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Activated Carbon from Palmyra Palm Peel as an Alternative Adsorbent for Removing Heavy Metal Ions Fe(III) and Cr(VI) in Industrial Waste

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***Abstract*— Palmyra palm peel served as raw material for preparing activated carbon. In addition to its high cellulose content, palmyra palm shells are also easily found in Gresik and Tuban, East Java. Palmyra palm shell is also an abundant solid waste with low economic value, so using palmyra palm shells as raw material for activated carbon production is low cost to reduce the contaminant in liquid waste. This experiment aims to determine the effectiveness of palmyra palm peel as a bio-adsorbent for heavy metal ions Fe(III) and Cr(VI) in industrial waste. This research was conducted through 3 processes: chemical activation, carbonization, and adsorption. The methods used in this study consisted of pre-treatment, activation of raw materials, manufacture of standard solutions, calibration of standard solutions, and adsorption of heavy metals from textile waste. The carbons' activation was conducted at 600 and 700°C in the presence of KOH as the activating agent. The results are a water content of 17.50% and an ash content of 8.37%. The moisture content and ash produced results comply with the SII and SNI 06-3730-1995 standards. The carbon produced at 700°C has a better adsorption performance than that produced at 600°C. The maximum removal efficiency for Fe(III) was 95.25, and Cr(VI) was 89.7%. Two well-known equations, Langmuir and Freundlich, were used to correlate the experimental adsorption data. Langmuir equation could represent the data better than Freundlich with an R^2 value close to unity.**

***Keywords*— Adsorption; bio-adsorbent; palmyra palm peel.**

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I. INTRODUCTION

Environmental pollution is one of the big problems we face today and needs serious attention. One type of environmental pollution is water pollution. One of the causes of water pollution is the presence of heavy metals in wastewater [1]. Serious processing facilities are required before releasing those industrial effluents into the environment. The hazardous contents in the effluent could damage the environment and its ecosystem and cause severe problems to human health. Heavy metals generally found in the industrial effluents are Ag(I), Ni(II), Cu(II), Pb(II), Co(II), Cr(III)/Cr(VI), Zn(II), Hg(II), Fe(II)/Fe(III), Cd(II). These are toxic to human life and can cause various health problems [2]. Physical activation is carbon activation with pyrolyzed under an inert atmosphere, while chemical activation is activating process with chemical activator impregnation such as KOH, NaOH, $NaCO_3$, $ZnCl_2$, $AlCl_3$, H_3PO_4, H_2SO_4, and some acids [3].

Several treatment processes are available to remove heavy metal content from industrial effluents, including adsorption, coagulation, biodegradation, and photodegradation. Each method has advantages and disadvantages regarding the operation, cost, and design. Of the available processes, the adsorption process is the most efficient in terms of operating costs and ease of use.

The success indicator of the adsorption process in eliminating heavy metals depends on the correct choice of the adsorbent; usually, activated carbons are widely used as the adsorbents [4]. Activated carbons with high surface area and adsorption capacity are preferred since they can completely remove heavy metals from wastewater. Activated carbons can be made from various carbonaceous materials; their adsorption capacity depends on the structure of activated carbon, such as porous structure, crystalline structure, chemical structure, and applications of activated carbon [5]. In this study, the activated carbon was produced from the palmyra palm peel (Figure 1).

Fig. 1 Palmyra Palm Peel

Palmyra palm is a native plant commonly found in Southeast Asia (including Indonesia) and the Indian subcontinent. Palmyra palm usually grows in sunny and dry areas, including coastal areas. Palmyra palm plant also has characteristics: round, fan-shaped leaves; the fruit is large, round, and protected by a blackish-brown shell; when it is old, the shell will be harder and thicker [6]. The palmyra palm fruit contains sweet jelly seed sockets, the edible part of the fruit, and the rest are non-edible and usually end up as solid waste. The palmyra palm plant is only limited to the fruit and stems, while the shell or peel is a waste that has not been utilized optimally. On a dry basis, the composition of palmyra palm shell contains 89.2% cellulose, 5.4% water, 3.1% carbohydrates, and 2.3% ash [7]. In addition to its high cellulose content, palmyra palm shells are also easily found in Gresik and Tuban, East Java. Palmyra palm shell is also abundant solid waste with low economic value, so using palmyra palm shells as raw material for activated carbon manufacturing will grow the economic value of the waste [8]. The effectiveness of palmyra palm peel-activated carbon as a bio-sorbent of heavy metals is explored in this study.

