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*by Widi Astuti*

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# Preparation of Activated Carbon from Cassava Peel by Microwave-Induced H<sub>3</sub>PO<sub>4</sub> Activation for Naphthol Blue-Black Removal

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**Abstract.** The use of dyes in textile and batik industries can pose a serious environmental problem when the effluent is discharged into the stream without previous treatment. Adsorption is one of the efficient and reliable methods for dyes removal. In this sense, biomass-based activated carbon has been considered as a low-cost adsorbent. The purpose of this work was to prepare a microwave-induced H<sub>3</sub>PO<sub>4</sub> activated carbon from cassava peel for naphthol blue-black removal. The surface morphology of the activated carbon was analyzed using a scanning electron microscope while the presence of the functional group in the activated carbon surface was recorded using Fourier transform infrared spectrophotometer (FTIR). In the range of H<sub>3</sub>PO<sub>4</sub> concentration studied (40, 50, and 60%), the best-activated carbon was obtained by microwave-induced H<sub>3</sub>PO<sub>4</sub> activation using H<sub>3</sub>PO<sub>4</sub> concentration of 60%. In this sense, 99.7% of naphthol blue-black was adsorbed. The highest naphthol blue-black uptake was found at pH 3.0 and the equilibrium was attained after 180 min. The equilibrium data of naphthol blue-black adsorption onto cassava peel activated carbon was best fitted with the Freundlich isotherm with  $K_F$  of 3.871 and  $n$  of 0.659.

## INTRODUCTION

At present, the growth of textile and batik industries in Indonesia is increasing rapidly. The presence of dyes, which are extensively used for the dyeing process, can pose a severe environmental problem when the effluent is discharged into the stream without previous treatment. The dyes consist mainly of the aromatic structure with functional groups, such as carboxylic (-COOH), amines (-NH<sub>2</sub>), and azo (-N=N-), that is stable to photodegradation and biodegradation [15]. Additionally, several dyes are very toxic, carcinogenic, and mutagenic. Compared with conventional dye wastewater treatment technologies such as coagulation [2], aerobic and anaerobic microbial degradation [3,4], and ion exchange [5], adsorption is one of the most effective and economical methods for dye removal due to its simple design and easy to operate [6]. In this sense, the effectiveness of dye removal depends on the character of adsorbent used. Recently, biomass-based activated carbon is considered as an effective adsorbent due to its abundant availability and high functional group content. One of biomass is cassava peel containing hemicellulose (37.86%), cellulose (13.75%), and lignin (9.14%), which enable it as a precursor in the preparation of activated carbon (AC).

Generally, the preparation of biomass-based AC comprises two stages. First, the thermal decomposition of precursor through the carbonization process at a lower temperature (<800°C) to convert agricultural waste into char. Second, the activation process to increase char porosity [7]. Physical activation processes use high temperature with steam or carbon dioxide as an activating agent. While in chemical activation processes, chars are impregnated with chemical reagents such as NaOH [7], KOH [7], ZnCl<sub>2</sub> [8] and H<sub>3</sub>PO<sub>4</sub> [9]. Among them, H<sub>3</sub>PO<sub>4</sub> has been preferred due to it has low toxicological constraints and no metal residue left. In addition, H<sub>3</sub>PO<sub>4</sub> allows the developing of mesopores and micropores, thereby increasing the effectiveness of the adsorption process. In the chemical activation, conventional heating using an electric furnace was adopted. Recently, microwave heating has been considered a promising technique to replace conventional heating due to it has rapid temperature rise and energy saving. The purpose of this study was to prepare a microwave-induced H<sub>3</sub>PO<sub>4</sub> activated carbon from cassava peel

for naphthol blue-black removal. In this sense, the effect of  $H_3PO_4$  concentration on the adsorption capability of the activated carbon for naphthol blue-black was investigated.

## MATERIALS

Cassava peel used as a precursor was obtained from a traditional market in Gunungpati, Semarang, Indonesia. The cassava peel was washed and heated in an electric oven at  $110^\circ C$ . The dried sample was further blended to a powder and screened to obtain the fraction with particle size  $0.625 < D_p < 0.149$  mm [10]. The powder was entered into a furnace for the carbonization process at  $700^\circ C$  for one hour. While, all solutions including naphthol blue-black (supplied from U.D. Indrasari),  $H_3PO_4$  (supplied from Merck, Germany) and HCl (supplied from Merck, Germany) were prepared using distilled water to the required concentration. Naphthol blue-black was used as adsorbate, while  $H_3PO_4$  and HCl were used for chemical activation in the activated carbon preparation.

