances of prepared coconut shell activated carbons were observed and recorded.

Characterization of Prepared Coconut Shell Activated Carbons and Commercial Activated Carbons

The properties of prepared coconut shell activated carbons and commercial activated carbons such as moisture content, ash content, volatile matter content, bulk density and fixed carbon content were determined by American Society for Testing of Materials (ASTM 2854-70) method. The nature of prepared coconut shell activated carbons and commercial activated carbons were characterized by X-ray Diffraction (XRD) analysis. The X-ray diffractogram of prepared coconut shell activated carbons and commercial activated carbons were also compared.

Characterization of Distillery Wastewater

The physico-chemical characteristics such as temperature, colour, turbidity, pH, total solids, total suspended solids and total chemical oxygen demand (COD) of distillery wastewater were determined by colourimetric method using digit logging colourimeter (HACH-DR/890) and COD digester (HACH-DRB200). The analyses data were recorded.

Treatment of Distillery Wastewater by using Prepared Coconut Shell Activated Carbons and Commercial activated carbons

Adsorption properties of prepared activated carbon and commercial activated carbon were conducted using the parameters like temperature, initial pH, adsorbent dose and contact time on the adsorptive removal of total chemical oxygen demands in distillery wastewater.

Variation of Temperature of Wastewater

(50) mL of distillery wastewater of known initial pH and (1) g of prepared activated carbon from coconut shell were placed into a (100) mL beaker. The mixture was stirred with temperature controlled magnetic stirrer at a speed of 90 rpm. Batch experiments were conducted using various temperatures ranging from 30ºC to 70ºC for 30 minutes. After that, the treated distillery wastewater samples were withdrawn and analyzed for total chemical oxygen demand and the results are recorded.

Variation of Initial pH of Wastewater

(50) mL of distillery wastewater and (1) g of prepared activated carbon from coconut shell were placed into a (100) mL beaker. This mixture was stirred with temperature controlled magnetic stirrer at a constant speed of 90 rpm. Batch experiments were conducted using various initial pH ranging from 3 to 8 of wastewater sample at 30ºC for 30 minutes. The various pH range of distillery wastewater was adjusted by the addition of 0.1 N NaOH and 0.1 N HCl solution. The treated distillery wastewater samples were withdrawn and analyzed for total chemical oxygen demand and the results are recorded.

Variation of Adsorbent Dose

(50) mL of distillery wastewater (initial pH 5) and various amounts ranging from (1) g to (5) g of coconut shell activated carbon were placed into the (100) mL of beaker. This mixture was stirred with temperature controlled magnetic stirrer at a constant speed of 90 rpm. Batch experiments were conducted with various amount of activated carbon ranging from (1) g to (5) g at 30ºC for 30 minutes. The treated distillery wastewater samples were withdrawn and analyzed for total chemical oxygen demand and the results are recorded.

Variation of Contact Time between the Adsorbent and Wastewater

(50) mL of distillery wastewater (pH 5) and (4) g of prepared activated carbon from coconut shell were placed into a (100) mL beaker. This mixture was stirred with temperature controlled magnetic stirrer at a constant speed of 90 rpm. Batch experiments were conducted at 30ºC for various contact times ranging from 30 minutes to 120 minutes. The treated distillery wastewater samples were withdrawn and analyzed for total chemical oxygen demand and the results are recorded.

Results and Discussion

In this research work, the physico-chemical characteristics of coconut shells were determined. According to the results in Table (1), it was found that 8.81%w/w moisture content, 0.79 %w/w ash, 52.20 %w/w volatile matter, 0.64 kg m^{-3} bulk density and 38.20 %w/w fixed carbon contents present in coconut shell. In the preparation of coconut shell activated carbon, the effects of carbonization temperature and time on the appearance of prepared coconut shell activated carbons were determined. The results in Tables (2) and (3) show that increase the carbonization temperature and time gave the ash occurred on the outer surface of carbonized coconut shells. And then, lower carbonization temperature and time gave the incomplete carbonization of coconut shells. Therefore, 400ºC and 90 minutes for carbonization indicated that the bright black colour and good appearance of coconut shell activated carbons.

