Tabah Bamboo Biocharas Methylene Blue Adsorbent from Solution

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Abstract: ***Tabah bamboo is a rare bamboo that only grows in some regions of Bali. Bamboo is one of the carbon-rich plants worthy of being processed as a raw material for activated charcoal. This study aimed to determine the potential of Tabah bamboo charcoal inactivated and activated by $ZnCl_2$ as an adsorbent for methylene blue. The research was started by making bamboo charcoal labeled Bo and activated charcoal, B_A. The optimized adsorption parameters cover contact time, adsorbent mass, pH solution, initial concentration, and temperature for the best performance. The adsorbent characterization includes functional groups, morphology, surface area, and pore volume. The results showed the contact time of 90 minutes, a mass of adsorbent 0.2 g, and a pH of 10, for the Bo and BA charcoals. Adsorption isotherm models for both charcoals tend to follow Langmuir. The adsorption capacities were 70.22 mg/g and 79.24 mg/g for Bo and BA respectively. Characterization results showed that Bo and B_A have similar functional groups had a carbon content of 85.79% and 96.96%, and a surface area of 0.1879 m2/g and 5.1037 m2/g, respectively.***

Keywords: Adsorption, Activated Charcoal, Tabah Bamboo, Methylene Blue

1. Introduction

Governor of Bali Circular No. 4 of 2021 concerning the use of Balinese endek woven fabrics/traditional Balinese woven fabrics, which requires people to wear traditional Balinese endek clothing [1]. The regulation creates business opportunities for the textile industry in Bali. However, apart from increasing the economic value for the community, a new problem arises the liquid waste produced disrupts the environmental ecosystem. The textile industry produces dyes and heavy metal pollution which can affect levels of oxygen, pH, and the life of organisms in the waters [2]. Methylene blue is one of the dyes that are widely used as basic dyes. This dye gives a dark blue color, is stable, and is easily soluble in water. However, it is toxic and difficult to decompose. Dye waste can be handled using various methods: adsorption, photodegradation, electrophoresis, activated sludge, and so on (Eleta, et al; Iget, et al)[3-4]. According to LKIP of the Denpasar City Environment and Sanitation Service (2019), small and medium businesses are still having difficulties in building a wastewater treatment plant (IPAL) because it requires relatively expensive operational and maintenance costs [5]. A straightforward method that can be offered for the micro-scale textile industry is the adsorption method. The adsorption process can directly use biosorbents derived from agricultural waste or made into activated charcoal. There is an appropriate adsorption device for the micro and small-scale textile industry because the procedure is simple, has high adsorption power, and is easy to regenerate (Ma, et al., 2018) [6]. Researchers previously had successfully adsorbed methylene blue using sawdust [4], chitosan [6], bamboo [7], rapeseed, whitewood, seaweed [8], and mangroves [9]. In this research, activated charcoal was made from tabah bamboo, which grows in the Pupuan area of Bali [10].Production of activated charcoal with ZnCl2 activator-based tabah bamboo and study of the mineral composition has never been reported. The research was started by making charcoal, then activated with ZnCl2. Activated and inactivated charcoals are used to adsorb methylene blue from the solution. Adsorption parameters such as contact time, pH solution, initial concentration, and amount of adsorbent, including the Isotherm models also studied. Surface aspects such as functional groups, morphology, mineral composition, and pore volume were studied. They were using FTIR, SEM-EDX, and BET.

2. Experimental Section

2.1 Materials and Instrumentation

The materials used in this study were Tabah bamboo (*Gigantochloa nigrociliata*) taken from the Pupuan area, Tabanan, Bali. The chemicals used included: $ZnCl_2$ powder (p.a), methylene blue powder, 0.1 N HCl, 0.1 N NaOH, and KBr. The equipment used is a 200 mesh stainless filter, Thermolyne F48010 furnace, Shimadzu UV-Vis Spectrophotometer UV1800, FTIR Shimadzu Prestige-1, Jeol-JSM 6510 LA-SEM, SAA Quantachrome Novatouch LX -4

2.2 Procedure

2.2.1 Sample Preparation

Bamboo was cut to a size of 1 x 1 cm, washed, and dried in an oven at 110^0C. The carbonation process refers to the research of Manurung, (2019)[7].As much as 500 g of dry bamboo was carbonized for 90 minutes in a furnace at 600°C. The charcoal result was crushed and sieved with a 200-mesh sieve. Charcoal that passes the sieve is used in the following research. Charcoal was divided into two portions: inactivation labeled Bo and activated B_A. A total of 20 g of

Bo was put into a 100 mL beaker and then 50 mL of 0.03 M $ZnCl_2$ was added as an activator. The mixture was allowed to stand for 24 hours while stirring, then filtered and rinsed with distilled water until the pH was neutral.