## METHODS

### Activated Carbon Preparation

In this research, the chemical activation process using  $H_3PO_4$  was carried out. Char (10 g) was added to a  $120\text{ mm}^3$  solution of  $H_3PO_4$  with different concentrations (40%, 50%, and 60%) and stirred for 24 h to ensure  $H_3PO_4$  access to the interior of char. Afterwards, the mixtures were filtered using Whatman paper (No.5), and the resulting solids were dried in an electric oven at  $110^\circ C$  for 24 h [11]. A weighed amount of impregnated char was further placed in an alumina reactor and inserted in a microwave oven (2.45 GHz, 600 W). The sample was heated for 7 min under a flow of  $N_2$  of  $0.25\text{ dm}^3/\text{min}$ . The sample was then leached with 0.1 M HCl and washed with distilled water until pH 7. After a final drying stage in an electric oven, the AC was considered ready to characterize and use in the naphthol blue-black removal.

### Characterization

The specific surface area of AC was characterized through adsorption of  $N_2$  at 77 K using an automated gas sorption apparatus (Quantachrome, USA) with BET (Brunauer-Emmett-Teller) method. The morphological characteristic of AC was determined using a scanning electron microscope (SEM) (Phenom Pro X Desktop, UK). The functional groups on the AC surface were identified using Fourier transform infrared spectroscopy (FTIR) (Spectrum 100, PerkinElmer, USA) at wave number  $400\text{-}4000\text{ cm}^{-1}$ .

### Adsorption Performance

The obtained char and AC were used in the naphthol blue-black removal. The experiment was carried out in a batch mode at room temperature (approximate  $25^\circ C$ ). Firstly, a certain amount of adsorbent (0.2 g) was mixed with  $25\text{ mm}^3$  naphthol blue-black solution (concentrations  $100\text{ mg/L}$ ) in a 100 mL Erlenmeyer. The adsorption was carried out at various pH (1, 3, 5, 7, 9, and 11) with added 0.1 N HCl or 0.1 N NaOH. The mixture was then placed in the shaker and shaken at 200 rpm for 180 min. After the adsorption time was reached, the solution was filtered, and the filtrate was analyzed for residual naphthol blue-black using a UV-Visible spectrophotometer (Model Genesys 10 UV, Thermo Scientific, USA) at  $\lambda_{\text{max}} 595\text{ nm}$ . The effect of contact time on the amount of naphthol blue-black adsorbed was investigated at different contact times (10, 20, 30, 40, 50, 60, 90, 120, 150, and 180 min) at pH of 3 and concentration of  $100\text{ mg/L}$ . While, adsorption equilibrium was studied by varying concentrations (5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 200, 300, 400, 500 mg/L) at pH 3 for 180 min. The surface concentration of naphthol blue-black at equilibrium (mmol/g) was calculated by Eq. (1) [12].

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (1)$$

Where  $C_0$  is initial naphthol blue-black concentration in the liquid-phase ( $\text{mmol/dm}^3$ ),  $C_e$  ( $\text{mmol/dm}^3$ ) is naphthol blue-black concentration in the liquid-phase at equilibrium,  $M$  (g) is adsorbent dose, and  $V$  ( $\text{dm}^3$ ) is the volume of naphthol blue-black solution.



## RESULTS AND DISCUSSION

### Textural and Surface Characterization

The morphology of char and AC characterized using a scanning electron microscope (SEM) is described in Figure 1. Some fragments with small pores were observed in the char surface (Figure 1(a)) indicating the existence of tarry substance residues formed during the carbonization process. While activation with  $\text{H}_3\text{PO}_4$  60% resulted in the creation of more pores, widening the existing pores, and opening the blocked pores due to volatile matter removal [13]. It was indicated by the well-developed pores, which can be clearly observed on the AC surface, as described in Figure 1(b).  $\text{H}_3\text{PO}_4$  molecules diffused into the small pores of the char and increased the  $\text{H}_3\text{PO}_4$ -char reaction via acid hydrolysis processes. In this sense,  $\text{H}_3\text{PO}_4$  hydrolyzed the glycosidic linkage in cellulose and hemicellulose along with the aryl bond cleavage in lignin [14]. The char and AC have a specific surface area of 17.14 and 257.76  $\text{m}^2/\text{g}$ , respectively.