According to the results in Table (4) and Figure (1), it can be seen that the characteristics of prepared coconut shell activated carbon were closely the same as the commercial activated carbon. The nature of coconut shell activated carbons and commercial activated carbon possess graphitic character and low content of inorganic constituents. The physico-chemical characteristics of distillery wastewater are 29900 (Pt-Co) colour, 5050 FAU turbidity, 6.01 pH at 29.8ºC, 4000 mg/L total solids, 3950 mg/L total suspended solids and 29550 mg/L total chemical oxygen demand, as shown in Table (5).

The activated carbons were used for removal of total chemical oxygen demand in distillery wastewater. The results in Table (6) and Figure (2) indicate that an increasing the temperature, the percent removal efficiency of total chemical oxygen demand (COD) in distillery wastewater was decreased. It was observed that the COD removal rates by prepared

coconut shell activated carbons are higher than the commercial activated carbons. From the results in Table (7) and Figure (3), it can be observed that the COD removal efficiency from distillery wastewater increases as pH increased from 3 to 5. Thereafter, the COD removal efficiency decreases with further increase in pH from 5 to 8.

From the results in Table (8) and Figure (4), it was observed that the percentage of COD removal efficiency increases with increase in the adsorbent dose from 1 to 4. Thereafter, the COD removal efficiency slows down with further increase in adsorbent dose. The rate of adsorption increases with increase in adsorbent dosage because of increase in surface area of the adsorbent. The results in Table (9) and Figure (5) indicate that the percentage of COD removal efficiency increases with increase in contact time from 30 minutes to 70 minutes. Thereafter, the rate of adsorption increases with increase in contact time and sometimes it remains constant due to equilibrium condition.

Table (1) Physico-chemical Characteristics of Coconut Shell

Sr. No.	Properties	Experimental Value	Literature Value*
1	Moisture content, %w/w	8.81	8-9
2	Ash content, %w/w	0.79	0.5-0.7
3	Volatile matter content, %w/w	52.20	53-50
4	Bulk density, kg/m^3	0.64	0.55-0.8
5	Fixed carbon content, %w/w	38.20	40-45

* Nurul'Ain Binti Jabit, 2007

Table (2) Effect of Carbonization Temperature on the Appearance of Prepared Coconut Shell Activated Carbon

Time for 95% H_2SO_4 treatment = 5 minutes

Sample No.	Coconut Shells (g)	H_2SO_4 (Conc:) (mL)	Carbonization		Appearance of Activated Carbons (Texture)
			Temp. (ºC)	Time (min)	
1	10	10	300	120	Outer surface soft, black colour and interior hard
2	10	10	400*	120	Soft, porous surface and bright black colour
3	10	10	500	120	Soft, porous surface covered with ash layer
4	10	10	600	120	Soft, porous surface covered with ash layer

* The most suitable temperature

Table (3) Effect of Carbonization Time on the Appearance of Prepared Coconut Shell Activated Carbon

Time for 95% H_2SO_4 treatment = 5 minutes

Sample No.	Coconut Shells (g)	H_2SO_4 (conc:) (g)	Carbonization		Appearance of Activated Carbons (Texture)
			Temp. (ºC)	Time (min)	
1	10	10	400	60	Outer surface soft ,black colour and interior hard
2	10	10	400	90*	Soft, porous surface and bright black colour
3	10	10	400	120	Soft, porous surface and bright black colour
4	10	10	400	150	Soft, porous surface covered with ash layer

* The most suitable time

Table (4) Physico-chemical Characteristics of Prepared Coconut Shell Activate Carbon and Commercial Activated Carbon

Sr. No.	Properties	Activated Carbon	
		Coconut Shell	Commercial (made in England)
1	Moisture content, %w/w	2.75	3.68
2	Ash content, %w/w	10.39	19.60
3	Volatile matter content, %w/w	5.33	4.37
4	Bulk density, kg/m^3	0.64	0.3
5	Fixed carbon content, %w/w	81.53	72.35