Several studies have applied palmyra palm choir as the raw material for activated carbon production, and the results were subsequently used as the adsorbents for hazardous contaminants removal. The use of palmyra palm fiber as the activated carbon has been studied by Arifianti *et al*. [9]. In her study, she used H_3PO_4 as the activator. This solvent was used since it was able to produce a higher area than NaOH and KOH solvents based on the earlier study. The surface area of activated carbon produced by H_3PO_4 solvent is 373.5675 m^2g^{-1}. Meanwhile, KOH and NaOH got a smaller area of 218.8230 m^2g^{-1} and 33.87878 m^2g^{-1}.

Besides, another study by Maia et al. [10] developed the utilization of activated carbon from the palm fibers to remove methylene blue. The resulting adsorption efficiency in removing methylene blue was 89.88% and 95% at 25 mg/L with the sheath and stem's activated carbon. The effect of contact time in removing Methylene Blue was also studied. It was found that with the shorter contact times, activated carbon can remove Methylene Blue faster, with the efficiency from the sheath being 65.26% in 10 min and 53.21% in 40 min from the stem.

The high-yield adsorption behavior with activated carbon from demineralized low-range coal (Rawdon) for methylene blue and phenol was investigated by Gokce et al. [11]. He learned about the effects of demineralization on the ACs adsorption capacities using common water contaminants, methylene blue (MB), and phenol adsorbates. The BET (Brunauer-Emmett-Teller) surface area of the AC obtained from the demineralized sample (1869 m^2g^{-1}) was found to be much higher than those obtained from the raw coal (1059 $m^2 g^{-1}$). The pre-carbonization also caused an increase in the surface areas of the samples. Adsorption tests were evaluated using The Langmuir and Freundlich models. The ACs were characterized in terms of total surface area, particle and pore size distribution, XRF (X-ray Fluorescence) analyses, FTIR (Fourier Transform Infrared Spectroscopy), and XRF (X-ray Fluorescence) analyses. The adsorption capacity of the same AC reached the highest values for MB (841.93 $m.g^{-1}$) and phenol (549.6 $mg.g^{-1}$). The surface area of the AC prepared from the demineralized and pre-carbonized sample was determined as 1951 m^2g^{-1}. [11].

II. Materials and Method

The materials used in this study were the palmyra palm peel, KOH, $FeCl_3$, and $K_2Cr_2O_7$. The palmyra palm peel waste was obtained in Gresik and Tuban Regencies, East Java. Merck purchased all the chemicals used in this study as the analytical grade. The methods used in this study include pre-treatment of raw materials, activation of raw materials, manufacture of standard solutions, calibration of standard solutions, and adsorption of heavy metals from textile waste.

The following procedure was employed to pre-treatment raw materials: the palmyra palm peel waste was separated from the rest of the fruit and shell using a cutter or knife. Subsequently, the palmyra palm peel was dried in an oven to remove some free moisture content—the drying process was conducted for 5 hours at a temperature of 110^{o}C. Dried palmyra palm peel was kept in the desiccator for about 1 hour. The particle size of dried palmyra palm peel was reduced to smaller sizes using scissors [12]

In the chemical activation process, palmyra palm peel was soaked in KOH solution with a concentration of 1 M. In this process, 25 grams of palmyra palm peel was soaked with 500 ml of KOH solution in a glass beaker. The ratio between palmyra palm coir and the activator solution was 1:20. The soaking was conducted without stirring, and the mixture was allowed to stand for 24 hours. After 24 hours, the palmyra palm coir was put into the oven at 110^{o}C for 1 hour [13] and stored in a desiccator for further use.

