Preparation and characterization of Gigantochloa robusta activated carbon to reduce COD levels of pharmaceutical waste

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Abstract. Activated carbon prepared from Gigantochloa robusta was studied to reduce Chemical Oxygen Demand (COD) levels in pharmaceutical-liquid waste. Physical activator was used to produce Activated Carbon (AC) in temperature regulation of 750°C retort for 60 minutes. Dosage of AC powder were 0.25%, 0.5%, and 0.75% of 250 mL of pharmaceutical waste, respectively. Retention time variations of 30, 60, and 90 minutes were used in laboratory scale experiments. Proximate analysis including volatile substances, water content, ash content, fixed carbon and I₂ adsorption were observed to characterize AC. This research shows that the produced AC successfully meets the SNI 06-3730-1995 standards. The study results show variations in COD removal between 9-23%. The best result was performed by the AC dosage of 0.75% over a 60 minutes retention time.

1. Introduction
The presence of effluents in water causes a negative influence on aquatic ecosystems. Various methods have been offered to overcome the pollution of aquatic ecosystems, one of which is activated carbon, both in powder or granules. Even before being activated, charcoal form, which has been used to prevent environmental degradation [1]. Charcoal or activated carbon displayed as an alternative by considering its inherent character. The pore volume of activated carbon is believed to be responsible for improving adsorption properties. In addition to water treatment, activated carbon can be used in handling air pollution. While the requirements for the nature of pore volume against liquid phase adsorption applications are not as strict as for the gas-phase purification. The pore volume radius for gas adsorption is estimated from micro (< 20 Å) to macropores (> 500 Å), while liquid adsorption applications can be greater than that interval [2]. Another fact is the precursor of activated carbon from biomass or biomass waste becomes an advantage in reducing the cost of restoration of aquatic ecosystems. Activated carbon can be synthesized from green precursors such as coconut shells [3], oil palm shells [4], candlenut shells [5], corn cobs and bamboo betung [6].

Activated carbon has been noted for reducing level of COD. Effectiveness of COD removal varies based on the nature and dosage of activated carbon against effluent-liquid waste. The percentage of removal in COD levels was recorded between 47% and 68%, namely: 47.31% (200 g/L), 55.22% (300 g/L), 62.33% (400 g/L) and a value of 68.37% at of 500 g/L [7]. Another phenomenon, even with better
effectiveness, was found in textile effluent treatment. The application of activated carbon from teak sawdust was declared successful in reducing COD of the liquid waste. The result demonstrated a decrease down to 84% when 1.15 grams of activated carbon adsorbent was used [8]. Adjacent study noted the consistency of activated carbon adsorption properties. It was stated that COD and BOD reduction levels were 78.96% and 33.51%, respectively, by activated carbon of coffee grounds [9]. A more recent study was carried out by including the retention time of activated carbon with effluents. Corn cob precursor was involved in the production of activated carbon. It successfully reduced the COD and BOD levels in domestic wastewater [10]. The emphasis is then directed towards pharmaceutical industry, a sector that has abundant chemical effluent. Advantage of activated carbon in the handling of pharmaceutical effluent has been pronounced through various technologies and application of activated carbon [11]. The general objective of this work is to investigate the properties of activated carbon from green precursor of bamboo mayan (*Gigantochloa robusta* Kurz.) and its effectiveness against COD removal of pharmaceutical effluent.

2. Materials and Methods

2.1. Materials

Mayan bamboo (*Gigantochloa robusta* Kurz.) was obtained from Sukabumi, while pharmaceutical effluents were obtained from crude-pharmaceutical waste. Na$_2$S$_2$O$_3$ (Merck type, CID 24477), HgSO$_4$ (CID 24544), H$_2$SO$_4$, K$_2$Cr$_2$O$_7$ 0.25 N, 0.25 N ferrous ammonium sulphate solution, iodine (I$_2$), indicator (starch, ferroin) pH paper, distilled water and filter paper was also used for testing material. Apparatus used were analytical balance, oven, reactor, desiccator, furnace (VULCAN A-550), laboratory sieve (BBS), beaker, chalice, Erlenmeyer, funnel and burette.

2.2. Methods

2.2.1. Activated carbon preparation

The initial stage of activated carbon production was the carbonization procedure. Each 100 mm long of *Gigantochloa robusta* Kurz stack were fed into a stainless-steel reactor. The carbonization duration was 5 hours with the applied temperature of 500°C. The activated carbons were prepared by one-step pyrolysis of *Gigantochloa robusta* Kurz, in the presence of steam. The treatment temperature was set at 750°C, and held for 60 minutes at the final temperature. After the treatment, the samples were left to cool down. The activation product was then ground with mortar and pastel, manually. The powder form was then retained by a 60 mesh filter system. The sample was ready to be tested for pharmaceutical COD removal.