2.2 Optimization Parameters

2.2.1 Contact Time

The contact time was determined by preparing a solution of 25 mL of 50 ppm methylene blue in a 100 mL beaker and 0.1 g of adsorbent. The mixture was stirred with a magnetic stirrer for 30, 60, 90, 120, 150, 180, and 240 minutes. Filtered and their absorbances were measured at the wavelength 650 nm (prepared before).

The Effect of mass adsorbent was determined by varied adsorbent mass from 0.1, 0.2, 0.3, 0.4, and 0.5g.The effect of the pH solution was studied by adjusting the pH through the addition of 0.1N HCl or 0.1N NaOH so that the pH of the solution was 2, 4, 6, 8, and 10, the next step carried out was determining the contact time. The amount of methylene blue absorbed at any time is calculated by equation 1 and in equilibrium with equation 2. The efficiency of adsorption by equation 3.[11]

$$Qt = \frac{(Co-Ct)V}{w} \quad (1)$$

$$Qe = \frac{(Co-Ce)V}{w} \quad (2)$$

$$\text{Efficiency} = \frac{Co-Ce}{Co} \text{ x100 \%} \quad (3)$$

$$Qe = \frac{qm.Kl.Ce}{1+Kl\ Ce} \rightarrow \frac{Ce}{Qe} = \frac{1}{Qm.Kl} + \frac{Ce}{Qm} \quad (4)$$

$$Qe = K_f\ .Ce^{1/n} \rightarrow \ln Qe = \ln Kf + \frac{1}{n} \ln Ce \quad (5)$$

2.3 Isotherm Pattern

Adsorption isotherm data is needed to determine the adsorption capacity and type of isotherm, which is usually checked against the Langmuir and Freundlich (equations 4, and 5) models[12]. This is carried out by preparing a solution of 25 mL of methylene blue, and 0.2 g of adsorbent at optimum conditions and equilibrium. The mixture was stirred with various concentrations of 50, 100, 150, 200, 300, 500, and 1000 ppm, then according to procedure 2.1.Plot between Ce/Qe vs Ce or lnQevs ln Ce (equations 4 and 5)in the form of a straight line.

2.4 The Effect of Temperature

The effect of temperature on the adsorption capacity was carried out by preparing a solution of 25 mL of 50 ppm methylene blue in a 100 mL beaker glass and 0.2 g charcoal Bo. The mixture was soaked in a water bath with temperature variations of 30, 45, and 60^{0}C, then followed procedure 2.1.The same treatment is carried out on activated charcoal, BA.

2.5 Surface Characterization

The functional groups of Bo and B_A charcoal were analyzed using FTIR, and the surface image, and composition of metal oxides were analyzed with SEM-EDX. The surface area was tested with BET

3. Discussion and Result

A total of 1000 g of carbonized dry bamboo produces 227.62 g of charcoal and the yield is 22.76%.The yield obtained depends on the type of raw material, pyrolysis temperature, and time [13, 14]. Figure 1 shows that the adsorbents Bo and B_A have the same optimum contact time of 90 minutes. The adsorption efficiency of 94.33% and 96.94% with adsorption capacities of 11.79 mg/g and 12.12 mg/g respectively. Gulec *et al.* (2020), stated that seaweed charcoal has an optimum contact time of 60 minutes and absorption efficiency for methylene blue about 94%.[8]