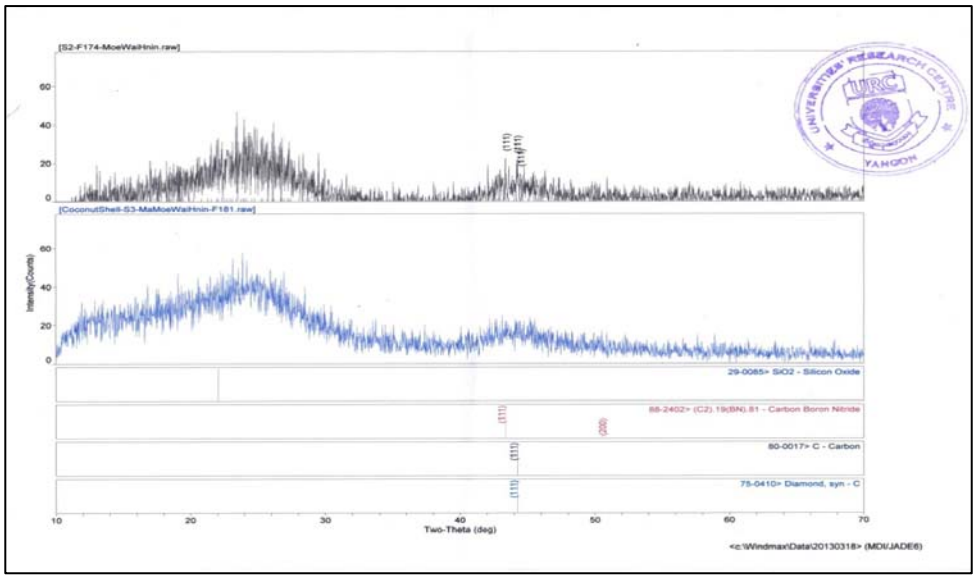

Figure (1) X-ray Diffractogram of Commercial Activated Carbon and Prepared Coconut Shell Activated Carbon

Table (5) Physico-chemical Characteristics of Distillery Wastewater

Sr. No.	Characteristics	Experimental Value
1	Temperature, ºC	29.8
2	Total solids, (mg/L)	4000
3	Total suspended solids, (mg/L)	3950
4	pH	6.01
5	Colour, (Pt-Co)	29900
6	Turbidity, (FAU)	5050
7	Chemical oxygen demand (COD), (mg/L)	29550

Table (6) Effect of Temperature on the Removal of Total Chemical Oxygen Demand in Distillery Wastewater by Coconut Shell Activated Carbon

Initial COD in wastewater = 29550 mg/L
pH of wastewater = 6
Stirring speed = 90 rpm
Contact time = 30 min

Sample No.	Volume of Wastewater (mL)	Activated Carbon Dose (g)	Temp. (ºC)	Residual COD (mg/L)		COD Removal (%)	
				CSAC	CAC	CSAC	CAC
1	50	1	30*	18109	12138	72.5	58.9
2	50	1	40	16575	20355	43.9	31.1
3	50	1	50	18750	21178	36.5	28.3
4	50	1	60	20875	21985	29.3	25.6
5	50	1	70	21536	22730	27.1	23.1

* The most suitable temperature

Note: COD = Chemical oxygen demand
CSAC = Coconut shell activated carbon
CAC = Commercial activated carbon

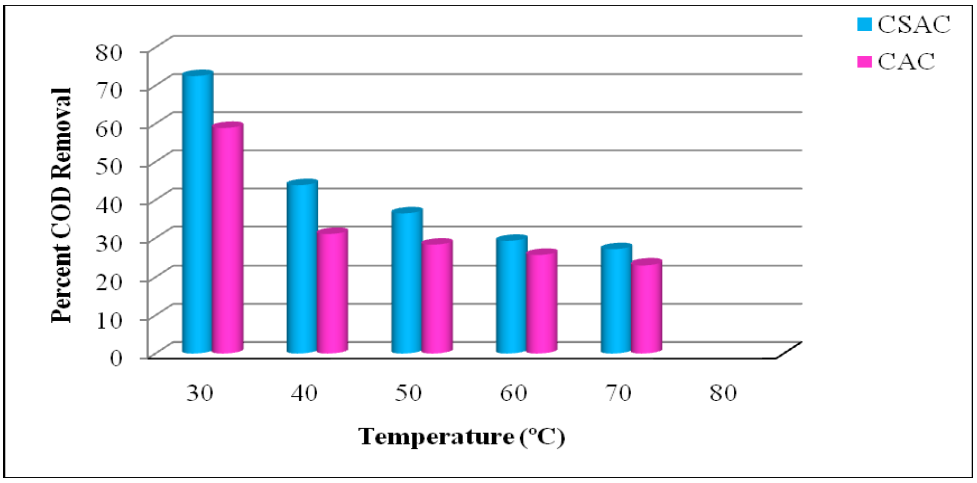

Figure (2) Percent Removal Efficiency of Chemical Oxygen Demand in Distillery Wastewater by Activated Carbons at Different Temperatures

