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To cite this article: J Phuriragpitikhon *et al* 2023 *IOP Conf. Ser.: Earth Environ. Sci.* **1139** 012003

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Conversion of cassava rhizome into efficient carbonaceous adsorbents for removal of dye in water

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Abstract. The development of sustainable adsorbent materials for removing pollutants from water is of great interest for environmental remediation and human safety. This work presents the beneficial use of cassava rhizome (CR) to produce economical and efficient carbonaceous adsorbents for dye removal. CR derived activated carbons (AC) and magnetic AC were fabricated by one-pot hydrothermal carbonization in the presence of ZnCl_2 and $\text{ZnCl}_2/\text{FeCl}_3$ respectively, followed by pyrolysis at 800 °C. Porosity tuning and degree of magnetization of as-prepared carbons were achieved by varying the amount of ZnCl_2 and FeCl_3 as an activating agent and source of magnetic particles respectively. The result indicated that the optimal condition to obtain ACs with a high S_{BET} (1405 m^2/g) and the largest mesopore volume (0.54 cm^3/g) was using the weight ratio of ZnCl_2 to CR of 3 (3AC). While, the magnetic mesoporous carbon with the highest S_{BET} (554 m^2/g) was yielded when addition of FeCl_3 into the reaction mixture with a FeCl_3 to ZnCl_2 weight ratio of 1 (3AC-1M). 3AC and 3AC-1M were tested as adsorbents to investigate the adsorption efficiency of methylene blue (MB) in water. The adsorption behavior of MB on 3AC and 3AC-1M were monolayer adsorption based on the Langmuir isotherm model with R^2 of 0.9993 and 0.9953, respectively. 3AC showed the MB adsorption capacity of 274 mg/g which is comparable to that of commercial ACs. 3AC-1M that adsorbs MB with the maximum capacity of 102 mg/g , was separated from aqueous solution easily using an external magnet. This study demonstrates that the carbon precursors from biomass waste as CR can be conveniently converted into efficient carbonaceous adsorbents and functionalized for example by adding magnetic properties into the carbon structure to enable manipulations in industrial operations.

1. Introduction

Various dyes have been widely used in textile, printing, dyeing, and papermaking industries. The wastewater containing dyes is discharged into water bodies resulting in environmental damages even at low concentrations. That causes harm to human health and negatively affects the living things in the surrounding aquatic environments [1]. Therefore, it is necessary to search for simple, efficient, and economical ways to treat dye wastewater before releasing into public water. Currently, numerous methods have been employed to removal dyes from wastewater including adsorption [2,3], membrane filtration [4,5], advanced oxidation processes [6,7]. Among these methods, adsorption is one of the most popular methods for water purification due to its simplicity, cost effectiveness, high efficiency and no



other chemical required to promote the reaction rate between the adsorbent and adsorbate. An activated carbon (AC) is the most extensively used as adsorbent for water and wastewater treatment because of its high surface area, high porosity with tailored pore sizes as well as surface functionalization [8,9], high thermal/chemical stability, and widely available carbon precursors [10]. Since commercially available activated carbon is still expensive, *there* have been many attempts to explore low-cost activated carbon. Therefore, the use of biomass as carbon precursors to produce activated carbons is attracting a lot of interest. A large number of activated carbons have been successfully prepared from a board range of biomass materials and used for adsorption applications such as corn stalk [11], poultry litter [12], peanut shell [13], lignin [14] and tannin [15]. However, the use of AC in the fine powder form makes the whole process slow and the separation from water difficult due to the sedimentation process, that limit its applications on a large scale [16]. To overcome these limitations, the introduction of magnetic particles to the carbon adsorbents to obtain magnetic carbons have attracted lots of attention owing to their rapid and simple separation from water by using an external magnet after completed adsorption [17].

In an agricultural country where agricultural sector plays a very big role, it is good way to find out agricultural residues derived adsorbents to be used in this field. Therefore, the present work demonstrates the utilization of cassava rhizome, one of abundant sources of biomass wastes in Thailand, as a carbon precursor to prepare activated carbons and magnetic activated carbons through one-pot hydrothermal carbonization in the presence of ZnCl_2 and $\text{ZnCl}_2/\text{FeCl}_3$ respectively which is combining methods between activation and magnetization into one step [18]. Both carbon samples were conducted on the removal of dyes in aqueous solution to demonstrate the adsorption performance. Methylene blue (MB), which is toxic and carcinogenic compound, is selected as model pollutant to perform the adsorption experiments.

2. Experimental section

2.1. Materials

Cassava rhizome, provided by Sapthip Co., Ltd., was used as carbon precursor. Methylene blue hydrate ($\geq 95\%$, calc. to the dried substance), ferric chloride hexahydrate (ACS grade) and zinc chloride (Reagent grade, $\geq 98\%$) were obtained from Sigma Aldrich. Hydrochloric acid fuming 37% (ACS grade) was purchased from Merck. Deionized water was used in all experiments.

