COD Reduction of Electroplating Wastewater Using Activated Carbon Prepared from Sugarcane Bagasse

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***Abstract*—Commercially prepared activated carbon can be very expensive, thus, research on low cost adsorbents are vastly studied especially by using waste as the raw materials. The significance of the study is to prepared low cost adsorbent using waste that is abundant and renewable. Other than that, COD parameter is very important in determining the amount of contaminants in the wastewater and the reduction of the said parameter is very crucial since higher COD can be dangerous to the aquatic life. The aim of the study is to prepare and characterize the sugarcane bagasse activated carbon (SBAC) using physiochemical activation method where both chemical and physical activation are both performed for the activation step. The characterization analysis is conducted, focusing on the pore structure of SBAC by Thermogravimetric Analysis (TGA), Fourier Transform Infrared Analysis (FTIR) and Brunauer- Emmett- Teller (BET). In addition to that, the SBAC is tested in a batch adsorption study for COD reduction. Batch adsorption study for COD reduction is performed using COD system reactor and colorimeter. The experiment is conducted with time and adsorbent dosage as the manipulative variables. From the findings, the highest COD percentage removal achieved is 91% with 10 g of adsorbent per 100 mL of water sample, with 240 rpm mixing speed and 1 hour contact time.**

Keywords*— *Activated carbon, adsorbent, chemical oxygen demand (COD), sugarcane bagasse, industrial wastewater

I. INTRODUCTION

Nowadays, agricultural by- products have been evoking the interest due to their renewability, low cost and wide prevalence to produce activated carbon [1]. Agricultural by- products mostly consists of lignin, cellulose and hemi- cellulose. Other than that, they also contain some proteins that make them a very potent adsorbents [2]. The studies on preparing activated carbon using agricultural by- products have been done vastly. Some of the agricultural by- products that have been used includes bamboo [3, 4], sugarcane bagasse and rice husk [5-8], corncobs [8], coconut shells [9], apricot stone [10], Paulownia wood [11] and many more.

In this study, sugarcane bagasse is the raw materials chosen as the precursor in preparing activated carbon. Sugarcane is one of the herb's species that belongs to the grass family where it is officially classified as *Saccharum officinarum*. Sugarcane comes from family of Gramineae [12]. Some of the uses of sugarcane includes sugar production, Falernum, molasses, rum, soda and fuel (ethanol) [12]. In addition, 75% of sugarcane is used to produce sugar in sugar industry [13]. In sugar production industry, the by-products are bagasse, filter cake and molasses where the major by-products are bagasse followed by filter cake and molasses [14, 15]. Most of the sugarcane bagasse produced comes from sugar and alcohol industries and is one of the major by- products produced from the use of sugarcane [15]. Even though sugarcane bagasse is usually burned for generation of bioelectric, the proper use of sugarcane bagasse are till encouraged. Further treatment of the sugarcane bagasse has potential to be utilized in the wastewater treatment sector.

In wastewater treatment industry, activated carbon (AC) is a very common adsorbent used due to the highly porosity and large surface area but it can be an expensive type of treatment. The application of activated carbon in reduction of chemical oxygen demand (COD) through carbon adsorption is very common type of treatment. COD provides a standard measure of oxygen consuming characteristics and the COD adsorption is regularly studied as it evaluated the overall adsorption behavior. High level of COD in wastewaters means high level of oxidable components resulting to high oxygen consumption. This will cause other aquatic life to not be able to consume oxygen. High COD level in wastewater is very harmful to the aquatic life.

These days, the demand for a low- cost adsorbent, specifically by using waste materials that are high in carbon is much needed. Therefore, this has led to anticipate a study on preparing activated carbon derived from agricultural by- products, in this case sugarcane bagasse. Adsorption via activated carbon is highly efficient due to the characteristics of the AC itself. AC has highly developed pores with enormous surface area (3000 m^2 g^{-1}), changeable characteristics of the surface chemistry and high reactivity of surface [3, 16-18]. A study on preparation of activated carbon from bamboo waste and tested on the real textile wastewater where from the study, it is observed that COD removal is possible by using bamboo waste- activated carbon and that increase in both activation temperature and impregnation ratio resulted to higher removal of COD from the wastewater. Other than that, a study on the adsorption efficacy using activated carbon derived from agricultural waste such as coconut shell and fiber and rice husk in reducing COD reading have been also conducted [19] . Based on the result, the percent reduction of COD for each raw materials are in the range of 47- 72% for coconut shell fiber carbon, 50- 74% for coconut shell carbon and 45- 73% for the rice husk [19].

