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PREPARATION OF ACTIVATED CARBON FROM PALM SHELL BY CHEMICAL ACTIVATION WITH KOH AS AN ADSORBENT DYE

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Abstract

Activated carbon is a well known as porous material, with large specific surface area, which is useful in adsorption of both gases and solutes from aqueous solution. In this research, prepraration of activated carbon was through chemical activation of palm shells using KOH as an activator. Preparation of carbon from palm shell were the first carbonized in a furnace at temperature of 400 - 500 °C. Carbon is soaked in KOH activator solution for 8 hours and then carbonized at a temperature of 600 - 800 °C. The activated carbon obtained was characterized by XRD, SEM and FTIR. Characterization using SEM shows that activated carbon has pores and using FTIR shows the presence of several functional groups such as hydroxyl (-OH), carboxyl (-COOH), carbonyl (-C=O) that can be used for adsorption. Adsorption studies were carried out using batch experiments with synthetic naphthol and remazole dye solutions. These results indicate that palm shell activated carbon has the potential used as an adsorbent for removing naphthol and remazole dyes from the solution.

INTRODUCTION

Dye waste release from various industries into water bodies will have a negative impact on aquatic life because dyes are toxic and carcinogenic [1]. As a result, the presence of dye waste in environmental water bodies is an increasing concern for environmentals. The long-term sustainable and efficient dye waste treatment methods must be created to eliminate this problem. Dye wastewater must be treated before disposal to minimize its negative impact on the environment and living creatures. Adsorption found to be very effective and cheap method among the all available dye removal methods. Dyes from the industrial waste water effluents are effectively separated by using adsorbent such as activated carbon however its cost restricts the use in large scale applications [2].

It is important to treatment the removal dye waste before it is released into body of water. The stability of synthetic dye molecules is different from natural dyes. They can be classified into cationic, anionic and non-ionic dyes due to their properties [3]. Remazol yellow and indigoszol are cationic dyes that are commonly used and are the main dyes that make up wastewater from the textile and batik industry. Dyes containing waste can hamper the survival of aquatic ecosystems because they are carcinogenic and difficult for the natural degradation process [4-5].

There are several technologies for processing textile dye waste, such as coagulation, membrane separation [6], oxidation and adsorption processes [1]. Adsorption using activated carbon is a preferred separation technique compared to physico-chemical methods for dye removal because it is rich in surface active sites and has wide pores [7]. However, commercial activated carbon precursors are non-renewable, while regeneration of used activated carbon is relatively expensive. Therefore, the search for new alternative carbon precursors that are abundantly available and low cost must be continue. One promising candidate is palm shells.

Activated carbon is carbon that has a high surface area and broad porosity so it can be used for wastewater and industrial gas treatment applications. Activated carbon is an adsorbent with a large porous surface area, has pores with a controllable structure, thermalo stability and low acid/base reactivity [8].

Chemical activation has been shown as a efficient method to obtain carbons with high surface area and narrow micropore distribution. Although it is a frequently used to prepare activated carbons, the general mechanism of chemical activation is not well understood, and the various interpretations found in literature underline the process complexity. In general terms, chemical activation by alkalis consists in solid–solid or solid–liquid reaction involving the hydroxide reduction and carbon oxidation to generate porosity [9].

The aim of this research is the preparation of activated carbon from palm shell with various activator agent KOH. The structural, functional and elemental characterization of the adsorbent characterized using SEM-EDX, FTIR, XRD.

EXPERIMENT

Material

The material used are palm shells, HNO_3 (Merck), KOH, NaOH, HCl (Merck), buffer solution pH 4.7 and 10, Remazol yellow, Indigozo, distilled water. All reagen used were of analtycal grade and and used without any further purification.

Instrumentation

Spectrophotometer Shimadzu, Termofisher Scanning Electron Microscopy (SEM), Benchtop pH meter, Xpert Pro Powder Pw3040/60 PANalytical X-Ray Diffraction (XRD), FTIR 600 series Agilent Technologies, Wish Term Furnace, AE Adam Equipment Analytical Balance, Sieve mesh 10 merk ABM.

