

# SYNTHESIS OF ACTIVATED CARBON FROM PET PLASTIC WASTE BY $H_3PO_4$ ACTIVATOR FOR ENVIRONMENTAL TREATMENT APPLICATION

CHẾ TẠO CARBON HOẠT TÍNH TỪ CHẤT THẢI NHỰA PET BẰNG CHẤT KÍCH HOẠT  $H_3PO_4$  ỨNG DỤNG TRONG XỬ LÝ MÔI TRƯỜNG

Lai Thi Hoan<sup>1</sup>, Tran Van Chinh<sup>2</sup>, Le Quoc Anh<sup>3</sup>,  
Nguyen Phuong Lam<sup>3</sup>, Ho Phuong Hien<sup>3</sup>, La Duc Duong<sup>2</sup>,  
Tran Thuy Nga<sup>1</sup>, Nguyen Manh Ha<sup>4,\*</sup>

DOI: <http://doi.org/10.57001/huih5804.2024.388>

## ABSTRACT

The increasing environmental problems caused by plastic waste necessitate innovative waste management and resource utilization methods. This study introduces a single-step approach to synthesizing activated carbon from Polyethylene Terephthalate (PET) plastic waste, utilizing phosphoric ( $H_3PO_4$ ) as the activating agent. Optimal activation conditions of the experiment were identified with 20g PET soaked with 30mL  $H_3PO_4$  and an annealing temperature of 800°C for 60 minutes. Comprehensive characterization of the materials was conducted using SEM, XRD, BET, and FTIR techniques. The resulting activated carbon exhibited a mesoporous structure with a high surface area of 655.595m<sup>2</sup>/g. This activated carbon demonstrated a good removal efficiency for MB dye in aqueous solutions, with a maximum adsorption capacity of 77.64mg/g. The effective conversion of plastic waste into a valuable resource highlights the significance of innovative strategies in combating environmental degradation. The synthesized activated carbon's performance in adsorption-based remediation techniques confirms its potential in addressing various pollution challenges, thereby offering new opportunities for the utilization of plastic waste.

**Keywords:** Plastic waste, activated carbon, polyethylene terephthalate, environmental treatment, chemical activation.

## TÓM TẮT

Các vấn đề môi trường ngày càng gia tăng do rác thải nhựa gây ra, đòi hỏi cần phải có các phương pháp quản lý và sử dụng sáng tạo. Nghiên cứu này trình bày phương pháp đơn giản chế tạo than hoạt tính từ chất thải nhựa PET sử dụng  $H_3PO_4$  làm tác nhân hoạt hóa. Điều kiện hoạt hóa tối ưu được xác định là: 20g PET ngâm với 30mL  $H_3PO_4$ , nhiệt độ nung ở 800°C trong 60 phút. Tính chất của than hoạt tính được đánh giá bằng các kỹ thuật như SEM, XRD, BET và FT-IR. Than hoạt tính có cấu trúc xốp với diện tích bề mặt riêng là 655,595m<sup>2</sup>/g. Than hoạt tính có khả năng loại bỏ tốt thuốc nhuộm MB trong dung dịch với dung lượng hấp phụ cực đại là 77,64 mg/g. Việc chuyển đổi hiệu quả chất thải nhựa thành vật liệu mới làm nổi bật tầm quan trọng của các chiến lược đổi mới chống suy thoái môi trường. Hiệu quả hấp phụ của than hoạt tính khẳng định tiềm năng ứng dụng giải quyết các thách thức ô nhiễm, từ đó mang lại cơ hội mới cho việc sử dụng chất thải nhựa.

**Từ khóa:** Chất thải nhựa, than hoạt tính, PET, xử lý môi trường, kích hoạt hóa học.

<sup>1</sup>Department of Fundamental Science, University of Transport and Communications, Vietnam

<sup>2</sup>Institute of Chemistry and Materials, Vietnam

<sup>3</sup>Hanoi National University of Education, Vietnam

<sup>4</sup>Hanoi University of Industry, Vietnam

\*Email: nmhacnh@gmail.com

Received: 08/7/2024

Revised: 30/9/2024

Accepted: 28/11/2024

## 1. INTRODUCTION

Due to escalating environmental challenges linked to the accumulation of plastic waste, extensive research has focused on innovative and sustainable management approaches. The persistence and ecological harm caused by non-biodegradable plastic waste, such as polyethylene (PE), polypropylene (PP), polyethylene terephthalate (PET), and polyvinyl chloride (PVC), pose significant concerns [1-3]. Among these, PET waste, with its high carbon content, remains inert in the environment for approximately 180 years, presenting a serious environmental threat [4, 5]. Recently, value-added recycling methods for plastic waste have gained considerable attention. The production of activated carbon from plastic waste offers a creative and eco-friendly solution to the demand for versatile adsorbent materials and the growing plastic waste problem. Activated carbon is increasingly popular due to its high surface area, porosity, electronic conductivity, rich and adaptable surface chemistry, and thermal stability. Consequently, carbon-based materials are being progressively utilized across various sectors, including energy, biomedical, environmental, analytical, and electrical applications, as well as catalysis [6-8].