The main purpose of this study is to prepare activated carbon derived from sugarcane bagasse that will be used to reduce COD value in electroplating wastewater by varying some experimental conditions which are the contact time as well as the adsorbent dosage.

II. METHODOLOGY

A. Materials

Sugarcane bagasse is obtained at Seksyen 7, Shah Alam. The chemical used for the activation step is zinc chloride ($ZnCl_2$). Other that, 0.1 M HCl and distilled water are used to wash and rinse the prepared activated carbon. The wastewater is taken from electroplating industry.

B. Methods

1) Adsorbent Preparation

The sugarcane bagasse is first washed with water thoroughly to remove any impurities. Then the sugarcane bagasse is dried in an oven for 6 hours at 110˚C and later is ground using cutter mill and sieved. Next, the dried and ground bagasse is impregnated with zinc chloride ($ZnCl_2$). Ten (10) grams of dried sugarcane bagasse is well mixed with distilled water to obtained 100 mL concentrated solution containing 10 grams of $ZnCl_2$. For this study, the impregnation ratio is set to be 3:1 with 30 grams of $ZnCl_2$ and 10 grams of sugarcane bagasse [20]. The mixed sugarcane bagasse is heated in a furnace for 1 hour at 600˚C and later is cooled to room temperature. Once it reached to room temperature the precursor is washed with 0.1 M hydrochloric acid (HCl) and rinsed repeatedly with distilled water until filtrate reached to neutral pH. Then the precursor is subjected to drying at 110˚C for 6 hours in an oven.

2) Characterization of Adsorbent

a) Thermogravimetric Analysis (TGA)

Thermal stability of raw sugarcane bagasse, char and activated carbon is prepared by subjecting each sample to thermogravimetric measurements performed under the following operational conditions: heating rate of 10˚C/ min, at atmosphere of nitrogen gas at 100 mL/ min in a range of temperature of 0- 700˚C.

b) Fourier Transform Infrared (FTIR) Analysis

The sample for raw dried sugarcane bagasse (SB), sugarcane bagasse char (SBC) and sugarcane bagasse activated carbon (SBAC) were characterized using Spectrum One FTIR Spectrophotometer at 4000- 515 cm^{-1} and the spectra bands have been compared for the three samples in order to determine the functional group presence in the samples.

c) Brunauer- Emmett- Teller (BET) Analysis

Surface area analysis of raw sugarcane bagasse (SB), sugarcane bagasse char (SBC) and sugarcane bagasse activated carbon (SBAC) are carried out using Brunauer- Emmett- Teller (BET) analysis. Other than that, pore volume and pore size distribution properties are all measured by BET method. The sample pre-treatment is to degas using nitrogen gas for 5 hours under 200˚C.

3) Batch Adsorption Studies

a) Effect of contact time

Batch experiments are carried out by placing 150 mL of wastewater sample in a beaker. Initial pH, COD and turbidity of the water sample are recorded beforehand. For each 100 mL of wastewater, 4.5 g of activated carbon is used as the adsorbent. The mixture of activated carbon and water sample is constantly mixed at rpm of 240. The reading for COD, turbidity and pH are taken at interval of 20 minutes for 3 hours. The data is recorded and plot of COD versus time, turbidity versus time and COD versus turbidity are plotted to help with the result and discussion.

b) Effect of adsorbent dosage

The effect of adsorbent dosage on COD reduction is observed by varying adsorbent; 1, 4.5 and 10 g for 100 mL of wastewater sample. Batch experiments are carried out by placing 150 mL of wastewater sample in a beaker. Initial pH, COD and turbidity of the water sample are recorded beforehand. The mixture of activated carbon and water sample is constantly mixed at rpm of 240. The reading for COD, turbidity and pH are taken at interval of 20 minutes for 3 hours. The data is recorded and plot of COD versus time, turbidity versus time and COD versus turbidity are plotted to help with the result and discussion.