2.2.2. Proximate analysis

The proximate analysis is based on the principles and procedures of SNI 06-3730-1995 concerning Technical Active Carbon which is not used as medicines raw material. The technical quality requirements for activated charcoal referred to this study consist of: volatile substances, water content, ash content, adsorption index I$_2$ and fixed carbon.

2.2.3. Effectiveness of pharmaceutical COD effluent removal

A total of 250 mL of pharmaceutical effluent was added to the Erlenmeyer. The activated carbon was added and stirred for 10 minutes, then filtered to get the filtrate. Three treatments were applied for activated carbon concentration (% w/v) and contact time (minutes), respectively. The active carbon concentration variations were 0.25%, 0.5%, and 0.75% while the retention time variations were 30 minutes, 60 minutes and 90 minutes.

As much as 0.4 gram of HgSO$_4$ crystal was added into 250 mL of filtrate, followed by the addition of 5 mL of concentrated H$_2$SO$_4$ to dissolve HgSO$_4$. The mixture was then cooled. Approximately 25 mL of K$_2$Cr$_2$O$_7$ 0.25 N solution and 30 mL H$_2$SO$_4$ were added subsequently. The mixture was refluxed for 2 hours. Afterward, the mixture was cooled. Further, approximately 50 mL of distilled water and 3 drops of the indicator ferroin were added into the mixture. The 0.1 N ferrous ammonium sulphate solution was for the titration process until the colour changes from green-blue to exactly red-brown.
3. Results and Discussion

3.1. the proximate analysis

Gigantochloa robusta Kurz. was cut 100 mm in size, serves to increase effectiveness of the carbonization process. Previously, pieces of material were cleaned from dirt and dried in room for a week until moisture content reached a value of less than 40%. Steam activation is responsible for the formation of activated carbon pores. The endothermic reaction was involved in the activation process carried out at 750°C [12].

\[
\begin{align*}
\text{CO} + \text{H}_2\text{O} & \rightarrow \text{H}_2 + \text{CO}_2 \quad (1) \\
\text{C} + 2\text{H}_2 & \rightarrow \text{CH}_4 \quad (2) \\
\text{C} + \text{H}_2\text{O} & \rightarrow \text{H}_2 + \text{CO} \quad (3)
\end{align*}
\]

Granular activated carbon was then made into powder with a size of 60 mesh. The activated carbon powder was analysed by proximate-analysis before being applied to pharmaceutical effluent. Table 2 shows that activation process has successfully met the quality requirement for parameters: volatile, moisture content, ash content, adsorption capacity of I\(_2\) and fixed carbon. The present study is consistent with Lam et al. [13] who observed dramatic increase of carbon content and activated carbon increase dramatically, as well as the significant decrease of the volatility properties.

**Table 1.** The proximate analysis of activated carbon compared to standard [14]

| No | Parameter         | Unit | Gigantochloa robusta Kurz\(^a\) | Present study | Standard |
|----|-------------------|------|---------------------------------|---------------|----------|
| 1  | Volatile          | %    | 10.30                           | Max 25        |
| 2  | Moisture content  | %    | 9.68                           | 7.40          | Max 15   |
| 3  | Ash content       | %    | 2.67                          | 8.80          | Minimal 10 |
| 4  | I\(_2\) Adsorption| Mg/g | 796.98                         | Minimal 750   |
| 5  | Fixed carbon      | %    | 80.80                          | Minimal 65    |

3.1.1. Volatile matter

Volatile matters were the dominant substances found in precursor, around 55-84% and decreased dramatically to 4-15% [15], 73% to 5-6% [16], and 84% to 16-22% [12] after carbonization. Volatile content indicates the amount of substance that evaporates at the treatment temperature. Volatile content, thus, can be an indicator of the success degree of treatment temperature. The higher level of activated carbon volatile substances is, the adsorption properties will be lower. High or low levels of volatile substances affect the porosity of charcoal or activated charcoal produced. The release of volatile biomass in carbonization stage enriched carbon composition and form porosity [17], even the activation process increases this phenomenon. Furthermore, activated carbon with volatile values and a low O/C ratio makes material more resistant to the oxidation processes. The atomic ratios of O/C and H/C were affected by the decarboxylation reaction, the loss of carboxyl groups and the release of CO\(_2\), the H/C ratio notified the double bonding of hydrocarbon groups [12]. Multiple bonding (double/triple/etc) causes strong-stable bonds that can only be broken at high temperatures. Volatile matters generally burn at 530°C [18]. The mass left after volatile evaporation are pure carbon and ash. Volatile content in this study was 10.3% and fulfilled SNI 06-3730-1995 standard. This fact shows that organic matter evaporates during the temperature manipulation, while non-volatile ash remains in the product.

