Treatment of Industrial Wastewater for Removal of Organic Micro-pollutants Using Processed Activated Carbon from Some Agricultural Wastes

Thwe Linn Ko, Khin Thet Ni and Aye Aye Mar*
Department of Industrial Chemistry, University of Yangon

Abstract

The present research work was to study the effect of processed activated carbons in wastewater treatment. Environmental friendly activated carbons from corn-cob, rice straw and sugar cane bagasse were processed by chemical activating method. The highest fixed carbon content 71.5%, 63.1% and 64.43% were obtained for corn-cob activated carbon, rice straw activated carbon and sugar cane bagasse activated carbon. Carbonization temperature at 300°C for 120 min gave the highest fixed carbon contents for processed activated carbons. Yield percentages were between 25% to 28%.. The largest surface area, 1503 m^2/g was obtained from corn-cob activated carbon with the highest iodine sorption capacity 63.6% and methylene blue number 348mg/g. Distillery effluent from Royal Distillery Plant, Shwe Pyi Thar Township,YangonRegion was subjected to treat with processed activated carbons. The physico-chemical properties such as pH, total solids, suspended solids, dissolved solids, color, turbidity, chemical oxygen demand and biological oxygen demand (BOD) of wastewater before and after treatment were determined. In batch type treatment, effect of reduction of chemical oxygen demand (COD) upon temperature, dosage of activated carbons and contact time in the treatment of distillery effluent were studied. The highest removal efficiency of processed activated carbons for COD and BOD were 94.7% and 83.86% by using 0.07:1 ratio of activated carbon to distillery effluent at 30°C for 120 min.

Keywords: activated carbon, carbonization, distillery effluent,fixed carbon content, removal efficiency

1. Introduction

Among various adsorbents used, activated carbon (AC) is well known for its high adsorption capacity due to large surface area and pore volume. In recent years, immerse research has been focused on converting the agricultural or lignocellulosic wastes into activated carbon, since this technology not only solve the waste disposal problem but also convert a potential waste into a valuable product.Activated carbon can be produced from any carbonaceous materials, both naturally occurring and synthetic. Process economics dictates the selection of readily available and cheaper precursors for the production of commercial Acs. Biomass precursors offer most economical service because they are renewable with low mineral content and appreciable hardness (John, W.H., 1974).

This research has been mainly organized in four parts. Partone is an introductory. Part two includes all the materials and methods involved in the work. Part three contains the results and discussion of conducted research work and the last part is conclusion. AC was prepared from agricultural wastes by using chemical activating agents like 25% calcium chloride solution.Influence of carbonization temperature and holding time on the characteristics of AC was investigated. The prepared samples were characterized by physico-chemical characteristics and adsorption capacity. The prepared ACs were used to treat the distillery effluent of a wide range of process parameters. Batch type treatment method was conducted to postulate the micro pollutant removal effectiveness of processed activated carbons. Influence of operating parameters such as adsorbent dose, contact time and

*Aye Aye Mar, Department of Industrial Chemistry, University of Yangon

temperature on the rate of adsorption was explored. Removal efficiency of among various processed activated carbons were comparatively studied.The overall objective of this study is to prepare activated carbon from lignocellulosic waste materials for the efficient removal of organic pollutants from distillery effluents.

2. Materials and Methods

Materials

Corn-cob, rice straw and sugar cane bagasse were collected from Hlaing Bazzar, Hlaing Township, Yangon Region. Rice straw was obtained from Myin Zu Village, Taungdwingyi Township, Magwe Region.

Distillery wastewater from Royal Distillery Plant situated in Shwe Pyi Thar Township, Yangon Region was collected to treat with processed activated carbon.

Calcium chloride (Analytical grade, England made) was purchased from Golden Lady chemical sale center, (28^{th}) Street, Pabedan Township, Yangon Region.

Preparation of raw materials

Selected agricultural wastes were washed with tap water and sun dried for 24 hr. The dried rice straw and sugar cane bagasse were cut into small pieces (1cm x 0.5 cm) but corn cobs were cut into (2.5 cm in diameter and 1cm in thickness).