2.2. Synthesis of cassava rhizome derived activated carbons (CR-AC) using ZnCl_2 as activating agent

First, cassava rhizome (CR) was collected, washed in order to remove impurities, and then dried in an oven at $105\text{ }^\circ\text{C}$ for 48 h. The dried CR was cut into smaller pieces. To investigate the effect of ZnCl_2 on porosity tuning, different amounts of ZnCl_2 were incorporated into the solution during the synthesis through a slightly modified one-pot hydrothermal carbonization (HTC) method as proposed by Li et al. [19]; namely, 5 g of CR, and varying amounts of ZnCl_2 (5.0 g, 10 g, and 15 g) were added into 50 mL of deionized water. The resulting mixture was stirred at room temperature for 2 h. The mixture was then heated at $150\text{ }^\circ\text{C}$ for 24 h under a static condition in a Teflon-lined autoclave. After that, the solid product was separated from the mixture and heated in an oven at $80\text{ }^\circ\text{C}$ for drying, followed by pyrolysis at $800\text{ }^\circ\text{C}$ under flowing nitrogen for 1 h. The resulting sample was collected, washed with 0.1 N of HCl until the pH was neutral. The obtained AC were designated as “xAC”, where “x” indicates the weight ratio of ZnCl_2 to CR in the synthesis mixture. For example, $x = 1, 2$ and 3 refer to 5 g, 10 g and 15 g of ZnCl_2 added, respectively.

For the purpose of comparison, pyrolyzed hydrochar was also prepared through the same procedure as AC but without the addition of ZnCl_2 . This resultant material was labeled as PHC.

2.3. Synthesis of CR derived magnetic activated carbons (CR-AC-M)

Magnetic activated carbons (AC-M) were made by the same process as AC except that there was the addition of FeCl_3 from 1 to 3 weight ratios in relation to ZnCl_2 into the mixture. A subset of the AC-M

was labeled as xAC-yM, where “x” indicates the weight ratio of ZnCl₂ to cassava rhizome of the selected activation condition and “y” refers to the weight ratio of FeCl₃ to ZnCl₂ in the synthesis mixture

2.4. Characterization

Nitrogen adsorption-desorption isotherms were conducted at -196 °C. Prior to the N₂ adsorption measurements, all samples were degassed under vacuum for 15 hours at 200 °C. Powder X-ray diffraction (XRD) analysis was performed on Bruker D8 Advance X-Ray diffractometer using Cu K α radiation operating at current and voltage of 40 mA and 40 kV, respectively. The scanning was set in a continuous mode with step size of 0.020°. Scanning electron microscopy (SEM) images and Energy dispersive spectroscopy (EDS) examinations were carried out on a JEOL JSM-6610 LV scanning microscope operated under 20 kV of accelerating voltage. The measurement of absorbance of all solution was performed on UV-2700 UV-Visible Spectrophotometer. The magnetic property of as-synthesized adsorbents was investigated using vibrating sample magnetometer (VSM), Lakeshore Model 7404.

2.5. Batch adsorption studies

Methylene blue (MB) was chosen as a model pollutant to perform the adsorption experiments of as-synthesized ACs. 70 mg of the carbon samples were dispersed in 250 ml of various concentrations of MB solutions (20, 50, 100, 150 and 200 mg/L) in 500 mL Erlenmeyer flasks. The mixture was shaken at 250 rpm at 25 °C to reach equilibrium without any pH adjustment. At different time intervals, the mixture was taken, followed by centrifugation and the left supernatant solution was immediately determined using UV-vis spectrophotometer at wavelength of 664 nm. All experiments were carried out twice to check the reproducibility of results [15]. The value presented is the means of duplicate experimental results

The adsorbed amount of MB (q_e) on carbon adsorbents was calculated with equation (1) as follows

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

where C_0 and C_e stand for the initial and equilibrium concentrations of MB (mg/L), respectively, m is mass of the sample (g), and V is the volume of the MB solution (L)

Two famous isotherms equations including Langmuir and Freundlich isotherms were applied to fit the experimental isotherm data of MB adsorption on carbon adsorbents. These equations can be written as:

Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (2)$$

Freundlich isotherm:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (3)$$

3. Results and discussion

3.1. Structure characterization

The microstructure of one CR-AC (3AC) and one CR-AC-M (3AC-1M) were examined by X-ray diffraction. The XRD pattern of 3AC is shown in Figure 1A presenting two broad peaks centered around $2\theta = 23.7^\circ$ and 43.4° which correspond to the (0 0 2) and (1 0 0) planes, respectively [20]. The resulting XRD pattern indicates a typical of turbostratic carbon structure with a very low degree of crystallinity. XRD peaks at $2\theta = 35.1^\circ$, 42.6° , 56.3° and 62.5° were observed on a XRD pattern of as-prepared magnetic carbon, 3AC-1M as illustrated in Figure 1B. These peaks correspond to the four indexed planes

(311), (400), (511), and (440) of magnetite particles (Fe_3O_4) [21]. This could confirm a successful conversion of Fe ions into Fe_3O_4 by one-pot HTC coupled with a subsequent pyrolysis.