III. RESULTS AND DISUSSION

1) Characterization of Adsorbent

a) Thermogravimetric Analysis (TGA)

For raw sugarcane bagasse, char and activated carbon, all plot showed obvious weigh losses and some corresponding valleys. For both the char and activated carbon there are only one valley observed in the plot. The valley existed at around 50 and 85˚C for char and activated carbon respectively, which the valley is attributed for the evaporation of water [21, 22].

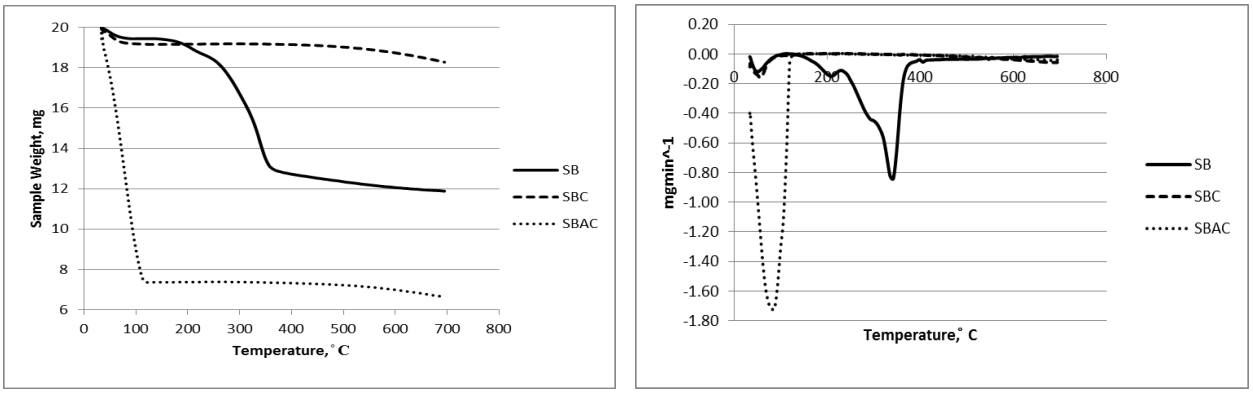
For sugarcane bagasse sample, three valleys with three corresponding phase are observed with also same decreasing pattern in the mass as the char and activated carbon. The first phase happened at around 52˚C with 7.7% of weight loss compared to original weight. The graph showed constant plot after that and at around 198˚C, the second phase take place with also very small decrease as the first phase. At 321˚C, the weight is reduced to 12.6 g, 37% reduced from the original weight. The graph kept decreasing in slow pace until 700˚C.

From the plot as well, we can observed the difference between both char and activated carbon in the weight losses. There is a rapid weight loss for the activated carbon compared to char. Furthermore, activated carbon losses around 63% of the original weight at around temperature of 70˚C while char losses only 4% of the original weight at around 52˚C. Constant weight is observed for the char sample until it reached around 450˚C where the weight started to decrease again. The sugarcane bagasse activated carbon is observed to be the least thermal stability with total weight loss of 63% while the most thermal stability is the char with only 4% of weight loss. This results can be supported from a study of activated carbon using supercritical extraction of hops where the char when compared to activated carbon would have more thermal stability [22]. However the study conducted only encountered 37% of weight loss whereas the sugarcane bagasse activated carbon encountered twice of the weight loss. From this comparison we can say that plant- based activated carbon has less thermal stability than chemical- based activated carbon [22].

We can say from all three graphs, that all the samples weight are reduced within the heating range of 0 to 700˚C. Char left the most residue at the end of heating while activated carbon left a very little amount (~6 mg). So, from thermogravimetric analysis we observed that the prepared activated carbon has the least thermal stability where the SBAC started to losses weight at around 80˚C while char (SBC) is observed to be most thermally stable. With the results, it is can be concluded that activated carbon (SBAC) losses the most weight due to activation process which leads to decomposition of surface functional group thus shows drastic weight loss [22]. In addition, the weight loss can also be from partial gasification of the least thermally stable piece of the structure of the carbon [22].