Physico-chemical characteristics of dried raw materials

Physico-chemical characteristics such as moisture content, ash content, total solids content, volatile matter content, bulk density and fixed carbon content of dried raw materials were determined. (American Society for Testing and Materials Standard test methods for activated carbon, 1990, ASTM –D1762)

Preparation of activated carbon

Chemical activation of dried raw materials

5 g of cleaned and dried precursors were separately impregnated in 10 mL of 25% $CaCl_2$ solution and then soaked for overnight at room temperature. Then each of samples was sieved with a stainless steel sieve and put individually into the respective crucible and dried in a hot air oven at 105°C for 30 min.

Carbonization of chemically activated raw materials

5 g of chemically activated raw materials were carbonized in a muffle furnace at various temperatures 200°C, 250°C, 300°C, 350°C, 400°C, 450°C and 500°C for 60 min carbonization period and the most preferable carbonization temperature was chosen. To get the most suitable and economically reliable carbonization period, raw materials were again carbonized at chosen temperature for 30 min, 60 min, 90 min, 120 min, 150 min and 180 min for all three samples.

Washing of activated carbon

After carbonization, activated carbon was cooled down and washed with water for six times. Then it was kept on the tray for draining water and transferred into the oven at 110°C for 3 hr.

Determination of yield percentage of activated carbon

The yield of activated carbon was calculated on a chemical-free basis and can be regarded as an indicator of the process efficiency. It was calculated as the percentage weight of the resultant activated carbon divided by weight of dried precursor used.

Characterization of activated carbon

Physico-chemical characteristics of activated carbon

Physico-chemical characteristics of prepared activated carbons such as moisture, ash, volatile matter and fixed carbon content were determined.

Determination of surface area, porosity and pH of activated carbon

Surface area, iodine sorption capacity, methylene blue number and pH of processed AC-$CaCl_2$ at their most suitable conditions were determined.

Physico-chemical characteristics of wastewater

The water sample was taken to the laboratory within 3 hr and physico-chemical characteristics such as chemical oxygen demand (COD), biological oxygen demand (BOD), pH, colour, turbidity, total solids, dissolved solids and suspended solids were determined.

Treatment of wastewater by using processed activated carbon

Adsorption properties of processed activated carbons were conducted using the parameters like temperature, adsorbent dose and contact time on the adsorptive removal of chemical oxygen demand (COD) in distillery effluent.

Effect of temperature on the removal of chemical oxygen demand

100 mL of wastewater and 1g of processed activated carbon were placed into a beaker and agitated at a constant speed of 100 rpm. Batch experiments were conducted using various temperatures ranging 30°C to 90°C for 30 min contact time. After 30 min contact time, the treated samples were analyzed for chemical oxygen demand.

Effect of adsorbent dose on the removal of chemical oxygen demand

For the optimum amount of adsorbent dose, the different ratios of wastewater to adsorbent mixture were used. The mixture was agitated at a constant speed of 100 rpm at 30°C for 30 min and analyzed for chemical oxygen demand.

Effect of contact time on the removal of chemical oxygen demand

The mixture which contained 0.07:1 ratio of adsorbent to wastewater was agitated at a constant speed of 100 rpm. Batch experiments were conducted at 30°C for various contact time (30 min, 60 min, 90 min, 120 min and 150 min) and analyzed for chemical oxygen demand.

Physico-chemical characteristics removal percentage of treated wastewater

Physico-chemical characteristics of treated wastewater such as chemical oxygen demand (COD), biological oxygen demand (BOD), pH, colour, turbidity, total solids, dissolved solids and suspended solids and their removal percentage at the most suitable treatment condition were determined.

3. Results and Discussion

The characteristics of activated carbon mainly depend upon the physic-chemical properties of raw material. As described in Table (1) that the fixed carbon content of corn-cob, rice straw and sugarcane bagasse are 70.78%, 59.3% and 64.24% respectively. The fixed carbon content of these materials was rather high. The high fixed carbon content and low ash content indicate that these materials were suitable sources for preparation of activated carbon. Volatile matter content of corn-cob was 0.97%, rice straw and sugarcane bagasse had 9.9% and 0.98%. The bulk densities were 0.19 kg/m^3, 0.01 kg/m^3 and 0.11 kg/m^3. The bulk density values can vary with raw material sources. Raw materials for activated carbon preparation should have high bulk density and sufficient volatile matter content. The selected agricultural wastes can be available in abundantly throughout the whole season in our country. Thus these agricultural wastes were reasonable and effective for preparation of activated carbon.