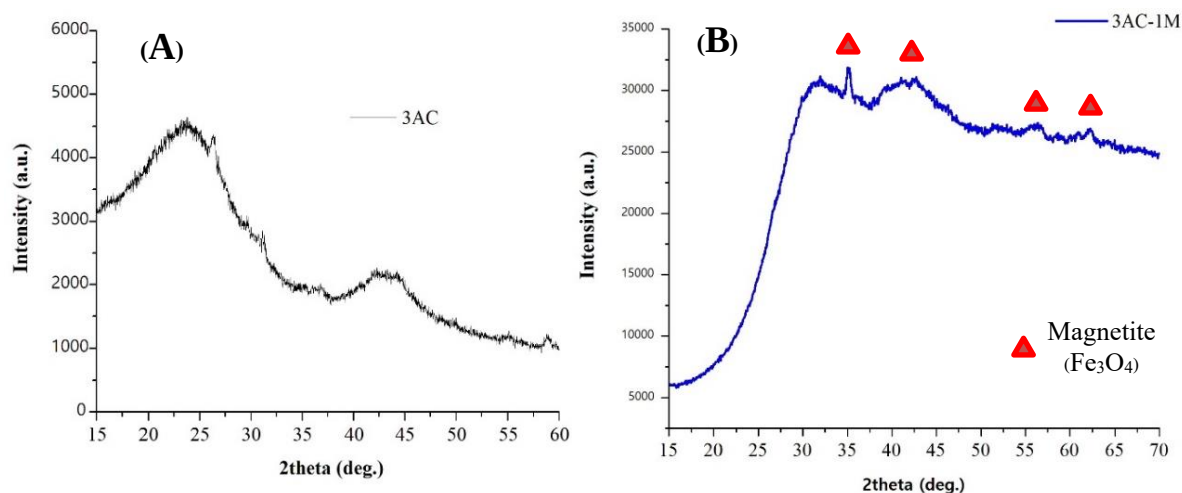


Figure 1. The XRD analysis of (A) 3AC and (B) 3AC-1M

The surface morphology of PHC, 3AC and 3AC-1M were compared using SEM analysis to investigate changes resulted from the activation and magnetization as seen in Figure 2A, 2B and 2C, respectively. In comparison with PHC prepared without the addition of ZnCl_2 , the outer surface of 3AC showed more cratered and cavities as depicted in Figure 2A and 2B. This may result from the evaporation of ZnCl_2 during the carbonization at 800 °C leaving the space previously occupied by the activating agent [22]. Moreover, the carbonization contributed to the release of volatiles resulting in the development of vacant sites at the pores. Finally new small pores were generated [23] that result in an increase in the BET surface area and micro-mesopore volume as seen in Table 1 for all activated carbon in comparison with PHC. A SEM image of a representative magnetic carbon, 3AC-1M was illustrated in Figure 2C showing the random distribution of small particles on the outer carbon surface. Such particles probably are Fe compounds generated during one - pot $\text{ZnCl}_2/\text{FeCl}_3$ co-HTC. Given the EDS result of 3AC-1M in Figure 2D, it is evident that the particles are Fe (9.79%) attributed to the iron oxide precipitated on the carbon surface during the magnetization [24]. The presence of iron oxide on the carbon surface is consistent with the former analysis results of XRD (Figure 1B). While, the existence of Zn and Cl in the sample were attributed to the use of ZnCl_2 as the chemical activating agent and the presence of inorganic elements such as Al, S and K could be directly from CR precursor. The percentage yield of the final nonmagnetic and magnetic ACs obtained by the proposed preparation method is between 20-28% as listed in Table 1.