Procedure

Sample Preparation

Palm shells were collected from palm oil mills Ma. Jambi District. It is washed with tap water to remove dirt and washed again with distilled water, dried in the sun for 4 hours to reduce humidity, dried in an oven for 48 hours at 110 °C to remove water. Dried palm shells were ground into small pieces (5 mm) using a grinder and stored in a closed container [10].The first preparation of activated carbon : typical carbonization run began by changing 100 g of impregnated sample in the reactor and heated up to the carbonization temperature in flowing stream of nitrogen [11]. The temperature of reactor was increased at the rate of 10 °C/min, until it reached the final carbonization temperature. The carbonization temperature was varied from 600-800 °C with activation duration of 2 h, finally with cold distilled water to remove residual chemicals [12-14]. Activated carbon tested for analysis proximately (ash content, water content, yields and pH).

Carbon Chemical Activation

The carbon chemical activation of carbon in KOH can follows this procedure : preparation activated carbon was done by soaking carbon powder in KOH solution (0, 2, 4, 6 dan 8M) with a weight ratio of 1:10. The biochar soaked in KOH solution was stirred and heated using a hot plate at a temperature of 80 °C until evenly mixed to form a slurry for 2 hours. Heating was stopped, and the slurry was left for 24 hours. The slurry is filtered to obtain activated carbon with KOH [15]. Activated carbon marker are (AC-1, AC-2, AC-3, AC-4 and AC-5).

Characterization of Activated Carbon (AC) KOH Activator

Characterization of AC-KOH activator was carried out using FTIR to analyze the functional groups of AC, Scanning Electron Microscope Energi Dispersive X-Ray SEM-EDX to determine the surface morphology and elemental composition of AC, XRD used analyzed the structure of crystalline or amorphous materials [14].

Remazol and Indigozol Dyes Adsorption Process

The efficiency and adsorption capacity of remazol and indigozol dyes using AC-KOH activator were carried out using the following treatment. A total of 20 mL of each dye solution of indigozol and remazol at an initial concentration of 10 mg/L was prepared in a 150 mL Erlenmeyer flask, then mixed with 0.1 g of AC-KOH each. The solution was shaker at 150 rpm for 15 minutes. The solution was filtered using Whatman filter paper, each filtrate from the dye was analyzed using UV-Vis spectrophotometry at respective wavelengths for naphthol and remazole dyes [15]. The dye of adsorption parameter that will be studied is the effect of pH studied at pH 3-9, contact time 15-200 minutes, initial dye concentration of 10 mg/L-250 mg/L, and adsorbent mass AC-KOH.

Adsorption of Remazol And Naphthol Dye

Samples of remazol and naphtol dye were prepared as much as 50 mL each into a 100 mL Erlenmeyer flask. Activated carbon adsorbent with a mass of 0.1 g was put into 50 mL of 10 ppm remazol solution. The adsorption process was carried out for 5 minutes at room temperature accompanied by stirring at a shaker speed of 200 rpm [16].

RESULT AND DISCUSSION

Preparation of Activated Carbon

Activated carbon (AC) is a carbonaceous substance produce from carbon-rich organic materials, including palm shells. The initial step preparation of activated carbon from palm shell was carbonation, the process of water evaporation and decomposition of several components of organic compounds contained in palm shells such as cellulose, hemicellulose and lignin [14]. The hemicellulose decomposition process takes place temperature a 250 - 400 °C, marked by the release of thin white smoke from the furnace chimney. The cellulose decomposition process begins at a temperature of 280 °C and ends at a temperature between 300 - 350 °C. This stage is characterized by the release of smoke that is thicker and darker in color, brownish black. The process of decomposing lignin begins at a temperature of between 300 - 450 °C [21]. The activated carbon was made using KOH activator with varying concentrations of 2, 4, 6, 8 M.

Table 1. Characteristics of activated carbon from palm shell.