Table 1: **Thermogravimetric Analysis for Sugarcane Bagasse (SB), Sugarcane Bagasse Char (SBC) and Sugarcane Bagasse Activated Carbon (SBAC)**

Sample	1st Mass Loss (%)	DTG Peak at 1st Mass Loss (℃)	2nd Mass Loss (%)	DTG Peak at 2nd Mass Loss (℃)	3rd Mass Loss (%)	DTG Peak at 3rd Mass Loss (℃)	Total Mass Loss (%)
SB	2.7073	51.52	3.2872	203.96	29.8915	339.45	35.886
SBC	3.6821	56.96	0.1419	-	-	-	3.824
SBAC	62.766	80.13	-	-	-	-	62.766



(a) (b)

Fig. 2: Thermogravimetric Analysis for Sugarcane Bagasse (SB), Sugarcane Bagasse Char (SBC) and Sugarcane Bagasse Activated Carbon (SBAC) (a) Weight Loss (b) DTG Curve

b) Fourier Transform Infrared Analysis (FTIR)

(1) Comparison between Sugarcane Bagasse (SB), Sugarcane Bagasse Char (SBC) and Sugarcane Bagasse Activated Carbon (SBAC)

The FTIR analyses of the samples are conducted in order to determine the functional group presence on the surface of the samples. Figure 6 visually have shown the broad peak at around 3256.84 cm^{-1} that can be indicated as the presence free and intermolecular bonded hydroxyl group (O-H) including hydrogen bonding [21, 23, 24]. The peak is also a result caused by the presence of chemisorbed water on the surface of the activated carbon [23]. This can also be supported as Nejadshafiee and Islami in their studies also stated that the hydroxyl group is precensed on a surface of activated carbon at (~ 3442 cm^{-1}) [25]. In placing more emphasis on the findings, Akçakal, Şahin et al. also found the precense of hydroxyl group stretching where the stretching band is at around 3630 cm^{-1} [26]. The peaks at 2167.39 cm^{-1} show the stretching vibration of the C-H group on SBAC. Similar findings have also been found from Naga, Saied et al. where in the study, the aliphatic C-H stretching is found at peaks of 2923 and 2820 cm^{-1} [23]. Furthermore the finding is also supported from a recent study that found that the stretching vibrations of C-H in $–CH_3$ or $–CH_2$- at peak of 2943 cm^{-1} [27].

Comparing to the raw sugarcane bagasse where the C-H stretching vibration is at peak of 2358 cm^{-1}, the stretching from SBAC has decreased to (~2167 cm^{-1}) indicating that the hydrogen element was removed from the raw material due to activation of the material [3]. It is also observed that for the sugarcane bagasse precursor, there is a series of complex bands presence, which were significantly reduced after activation. In Table 2, the bond peaks are tabulated for all three samples.

(2) Comparison between Sugarcane Bagasse Activated Carbon (SBAC) Before and After Use

Both activated carbons showed the presence of O-H bonding. For activated carbon after being used there are two O-H bonds detected where the peak at 3749.90 referred to stretch, H-bonded type of vibration while peak 3271.40 is stretch, free type of vibration. Comparing to the activated carbon before use where the C-H stretching vibration is at peak of 2167 cm^{-1}, the stretching from activated carbon has decreased to none indicating that the hydrogen element was removed from the raw material due to adsorption for COD removal from the wastewater sample [3]. Other than that, the IR spectra for both before and after activated carbon being used for adsorption shows no significant difference that indicates that the prepared activated carbon does not degrade or change in the surface chemical properties after being used for COD reduction process through adsorption. The results are shown in **Fig. 5** and are tabulated in **Table 3**.