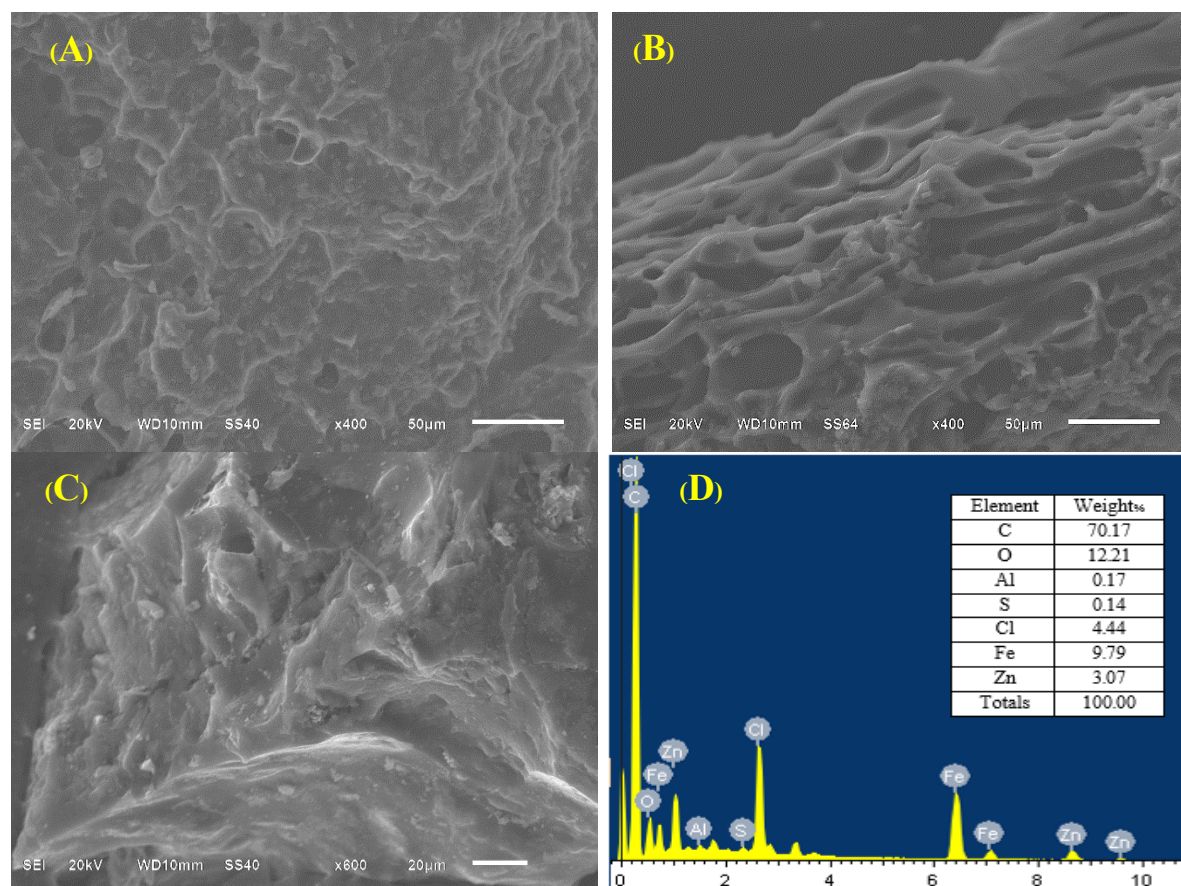


Figure 2. SEM images of (A) PHC (x400), (B) 3AC (x400), (C) 3AC-1M (x600) and (D) the EDS spectrum of 3AC-1M and its elemental compositions.

3.2. Textural properties

3.2.1. AC obtained through one – pot HTC of CR in the presence of ZnCl_2 as an activating agent.

The structural parameters of the obtained carbon samples calculated from nitrogen adsorption are listed in Table 1. The values of S_{BET} of these sample series range from 207 to 1425 m^2/g , V_t values range from 0.09 to 0.96 cm^3/g , V_{mi} and V_{me} values range from 0.08 to 0.49 cm^3/g and 0.01 to 0.54 cm^3/g , respectively. Figure 3A and 3B displayed nitrogen adsorption isotherms and differential pore size distributions (PSDs) up to 6 nm for PHC, 1AC, 2AC and 3AC, respectively. It can be seen in Figure 3A, the pyrolyzed hydrochar, PHC exhibits type I isotherm according to the IUPAC classification suggesting a presence of mainly micropores (pore size <2 nm) [25]. In addition, a PSD for this sample shows the sharp distributed micropore size of 0.95 nm. The values of S_{BET} increase with increasing amount of ZnCl_2 added for up to a 2 weight ratio to CR due to the enlargement of the volume of micropores. Moreover, the generation of mesopores can be observed when addition of ZnCl_2 during the preparation of all ACs confirming by the existence of hysteresis loops of nitrogen adsorption isotherms.