In numerous countries, the textile industry has emerged as a significant economic contributor. This expansion is accompanied by concerns regarding the polluted wastewater generated, which contains dyes and various pollutants that are challenging to degrade [9]. Methylene blue (MB), a commonly used dye, can cause adverse health effects such as nausea, vomiting, anemia, and hypertension with prolonged exposure [10]. Various techniques are employed to treat wastewater containing these contaminants; however, traditional adsorption using activated carbons remains a crucial strategy due to its effectiveness, low initial and setup costs, and reduced operational expenses [11].

Activated carbon can be prepared through physical or chemical activation processes. Physical activation involves two stages: carbonization of raw materials followed by activation using steam or  $\text{CO}_2$ . In contrast, chemical activation combines the activation and carbonization steps into a single process. Chemical activation offers several advantages over physical activation, including higher yield, a one-step process, and lower activation temperatures [12-24]. Numerous studies have synthesized activated carbon from plastic waste using various chemical activators, such as KOH, NaOH [4,

15];  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{PO}_4$  [16, 17];  $\text{ZnCl}_2$  [18]; and  $\text{K}_2\text{CO}_3$  [19, 20]. According to the literature reports, chemical activation by  $\text{H}_3\text{PO}_4$  offers many advantages over the other methods, including a lower activation temperature, a higher carbon yield, and the creation of structures that are primarily mesoporous [21, 22].

This study aimed to synthesize highly porous activated carbon (AC) from polyethylene terephthalate (PET) plastic using the  $\text{H}_3\text{PO}_4$  - activation process. The effects of various factors, including the PET mass ratio, activation temperature, and activation time on the iodine number and yield of the activated carbon, were investigated. Additionally, the synthesized activated carbon was evaluated for its efficacy in removing methylene blue (MB) dye from aqueous solutions.

## 2. MATERIALS AND METHODS

### 2.1. Materials

The primary precursor for producing activated carbon is polyethylene terephthalate (PET) derived from plastic bottles typically used for mineral water. Following thorough washing and oven drying, the plastic bottles were fragmented into small particles approximately 2 - 3mm in size. Chemical reagents including  $\text{H}_3\text{PO}_4$  (85%), HCl (98%), iodine solution, sodium hydroxide, sodium thiosulfate, and methylene blue (analytical grade) were sourced from Xilong Chemical Company (China). Distilled water served as the solvent throughout all experimental procedures. All chemicals were utilized without additional purification.

### 2.2. Synthesis of the activated carbon (AC)

The PET pieces were impregnated with acid  $\text{H}_3\text{PO}_4$  at a ratio of 20g:30mL. Subsequently, the PET was dried at  $120^\circ\text{C}$  for 12 hours, transferred to a ceramic crucible, and heated in a furnace at various activation times and temperatures, with a heating rate of  $10^\circ\text{C}/\text{min}$  under an inert  $\text{N}_2$  flow of 100mL/min. The samples were then washed with distilled water to a neutral environment. The activated carbon was dried at  $100^\circ\text{C}$  for 8 hours, ground for characterization, and utilized in experiments.

### 2.3. Determination of iodine values

The iodine number, a metric indicating the amount of iodine adsorbed per unit mass of activated carbon (mg/g), was used to optimize activation conditions. In a standard procedure, 0.05g of activated carbon was placed in separate 100 mL glass beakers, followed by the addition of 20mL of 0.025M  $\text{I}_2$  solution to each beaker. The mixtures were stirred for 15 minutes and then filtered

using filter paper. The filtrate was titrated from orange to pale yellow, followed by the addition of a few drops of starch solution. Titration was continued until the solution became colourless. The volume of 0.05M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution used was recorded. This titration process was repeated three times, and the average result was calculated.