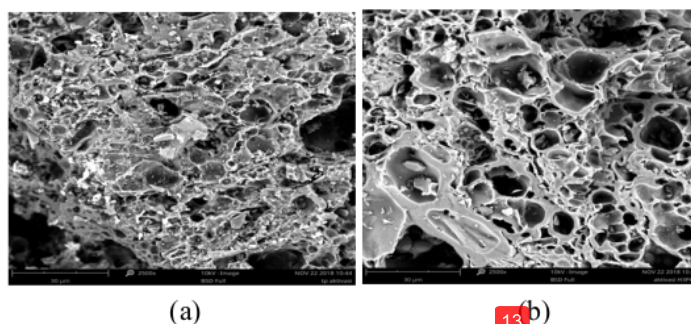


FIGURE 1. SEM micrographs of (a) char and (b) activated carbon

The functional groups on the char and activated carbon surface were characterized using FTIR spectra, recorded between 4000-400  $\text{cm}^{-1}$ , as can be seen in Figure 2. The spectra of char (Figure 2(a)) shown the presence of absorption peaks at 3843.28 and 3740.25  $\text{cm}^{-1}$  indicated the O-H stretching of hydroxyl groups from phenols, carboxyls or alcohols derived from cellulose and lignin [15]. The absorption peak at 2365.84  $\text{cm}^{-1}$  indicated the presence of C=C vibrations in alkenes and methylene groups [15]. While, a high band at 1526.91  $\text{cm}^{-1}$  may be attributed to the presence of C=C stretching vibrations in aromatic rings, especially for carbon material. The absorption peak at 1042.1  $\text{cm}^{-1}$  indicated the presence of C-O stretching groups in primary alcohols [15]. After the activation process with  $\text{H}_3\text{PO}_4$  60% (Figure 2(b)), the O-H group shifted from 3843.28 to 3841.83  $\text{cm}^{-1}$  and from 3740.25 to 3741.03  $\text{cm}^{-1}$ . Peak around 2365.84  $\text{cm}^{-1}$  shifted to 2369.08  $\text{cm}^{-1}$  while the peak at 1526.91  $\text{cm}^{-1}$  shifted to 1529.35  $\text{cm}^{-1}$  followed by a decrease in intensity, which indicated the decrease of lignin and cellulose content. Figure 2(b) has also confirmed the existence of a new peak around 1250-1170  $\text{cm}^{-1}$  assigned to the O-C stretching vibrations in the P-O-C linkage, hydrogen-bonded P=O, and P=OOH of phosphorus species after the activation process.

### Adsorption Performance

Solution PH affects the surface charge of the adsorbent. Therefore, it is one of the most significant factors in controlling the adsorption process on activated carbon. Adsorption of naphthol blue-black onto activated carbon prepared with  $\text{H}_3\text{PO}_4$  40, 50, and 60% is described in Figure 3(a). In the range of pH studied (between pH 1 and 11), adsorption of naphthol blue-black onto AC was pH-dependent, especially between pH 5 and 11. At low pH, the AC surface has a net positive charge. The increase in pH causes a decrease in the net positive charge and an increase in the net negative charge. Naphthol blue-black is an anionic dye having a negative charge. It causes the electrostatic attraction between active sites (i.e., functional groups) in the AC surface and adsorbate molecules (i.e., naphthol blue-black) at pH 3. While at pH>5, the electrostatic repulsion between the active sites and naphthol blue-black molecules prevails. Therefore, it can be concluded that the pH value of 3 was selected for naphthol blue-black adsorption onto cassava peel activated carbon. On the other hand, the amount of naphthol blue-black adsorbed by

activated carbon using  $H_3PO_4$  60% was higher than that of using  $H_3PO_4$  40% due to more pores created, although at pH 1-3 the difference was insignificant.

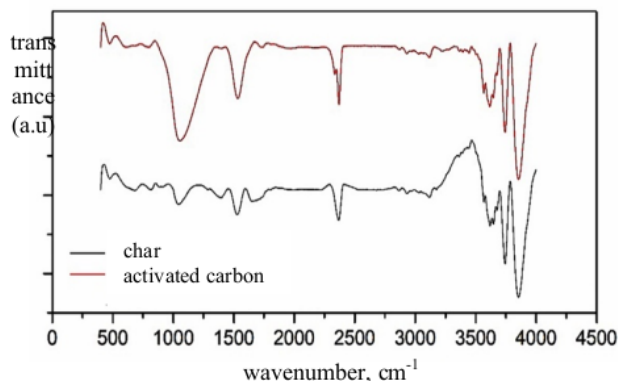


FIGURE 2. FTIR spectra of (a) char and (b) activated carbon using  $H_3PO_4$  60%