The nitrogen adsorption isotherm obtained from 1AC was a combination of type I and IV. Its initial part presents the characteristic of Type I indicating the existence of micropores, while at higher p/p_0 values appeared as Type IV with a narrow hysteresis revealing the existence of small mesopores [3]. While, the nitrogen adsorption isotherms of 2AC and 3AC are type IV with obvious hysteresis loops confirming the existence of mesopores as depicted in Figure 3A and 3B. A further increase of the weight ratio of ZnCl_2 to cassava rhizome to some extent as in the case of 3AC leads to a decrease in S_{BET} due to the partial collapse of porous structure resulting in reduced microporosity as seen in Figure 3B [15].

These findings prove that the porosity can be tuned to some extent by varying amounts of activating agent as ZnCl_2 . Herein, the activation of the sample with a ZnCl_2 to CR weight ratio of 3 was chosen as the condition used in preparing magnetic- carbonaceous adsorbent because this condition produce a carbon sample with the high value of S_{BET} as well as the highest mesopore volume and a wide range of mesopore size (Figure 3B) which are a good candidate for adsorption of dye [26]. In addition, it was found that CR-ACs prepared through one pot-HTC have higher S_{BET} and overall porosity that those of commercial activated carbons (labeled as Com-PAC in Table 1) which presents the potential of CR as an effective carbon precursor for the production of mesoporous activated carbons

Table 1. Yield and adsorption parameters for samples studied

Sample	Yield (%)	S_{BET} (m^2/g)	V_t (cm^3/g)	V_{mi} (cm^3/g)	V_{me} (cm^3/g)	Mesoporosity (%)
PHC	-	207	0.09	0.08	0.01	11
1AC	28	1172	0.54	0.44	0.10	19
2AC	23	1425	0.83	0.49	0.34	41
3AC	21	1405	0.96	0.42	0.54	56
3AC-1M	21	554	0.62	0.14	0.48	77
3AC-2M	22	454	0.53	0.12	0.41	77
3AC-3M	20	440	0.61	0.10	0.51	84
Com-PAC	-	1078	0.49	0.45	0.04	8

Notation: S_{BET} = specific surface area; V_t = total pore volume; V_{mi} were obtained from the cumulative pore volume calculated using the 2D-NLDFT method for pores smaller than 2 nm; and V_{me} represents the volume of mesopores (pore size between 2 and 50 nm) calculated by $V_t - V_{\text{mi}}$; mesoporosity – percentage of the volume of mesopores in the total pore volume; The percentage yield was calculated as follows:

$$\text{Yield (\%)} = \frac{m_{\text{CR-AC}}}{m_{\text{CR}}} \times 100$$

where $m_{\text{CR-AC}}$ and m_{CR} are the weight of the CR-AC (in grams) and the CR before activation (in grams), respectively.

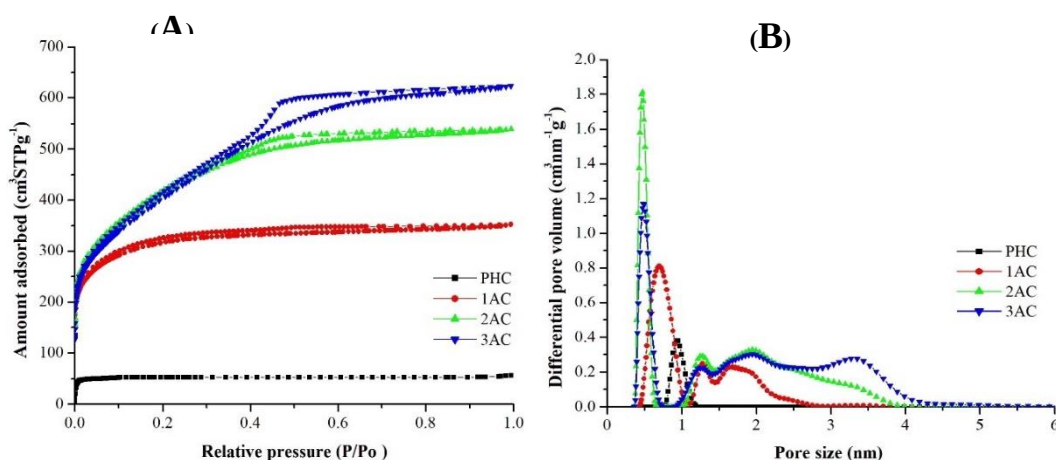


Figure 3. (A) Nitrogen adsorption isotherms for PHC, 1AC, 2AC and 3AC and (B) Differential pore size distributions (PSDs) up to 6 nm calculated using the 2D-NLDFT method for PHC, 1AC, 2AC and 3AC