Table (7) Effect of pH on the Removal of Total Chemical Oxygen Demand in Distillery Wastewater by Coconut Shell Activated Carbon

Initial COD in wastewater = 29550 mg/L
Temperature of wastewater = 30º C
Agitation speed = 90 rpm
Contact time = 30 min

Sample No.	Volume of Wastewater (mL)	Activated Carbon Dose (g)	Initial pH	Residual COD (mg/L)		COD Removal (%)	
				CSAC	CAC	CSAC	CAC
1	50	1	3	12505	14215	57.6	50.8
2	50	1	4	8372	10976	71.6	62.8
3	50	1	5*	7130	9255	75.8	68.6
4	50	1	6	8109	12138	72.5	58.9
5	50	1	7	13073	14860	55.7	49.7
6	50	1	8	15218	18375	48.5	37.8

* The most suitable pH

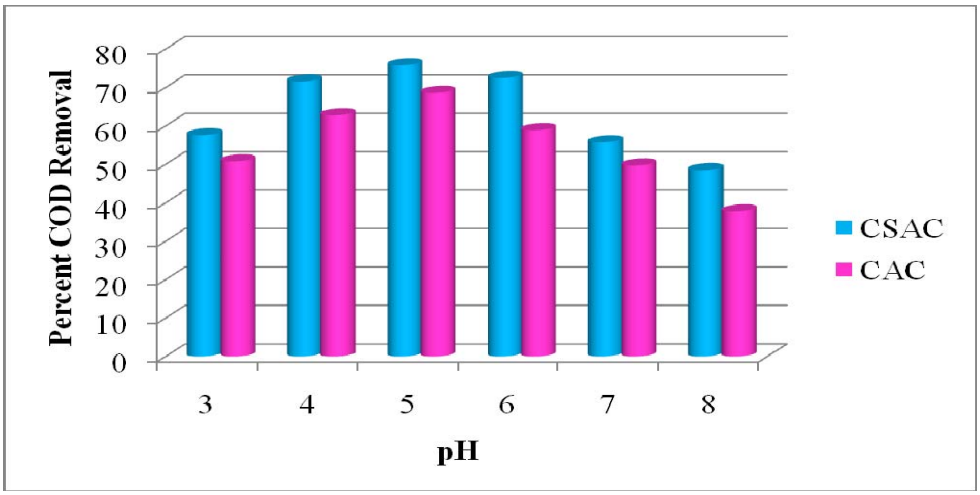

Figure (3) Percent Removal Efficiency of Chemical Oxygen Demand in Distillery Wastewater by Activated Carbons at Different pH

Table (8) Effect of Adsorbent Dose (Coconut Shell Activated Carbon) on the Removal of Total Chemical Oxygen Demand in Distillery Wastewater

Initial of COD in wastewater = 29550 mg/L
Heating temperature = 30º C
Agitation speed = 90 rpm
Contact time = 30 min

Sample No.	Volume of Wastewater (mL)	pH	Activated Carbon Dose (g)	Residual COD (mg/L)		COD Removal (%)	
				CSAC	CAC	CSAC	CAC
1	50	5	1	7130	9255	75.8	68.6
2	50	5	2	6348	8116	78.5	72.5
3	50	5	3	5435	7123	81.6	75.8
4	50	5	4*	4873	6319	83.5	78.6
5	50	5	5	4670	5820	84.1	80.3