The chemically activated palmyra palm peel was then physically activated in a tube furnace with N_2 gas flowing at a flow rate of 1.5 l/min. Five grams of chemically activated palmyra palm were put into a crucible cup and placed in the tube furnace. The furnace was then heated at a temperature variation of 600°C and 700°C. After this process was completed, the solid product was called palmyra palm activated carbon [14].

A total of 1 gram of palmyra palm activated carbon was put into 2 Erlenmeyer. In each Erlenmeyer, textile waste was added with aquadest with a waste ratio and distilled water of 4:6 (v/v) with a mixed volume of 50 mL. Subsequently, the mixture was stirred, and some of the solutions were taken every 20 min (20, 40, 80, 100, 120 min). The metal concentration in the filtrate was measured using a UV-Vis Spectrophotometer. The adsorption experiment was conducted isothermally.

The Langmuir and the Freundlich model were used to representing the experimental isotherm adsorption. The Langmuir isotherm equation parameters were obtained using the linear method of least squares. The linear form of the Langmuir model is shown as follows:

$$\frac{Ce}{Qe} = \frac{1}{Qm} Ce + \frac{1}{Qm.KL} \quad (1)$$

Where Qm is the maximum adsorption capacity (mg/g); KL is Langmuir's constant (mg/L); Qe is the amount of adsorbed substance per unit mass of adsorbent (mg/g), and Ce is the equilibrium concentration (mg/L). The linear form of the Freundlich isotherm equation is written as follows:

$$E(F) = E(0) + \sum_i \left(\frac{\delta E(F)}{\delta F_i}\right)_0 F_i \quad (1)$$

$$Ln\ Qe = Ln\ Kf + 1/n\ \ Ln\ Ce \quad (2)$$

The adsorption equations could represent the experimental data if the value of R2 is close to unity. The adsorption capacity of the adsorbent was determined from the Langmuir and Freundlich constant (Qm for Langmuir and Kf for Freundlich) [15], [16].

III. Results and Discussion

Adsorption is the process of the accumulation of adsorbate on the surface of the adsorbent due to the attraction force between the adsorbent and adsorbate [17]. This study used palmyra palm peel waste to manufacture activated carbon to remove metal ions and dye ions from textile waste. The manufacture of activated carbon from palmyra palm peel is carried out through several processes steps, such as the wastewater content of the palmyra palm peel, activated carbon ash content, calibration curves of $K_2Cr_2O_7$ and $FeCl_3$, and the process of adsorption of heavy metal ions Fe(III) at various concentrations and activation temperatures [18].

KOH is a suitable activator for the production of activated carbon from palmyra palm peel waste. The presence of KOH in the activation process can reduce the activation temperature, retard the release of volatile matter, and create a substantial amount of pores in the resulting carbon [19]. The bio-adsorbent carbonization process was carried out at a temperature of 600°C and 700°C for 1 hour. This carbonization process aims to break down organic materials into carbon [20].

Several phenomena occurred during the carbonization and activation process of the palmyra palm peel. The release of free moisture content and bound water occurs at temperatures of 100 to 200ºC. Further increase of carbonization temperature to 300ºC resulting in the breakdown of hemicellulose into lower molecular weight substances (gaseous and low molecular weight hydrocarbons). At temperatures up to 360ºC, the thermal degradation of cellulose happens. The thermal decomposition of lignin happens at a temperature on top of 360ºC. Carbon formation will occur at a temperature of 400 - 600ºC [21].

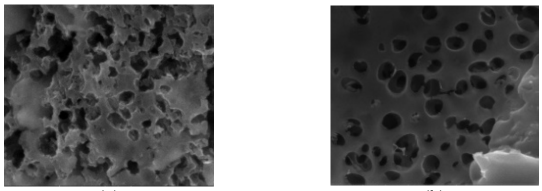
According to SNI, the properties of activated carbon made from palmyra palm peel were determined. The water content is based on Standards from the Institute for Industrial Research and Consulting. The water content was analyzed to determine the hygroscopic nature of activated carbon [21][22]. The moisture content of activated carbon produced from palmyra palm peel waste is shown in Table 1. The quality of activated carbon is also affected by ash content [23]. The ash content of activated carbon was determined based on Indonesian National Standard (SNI 06-3730, 1995), and the result is presented in Table 1.