3.2.2. Magnetic-AC obtained by one - pot $\text{ZnCl}_2/\text{FeCl}_3$ co-HTC.

The nitrogen adsorption isotherms and PSDs obtain from magnetic carbon series, 3AC-yM are illustrated in Figure 4A and 4B, respectively. Their structural parameters are listed in Table 1. From the results in Figure 4A, all N_2 adsorption -desorption isotherms were clearly of type IV with hysteresis loops in accordance with the IUPAC representing the characteristic of mesoporous materials. It implies that the magnetization of CR-AC through one-pot HTC did not destroy the mesoporous structure of obtained samples. The values of S_{BET} range from 440 to 554 m^2/g , V_t values range from 0.53 to 0.62 cm^3/g , V_{mi} and V_{me} values range from 0.10 to 0.14 cm^3/g and 0.41 to 0.51 cm^3/g , respectively. All magnetized samples, 3AC-yM, obviously exhibited a smaller S_{BET} , V_t and V_{mi} than those for a non-magnetic activated carbon, 3AC due to the coverage of iron particles on the surface and pore clogging during the magnetization process [24].

In addition, when the weight ratio of FeCl_3 to ZnCl_2 increased from 1 to 3, a reduction of S_{BET} and V_t values of magnetic activated carbons were observed (Table 1). These findings can be ascribed to various mechanisms; an excessive amount of FeCl_3 leads intensely catalyze the activation reaction contributing to pore merging. Moreover, higher amounts of FeCl_3 can result in bigger iron oxide particles acting as a template for the formation of mesopores. Therefore, an increase in V_{me} of 3AC-yM was observed with increasing FeCl_3 amount along with a reduction of S_{BET} and V_{mi} as displayed in Table 1 and Figure 4B [27, 28].

Thus, 3AC and 3AC-1M was selected as the representatives of nonmagnetic activated carbons and magnetic activated carbons for the further dye adsorption studies due to the relatively high values of S_{BET} , V_{mi} and V_{me} adsorption among each series of samples which are the crucial structural parameters for dye.

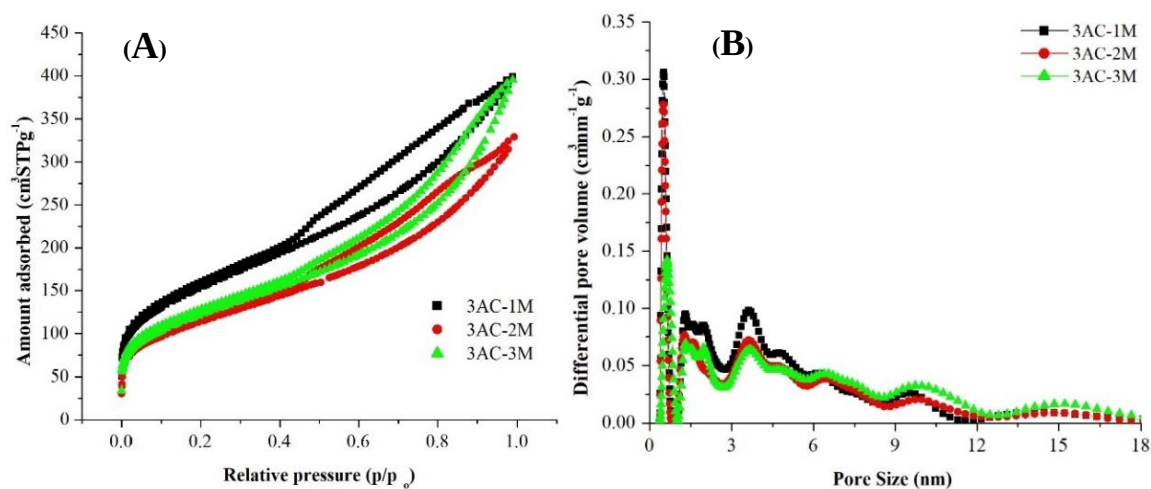


Figure 4. (A) Nitrogen adsorption isotherms for 3AC-1M, 3AC-2M and 3AC-3M and (B) Differential pore size distributions (PSDs) up to 18 nm calculated using the 2D-NLDFT method for 3AC-1M, 3AC-2M and 3AC-3M

3.3. Adsorption Studies

3.3.1. Effect of contact time.

The adsorption contact time is an essential factor in adsorption control process onto the adsorbents. Figure 5 shows the effect of contact time on methylene blue (MB) adsorption by 3AC and 3AC-1M investigated with the initial concentration of 100 ppm MB solution (pH 8, without any pH adjustment). The adsorption experiment was approximately carried out for 6 h to determine the adequate adsorption time. As seen in Figure 5, the amount of the adsorbed MB (q_t) onto all samples rapidly increased with the contact time in the first 35 min and it reached a constant value after 210 and 240 min for 3AC and 3AC-1M respectively. The fast initial adsorption of dye is mainly due to the abundant vacant sites on the surface of the adsorbent [29]. During adsorption, the adsorption rate slowly increased because of the saturation of the active sites and diffusing of dye from the outer surface to the pores of adsorbents [30]. As can be seen in Figure 5 and Table 1, the adsorption equilibrium time for 3AC and 3AC-1M were quite similar although the S_{BET} value of 3AC-1M is very smaller than that of 3AC. It is probably because a relatively higher fraction of mesopores in 3AC-1M ($\% V_{me}=77$) than that for 3AC enhances the diffusion rate of adsorbates from the external surface to porous structure in carbon framework [31].