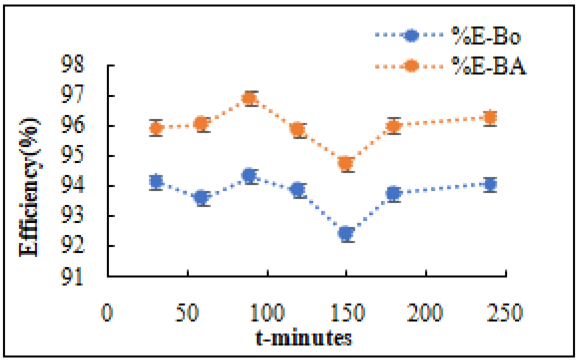


Figure 1: Relationship between efficiency and times

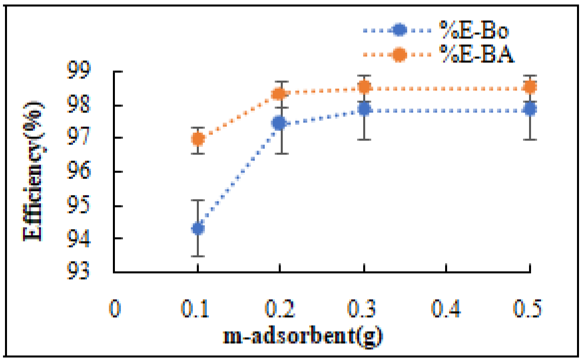


Figure 2: Relationship between efficiency and adsorbent mass

Based on Figure 2, an increase in the adsorbent's mass increases adsorption efficiency for both charcoals, Bo at a mass of 0.1-0.2 g has an increase in efficiency from 94.3% to 97.4%, and B_A from 96.6% to 98.3%. The increase in mass at 0.3 and 0.5 g did not give a significant change and tended to be stable. Increasing the adsorbent mass gives a very good adsorption effect because will increase the surface area and active sites and increase the conglomeration of the adsorbent [13, 15]. This is presumably due to the adsorption-desorption process that occurs continuously so that the absorption effect is not possible to reach 100%.Zakaria et al. (2021) showed that the adsorption of methylene blue increased from 55.3 to 97.3% with an increase in the adsorbent dose of 0.5-1.0 g[9]. Then the mass of the adsorbent used for further research is 0.2 g. The contribution of pH to adsorption capacity is presented in Figure 3.Adsorption of methylene blue dye at low pH was relatively low, while at high pH, the adsorption capacity of quite large.

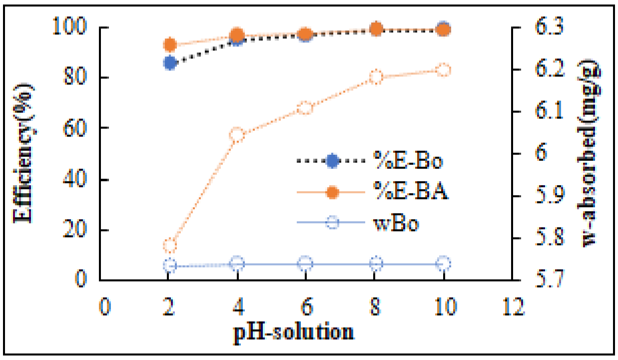


Figure 3: Correlation between efficiency solution with efficiency Bo (blue), BA (red) as well as

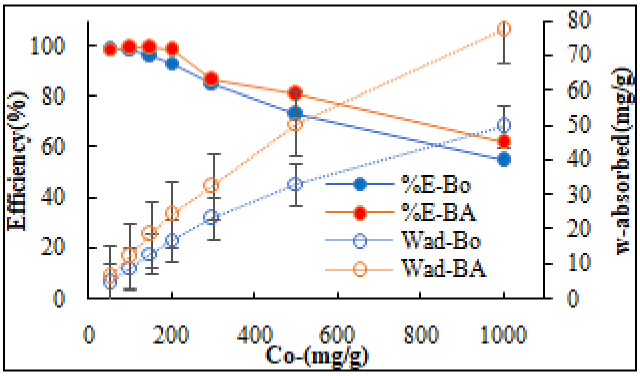


Figure 4: Correlation between concentration efficiency to pH- absorbed dye Bo (green), and BA (red).