The iodine number of the activated carbon sample was determined as follow:

$$Q = (C_1 \cdot V_1 - C_2 \cdot V_2) \cdot \frac{M_{I_2}}{m_{AC}} \quad (1)$$

Where Q is the iodine number (mg/g); C<sub>1</sub> (mol/L), V<sub>1</sub> (L) are the initial concentration and volume of the solution, respectively; C<sub>2</sub> (mol/L), V<sub>2</sub> (L) are the iodine concentration and volume of the solution after adsorption (L), respectively; M<sub>I<sub>2</sub></sub> is the molecular weight of iodine (M = 254g/mol), and m<sub>AC</sub> is the mass of activated carbon used (g).

#### 2.4. MB adsorption experiments

All adsorption studies were conducted in batch mode using activated carbon (AC) as the adsorbent. A stock solution of methylene blue (MB) was prepared by dissolving 1g of the dye in 1L of distilled water. In a typical experiment, 0.03g of AC was added to separate flasks containing 10mL of MB solutions with concentrations ranging from 30 to 100mg/L. The mixtures were shaken at 100rpm at room temperature. Post-adsorption, the MB solutions were centrifuged for 15 minutes at 5000rpm, and the concentration of MB was determined using an Agilent 8453 UV-Vis spectrometer (USA) at the maximum absorbance wavelength (664nm). The adsorption capacity (q<sub>e</sub>) was determined by the following equation:

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \quad (2)$$

where C<sub>0</sub> is the initial concentration of MB (mg/L), C<sub>e</sub> is the final concentration of MB (mg/L), V is the volume of MB solution (L), and m is the mass of AC (g).

#### 2.5. Characterization

The crystal structure of the activated carbon was analyzed via X-ray diffraction (XRD) using a D8-Advance diffractometer (Bruker) with a Cu Kα radiation source (λ = 0.15405nm). The morphology and the elemental compositions of the adsorbent were examined using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) with a Hitachi S-4600 microscope. Fourier-transform infrared (FT-IR)

spectroscopy (Tensor II, Bruker) was employed to record the bond vibrations within the activated carbon structure, covering a spectral range from 400cm<sup>-1</sup> to 4000cm<sup>-1</sup>. The surface area and porosity were determined by nitrogen adsorption-desorption using the NOVA Touch 2LX/Quantachrome instrument.

### 3. RESULTS AND DISCUSSION

#### 3.1. Activated carbon synthesis

The effect of the activation parameters on the AC yield, and iodine number were investigated, and the results are presented in Table 1.

Table 1. Effect of the activation parameters on the iodine adsorption number, and surface

Sample	Temperature, °C	Yield (wt. %)	Specific surface area (m <sup>2</sup> /g)	Iodine number (mg/g)
AC0	400	18.09	36.559	125.27
AC6	600	14.37	138.039	335.92
AC7	700	15.72	197.118	446.43
AC8	800	13.29	655.595	965.74
AC9	900	13.99	624.308	844.21
Activation time: 60 minutes; PET:H <sub>3</sub> PO <sub>4</sub> = 20g:30mL				
Sample	Time, min.	Yield (wt. %)	Specific surface area (m <sup>2</sup> /g)	Iodine number (mg/g)
AC30	30	14.62	505,441	872.83
AC60	60	13.29	655,595	965.74
AC90	90	13.94	609,468	912.38
Activation temperature: 800 °C; PET:H <sub>3</sub> PO <sub>4</sub> = 20g:30mL				

According to the results of Table 1, the efficiency of activated carbon recovery from PET plastic when carbonized at 400°C is the highest at 18.09%, when activated at temperatures from 600 - 900°C, the recovery efficiency fluctuates, in the range of 13 - 16%. This shows that carbonization at 400°C has not completely lost the organic compounds in PET plastic, and this happens when the heating temperature is raised above 600°C. The amount of activated carbon obtained when calcined at temperatures greater than 600°C is about 13 - 16%, due to the amount of carbon blown away during the calcination process and lost during the filtration process.