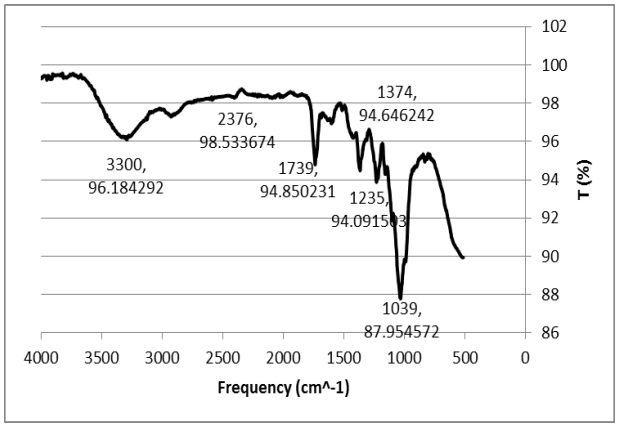


Fig. 2: IR Spectra for Raw Sugarcane Bagasse (SB)

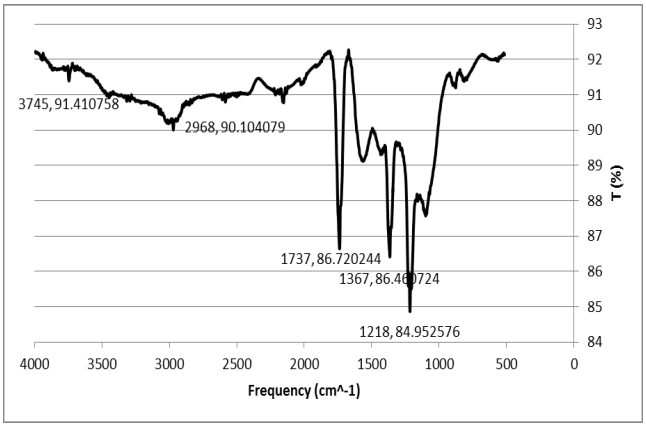


Fig. 3: IR Spectra for Sugarcane Bagasse Char (SBC)

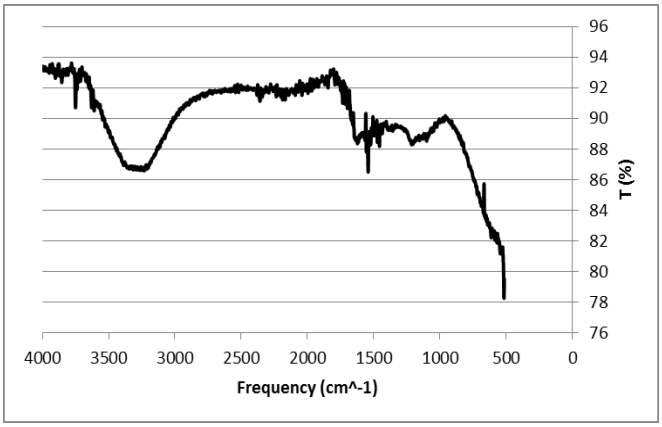


Fig. 4: IR Spectra for Sugarcane Bagasse Activated Carbon (SBAC)

Table 2: IR Spectra of Sugarcane Bagasse (SB), Sugarcane Bagasse Char (SBC) and Sugarcane Bagasse Activated Carbon (SBAC)

Sample	Frequency (cm^{-1})					
	0-H	**C-H**	**C=O**	**C=C**	**C-O**	**C-H bending**
SB	3300 (*s*)	2376 (*s*)	1739 (*s*)	1374 *(m to w)*	1039 (*s*)	1235 *(v)*
SBC	3745 (*s*)	2968 (*s*)	1737 (*s*)	1367 *(m to w)*	-	1218 *(v)*
SBAC	3296 (*s*)	2364 (*s*)	1541 (*s*)	-	-	668 *(s to m)*