Activated carbon treatment	Temperature (°C)	Yield (%)	Ash content	Water content	pH
AC-1	500	31	1.85	6.0	5.1
AC-2	500	38	2.35	5.2	4.9
AC-3	500	39.5	2.5	5.8	4.5
AC-4	500	44.5	2.85	6.1	4.2
AC-5	500	41.5	3.25	8.2	3.2

Table 1 shows the use of various KOH precursor variables as activator substances. According from **Table 1**, it can be seen that the amount of KOH present has a significant impact on the production of palm shell active carbon [1]. The results obtained varied between the range of 31 to 44.5%. **Table 1** shows that the production of palm shell activated carbon from palm shell that activated using with KOH increases with increasing KOH concentration. These results are in accordance with experiments on making activated carbon from lignocellulosic waste activated with KOH [2-4]. It can be seen that if base concentration is increased, mass transfer into the substrate will be increased. This will speed up the activation process, resulting in a higher yield percentage. In **Table 1**, it is observed that AC-1 is 31% while AC-4 shows the highest yield percentage of 44.5%. However, high amounts of KOH will cause damage to the carbon surface and destruction of the carbon surface, there by reducing the percent yield of active carbon. This is because the chemical activator alkaline can be use to maintain carbon formation. At high alkaline concentrations, the polymer structure of palm shells is damaged at high temperatures and most of the non-carbon elements are released.

Characterization of FESEM

Field Emission Scanning Microscopy (FESEM) is widely used to examine the surface morphological structure of activated carbon. The morphology of activated carbon palm shell show that in **Figure 1**.

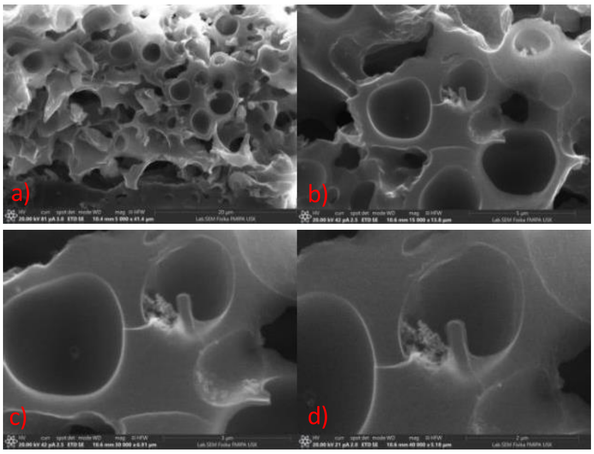

Figure 1. SEM images of activated carbon using on KOH as an activator: (a) 2M, (b) 4M, (c) 6M, (d) 8M.

The scanning electron microscopy (SEM) images of surface morphology of activated carbon palm shell using KOH activator shows that in **Figure 1.** **Figure 1** presents surface morphology activated carbon, where the higher KOH concentration, surface pores of activated carbon will increase with increasing concentration KOH. This is because particles in carbon are broken into smaller parts. A good pore structure can facilitate electrolyte ions to diffuse on the carbon surface [8]. It can be seen that the pores of the activated carbon are open because the remaining impurities and organic compounds are lost after activation with alkaline. The activation process will open the pores of the activated carbon by breaking the hydrocarbon bonds and it will adsorption capacity increase.

XRD Characterization

The physicochemical properties of activated carbon from oil palm shells were characterized using XRD show that crystallite structure of a material. The crystallite structure of activated carbon from oil palm shells is shown by the diffraction pattern in **Figure 2**.

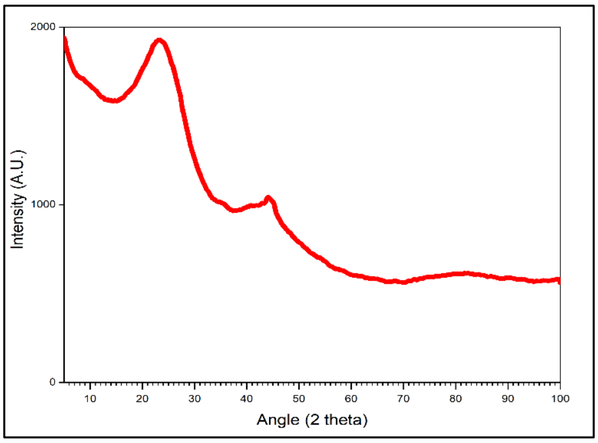

Figure 2. XRD analysis of activated carbon palm shell.