At acidic pH, the content of H^+ ions in the solution is high enough so that it easily diffuses onto the surface of the adsorbent becomes positively charged, and will repel methylene blue ions from a solution that also has the same charge. The alkaline pH conditions, especially at a pH greater than 8 the surface of the adsorbent to be surrounded by OH^- ions resulting in an electrostatic attraction force with cationic methylene blue ion. The optimum condition was reached at pH 10 with an efficiency of 98.70% for Bo and 99.14% for B_A. This is in accordance with the research of Alam *et al*. (2022), where at a pH of 5.7 banana leaf ash had an adsorption capacity of methylene blue of 143.55 mg/g and increased to 166.97 mg/g at a pH of 11.7[13]. In this research, the efficiency of methylene blue absorption decreases due to an increase in the initial concentration of dye. When the concentration of 100 mg/g, the adsorption efficiency of Bo and B_A is 99.05% and 99.18%, respectively. At a methylene blue concentration of 200 mg/L, Bo efficiency decreased to 93.53% and BA tended to stable with an efficiency of 98.59%. Even at a concentration of 1000 mg/g, the efficiency of Bo and BA charcoal decreased to 54.78% (68.48 mg/g) and 62.10% (77.62 mg/g) respectively (Figure. 4). The activation process can increase the adsorption capacity by 13.35% (9.14 mg) only. The efficiency of adsorption decreases as the initial concentration increases because the high abundance of adsorbate in the solution causes interactions among adsorbates and blocks dye diffusions to the surface, so that the amount of dye that reaches the surface is small. At the same time, the abundance of dye in the solution also increases, as a result, efficiency decreases. (Tay, et al., 2022) [11].

Adsorption Isotherm

The linearity correlation value R^2 at Bo indicates that the adsorption process of bamboo charcoal Bo can take place in a Langmuir or Freundlich manner, whereas in BA it tends to follow the Langmuir adsorption process. Table 1 shows that the adsorbents Bo and BA comply with the Langmuir isotherm rules marked with the correlation coefficient R^2 values of 0.9766 and 0.9757 respectively. This is also supported by the value of the equilibrium parameter (R_L), with a value of $0 < R_L < 1$, which means it is more in line with the Langmuir model. Similar results were also reported by Gulec, et al. (2022), using seaweed charcoal [8]. The adsorption capacity (qm) of Bo and BA was 70.22 mg/g, and 79.24 mg/g respectively. The difference value between qexp and theoretical (qm) for Bo is about 1.75 mg (2.55%) and 1.62 mg (2.09%) for BA charcoal. The value of *n* in Table 1 shows that the Bo and BA adsorption process can occur according to Freundlich's rule which forms a multilayer layer on the adsorbent surface. The value of the heterogeneity factor *n* indicates that the adsorption process is physical. In this research, the value of n > 3 indicates that the interaction intensity between surface and methylene blue is intensive, supported by a large K_F value (Table-1).

Table 1: Data Isotherm Pattern Langmuir and Freundlich for Bo and BA Charcoals

Adsorbents Charcoal		Langmuir		
	qm (mg/g)	K_L (L/mg)	R^2	R_L
Bo	70.224	0.0369	0.9719	0.0264-0.3515
BA	79.239	0.0507	0.9709	0.0193-0.2829
		Freundlich		
	n	Kf (mg/g) $(L/mg)^{1/n}$	R^2	
Bo	3.083	9.6141	0.9475	
BA	3.334	12.9544	0.8676	

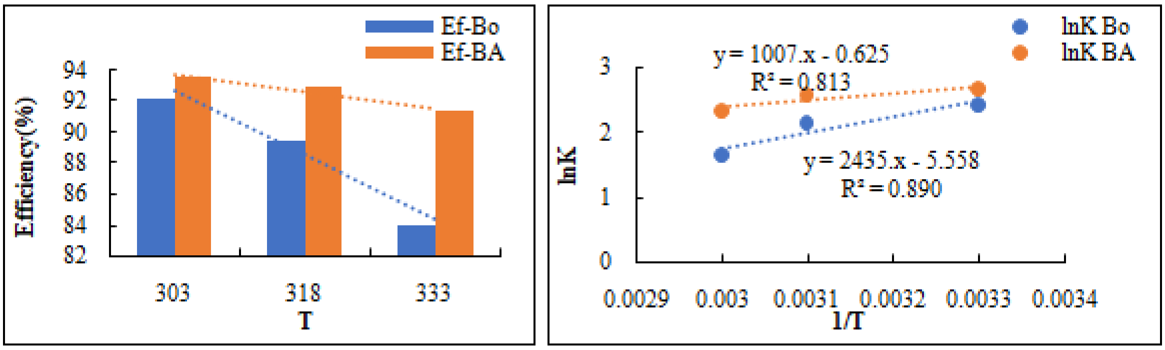
Adsorption thermodynamic parameters were obtained by measuring the adsorption capacity at various temperatures, namely from 30.45 and 60 at the optimum conditions. The adsorption equilibrium constant is considered to be the ratio between the adsorbed methylene blue dye and the remaining in the solution. Van Hoff (equation 6) and Gibbs (equation 7) determine the thermodynamic parameter value. The combination of equations 6 and 7 at equilibrium produces equation 8. [16- 18]