Note: *s*- strong, *m*- medium, *w*- weak, *v*- variable

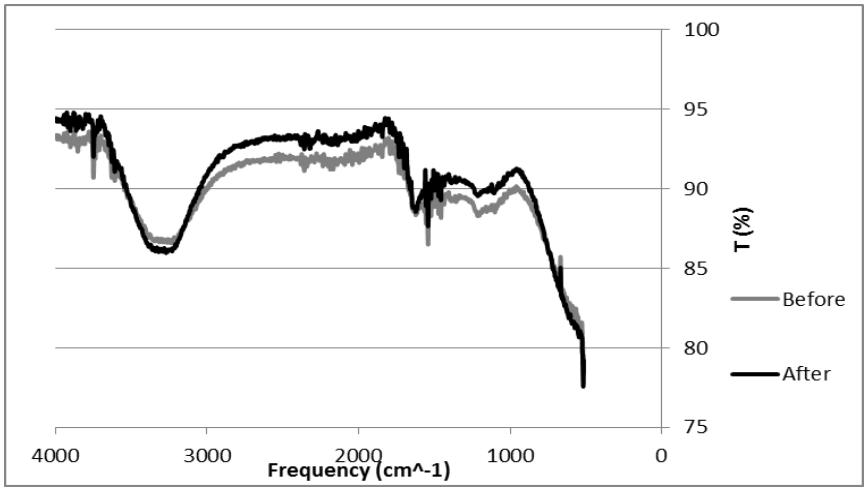


Fig. 5: IR Spectra for Sugarcane Bagasse Activated Carbon (SBAC) Before and After Being Used for Adsorption

Table 3: IR Spectra of Sugarcane Bagasse Activated Carbon (SBAC) Before and After Use

Sample	Frequency (cm^{-1})					
	O-H	**C-H**	**C=O**	**C=C**	**C-O**	**C-H bending**
SBAC (Before Use)	3296 (*s*)	2364 (*s*)	1541 (*s*)	-	-	668 *(s to m)*
SBAC (After Use)	3749.90 (*s*) 3271.40 (*s*)	-	1623.01 (*s*)	1541.49 *(m to w)*	-	668.00 (*s*)

Note: *s*- strong, *m*- medium, *w*- weak, *v*- variable

c) Brunauer- Emmett- Teller (BET) Analysis

From **Table 4**, the compiled results are shown which are obtained from Brunauer- Emmett- Teller (BET) Analysis for three samples which are sugarcane bagasse (SB), sugarcane bagasse char (SBC) and sugarcane bagasse activated carbon (SBAC). The activated carbon prepared from sugarcane bagasse contained large surface area and total pore volume when is set side by side with the commercially available activated carbons such AC AK 7080 with surface area of 1200 m^2/ g and 0.078 cm^2/ g respectively [28]. These values when compared to the prepared activated carbon are relatively smaller. Activation of the char using $ZnCl_2$ as the activating agent resulted to high BET surface area, total pore volume and pore developments [4]. This is also resulted to a significant difference in the values for BET surface area, Langmuir surface area, and total pore volume and pore diameter for the three samples.

Both sugarcane bagasse and the char are carbon components without activation while the prepared activated carbon is the char that have been going through activation process. Pores are formed in a carbon component when they are activated with activated agents but too high of concentration of activating agent can cause distortion on the carbon component [29]. From **Table 4,** it has shown that very small BET surface area (1.4652 m^2/ g) and a negative value of pore volume (-0.0006 cm^3/ g) for the raw material sugarcane bagasse. Limited pores present in the sample might be the cause for the small BET surface area. Limited pores available is because the sample has not been chemically activated [29]. Moreover, the micropores area is not reported for the said sample because the micropores volume is negative. The micropores volume is observed because the fact that a negative y- intercept is observed in the t- plot.

The average pore diameter for sugarcane bagasse activated carbon is found to be 18.63 Å which equivalent to 1.863 nm, implying that the prepared activated carbon is in micropores region. Pore that has a diameter less than 2 nm is characterized as micropores, pores are classified as mesopores if the diameter is around 2 to 50 nm and macropores is characterized to sample that has pore diameter more than 50 nm according to the International Union of Pure and Applied Chemistry (IUPAC). This statement is also is supported in the study conducted by [30]. According to Anisuzzaman, Joseph et al. the pore diameter of a commercial activated carbon, AC AK 7080 which is a coconot shell- based activated carbon is analysed to be 20.8 Å [28].