The image shows a wide peak at an angle of 2 theta at 26.62 and 44.5672º which shows that the structure of activated carbon from oil palm shells is amorphous. Activated carbon that has been activated with acid shows two main diffraction peaks at an angle of 2θ around 26.6200º and 44.5627º. The expected crystal structure has a diffraction peak at an angle of 2 theta, and 26.555º with a miller index (HKL) of 002 and at an angle of 44.572º has a miller index (HKL) of 101, this is indicates that the characteristics of the hexagonal crystal [17]. The most of the alkaline -activated activated carbon material will maintain the desired hexagonal crystal structure. To calculate the average diameter size of the crystal from KOH-activated activated carbon, the Scherrer equation is used. Based on the half- maximum width (FWHM) of the main diffraction peak at an angle of 26.6200º and 44.5672º, the same FWHM value is obtained, at 0.3897 where the calculation results in an average crystal size of 21 nm [18].

FTIR Characterization

Fourier Transform Infrared (FTIR) (Perkin Elmer Spectrum One) was used to determine the surface of organic functional groups. The FTIR spectrum was recorded from the wave number range of 500 - 4000 cm^{-1}. The resulting activated carbon has a peak of 2919.509 cm^{-1} indicating the absorption of the C-H stretching group in the range of 2970 - 2850 cm^{-1}. The C-H groups on activated carbon after activation is caused by the release of water molecules in the activated carbon. The wave number of 3332.182 cm^{-1} in the range of 3600 - 3200 cm^{-1} is identified as the O-H stretching functional groups and the wave number of 1205.17 cm^{-1} in the range of 1300 - 1050 cm^{-1} is identified as the C-O stretching functional group. The wave number of 1588.87 cm^{-1} was identified as the C=C stretching functional group of aromatic compounds in the range of 1600 – 1500 cm^{-1} [19].

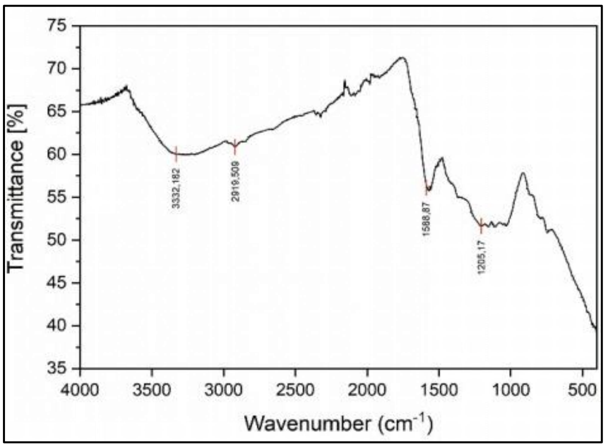

Figure 3. Spectra FTIR activated carbon from palm shell.

Standar calibration curve of remazom and Naphtol

The standard calibration curve of remazol and naphthol solutions shown in **Figure 4**. In

Figure 4, shown the correlation coefficient (r^2) is the degree of correlation between the absorbance value and the concentration value [20]. The calibration curve obtained from the remazol and naftol solution has a linear regression of y = 0.0225X + 0,001 with a correlation coefficient (r^2) of 0.9692 and 0.0199x – 0,0019 with correlation coefficien (r^2) of 0.959.

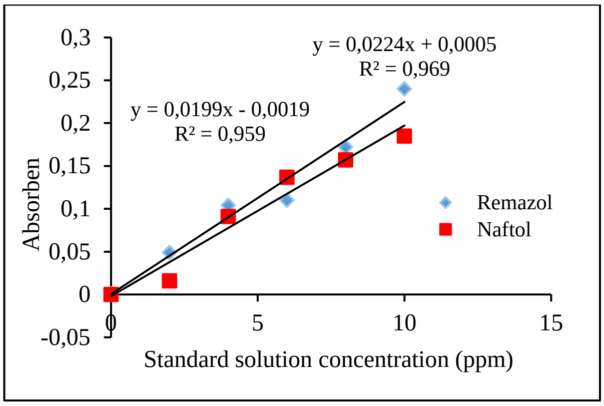

Figure 4. Standard calibration curve of remazol and naftol.