3.1.2. Moisture content

Moisture content is related to the hygroscopic properties of surface and activated carbon pores. Superior quality activated carbon was characterized by having a low moisture content, thus the activated carbon is very suitable for liquid effluent treatment [13,17], given that the surface was not clogged and an activated carbon pore has been formed. The evaporation of water begins immediately after reaching its
boiling point, marked by the significant loss of mass. The evaporation of water began after the carbonization temperature reaches 120°C, followed by rapid volatile evaporation at temperatures of 400°C [16]. The result shows the moisture content of the sample of this study was 7.4%, which met the SNI 06-3730-1995 standard.

3.1.3. Ash content
Ash is composed of minerals contained in activated carbon precursors. Ash content is one of the parameters that determine the profile characteristics and adsorption performance. Mineral content may cover activated carbon pore thereby reducing activated carbon adsorption properties. Mineral properties trigger catalytic processes caused by inorganic content and reduce the adsorption of surface activated charcoal [17]. Temperature treatment does not burn precursor minerals and remains in the product. Increasing of ash content actually occurs after the carbonization stage is implemented. Ash content increased from 2% to 5% after carbonization [16], 10-25% to 28-51% [15] and relatively 4% to 3-4% [12]. Treatment of strong acids before activation is one of the efforts to reduce ash content. Typically, solutions of up to 10% H2SO4 were applied in the immersion of precursor (charcoal), in anticipation of acid washing by water. Ash content in this study was 8.8% and fulfilled SNI 06-3730-1995 standard.

3.1.4. Fixed carbon
The nature profiles of the precursors are low carbon content and high volatility. Research by Gonsalvesh et al. [15] showed an increasing fixed carbon after the carbonization stage, from 5-20% to 43-67%. Other study notified changes in the carbon content from 24% to 87-89% [16] and 34% to 73-77% [12]. Fixed carbon content is a component of the carbon fraction (C) contained in activated carbon material. Fixed carbon content is influenced by the value of ash content and volatile levels of activated carbon. The lower the ash content and volatile matter is, the higher the fixed carbon content us, in addition, it is also influenced by the content of cellulose and lignin. The decrease in elements (H,N,O) and volatile material caused by the degradation of cellulose, hemicellulose and lignin into organic molecules such as CO2 and CH4 [13]. The typical peaks of organic chemical degradation were 220, 300, and 350°C [16]. The general temperature to pure the fixed carbon was above 650°C [18]. The fixed carbon in this study was 80.8%, above the SNI 06-3730-1995 standard. The facts show that the carbonization and activation cause an increase in C content and decrease other content.

3.1.5. Iodine (I2) number
The determination of I2 index was conducted to observe the adsorption ability of activated carbon. The adsorption value of activated carbon indicates the amount of pore volume of activated carbon. The determination of iod adsorption used iodometric titration method. The activated carbon powder of Gigantoclhoa robusta Kurz. reacted with iodine solution. At first Na-thiosulfate reacted with iodide ions to produce sodium iodide and sodium tetrathionate as below.

\[ 2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6 \] (4)

The reaction between Na-thiosulfate with iodide ion produced a yellow colour. After the addition of the starch indicator, the titration with Na-thiosulfate produces a clear colour. Figure 1 shows the colour changes before and after the titration. The iodine adsorption by the activated carbon in this study amounted to 796.981 mg/g.

Gigantoclhoa robusta Kurz. has solubility characteristics in different solvent as follow: in benzene alcohol 3.24%; 9.63% hot water; cold water 6.68%; and NaOH (1%) 23.95%; cellulose 57.55%; holocellulose 63.32%; lignin 31.66%; pentosane 18.60%; starch 9.42%; water content 9.68%; ash 2.67%, and silica 1.48% [14].
3.2. Chemical Oxygen Demand Analysis

3.3. The COD analysis was carried out by using 3 dosages of activated charcoal, which were 0.25%, 0.5% and 0.75% of the 250 mL effluent of pharmaceutical waste. The determination of COD index involves HgSO4 compounds to eliminate chloride disturbance in pharmaceutical wastewater and potassium bichromate as an oxidizing agent (source of oxygen) titration with ammonium sulphate. The colour alteration from orange to brighter colour indicates a decrease in COD index (Figure 2). The highest COD removal was 23.3% which was provided by 0.75% activated carbon for 60 minutes retention time (Figure 3). The trend of COD removal by activated carbon and retention time in this study occurred gradually. Increasing the dosage and retention time caused the decrease in COD.