The porous properties of activated carbon samples were evaluated through the specific surface area and iodine index. It was found that for the AC0 sample, when

only carbonized, the specific surface area and iodine index were the lowest, respectively 36.559m<sup>2</sup>/g and 125.27mg/g respectively. When activated by H<sub>3</sub>PO<sub>4</sub> at temperatures above 600°C, the specific surface area and iodine index of the samples increased significantly, gradually increasing with increasing calcination temperature. Thus, the carbonization process simply causes the loss of organic compounds in PET plastic. When activated with H<sub>3</sub>PO<sub>4</sub>, it develops porous channels on the surface of the carbon, leading to an increase in the specific surface area and adsorption index I<sub>2</sub>. At a calcination temperature of 800°C, the specific surface area and iodine adsorption index are the highest at 655.595m<sup>2</sup>/g and 965.74mg/g, respectively; These values decrease when increasing the calcination temperature to 900°C to 624.308m<sup>2</sup>/g and 844.21mg/g, respectively. At a temperature of 800°C, the pore system is most complete. At 900°C, it begins to transit to the ashing stage and the capillaries break down, so the specific surface area and adsorption index I<sub>2</sub> have decrease. Thus, we choose the optimal calcination temperature of 800°C.

It is seen that, in Table 1, when activated for 30 minutes, the activated carbon sample only has a specific surface area and an iodine index of 505.441m<sup>2</sup>/g and 872.83mg/g. During this time, the capillary system is gradually being completed and when activated for 60 minutes, the activated carbon capillary system is most complete with a specific surface area of 655.595m<sup>2</sup>/g and an iodine index 965.74mg/g. When increasing the activation time to 90 minutes, there was a slight decrease in the porosity of activated carbon. Thus, we choose the optimal calcination time of 60 minutes.

**3.2. Characterization of activated carbon**

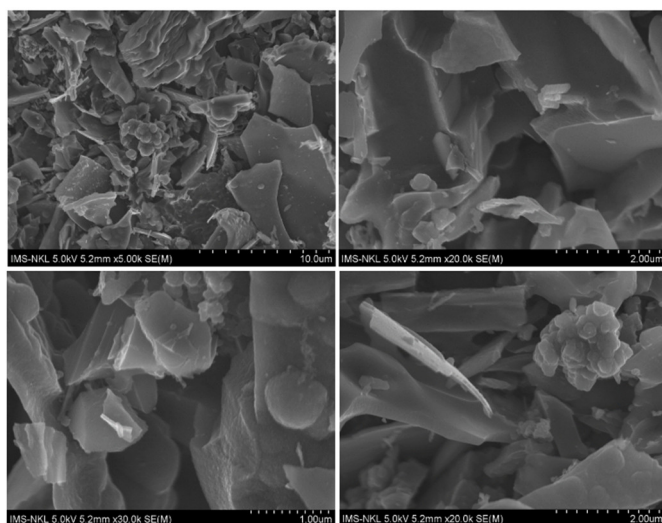
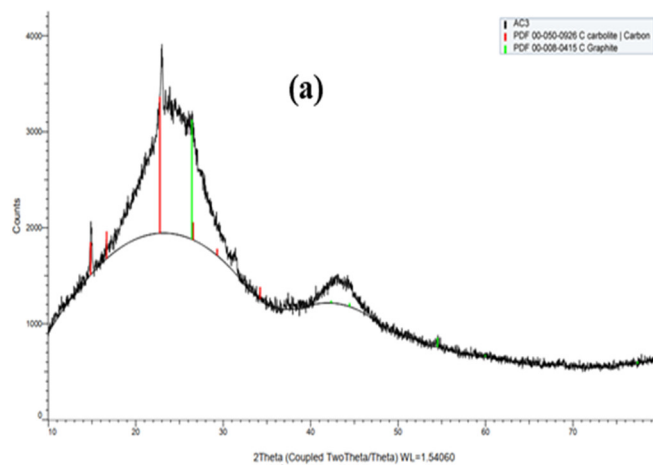


Figure 1. SEM of the activated carbon

The surface morphology of activated carbon synthesized under optimal conditions (activation time: 60 minutes; activation temperature: 800°C) was characterized by scanning electron microscopy (SEM). As shown in Fig. 1, the produced activated carbon structure has many grooves and pores, showing that the activated carbon surface is a porous capillary system developed. For low-resolution SEM images, we observe granules and fragments of activated carbon, and for high-resolution SEM images, we see that the surface of activated carbon is quite smooth. These observations indicate that the activating agent reacted progressively with the material, affecting not only the surface but also penetrating the inner parts of the carbon material.