Effect of pH adsorption

The effect of pH on adsorption remazol and naftol shown in **Figure 5**. Adsorption of remazol and naftol on activated carbon occurred at pH 6 and pH 10 with adsorption capacity of 1.121 mg/g and 1.133 mg/g respectively. The influence of pH adsorption is in accordance with that carried out Arya et al [22], with optimum pH adsorption of remazol at pH 6. At H 6 conditions remazole is negatively charged so that it can form interactions with the -CH group on active carbon. Adsorption of naphtol occurs at pH 10 with an adsorption capacity of 2.796 mg/g. at pH 11-12 the adsorption capacity decreased to 2.764. This is due to ionization of the hydroxyl group (-OH) in activated carbon.

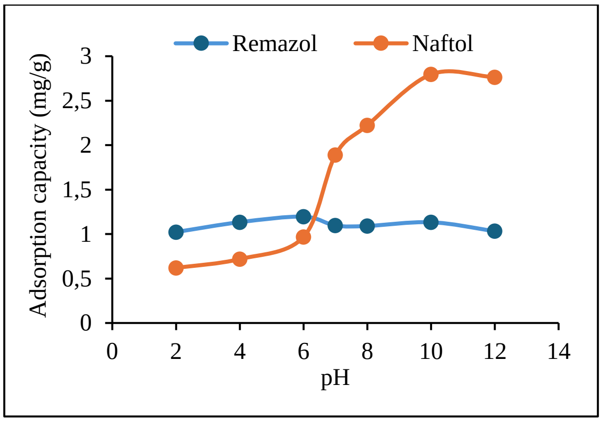

Figure 5. The effect of pH remazole and naphtol on adsorption capacity.

Effect of contact time

The effect of contact time on the absorption of remazole and naphthol on KOH-activated activated carbon is shown in **Figure 6**. Absorption occurred at an interval of 30-60 minutes with an absorption capacity of remazole of 0.085 mg/g and naphtol of 2.771 mg/g. The significant increased in the initial period occurred because more active site space was available on the adsorbent surfac and adsorption capacity increased. At 90 minutes adsorption capacity remazole and naphtol decreased. The decreased adsorption capacity of naphthol on activated carbon can also be seen in of activated carbon from orange peel [16]. The inceasing contact time an increase in contact time causes a decrease in adsorption capacity, this is due desorption of the adsorbate into the solution, causing the adsorption rate to decreased.

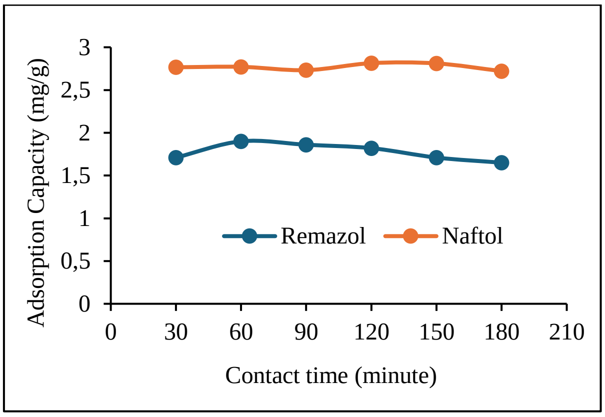

Figure 6. The effect of contact time on adsorption capacity remazol and naftol.

CONCLUSION

Activated carbon palm shell in has been potential to used as an adsorbent for naphthol and remazol dyes. The results of scanning electron microscopy (SEM) characterization shown that surface morphology has shows a porous surface morphology, XRD analysis shown that activated carbon has crystallinity form and FTIR analysis shown several functional groups such as hydroxyl (-OH), carbonyl (C=O) and CH which are expected to interaction with remazol and naftol dye during in adsorption process. The effect of parameter adsorption occur of pH at 6 and 10 for adsorption remazol and naftol, contact time on

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