* The most suitable amount of adsorbent dose

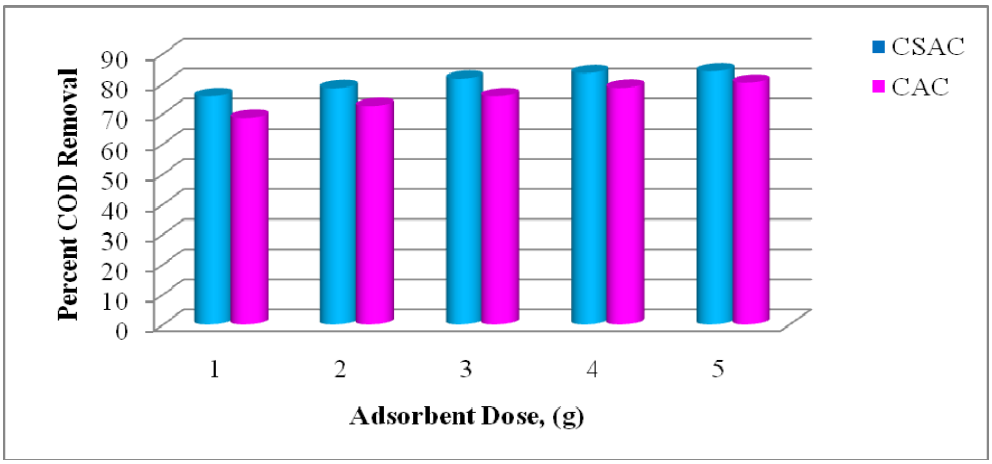

Figure (4) Percent Removal Efficiency of Chemical Oxygen Demand in Distillery Wastewater by using Different Adsorbent Doses

Table (9) Effect of Contact Time on the Removal of Total Chemical Oxygen Demand in Distillery Wastewater by Coconut Shell Activated Carbon

Initial of COD in wastewater = 29550 mg/L
Heating temperature = 30ºC
Agitation Speed = 90 rpm
Initial pH = 5

Sample No.	Volume of Wastewater (mL)	Activated Carbon Dose (g)	Contact Time (min)	Residual COD (mg/L)		COD Removal (%)	
				CSAC	CAC	CSAC	CAC
1	50	4	30	4873	6319	83.5	78.6
2	50	4	50	4556	5379	84.5	81.7
3	50	4	70*	3385	4632	88.5	84.3
4	50	4	90	3090	4478	89.5	84.8
5	50	4	120	3019	4329	89.7	85.3

* The most suitable contact time

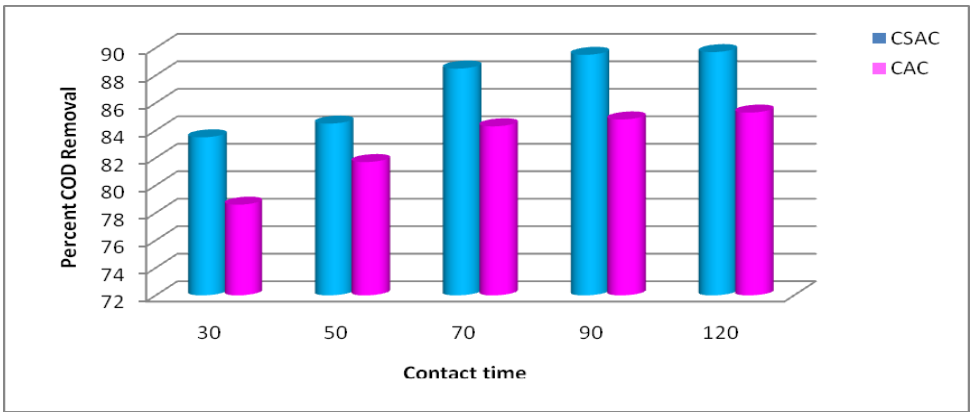

Figure (5) Percent Removal Efficiency of Chemical Oxygen Demand in Distillery Wastewater at Different Contact Times

Figure (6) Prepared Coconut Shell Activated Carbon

Figure (7) Commercial Activated Carbon (Made in England)

Conclusions

In this research work, the coconut shell activated carbons were prepared from acid treated coconut shells by carbonization method. The results indicated that acid treated coconut shells could produce activated carbons of high adsorptive capacity comparable with that of commercial product. Effective and full carbonization of coconut shells is found to be 400ºC for 90 minutes. At the temperature lower than the above condition, there is an inefficient carbonization due to incomplete removal of volatile products resulting in pore blockade and less adsorptive capacity. And also at temperature higher than 400º C for 90 minutes the carbonized products are generally reduced in activity due to excessive or post-carbonization process. The activated carbons were used for removal of total chemical oxygen demand (COD) in distillery wastewater. According to the collected results, it was found that an increasing the temperature, the percent COD removal efficiency of distillery wastewater was decreased whereas the percent COD removal efficiency of distillery wastewater was increased with the increased in pH, adsorbent dose and contact time of wastewater.

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