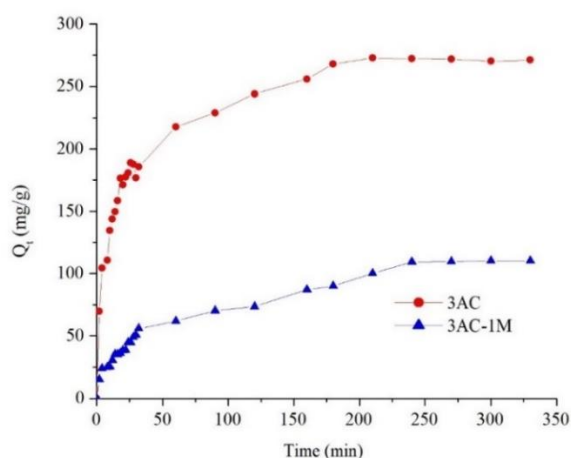


Figure 5. Adsorption kinetics of MB onto 3AC and 3AC-1M (Adsorption conditions: initial concentration 100 mg/L, temperature 25 °C, the solution pH 8 and adsorbent dosage 0.07 g, 250 rpm)

3.3.2. Adsorption isotherm study.

To examine the adsorption isotherm, the collected data were fitted with non-linear equation of Langmuir and Freundlich adsorption models (Figure 6). With respect to correlation coefficients (R^2) as shown in Table 2, it indicates the isothermal data was better fit the Langmuir model with R^2 of 0.9993, 0.9953 and 0.9996 for 3AC, 3AC-M and Com-PAC respectively. Based on this result, the adsorption behaviors of MB on all carbon samples were monolayer adsorption. The maximum adsorption capacities (q_m) as calculated according to the Langmuir model for 3AC, 3AC-M and Com-PAC are 274, 102 and 260 mg/g respectively. The result showed that q_m of CR-AC is comparable to that of Com-PAC.

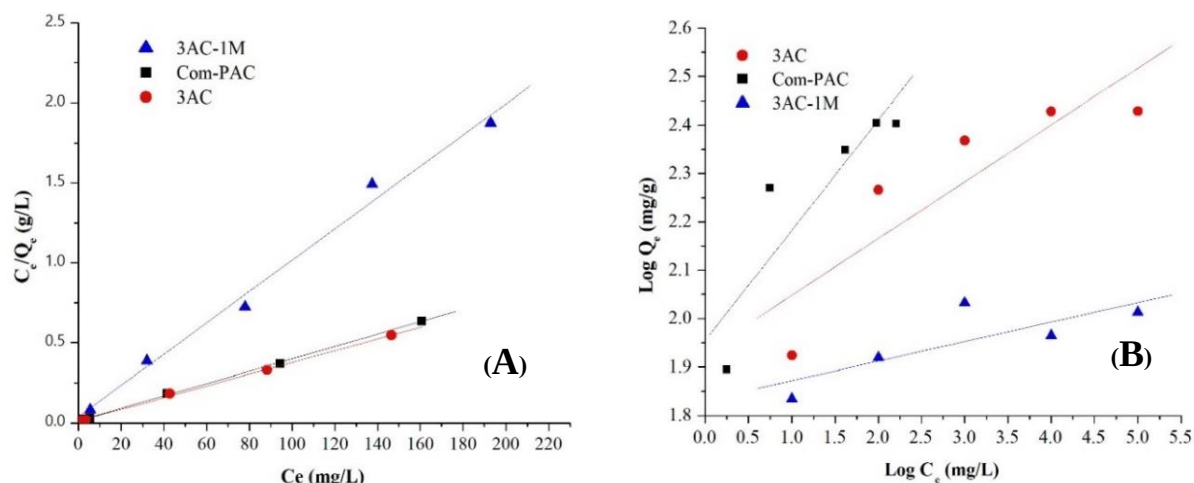


Figure 6. The linear regression by fitting the equilibrium adsorption data with (A) the Langmuir adsorption isotherm model and (B) Freundlich adsorption isotherm model

Table 2. Adsorption isotherm parameters of the Langmuir and Freundlich model for the MB adsorption on the sample studied

Sample	Langmuir model			Freundlich model		
	K_L (L/mg)	q_m (mg/g)	R^2	K_F (mg/g)	$1/n$	R^2
3AC	0.294	274	0.9993	103.72	0.2097	0.9037
3AC-1M	0.247	102	0.9953	56.92	0.1152	0.8948
Com-PAC	0.259	260	0.9996	90.18	0.2278	0.8876

The magnetic property added to CR-AC by one-pot HTC made the carbon sample as in the case of 3AC-1M more novel owing to the fast and easy separation from water by using an external magnet after completed adsorption [17]. This work presents that CR is a potential low-cost carbon precursor to produce multifunctional carbon adsorbents for dye removal.