Effect of carbonization temperature on the physico-chemical properties of activated carbon by using impregnation agent, $CaCl_2$are described in Tables (2), (3) and (4). Fixed carbon content increased with increase of carbonization temperature up to certain value and then it was decreased. The highest fixed carbon content 67.5%, 61.3% and 60.43% were obtained for corn-cob activated carbon, rice straw activated carbon and sugarcane bagasse activated carbon respectively at 300 °C for 60 min. Carbonization temperature is one of the influencing factors for the development of porosity / good adsorbent during activation process.In all cases, the yield of activated carbons also decreased with increase of temperature. This is mainly due to the promotion of carbon burn – off and tar volatilization at higher temperatures.

Effects of carbonization time on the properties of prepared activated carbons from selected agricultural wastes are explored and data are shown in Tables (5),(6) and (7). The carbonization period at that final temperature was varied in between 30 min to 180 min. Fixed carbon content increased with increase of carbonization period up to certain value and then gradually decreased. Whereas ash content decreased with increase of carbonization time up to certain value and then increased for all precursors. The highest fixed carbon content 71.5%, 63.1% and 64.43% were observed for corn-cob, rice straw and bagasse respectively and thus the most suitable carbonization period was 120 min, at their most suitable carbonization temperature.The appearance and texture of all of activated carbon samples are brittle, bright in colour and light weight. For all cases, the percentage yield of activated carbon was decreased at longer carbonization period because of the higher fixed carbon content and changed to form as char. It could be attributed to the release of more volatiles by keeping the sample precursor for larger duration at final carbonization temperature.

Physico-chemical characteristics of processed activated carbon at their optimum preparation conditions are described in Table (8). It was observed that fixed carbon contents of processed activated carbons were high together with low ash and moisture content. Bulk density is used to determine the weight of a fixed volume of activated carbon. These bulk density values depend on the shape, size and density of the individual particles. The characteristics of activated carbon thus depend on the physico-chemical properties of raw material used. Due to chemical activation, activated carbon can be carbonized at lower temperature and shorter carbonization time.

Iodine sorption capacityand methylene blue number presented micro pore and meso pore content of activated carbon. The surface area and micro and meso pore content are related because these two determination procedures indicated that processed activated carbon which had more micro and meso pores, more surface area they had.

The physico-chemical characteristics of wastewater are described in Table (10). The temperature of the collected sample water was 60°C. The biochemical oxygen demand (BOD) was found 10840 ppm. PH of wastewater was 6.0 with higher suspended solids and rather high dissolved solids. The total solids contentin wastewater was 20696 mg/L.Color of the wastewater was deep yellowish. The odour of effluent was offensive. It may be due to derivative of molasses during distillation. Chemical oxygen demand (COD) was 19200 mg/L.

The effects of processed activated carbon on the percentage reduction of chemical oxygen demand are shown in Tables (11), (12) and (13). It was found that reduction values of COD concentrations were absolutely depended upon temperature, contact time between activated carbon and wastewater and it was also depended upon amount of activated carbon used. It was found that efficiency of activated carbon was related directly with temperature of effluent because removal percentage of COD was declined as temperature was increased. The most suitable temperature was 30°C and removal percentage was 73.4% for corn-cob AC, 69.79% for rice straw AC, and sugarcane bagasse ACwas 72.92%.

Treatment procedure was continued by increasing dosage of activated carbon at the temperature 30°C. It was observed that the more activated carbon used the more reduction of COD concentration to a certain limit. The most suitable activated carbon dosage to wastewater ratio found at 0.07:1. The reduction percent of COD concentrations were 90.9%, 89.94% and 90.52% for ACs-$CaCl_2$ of corn-cob, rice straw, and sugar cane bagasse.