Information about the structure of activated carbon was analyzed by X-ray diffraction method, the result is shown in Fig. 2a. On the XRD pattern, peaks appear at position 2θ = 14.84°; 22.76° corresponds to the (110) and (120) faces, which characterize the carbon phase with hexagonal structure (standard card 00-050-0926) and the peaks at 2θ = 26.42°; 42.36°; 54.51° corresponds to the (002), (100) and (004) faces that characterize the hexagonal structure graphite phase (standard card 00-008-0415). After that, the diffraction peaks are weak. Gradually, because the structure of activated carbon is graphite, there are many defects on the wall, leading to a decrease in graphite crystallization of activated carbon. The desired activation process creates many defects on the carbon wall to increase the surface area of activated carbon to facilitate the adsorption process. In this case, the diffraction peak of activated carbon made from PET plastic is relatively weak, proving that the surface area of activated carbon is quite good. The XRD pattern with few peaks and disorder also shows that the active carbon is amorphous.



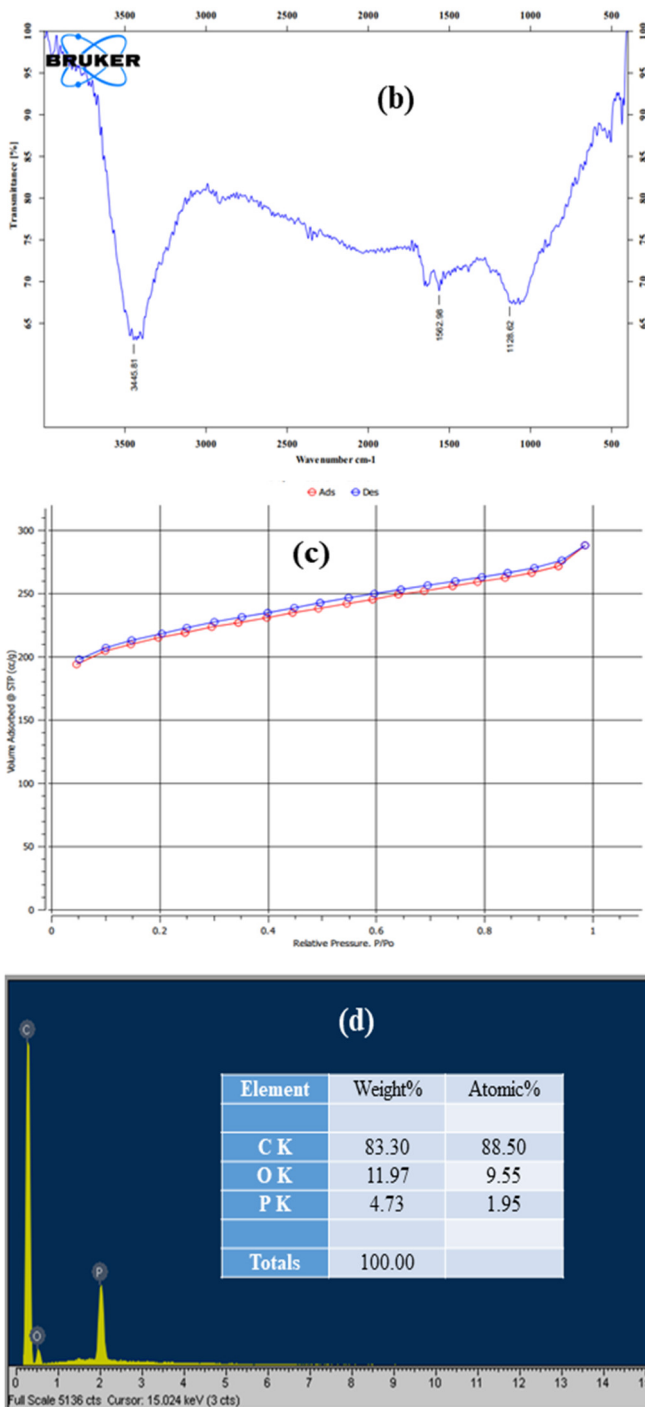


Figure 2. XRD pattern (a), FT-IR spectra (b), N<sub>2</sub> adsorption-desorption isotherm (c), and EDX spectra of activated carbon

According to Fig. 2a, the wave number at position 3445cm<sup>-1</sup> characterizes the vibration of the -OH functional group. The spectral fringe vibrating around 1562cm<sup>-1</sup> characterizes the C=C bond, and at 1128cm<sup>-1</sup> there is the appearance of the C-O stretching vibration. The resultant activated carbon's surface may have these functional groups, which could provide it an effective adsorption affinity for organic molecules.

As seen in Fig. 2c, the adsorption-desorption isotherm curve shape of activated carbon is of type II according to IUPAC's classification, which is a material with large capillaries (macroporous) and has a hysteresis loop of type H3, which is a flexible sheet-shaped material [23]. The specific surface area, capillary diameter and capillary volume of activated carbon are 655.595m<sup>2</sup>/g, 3.389nm and 0.120cm<sup>3</sup>/g, respectively.