TABLE I
WATER AND ASH CONTENT IN ACTIVATED CARBON PALMYRA PALM

Activated carbon sample	Moisture content (%)	Ash content (%)
I	16.33%	8.14%
II	18.67%	8.61%
Average	**17.50%**	**8.37%**
Maximum	18.90%*	10%**

* Standards from the Institute for Industrial Research and Consulting
** SNI 06-3730-1995

The results obtained indicate that the activated carbon has fulfilled the Indonesian national standard [23]. The surface topography of palmyra palm-activated carbons is depicted in Figure 2. This figure clearly shows that both activated carbons (activated at 600ºC and 700ºC) have a porous structure.



(a) (b)

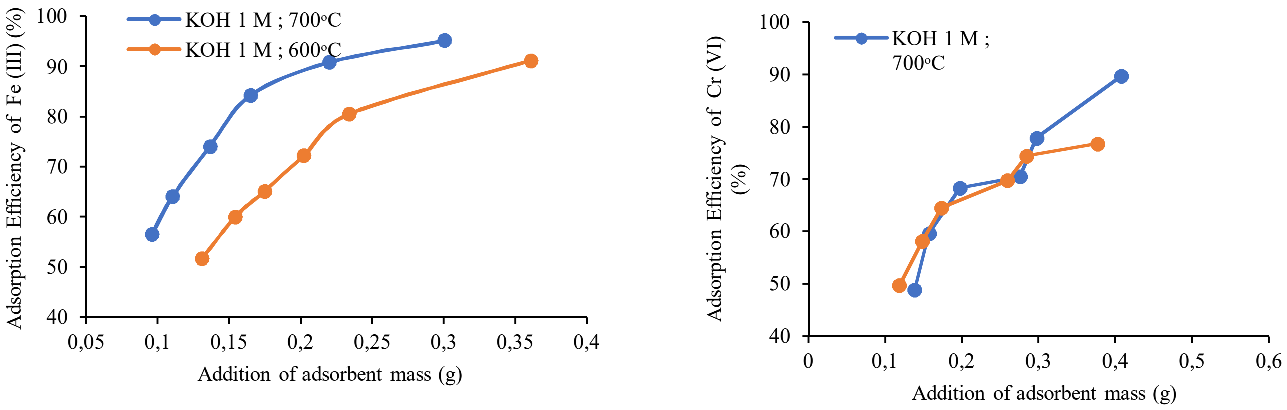
Fig. 2 a) KOH 1 M with T=600°C, b) KOH 1 M with T=700°C

Potassium hydroxide is a strong base, and its presence during the carbonization process will stimulate and catalyze oxidation reactions on the carbon surface, especially at high temperatures. At temperatures above 400ºC, the reaction between carbon dan KOH is as follows [24]:

6 KOH + 2C ↔ 2K + 3H2 + 2K2CO3 (3)

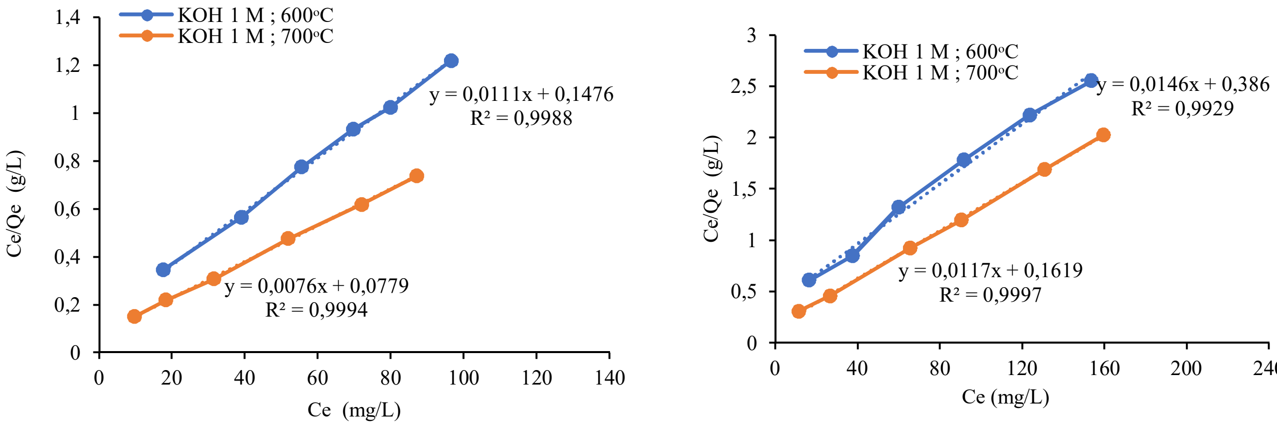
The formation of metallic K on the carbon surface will add the carbon matrix, leading to the widening of the areas between carbon atomic layers. This development creates new pores and will increase the pore volume of the carbon. Increasing the carbonization temperature from 600ºC to 700ºC increases the discharge of volatile matter from the carbon matrix and creates new pores, leading to higher pore development [25].

The palmyra palm-activated carbons' adsorption performance was tested to remove Fe(III) and Cr(VI). The adsorption efficiencies for the removal of Fe(III) and Cr(VI) are depicted in Figure 3. For both heavy metal ions, it can be seen that activated carbon carbonized at 700°C had a better removal efficiency than carbon carbonized at 600°C. The maximum removal efficiency for Fe(III) was 95.25%, while for Cr(VI) was 89.7%. This evidence indicates that activated carbons with larger pore sizes and volume (carbonized at 700ºC) performed better adsorption performance than the carbon carbonized and activated at 600ºC.



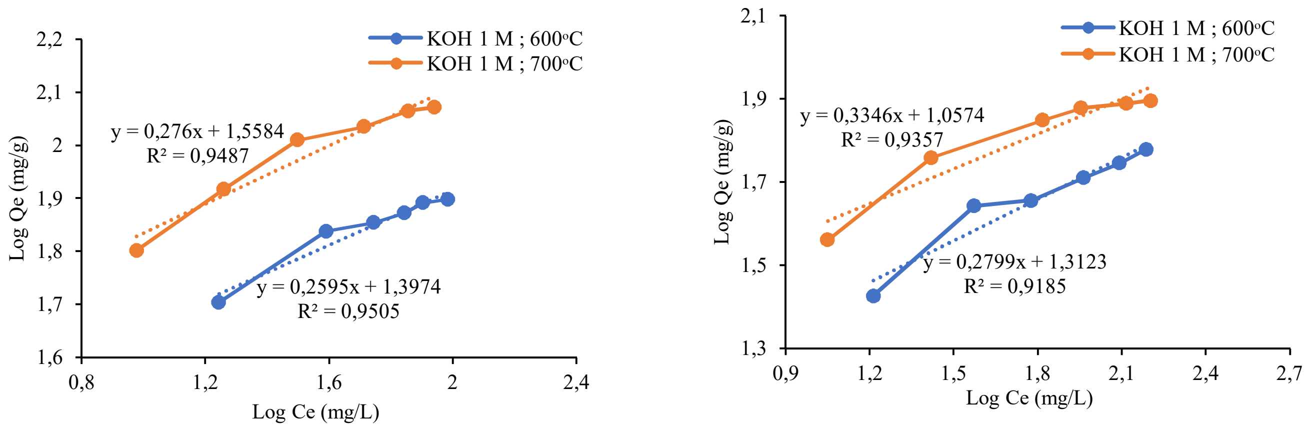
(a) (b)

Fig. 3 a) Adsorption Efficiency of Fe(III), b) Adsorption Efficiency of Cr(VI)



(a) (b)