Table 1. Physico–chemical properties of agricultural waste materials for preparation of activated carbon

Sr. No.	Physico-chemical Properties (w/w%)	Lignocellulosic Waste Materials		
		Corn cob	**Rice straw**	**Sugar cane bagasse**
1	Moisture content	26.05	23.00	34.83
2	Ash content	2.20	7.80	0.93
3	Volatile matter content	0.97	9.90	0.98
4	Fixed carbon content	70.78	59.30	64.24
5	Bulk density, (kg/m^3)	0.19	0.01	0.11

Table 2.Effect of carbonization temperature on the physico–chemical properties of activated carbon from corn-cob

Carbonization time = 1hr
Impregnation ratio of raw material and activating agent= 1: 2

Sr. No.	Carbonization temperature (°C)	Physico–chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Ash content (w/w%)	Volatile matter content (w/w%)	Fixed carbon content (w/w%)	
1	200	26.51	12.16	5.61	55.72	27.13
2	250	15.67	12.26	11.36	60.71	26.21
3	300*	13.94	14.95	3.61	67.50	26.10
4	350	12.50	15.65	7.50	66.35	24.14
5	400	13.46	16.56	4.89	67.09	22.12
6	450	14.55	16.78	5.60	63.07	20.19
7	500	15.29	16.79	6.54	61.38	18.90

*the most suitable carbonization temperature

Table3. Effect of carbonization temperature on the physico–chemical properties of activated carbon from rice straw

Carbonization time = 1hr

Impregnation ratio of raw material and activating agent= 1: 2

Sr. No.	Carbonization temperature (°C)	Physico–chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Ash content (w/w%)	Volatile matter content (w/w%)	Fixed carbon content (w/w%)	
1	200	12.30	8.84	20.54	58.32	26.78
2	250	11.00	9.85	19.08	60.07	24.58
3	300*	11.00	13.80	13.90	61.30	23.05
4	350	12.89	16.70	16.78	53.63	20.19
5	400	13.97	18.98	18.97	48.08	18.76
6	450	16.70	20.90	19.89	42.51	13.89
7	500	18.76	26.50	20.80	33.94	12.13

*the most suitable carbonization temperature

Table 4. Effect of carbonization temperature on the physico–chemical properties of activated carbon from sugar cane bagasse

Carbonization time = 1hr

Impregnation ratio of raw material and activating agent= 1: 2

Sr. No.	Carbonization temperature (°C)	Physico–chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Ash content (w/w%)	Volatile matter content (w/w%)	Fixed carbon content (w/w%)	
1	200	18.56	10.45	20.34	50.65	25.90
2	250	12.34	11.25	24.50	51.91	25.45
3	300*	11.93	12.98	13.66	61.43	24.60
4	350	6.78	19.07	13.45	60.7	19.76
5	400	8.94	23.67	12.34	55.05	17.09
6	450	9.90	26.78	23.45	39.87	12.80
7	500	12.34	28.90	4.50	54.26	10.76

*the most suitable carbonization temperature

Table 5. Effect of holding time on the physico–chemical properties of activated carbon from corn cob

Carbonization temperature = 300°C

Impregnation ratio of raw material and activating agent = 1: 2

Sr. No.	Holding time (min)	Physico–chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Ash content (w/w%)	Volatile matter content (w/w%)	Fixed carbon content (w/w%)	
1	30	16.57	13.67	4.56	65.2	27.54
2	60	13.94	14.95	3.61	67.50	26.10
3	90	14.56	14.99	7.67	68.78	25.99
4	120*	8.94	15.95	3.61	71.50	25.10
5	150	23.54	22.46	23.45	48.55	23.45
6	180	14.56	23.54	23.40	38.50	22.34

*the most suitable holding time

Table 6. Effect of holding time on the physico–chemical properties of activated carbon from rice straw

Carbonization temperature = 300°C

Impregnation ratio of raw material and activating agent = 1: 2

Sr. No.	Holding time (min)	Physico–chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Ash content (w/w%)	Volatile matter content (w/w%)	Fixed carbon content (w/w%)	
1	30	12.34	12.36	15.78	59.52	24.5
2	60	11.00	13.80	13.90	61.30	23.05
3	90	13.45	13.45	18.57	62.53	22.34
4	120*	9.00	14.56	13.34	63.10	21.23
5	150	7.65	17.89	14.56	59.90	15.67
6	180	6.74	20.76	13.45	59.05	12.34