Table 2 shows a comparison of the porous properties of prepared activated carbon with some other studies.

Energy-dispersive X-ray spectroscopy (EDX) was used to analyze the elemental composition of the activated carbon. The resulting figure (Fig. 2d) displays the distinctive peaks of C, O, and P. The appearance of C comes from the original PET plastic material. The activator, H<sub>3</sub>PO<sub>4</sub> acid, is responsible for P and O's appearance. The findings of the EDX analysis demonstrated the acid H<sub>3</sub>PO<sub>4</sub>'s activation ability and validated the elements present in the first material that was anticipated to be produced.

### 3.3. MB adsorption study

#### 3.3.1. Adsorption isotherm

To determine the adsorption capacity of prepared-activated carbon, the adsorption isotherm has been investigated to collect the adsorption equilibrium. The adsorption isotherm of MB on the AC was investigated by mixing the adsorbent with 10mL of MB solutions for 60 minutes at room temperature in which the MB concentration varied from 30 to 50mg/L at pH 7, adsorbent mass of 0.03g.

Adsorption isotherms for MB using activated carbon as adsorbents were investigated by the Langmuir and Freundlich models. The obtained data were fitted with the following equations:

$$\frac{C_e}{q_e} = \frac{1}{q_m \cdot K_L} + \frac{C_e}{q_m} \tag{3}$$

$$\log q_e = \log K_F + \frac{1}{n} \cdot \log C_e \tag{4}$$

where q<sub>e</sub> (mg/g), q<sub>m</sub> (mg/g), C<sub>e</sub> (mg/L), K<sub>L</sub> (L/mg) are the equilibrium adsorption capacity, the maximum capacity, the equilibrium MB concentration, and Langmuir constant, respectively. K<sub>F</sub> (L/g) is the Freundlich constant and n is the heterogeneity factor.

The curve intercept provides the Langmuir constant (K<sub>L</sub>), which represents the binding centers' affinity. The separation factor (R<sub>L</sub>) provides the characteristic of

Table 2. Porous comparison of prepared activated carbon with other reports

Type of waste	Activator	Optimum temperature, °C	Surface area, m <sup>2</sup> /g	Total pore volume, cm <sup>3</sup> /g	Pore diameter, nm	References
PET	H <sub>2</sub> SO <sub>4</sub>	600	537	0.36	1.67	[24]
PET	ZnCl <sub>2</sub>	800	682	0.47	2.7	[18]
Durian Shell Waste	H <sub>2</sub> SO <sub>4</sub>	600	348	0.153	4.38	[25]
Tire rubber	KOH	850	398.50	0.38	3.81	[26]
PET	KOH	500	335.307	0.29	3.27	[27]
Jujube Stones	H <sub>3</sub> PO <sub>4</sub>	700	794.5	-	-	[28]
PET	H <sub>3</sub> PO <sub>4</sub>	800	655.595	0.120	3.389	This work

Langmuir isotherms, making it an important dimensionless component. The formula that follows is used to determine R<sub>L</sub>.