$$\Delta G = \Delta G^o + R.T \ln K \rightarrow \Delta G^o = - R.T \ln K \quad (6)$$

$$\Delta G = \Delta H^o - T.\Delta S^o \quad (7)$$

$$\ln K = \frac{-\Delta Ho}{RT} + \frac{\Delta So}{R} \quad (8)$$

By drawing a curve between lnK and 1/T, and using equation 8 then the enthalpy (ΔH^o), entropy (ΔS^o), and Gibbs energy (ΔG^o) can be determined. The results obtained are shown in Figure 5 and Table-2



(a) (b)

Figure 5: Correlation between Temperature and Efficiency (a) and lnK to 1/T (b) for Bo and BA Charcoals

Table 2: The Equilibrium and Thermodynamic Data of Bo and BA Charcoals

Absorbent charcoal	T (Kelvin)	K	lnK	ΔH (KJ/mol)	ΔS (J/mol)	ΔG (KJ/mol)
Bo	303 318 333	11.41 8.51 5.23	2.43 4.14 1.65	-20.2504	-46, 2142	-6.2518 -5.6578 -4.5681
BA	303 318 333	14.50 13.14 10.39	2.67 2.58 2.34	-8.3690	-5.200	-6.7934 -6.8211 - 6.4784

The adsorption of Bo and BA charcoals on methylene blue from the solution is exothermic, so an increase in temperature will slow down adsorption and accelerate desorption, which is marked by a reduced equilibrium constant value. As a result, efficiency will decrease. The low value of reaction energy about20 KJ/mol indicates that Bo and BA charcoals' adsorption of methylene blue isa mixture of physical and chemical adsorption controls.[16, 17]

Characterization of Charcoals

Analysis of functional groups for Bo and BA charcoals using FTIR showed similar spectra, meaning that Bo and BA have similar functional groups (Figure 6). The high temperature in the heating process causes a cracking process, which changes the aliphatic structure to a united ring structure through aromatization which supported of activating $ZnCl_2$ (Chen et al., 2020)[19], but not change functional groups because the same based charcoal, stated by Mustofa, et.al., (2023) [16]. In this research found functional groups that is OH at 3500 cm^{-1}, C=C is loosely aromatic at (1573 and 1577) cm^{-1}, C=O carbonyl at (1650-1700) cm^{-1}, is tenuous aromatic at (1253 and 1261) cm^{-1}, C=C bending at (885, 808 and 883, 802) cm^{-1} and C-H aromatic bending at absorption range (675-445) cm^{-1}.

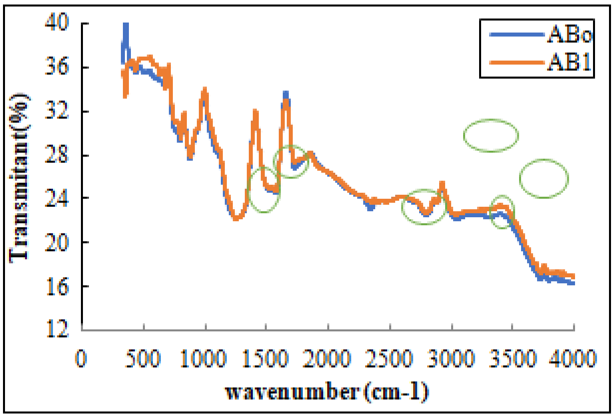


Figure 6: FTIR spectra of Bo (blue), BA (red)

SEM-EDX analysis Bo, BA, and BA1 charcoals to determine morphology surfaces and chemical composition like Figure 7 and Table 3.