*the most suitable holding time

Table 7. Effect of holding time on the physico–chemical properties of activated carbon from sugar cane bagasse

Carbonization temperature = 300°C

Impregnation ratio of raw material and activating agent= 1: 2

Sr. No.	Holding time (min)	Physico–chemical Properties				Yield percentage of activated carbon (w/w%)
		Moisture content (w/w%)	Ash content (w/w%)	Volatile matter content (w/w%)	Fixed carbon content (w/w%)	
1	30	12.43	11.45	14.33	61.79	25.13
2	60	11.93	12.98	12.66	62.43	24.60
3	90	10.13	13.00	13.67	63.20	23.99
4	120*	10.93	14.98	9.66	64.43	23.60
5	150	12.45	17.89	13.45	56.21	21.90
6	180	12.34	23.49	12.56	51.61	18.21

*the most suitable holding time

Table 8. Physico-chemical characteristics of prepared granular activated carbons carbonization temperature = 300°C

Carbonization time = 120 min

Sr. No.	Physico-chemical characteristics	Activated carbon			
		Corncob	Rice straw	Sugarcane bagasse	Literature value
1	Moisture content, (w/w%)	12.94	9.00	14,98	3-10 max*
2	Ash content, (w/w%)	11.95	14.56	9.66	$\leq$ 8 max*
3	Volatile matter content, (w/w%)	3.61	13.31	13.6	25*
4	Fixed carbon content, (w/w%)	71.50	63.10	64.43	3-90*
5	Bulk density, (kg/m^3)	0.16	0.01	0.07	0.50-0.56**
6	pH	6.29	6.29	6.29	1-8**

* = (http// www.pelagiaresearchlibrary.com)** = (Okibe, F.G., *et al.*, 2013)

Literature value of granular activated carbon was made from rice husk.

Table 9. Surface area and porosity of prepared activated carbons

Sr. No.	Parameter	Activated carbon			Literature value*
		Corn-cob	Rice straw	Sugarcane bagasse	
1	Surface area,(m^2/g)	1503	1433	1503	199-2105
2	Iodine sorption capacity, (w/w%)	63.60	54.50	59.0	76-84
3	Methylene blue number, (mg/g)	348	187	230	0-501

* = (Okibe, F.G., *et al.*, 2013)

Table 10.Characteristics of wastewater from royal distillery plant

Sr. No.	Parameters	Distillery Effluent
1	pH	6
2	Chemical oxygen demand (mg/L)	19200
3	Biochemical oxygen demand (ppm)	10840
4	Colour (Pt Co)	3500
5	Turbidity (NTU)	12200
6	Total solids (mg/L)	20696
7	Dissolved solids (mg/L)	846
8	Suspended solids (mg/L)	19850

Table 11. Effect of temperature on the removal of total chemical oxygen demand from distillery effluent by processed activated carbon

Initial concentration of COD in wastewater = 19200 mg/L
Contact time = 30 min
Activated carbon dosage to wastewater ratio = 0.01: 1

Sr. No.	Temperature (°C)	Residual COD_{Total} (mg/L)			COD Removal (%)		
		Corn-cob AC	Rice straw AC	Sugarcane bagasse AC	Corn-cob AC	Rice straw AC	Sugarcane bagasse AC
1	*30	5100	5800	5200	73.4	69.79	72.92
2	45	6800	6534	6530	64.5	65.9	65.99
3	60	12570	13580	16850	34.53	29.2	12.2
4	75	17000	16650	18650	11.45	13.3	2.86
5	90	18000	18840	19000	6.25	1.8	1.0

* The most suitable temperature

Table 12. Effect of adsorbent dosage on the removal of total chemical oxygendemand from distillery effluent by processed activated carbon

Initial concentration of COD in wastewater = 19200 mg/L
Temperature of wastewater = 30°C
Contact time = 30min