3.5. Effectiveness of activated carbon against COD removal

The COD analysis is often used to determine the amount of oxygen required to oxidize organic particles and dissolved compounds in aquatic. The COD analysis is very important related to the determination of the effect of disposal of industrial effluents into aquatic ecosystem. The COD index indicates the level of oxidized organic matter in water. The higher value of COD is, the higher the amount of oxidation of inorganic chemicals into nitrate/nitrite is at high level and potential to reduce dissolved oxygen (DO). Decreasing DO is responsible for triggering anaerobic conditions that alter the aquatic ecosystem.

Figure 1. (a) Colour alteration before titration and (b) after titration

Figure 2. The colour alteration of pharmaceutical effluent notifies COD decrease by activated carbon treatment in 250mL of each (a) Pharmaceutical effluent (b) activated carbon 0.25% (c) activated carbon 0.5% (d) activated carbon 0.75%
The principle of COD analysis is a strong oxidizing agent under the acidic condition to oxidize organic compounds into carbon dioxide and H₂O. The oxidizer used in this study was potassium dichromate (K₂Cr₂O₇) combined with boiling sulfuric acid (H₂SO₄) and mercury sulfate (HgSO₄). Cr₂O₇²⁻ to oxidize organic compounds into carbon dioxide and water. The intervention of oxidation by chloride ions is limited by HgSO₄ assisted by concentrated H₂SO₄ solvent.

![Graph](image)

**Figure 3.** Percent of COD removal by activated carbon dosage and retention time (minutes)

3.6. This study uses activated carbon with I₂ value of 796.98 mg/g, and measurement of COD alteration (ppm) was observed on pharmaceutical effluent. The result was then compared to the pharmaceutical effluent water quality standard based on the Minister of Environment Regulation No. 5 of 2014. This study noted a decreasing trend in COD index by activated carbon from *Gigantoelhhoa robusta* Kurz. (Table 2). Increasing in activated carbon concentration (% w/v) and retention time-intensity (minutes) positively correlated to COD index. An increasing amount of activated carbon and contact time will reduce the COD index [10]. The highest COD removal by 0.23% was attained in the present work (AC 0.75) leading this achievement by 60 minutes retention, equal to COD removal as much as 280ppm compared to crude pharmaceutical effluent. Although the present work does not comply with the standard, this effort demonstrates activated carbon adsorption property to purify water waste.

The optimum retention time for a decrease in the pharmaceutical effluent COD index in this study was consistent with Karunya et al. [19] and Sivakumar and Muthukumar [20], which was 60 minutes. An initial conclusion from these studies that, the first optimal time to remove pharmaceutical effluent COD occurs after 60 minutes. This phenomenon was predicted related to effluents which cannot be further degraded or the presence of OH⁻ and H₂O₂ oxidant [20]. Another result shows more distant retention with a better COD removal, between intervals of 180 and 210, which COD removal along 78-80% [21]. The present study was in line with previous studies, where the optimum contact time was achieved after 60 minutes. Activated carbon behaviour against waste water notifies an adsorbent function.

Effluent intermolecular adsorbed into active carbon pore reach a saturation point after 60 minutes. This process is possible through the electrostatic effect between surface particles of adsorbent and adsorbate in pore space of activated carbon: micro, meso and macro pores. The surface area is an
excellent absorbent, so that adsorbate rapidly, then into pore volume inside. The adsorption performance is dependent on the carbon profile of activation. However, the time limit and effectiveness of activated carbon may be modified using multilevel technology by additives such as hydrogen peroxide-$\text{H}_2\text{O}_2$, carbon tetrachloride-$\text{CCl}_4$, potassium dichromate-$\text{K}_2\text{Cr}_2\text{O}_7$[20], continuous reactors, or membrane coating technology.

The effect of activation carbon of 0.625 g, 1.25 g and 1.87 g against 250 mL pharmaceutical waste were 6-23%. A distinct dosage of 2 g/L (0.2%) successfully reduced the pharmaceutical effluent COD by 60% [20] for 120 minutes of retention. Higher concentration was applied to industrial effluents by 50 g/L, where the decline in COD index was around 56, 68 and 74% [21].