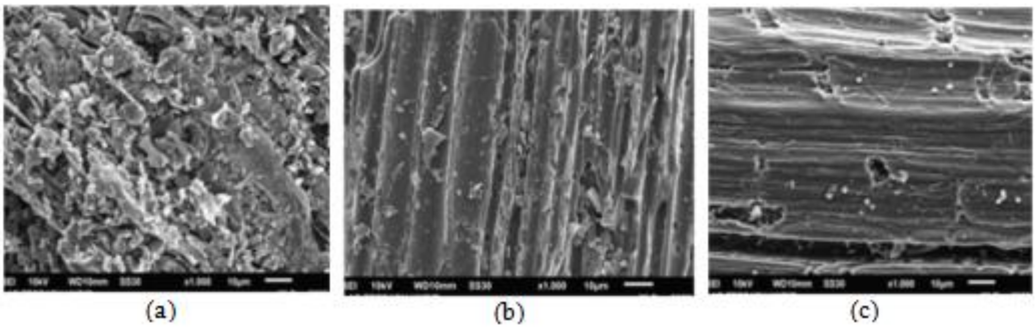


Figure 7: Spectrogram Surface morphology of Bo (a), BA (b), and BA1 (c) at 1000x magnification by SEM-EDX

Table 3: Chemical composition of Bo, BA, and BA1 samples in SEM-EDX analysis

Adsorbents	Compositions (%)						
	C	Na_2O	K_2O	MgO	SiO_2	Cl	ZnO
Bo	85.79	0.18	10.42	0.31	2.39	0.92	-
BA	96.96	-	1.87	-	0.88	-	0.28
BA 1	98.36	-	1.17	-	0.42	-	0.05

- = undetected ; BA1 is BA after adsorption.

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In Figure 7. It can be seen that Bo still has a lot of impurities covering the surface of the charcoal (a), in contrast to the clean surface morphology of BA (b). This means that activation with $ZnCl_2$ solution is able to clean impurities on the surface of the charcoal [13, 14]. SEM image shows BA1

(c) forming a thin layer that covers the surface of the charcoal and is thought to be a methylene blue dye that is absorbed evenly on the surface of the charcoal. There are several points on the surface of BA1 containing lumps which are thought to originate from methylene blue solution which forms multilayer layers due to saturation on the charcoal surface. The solution forms multilayer layers due to saturation on the charcoal surface. Based on Table 3, it is known that the results of the EDX test from Bo have far more minerals compared to BA and BA1. The results of activation with $ZnCl_2$ are proven to be able to remove mineral impurities on the surface of the adsorbent. The presence of Zn was able to remove Na and Mg oxides and chloride, reducing the levels of Si oxide from 2.39% to 0.88% and K oxide from10.42% to 1.17%. The loss of mineral content during the activation process is thought to be due to a substitution reaction by Zn ions, causing other minerals to dissolve, as the state of TaY, C.C, et al, [11]. The increase in the carbon content in BA1 charcoal to 98.36% was due to methylene blue molecules on the surface with the molecular formula $C_{16}H_{18}N_3SCl$. After adsorption, Zn oxide levels in the charcoal also decreased, presumably because Zn dissolved the other elements into the solution. The charcoal and methylene blue reaction involves the ionic and van der Waals process [16].The test results using BET obtained a surface area value of BA of 5.104 m2/g and an average pore size of 2.364nm, while Bo was 0.188 m2/g and an average pore size of -29.636 nm. The activation process can increase the surface area by 2, 615%. The high surface area causes activated charcoal better than inactivated charcoal Bo in the methylene blue adsorption. The activation process also increases the surface area as well as the diameter and pore volume, as reported by Negara, et al. (2018) activating bamboo charcoal with H_3PO_4, [21]and Meng, et al. (2023) activating bamboo charcoal with ammonium acetate particles.[22]

4. Conclusion

Tabah bamboo can be used as a potential source of charcoalas an adsorbent for methylene blue from solution. Activation of charcoal with ZnCl2 produces charcoal with better quality rather than inactivation. The optimum adsorption conditions were a contact time of 90 minutes, an adsorbent mass of 0.2 grams, pH 10, and adsorption capacities were 79.239 mg/g and 70.224 mg/g for activated and inactivated charcoals respectively. The adsorption isotherm for activated charcoal follows Langmuir while inactivated charcoal applies to Langmuir or Freundlich. The functional groups of activated and inactivated charcoals are the same: contain OH, -CO, aromatic, C=C, and C-O groups. Activator $ZnCl_2$ is able to improve the quality of charcoal and reduce mineral content through substitution reactions. The carbon contents in activated and inactivated charcoals are 96.96% and 85.79% respectively, and the surface area of each charcoal is 5.104 m2/g and 0.188 m2/g.

Thank You

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