Fig. 4 a) Langmuir Fe(III) Adsorption Isotherm Curve; b) Langmuir Cr(VI) Adsorption Isotherm Curve



(a) (b)

Fig. 5 a) Freundlich Fe(III) Adsorption Isotherm Curve; b) Freundlich Cr(VI) Adsorption Isotherm Curve

The adsorption isotherm study was carried out to determine Fe(III) and Cr(VI) adsorption mechanisms onto activated carbon produced from palmyra palm. The adsorption mechanisms provide crucial information for designing the adsorption plants [26]. In this study, two well-known adsorption equations (Langmuir and Freundlich) were used to correlate the experimental adsorption data. The Langmuir isotherm model explains that the adsorbate adsorbed onto the surface of the adsorbent form a monolayer, whereas the Freundlich model describes the multilayer adsorption [27]–[29].

The parameters of Langmuir and Freundlich were obtained through linear regression of the experimental data, as indicated in Figure 4 (Langmuir) and 5 (Freundlich). The Langmuir and Freundlich parameters for the adsorption of Fe(III) and Cr(VI) adsorption on palmyra palm activated carbon are summarized in Table 2. The suitability of the equation for correlating the experimental data was determined using the values of R^2. The equation could represent the experimental data if the values of R^2 are close to unity [30][31]. R^2 for the Langmuir adsorption isotherm is between 0.9976 - 0.9998, and for the Freundlich adsorption isotherm, the R^2 value is 0.9208 – 0.9505. This evidence indicates that Langmuir could represent the adsorption data better than Freundlich isotherm.

TABLE II
ADSORPTION ISOTHERM CALCULATION RESULT DATA

Model Isotherm	Parameter	KOH 600°C Fe (III)	KOH 700°C Fe (III)	KOH 600°C Cr (VI)	KOH 700°C Cr (VI)
Langmuir	qm (mg/g)	90.09	131.57	68.49	85.47
	kL (L/mg)	0.07	0.09	0.04	0.07
	R^2	0.99	0.99	0.99	0.99
Freundlich	Kf (mg/g)	36.17	24.96	11.41	20.53
	n	3.62	3.85	2.99	3.57
	R^2	0.94	0.95	0.94	0.92

The parameter qm within the Langmuir equation indicates the maximum adsorption capacity of the adsorbent, and also, the KL represents adsorption energy and the affinity constant [32], [33]. For the Freundlich equation, Kf and 1/n values indicate the adsorption capacity and heterogeneousness of the adsorbent, respectively. The worth of parameter n is between 1 to 10, and the upper value of n, is the additional heterogeneous adsorption system [34].

The parameter qm values of the Langmuir equation for the system KOH-700°C-Fe(III) and KOH-700°C-Cr(VI) were higher than systems KOH-600°C, which indicates that KOH-700°C has a higher adsorption capacity than KOH-600°C. Furthermore, greater KL values for KOH-700°C than for KOH-600°C imply that heavy metals were strongly adsorbed on the surface of KOH-700°C than on the surface of KOH-600°C. Since the Langmuir equation assumes monolayer adsorption, which is chemisorption behavior, it can be concluded that the adsorption of Fe(III) and Cr(VI) on palmyra palm activated carbon is chemisorption [7], [35].

IV. CONCLUSION

Activated carbon with high adsorption capability was synthesized through chemical activation (KOH) from the palmyra palm shell. The carbons were used as the adsorbent for Fe(III) and Cr(VI) removal. The activated carbon made at 700°C exhibited a higher porosity and adsorption capacity than the activated carbon made at 600°C. The adsorption performance of activated carbon from the palmyra palm shell was tested for Fe(III) and Cr(VI) removal. Langmuir adsorption equation could represent the experimental data better than the Freundlich equation.

NOMENCLATURE

Qm	maximum adsorption	mg/g
KL	Langmuir constant	mg/L
Qe	amount of adsorbed/ unit mass adsorbent	mg/g
Ce	equilibrium concentration	mg/L
Kf	Freundlich constant	mg/L

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