2) Batch Adsorption Studies

a) Effect of Contact Time

The experiment is started using 4.5 grams of activated carbon for 100 mL of wastewater sample [31]. In this part of experiment, the effect of contact time is analyzed for COD percentage removal, turbidity as well as pH reading. From **Fig. 6**,we can see rapid declining of plot for turbidity reading. The turbidity decrease from 85.80 NTU to around 0.5 NTU in the first 20 minutes of contact time. After that we can observed very stable readings of the turbidity throughout. A minimum turbidity of 0.30 mg/ L is achieved using activated carbon from sugarcane bagasse. From the results obtained, we can say that the addition of sugarcane bagasse activated carbon (SBAC) can reduce the turbidity of the wastewater sample. Turbidity content decrease with time. The same finding on turbidity treated using activated carbon in a past study also shows decrement of turbidity content with increasing time [32]. Next is pH parameter. **Fig. 6** shows the plot for pH shows consistency in reading which the highest pH is observed is 8.69 at initial and the.lowest pH obserbed is at minute 60 which is 6.23. Overall, the pH readings are in the range of 8 to 6 which still complied with Standard A and B for Malaysia Sewage and Industrial Effluent Discharge Standards.

For COD percentage removal, we can observed the highest peak at minute 140 where the percentage removal is around 70%. After that the percentage removal started to decrease. From this finding we can say that percentage removal of COD increase with increasing time but will later started to decrease with time. This is because adsorption site with increasing time, the availability of unoccopied adsorption site will decrease and later none will be available for adsorption to take place. From the result, we can see increasing of COD removal in early stage since the unoccupied activation site are still available. After minute 140, where the COD removal started to decrease, it is due to absent of unoccupied adsorption site. The adsorption started to slow down thus decreasing the percentage removal. From this part of experiment to observe the effect of contacct time we can deduced that COD removal percentage will increase with time but after saturation on the surface of activated carbon is reached, the adsorption will slow down, the percentage removal will decrease.

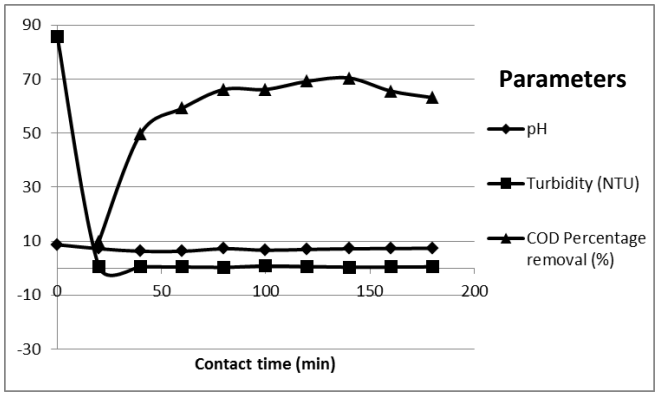


Fig. 6: Plot of pH, Turbidity and COD Percentage Removal versus Contact Time

b) Effect of Adsorbent Dosage

The numbers of adsorption sites as well as surface area available are determined by the amount and quantity of the adsorbent dosage. For this study, different adsorbent dosage which are 1g, 4.5g and 10g are investigated in reducing COD reading of a wastewater sample from copper electroplating industry. Plot of COD percentage removal against time for different adsorbent dosage is shown on **Fig. 7**. Overall view of the plot we can see that the COD percentage removal has increased after a certain amount of contact time where the plot started to decline. This finding can be further deliberate where adsorption of the activated carbon will reached maximum after certain amount of time due to decreasing adsorption capacity on the surface of the adsorbent [31].

For adsorption dosage of 1 gram, the plot shows that the COD removal efficiency plateaued at around 80 to 100 minute implying that the solution has reached equilibrium [31]. This is because the adsorption is rapid since there were many unoccupied adsorption site on the activated carbon [31].This can also be applied to other adsrobent dosage as we can deduce from the plot that the early stage shows rapid increase of COD removal percentage. The plot started to decline after 100 minutes, where it reached the lowest COD removal percentage at minute 180. This shows that once adsorption site on the surface have been occupied and there is no vacant site available for the adsorption place to take place anymore. Adsorption also becomes more difficult because of there is repulsive force happened between the adsorbed solute molecules and the adsorbate bulk liquid phase [31]. For adsorbent dosage of 4.5 grams, we also observed a quite similar result with the previous adsorbent dosage (1 gram). Rapid increase from the beginning to minute 140. Then the graph started to decline. The highest pecentage removal is observed at minute 140 with 70.39% COD removal. Comparing to dosage of 1 gram where the highest percentage removal is only 50.74%, we can say that higher adsorbent dosage give higher COD removal. This is because as adsorbent dosage increase, there will be more adsorbent surface the increases in adsorption site vacancies as well. This statement san be further supported by a study where in the study it is stated that the increment of adsorption is due to available vacancies of adsorption site on the adsorbent surface [31, 33].