$$R_L = \frac{1}{1 + K_L \cdot C_0} \tag{5}$$

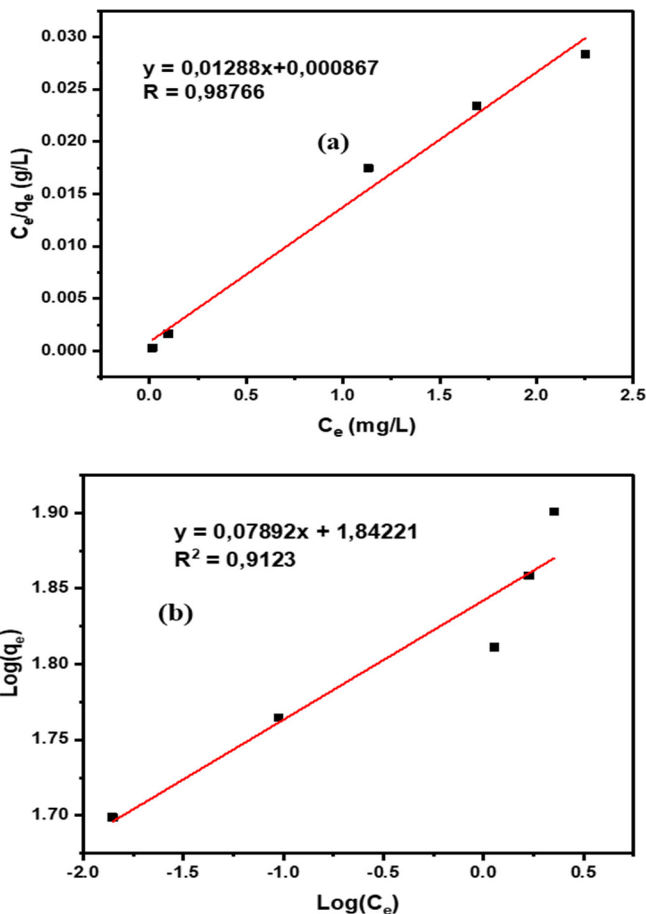


Figure 3. Linear plot isotherm data by (a) Langmuir and (b) Freundlich models

The linear plots of experimental data based on the Langmuir and Freundlich models are shown in Fig. 3a, b,

respectively, and adsorption isotherm parameters are presented in Table 3.

Table 3. Adsorption isotherms parameters of MB adsorption by activated carbon

	Langmuir model				Freundlich model		
	q <sub>m</sub> (mg/g)	K <sub>L</sub> (L/mg)	R <sub>L</sub>	R <sup>2</sup>	K <sub>F</sub> (L/g)	n	R <sup>2</sup>
MB	77.64	14.85	0.0013-0.0024	0.988	69.53	1.267	0.912

According to Table 3, the adsorption of methylene blue (MB) was more closely aligned with the Langmuir model (R<sup>2</sup> = 0.988) than with the Freundlich model (R<sup>2</sup> = 0.912). Additionally, the calculated separation factor (R<sub>L</sub> = 0.0013 - 0.0024) lies within the range of 0 to 1 (0 < R<sub>L</sub> < 1), suggesting that the Langmuir model is more favourable. The maximum adsorption capacity of activated carbon for MB, as calculated from the Langmuir model, is 77.64mg/g. In accordance with the Langmuir isotherm, MB exhibits monolayer coverage on the AC surface, indicating that adsorption occurs at specific homogeneous sites on the surface.

Table 4 shows the comparison of the activated carbon's maximum MB adsorption capacity with other adsorption capacities of the activated carbon made from various raw materials. From Table 4, it is clear that the activated carbon that was generated was a potent porous adsorbent that effectively removed the dyes from aqueous media.

Table 4. The maximum adsorption capacity of MB by different adsorbents

Adsorbents	q <sub>m</sub> (mg/g)	Reference
Activated carbon from PVC waste	97.10	[29]
Carbon nanotubes	64.70	[30]
Activated carbon from wood	59.92	[31]

Weeds activated biochar	92.60	[32]
ZnO-nano rods-activated carbon	89.30	[33]
Activated carbon from Durian Shell	57.47	[25]
Activated carbon from PET	77.64	This work

**3.3.2. Adsorption kinetic**

Adsorption kinetics are crucial to the adsorption process as they determine the rate-limiting step in surface adsorption, where physicochemical interactions between the adsorbate and adsorbent occur. The adsorption of methylene blue (MB) onto activated carbon was analyzed using pseudo-first-order and pseudo-second-order kinetic models. These models are represented by the following linear equations:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{6}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{7}$$

where  $k_1$  (L/min) is the rate constant of pseudo-first-order adsorption;  $q_e$  and  $q_t$  (mg/g) are the amount of MB adsorbed on the adsorbent at equilibrium and at time  $t$ , respectively;  $k_2$  (mg/g·min) is the rate constant of pseudo-second-order adsorption.