Table 2. the effects of activated carbon to COD removal by precursor, effluent, dosage and retention time

| Study/material                        | Effluent         | Dosage            | Retention time | COD removal (%) |
|---------------------------------------|------------------|-------------------|----------------|-----------------|
| The present study                     | Pharmaceutical   | 0.25%             | 10 min, 30 min, 60 min | 6; 8.67; 19.33  |
| Gigantochloa robusta Kurz             |                  | 0.5%              |                | 8; 10; 20       |
|                                       |                  | 0.75%             |                | 8.67; 12; 23.33 |
|                                         | Municipal        | 0.01-1.5 g/100 mL | 2 hours        | above 80        |
| Bansode et al. [22]                   |                  |                   |                |                 |
| Pecan shell                           |                  |                   |                |                 |
| Delgado et al. [23]                   | Carbamazepine,  | 100 mg/L          | 72 hours       | Promising agent |
| Vegetable origin                      | sildenafil citrate|                  |                | by 3 adsorption |
| Dalahmeh et al. [24]                  | carboxylic acid, metoprolol, ranitidine and caffeine | Filter | Over 22 weeks | isotherm model 7-99 |
| Commercial biochar                    |                  |                   |                |                 |
| Li et al. [25]                        | Sandwich filter  | 3 weeks           |                | 65.8            |
| Granular activated carbon             |                  |                   |                |                 |
| Karunya et al. [19]                   | DEET, paracetamol, caffeine and tri- closan | Pharmaceutical industry wastewater | 60, 90, 120, 150 and 210 min | Above 80% |
| Date seed                             |                  |                   |                |                 |
| Sivakumar and Muthukumar [20]         | Pharmaceutical Wastewater | 1, 1.5, 2, 2.5, 3 g/L | Interval 10 min | % significant of COD reduction until 60 minutes |
| Commercial activated carbon           |                  |                   |                |                 |
| Badmus and Audu [21]                  | Industrial wastewater | 20, 30, 40, 50, 60 and 70 g | 30-300 min | % significant of COD reduction until 50 minutes |
| Periwinkle shell                      |                  |                   |                |                 |
| Saleem [26]                            | Pharmaceutical Wastewater | 1 g/L | filtration method | Granular activated carbon was effectively used in the final stages of filtration |
|                                       |                  |                   |                |                 |

A decrease in the COD index of 23% by 1.875 gr/ 250 mL was obtained in this study. The ability of activated carbon adsorption in this study has successfully demonstrated a decrease in dissolved oxygen as a reducing factor for the COD index, in the context of the adsorption process. When a solution containing effluent materials contact with surface area, it triggers adsorption stage. Effluent organic molecules attach to activated carbon adsorbent and transform to adsorbates the surface area of product. This phenomenon alters activated carbon into a catalyst for oxidation of organic materials until it attains a saturation point. The efforts to increase the performance of COD removal by activated carbon adsorbents can be increased through the interpolation of 79% of $\text{H}_2\text{O}_2$, 83% of $\text{CCl}_4$ and 68% of $\text{K}_2\text{Cr}_2\text{O}_7$ respectively, and to COD removal by 60% [20]. Furthermore, Sivakumar & Muthukumar [20] stated that the decomposition of $\text{H}_2\text{O}_2$ into hydroxyl groups triggering the oxidation of adsorbates, while $\text{K}_2\text{Cr}_2\text{O}_7$ additive was responsible for pH improvement. The $\text{CCl}_4$ was a hydrogen atom binder and balances reaction of hydrogen atoms and hydroxyl radicals, following the formula [20]:


H₂O → •H + •OH

C_Cl₂ + •H → HCl + •C_Cl₂

The activated carbon ability was tested to improve chemical handling of pharmaceutical effluent. Activated carbon was applied in the final stage of filtration and successfully reduced the COD index to 71.1%, using continuous reactors [26]. The best achievement of this study succeeded in reducing COD by 23% but it was still above the regulatory requirements regarding the wastewater quality standard. Thus, the chemical content of the pharmaceutical effluent was suppressed by the adsorption properties of activated carbon in a single product application, without the aid of other catalyst additives or in filtration system. Saleem [26] filtration technique may be applied in the future studies.

4. Conclusion

This study has succeeded in producing activated carbon which satisfactorily meet the requirements according to SNI 06-3730-1995. The proximate analysis (MC, ash content, volatile, pure carbon) were within the standard. On the other hand, the adsorption capacity of I₂ was above the required standard of 796.98 mg/g. The activated carbon profile applies as an adsorbent function.

This work demonstrated the highest COD reduction of 23% with the dosage of activated carbon was 0.75%, although was still above the minimal requirement standard for pharmaceutical wastewater. The retention optimum in this study was 60 minutes, indicating activated carbon performance in short period. An effort to increase activated carbon profile and filtration technique including a sequence study is required in the future.

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