3.4. magnetic properties

The magnetic hysteresis curves evaluated by the vibrating sample magnetometer (VSM) at 25 °C and the magnetic behavior of the resulting magnetic carbon, 3AC-1M are illustrated in Figure 7 and 8, respectively. Magnetization curves demonstrated that the saturation magnetization value was 3.343 emu/g which is consistent with the presence of iron oxide in the XRD results and 9.79% of Fe found by the EDS analysis. As seen in Figure 8, 3AC-1M was easily separated from water by an external magnet after adsorption which would be beneficial for pollutant separation and adsorbent recycled with magnets.

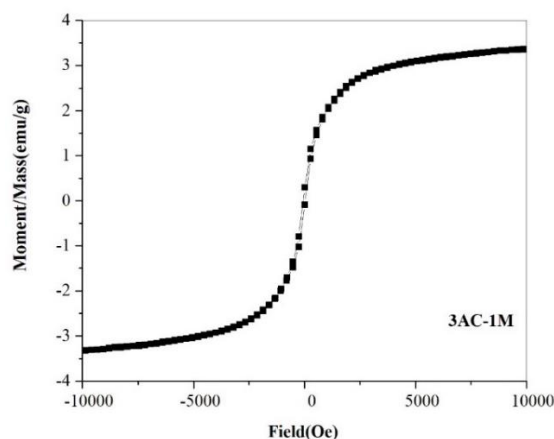


Figure 7. Magnetization curves obtained by VSM at 25 °C

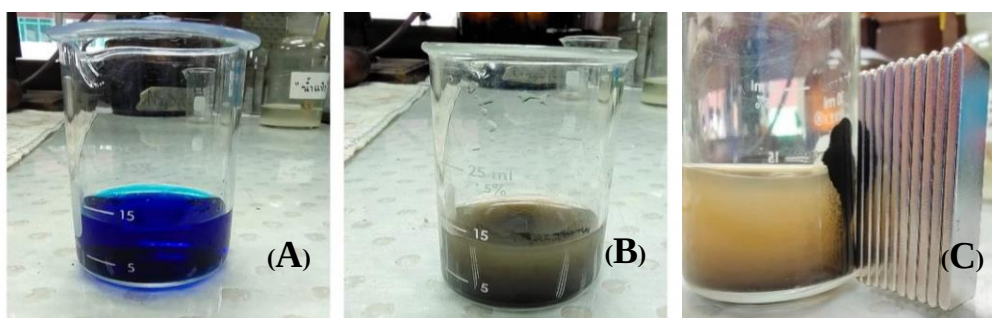


Figure 8. Photographs of (A) 3AC-1M dispersed in MB solution, (B) MB solution after adsorption by 3AC-1M for 2 min and (C) magnetic interaction between a magnet and dispersed 3AC-1M

Conclusion

Activated carbon (3AC) and magnetic carbon adsorbent (3AC-1M) were successfully prepared using cassava rhizome which is one of abundant agricultural wastes in Thailand as a carbon source. They are fabricated via a facile, one-pot HTC. The 3AC can be used to adsorb MB from aqueous solution with adsorption capacity of 274 mg/g which is comparable to the commercial activated carbons (260 mg/g). In addition, 3AC-1M shows the adsorption capacity of 102 mg/g MB with a saturation magnetization value of 3.343 emu/g. Such a synthesized magnetic carbon could be attracted by magnets, which would be beneficial for pollutant separation from the treatment media and adsorbent recycling. This work demonstrates that the carbon precursors from biomass waste as CR can be conveniently converted into efficient carbonaceous adsorbents and functionalized for example by adding magnetic properties into the carbon structure to enable manipulations in industrial operations.

Towards the circular economy point of view, the management of polluted water containing zinc which is a heavy metal after ZnCl_2 activation process should be also considered. In previous studies, various methods for recycling zinc from wastewater and turning it into new metal products or value materials have been extensively explored such as coupled coagulation and hydrothermal route producing highly purified zinc phosphate and adsorption coupled with in-situ sulfuring of zinc obtaining the highly-efficient photocatalyst for hydrogen evolution [32,33].

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