Next is adsorbent dosage of 10 grams. We can see from the plot that it shows the highest COD percentage removal compare to the other dosage amount. After one hour, the COD removal is already at 90% removal. Comparing to other adsorbent dosage we also can deduce that for 10 grams dosage, it reached the highest percentage removal the fastest. This result however is quite different from the study from Chingono, Sanganyado et al. where the optimum adsorbent dosage is 4.5 grams and in the study showed that increasing of adsorbent dosage more than 4.5 grams showed decrement in COD removal [31]. But the COD percentage removal achieved is almost similar which is 70% while in the study from Chingono, Sanganyado et al. it achived maximum of 68% [31].

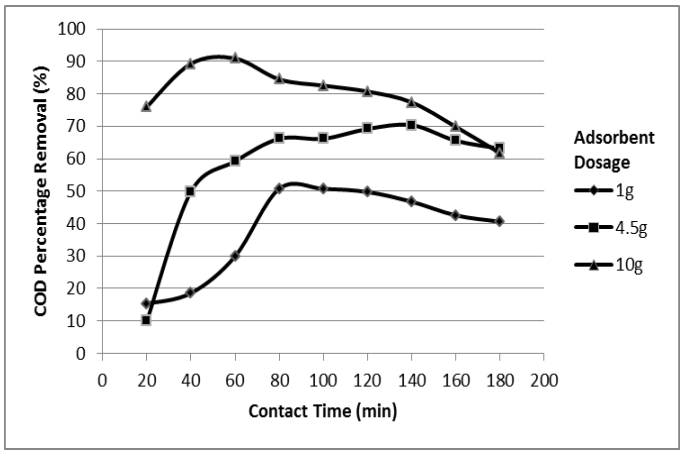


Fig. 7: COD Percentage Removal for Different Adsorbent Dosage

Table 4: Results for BET Analysis for SB, SBC and SBAC

	BET Surface Area (m^2/ g)	Langmuir Surface Area (m^2/ g)	Total Pore Volume (cm^3/ g)	Average Pore Diameter (Å)
Sugarcane Bagasse (SB)	1.4652	3.8735	-0.0006	83.7706
Sugarcane Bagasse Char (SBC)	91.9487	109.3364	0.0272	19.5271
Sugarcane Bagasse Activated Carbon (SBAC)	1492.3300	1933.2042	0.3092	18.6335

IV. CONCLUSION

The aims of this study are to prepare and evaluate the characteristic of activated carbon derived from sugarcane bagasse as well as to evaluate the performance of the prepared activated carbon in reducing COD and turbidity. For the characterization part of this study, the activated carbon derived from sugarcane bagasse is characterized using Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR) analysis and Brunauer-Emmett- Teller (BET) analysis. The prepared activated carbon

showed one valley existed at around 85˚C, which attributed for the evaporation of water. Rapid weight loss for the activated carbon happened starting the temperature 35˚C until 130˚C. For the infrared spectroscopy analysis, the activated carbon showed four peaks indicating the existence of free and intermolecular bonded hydroxyl group (O-H), stretching vibration of the C-H group, C-H bending is also presence on the surface of the activated carbon as well as C=O group. The prepared activated carbon also poses high BET and Langmuir surface area with 1492.33 and 1933.2042 m^2/ g respectively, pore volume of 0.3092 cm^3/ g and is categorized as micropores since the pore diameter observed which is 1.86 nm is smaller than 2nm. COD reduction experiment is conducted where it is observed that by increasing of adsorbent dosage, the percentage of COD removal also increase. It is also observed that increasing of contact time also will increase the COD percentage removal but after saturation of the adsorption sites have reached, the percentage started to decrease. The saturation is reached in the range of contact time of 60 to 140 minutes.

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