Sr. No.	Activated carbon dosage to wastewater ratio	Residual COD_{Total} (mg/L)			COD Removal (%)		
		Corn-cob AC	Rice straw AC	Sugarcane bagasse AC	Corn-cob AC	Rice straw AC	Sugarcane bagasse AC
1	0.01:1	5122	5680	5390	73.32	70.41	71.92
2	0.03:1	3508	3885	3754	81.73	79.77	83.6
3	0.05:1	2158	2456	2333	88.76	87.2	87.85
4	0.07:1*	1740	1930	1820	90.9	89.94	90.52
5	0.09:1	1740	1924	1815	90.9	89.9	90.5

* The most suitable dosage of activated carbon

Treatment procedure was conducted by varying contact time between processed activated carbons and distillery effluent. It was found that the much of contact time, the better adsorption capacity was. The most effective contact time was 120 min with removal COD percentageabove 92 %.The removal efficiency of processed activated carbons on the physico-chemical characteristics of effluent are presented in Table (14). pH of treated wastewater was increased to 6.3 andBOD removal percentages were above 82% by processed activated carbons. The highest colour and turbidity removal percentages were occurred by corn-cob activated carbon. Due to high porosities, from macro to microporous structures of efficient processed adsorbents which trapped low molecular weight chemicals such as metal ions, dyes and other organic compounds. Processed activated carbon did not effect on the dissolved solids removal percentage. There would be some chemical reaction between precursors and dissolved chemicals of effluent.Thus chemically activated processed activated carbons were effective for removal of micro pollutants from distillery effluent.

Table 13.Effect of contact time on the removal of total chemical oxygen demand from distillery effluent by processed activated carbon

Initial concentration of COD in wastewater = 19200 mg/L

Temperature of wastewater = 30°C

Dosage of activated carbon to wastewater ratio =0.07:1

Sr. No.	Contact time (min)	Residual COD_{Total} (mg/L)			COD Removal (%)		
		Corn-cob AC	Rice straw AC	Sugarcane bagasse AC	Corn-cob AC	Rice straw AC	Sugarcane bagasse AC
1	30	1544	2950	1890	91.96	84.64	90.16
2	60	1340	1964	1560	93.0	89.77	91.9
3	90	1050	1875	1250	94.5	90.23	93.6
4	120*	1020	1530	1060	94.68	92.03	94.5
5	150	1020	1530	1060	94.68	92.03	94.5

* The most suitable contact time

Table 14.Removal efficiency of processed activated carbons on distillery effluent

Sr. No.	Parameters	Removal efficiency, (%)			
		Corn-cob AC	Rice straw AC	Sugarcane bagasse AC	Literature value*
1	Chemical oxygen demand	94.68	90.89	94.45	36.4-80.6
2	Biochemical oxygen demand	83.86	82.24	83.87	-
3	Colour	65.71	77.14	71.43	68.5-82.7
4	Turbidity	92.07	94.1	92.17	-
5	Total solids	78.35	65.45	73.71	65.7-90.8
6	Dissolved solids	nil	nil	nil	63.6-93.3
7	Suspended solids	97.68	97.82	97.87	69.6-86.1

* (Naimita, T., *et al.,* 2006)

Figure 1. Processed activated carbons from: corn-cob, rice straw and sugarcane bagasse

Conclusion

It has attempted various lignocellulosic wastes such as corn-cob, rice straw and sugarcane bagasse due to the growing need for activated carbon in our society and the high cost of raw materials and production. In addition the choice of a cheap precursor for the production of activated carbon means both considerable saving in production cost and a way of making use of waste materials, thus reducing its disposal and environmental polluted problem.

Due to porous structure, organic nature and large scale availability, these were ideal precursors for preparing activated carbon which have been found to be effective for the removal of metal ions and organic pollutants. By thermodynamic point of view, prepared granular activated carbons had low combustion energy value, low carbonization temperature and period compare to other biomass residues.

It was clearly found that processed activated carbons could be used for treatment of distillery effluent, achieving 94.68% COD and 89.1% BOD removal efficiency of activated carbon to distillery wastewater ratio 0.07:1. This would be of immense benefit not only to the manufacturingindustry in terms of minimizing cost of COD and BOD treatment but also to minimize the impact on environment.

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