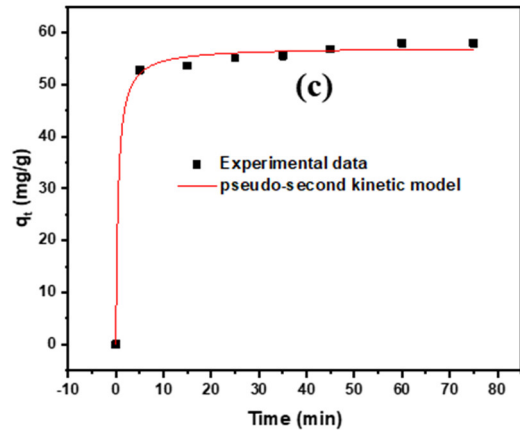
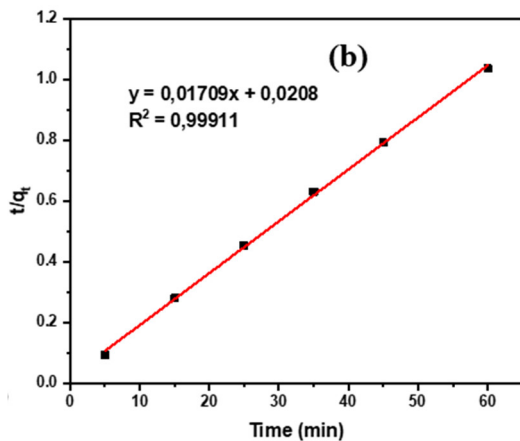
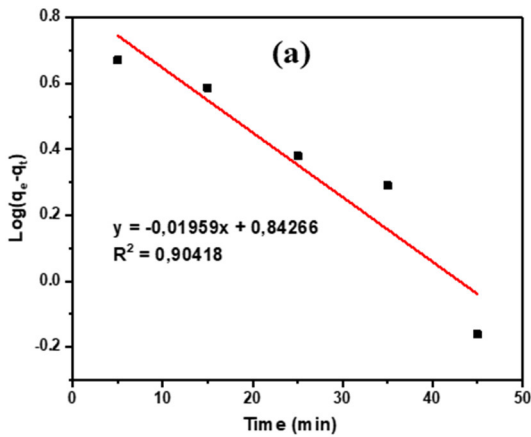


Figure 4. The pseudo-first-order (a), pseudo-second-order (b) kinetic model, and the calculated data from the pseudo-second-order kinetic model (c) for MB adsorption on AC

Figure 4 displays the obtained results based on these models, and the kinetic parameters are given in Table 5. Table 5 presents the results, which reveal that the pseudo-second-order kinetic model with  $R^2 = 0.999$  best describes the MB adsorption onto AC. Furthermore, the equilibrium concentration determined by the MB adsorption experiment was found to be almost equal to the  $q_{cal}$  values of the pseudo-second-order model. This suggests that the interaction between the MB and AC surface took the form of electron sharing or exchange.

Table 5. The kinetic parameters for MB adsorption on AC

The pseudo-first-order kinetic model			The pseudo-second-order kinetic model			
$q_e$ (mg/g)	$k_1$ (L/min)	$R^2$	$q_e$ (mg/g)	$q_{cal}$ (mg/g)	$k_2$ (mg/g·min)	$R^2$
6.872	0.036	0.932	58.478	57.941	0.014	0.999

Figure 4c shows that MB adsorption by activated carbon is a time-dependent process. The concentration of MB in the solution decreased with time,  $q_e$  increased with time. The adsorption rate of activated carbon to MB in the solution was very fast, and after about 20 minutes, the AC effectively reached the adsorption equilibrium.

**4. CONCLUSION**

In this paper, activated PET carbon was successfully synthesized by treatment of PET plastic waste with  $H_3PO_4$ -activation at  $800^\circ C$  for 60 minutes with a fixed PET:KOH of 20g:30mL. Synthesized activated carbon had a high surface area of  $655.595 \text{ m}^2/\text{g}$  with considerable pore volume and pore radius of  $0.120 \text{ cm}^3/\text{g}$  and  $3.389 \text{ nm}$ , respectively. AC was applied as an effective adsorbent for the removal of MB from aqueous media with a maximum adsorption capacity of  $77.64 \text{ mg/g}$ . Thus, using the

process this study suggests to produce activated carbon from used PET plastic waste can be considered both environmentally and economically.

### ACKNOWLEDGEMENTS

This research is funded by University of Transport and Communications (UTC) under grant number T2024-CB-008

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#### THÔNG TIN TÁC GIẢ

Lại Thị Hoan<sup>1</sup>, Trần Văn Chinh<sup>2</sup>, Lê Quốc Anh<sup>3</sup>,  
Nguyễn Phương Lam<sup>3</sup>, Hồ Phương Hiền<sup>3</sup>, Lã Đức Dương<sup>2</sup>,  
Trần Thúy Nga<sup>1</sup>, Nguyễn Mạnh Hà<sup>4</sup>

<sup>1</sup>Khoa Khoa học cơ bản, Trường Đại học Giao thông vận tải

<sup>2</sup>Viện Hóa học - Vật liệu, Viện Khoa học và Công nghệ Quân sự

<sup>3</sup>Trường Đại học Sư phạm Hà Nội

<sup>4</sup>Trường Đại học Công nghiệp Hà Nội