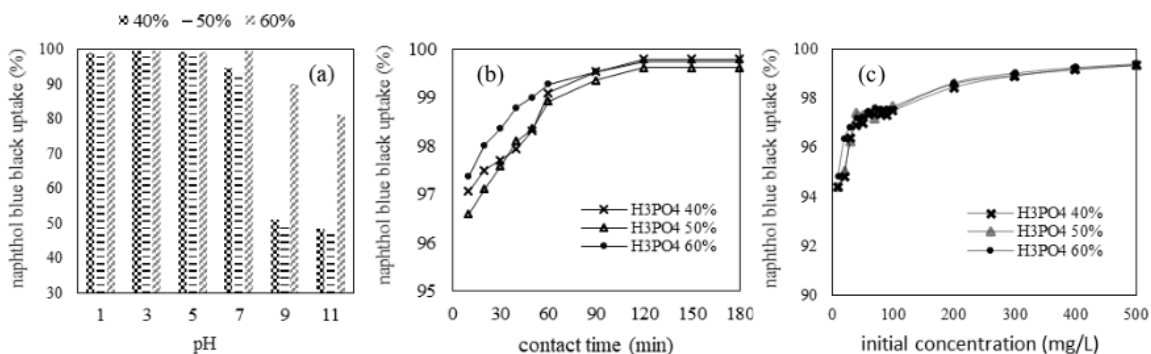


FIGURE 3. The effect of (a) pH (b) contact time and (c) initial concentration toward naphthol blue-black uptake at different  $H_3PO_4$  concentration (activated carbon dosage of  $0.2 \text{ g}/25 \text{ mm}^3$  dye solution)

Figure 3(b) depicts the effect of contact time toward naphthol blue-black uptake. According to the results, a sharp naphthol blue-black reduction occurred in the first 60 min of contact time. It may be due to a large number of active sites were available for the adsorption process at the initial stage. Furthermore, the naphthol blue-black uptake tended to constant until the equilibrium was attained.

The effect of initial naphthol blue-black concentration on the naphthol blue-black uptake is presented in Figure 3(c). The dye adsorption increased from 94.11 to 97.6% with the increase in dye concentration from 10 to 90  $\text{mg}/\text{dm}^3$ , and then it gradually slowed down. The initial dye concentration provides a driving force to overcome all mass transfer resistance in the adsorption process.

### Adsorption Isotherm Study

The equilibrium of naphthol blue-black molecules in the solid (i.e. activated carbon) and liquid phase is described by adsorption isotherm that is important for adsorption system design. Langmuir and Freundlich isotherms are the two most common isotherms describing dyes adsorption in the liquid system. In contrast to the Freundlich isotherm that is based on the assumption of multilayer adsorption on heterogeneous surface energy, Langmuir isotherm is derived from monolayer adsorption with homogeneous surface energy. The Freundlich equation is formulated by Eq. (2) while the Langmuir model is defined by Eq. (3) (Table 1). Eqs. (2), and (3) were solved by optimizing the correlation coefficient between the value of  $q_e$  from experimental data and the value of predicted  $q_e$ .

from Eq. (2) or (3). Parameters in Eq. (2) (e.g.,  $K_F$  and  $n$ ) or Eq. (3) (e.g.,  $q_m$  and  $K_L$ ) were determined by minimizing the difference between the experimental data and the model predicted data using the solver add-in feature of Microsoft Excel software. The results are presented in Table 1. The nonlinear correlation coefficient ( $r$ ) for the Freundlich isotherm was closer to unity (0.9779) than that of Langmuir isotherms (0.7222). It indicated that the Freundlich model best fitted to the equilibrium data.

TABLE 1. Isotherm parameters for naphthol blue-black-activated carbon system

Isotherm model	Value of the parameters						
	$q_e = K_F C_e^n$	(2)	$r$	$K_F$	$n$	$q_m$	$K_L$
Freundlich	$q_e = q_m \frac{K_L C_e}{1 + K_L C_e}$	(3)	0.978	3.871	0.659	-	-
Langmuir	$q_e = q_m \frac{K_L C_e}{1 + K_L C_e}$	(3)	0.811	-	-	107.385	-

$K_F$  [(mmol/g)(mmol/dm<sup>3</sup>)<sup>-n</sup>] is the Freundlich constant represented the adsorption capacity when  $C_e$  equals 1,  $n$  is the Freundlich constant indicated the degree of dependence of adsorption on equilibrium concentration,  $q_m$  (mmol/g) is the amount of naphthol blue-black adsorbed at complete monolayer coverage and  $K_L$  (dm<sup>3</sup>/mmol) is a Langmuir constant related to the adsorption heat [17]

## CONCLUSION

Activated carbon (AC) was prepared successfully from cassava peel with H<sub>3</sub>PO<sub>4</sub> 60% as activating agent. The optimum condition occurred at pH 3, and the equilibrium time was reached after 180 min. The Freundlich isotherm was more appropriate to the equilibrium data than the Langmuir isotherm.

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