



Synthesis of Cassava Rhizome Biochar for Methomyl Adsorption

Lalita Kamolklang¹, Pariyaporn Seekhumlek¹, Anusara Kaeokan¹ and Apichon Watcharenwong^{1*}

¹School of Environmental Engineering, Institute of Engineering,
Suranaree University of Technology, Nakhon Ratchasima 30000, Thailand

*E-mail : w.apichon@sut.ac.th

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Abstract

This research aims to study the synthesis of biochar from cassava rhizomes and the factors involved in the adsorption of methomyl. Methomyl is a carbamate pesticide. The factors of interest in the synthesis of biochar include the pyrolysis temperatures of 300, 400, and 500 °C and the pyrolysis time of 2.5 hours under nitrogen gas conditions, and the obtained biochar was modified with phosphoric acid to increase the efficiency of adsorption. The synthesized biochar was examined using various techniques, including CHN/O, BET, SEM, FTIR, and XTM. The factors of interest in the adsorption study include contact time, agitation speed, and pH value. The study found that the temperature and duration of pyrolysis affect biochar quality. The selected biochar was obtained at 500 °C for 2.5 hours with the highest %C of 78.149 and the lowest H/C of 0.026, similar to other research studies. The biochar is of high quality, has a stable C ratio, and a low H/C. A higher carbon content results in more stable biochar. Modifying biochar with phosphoric acid results in an increase in its physical and chemical properties. The specific surface area from BET measurement and the average pore diameter increased from 2.29 to 3.39 m²/g and 1.57 to 6.54 Å, respectively. For methomyl adsorption experiments, it was found that equilibrium was reached after 180 minutes. The rotational speed and pH value affected the adsorption efficiency. The optimum condition for methomyl adsorption was an agitation speed of 200 rpm because it achieved the highest adsorption efficiency of 19.7%. Further agitation speed experiments revealed that turbulence is critical in controlling the solid-liquid mass transfer mechanism. The pH condition that resulted in the best adsorption was pH 3, with an efficiency of 27.90% and the highest adsorption capacity (q_e) at 2.79 mg/g.

Keywords : biochar; cassava rhizomes; methomyl; pesticides; phosphoric acid

Introduction

Currently, the agricultural sector uses pesticides in farming, accumulating pesticides in the environment. When pesticide accumulation exceeds saturation, it will cause desorption from the soil. Pesticides are washed away by the leaching process of rain into natural water sources [1]. Pesticides contaminating natural water sources can accumulate in the food chain of living organisms in these water sources. Methomyl, a class I (restricted-use pesticide) according to the US EPA, is harmful to mammals, fish, and aquatic invertebrates. The high water solubility (57.9 g/L at 25 °C) [2] of methomyl and its low sorption affinity to soil contribute to a high likelihood of detecting methomyl in surface and groundwater [3]. This results in an increased risk to human health [4]. Methomyl has neurotoxic effects with symptoms such as headache, dizziness, and abnormal muscle function [5]. Exposure to methomyl can cause difficulty breathing and, in high doses, respiratory arrest [6], nausea, vomiting, and abdominal pain [7]. Methomyl has been found in groundwater and surface water at 10 µg/L and 30 µg/L, respectively. The standards for soil and surface water pesticides should not exceed 0.1 µg/L, as regulated by the EU and US EPA [3]. However, the amount of methomyl detected is still higher than the standard.

This research focuses on methomyl treatment. Previous research studies have used various technologies to treat methomyl, such as adsorption, photodegradation, and advanced oxidation processes. Adsorption is widely used to remove pesticides because it is easy to apply and inexpensive [3]. Biochar is a carbon-rich product used as an adsorbent to remove contaminants from water supplies due to its physical and chemical properties. Biochar production is also relatively low-cost, as most of the starting materials are waste products from the industrial and agricultural sectors, leading to interest in synthesizing biochar from biomass. Biochar is a product of the carbonization of biomass, characterized by a porous structure. Biochar has properties that help improve soil quality [8], sequester carbon in the soil [9], and reduce greenhouse gas emissions [10]. Biochar is therefore a material with the potential to promote

sustainability in agriculture and environmental management. In this research, the starting raw material for producing biochar is cassava rhizome because cassava is one of the economic crops of Thailand. In the study by Aup-Ngoen in 2020, it was found that cassava rhizomes have the highest percentage of carbon content compared to durian peel, pineapple peel, and corncob, making it a suitable starting material for producing biochar [11]. Adsorption of atrazine and imidacloprid using phosphoric acid-modified biochar from agricultural waste showed maximum adsorption efficiencies of 70.7% for atrazine and 77.8% for imidacloprid, respectively. The phosphoric acid treatment increased the adsorption of both pesticides [12].

Therefore, the objective is to study factors in biochar synthesis from cassava rhizomes and investigate the factors involved in the adsorption of methomyl using biochar derived from cassava rhizomes.

Methodology

Preparation and modification of biochars

Preparation of biochars: 10g of cassava rhizomes were pyrolysis at temperatures of 300, 400, and 500 °C for 2.5 hours with a temperature increasing rate of 5°C/minute under nitrogen gas conditions.

Modification of biochar: The size of the biochar was controlled by grinding it into powder form and analyzing the particle size using a laser scattering particle size distribution analyzer. The average particle size was 54 µm. And then 10 g were immersed in 100 mL 14% H₃PO₄ solution for 24 hours at 25°C, to enhance its surface area and introduce functional groups such as P=O and P-OOH, which improve the adsorption of pollutants [13]. After that, the phosphoric-modified biochars were washed with distilled water until the pH of the supernatants was stable. Subsequently, the supernatants were discarded, and the biochars were oven-dried overnight at 105°C [14].

Characterization of biochars

The elemental analyzer measured the total elemental composition, such as carbon, hydrogen, nitrogen, and oxygen. Brunauer–Emmett–Teller (BET) was used to detect specific

surface areas of biochars. Fourier transform infrared (FTIR) spectra of biochars were conducted by an FTIR instrument. The morphology of the biochar was determined via scanning electron microscopy (SEM) and Synchrotron X-ray tomographic microscopy (XTM) at beamline 1.2W was operated at 1.2GeV, 150 mA in Synchrotron Light Research Institute (SLRI) were characterized the porosity by Octopus Analysis software and rendered in 3D tomographic reconstruction by using Drishti software. A laser scattering particle size distribution analyzer was used to measure the size and particle distribution of the material. Point of zero charge with salt addition technique To determine the charge on the surface of biochar.

Adsorption experiments

Varying methomyl pH solutions (3, 5, 7, 9, and 11), the pH values in the study were all adjusted with HCL solution and NaOH solution, and varying agitation speeds (100, 150, 200, and 250 rpm) were also investigated in separate experiments. Batch experiments were conducted using an orbital shaker at an agitation speed of 200 rpm at room temperature. To an Erlenmeyer flask 250 ml filled with 10 mg/L methomyl (aq), Volume 100 ml, a specific mass of biochar (1 g/L) was added. Equilibrium studies were performed by shaking the suspension containing biochar and methomyl in a particular time interval, up to a maximum of 360 min. The samples were collected at 2 mL, poured into the vials at the specified time, and filtered through a 0.45- μ m Nylon filter before methomyl analysis. Methomyl removal efficiencies were measured in triplicates following a specific protocol for each condition. The filtrate was analyzed for pesticide concentrations using high-pressure liquid chromatography (HPLC) techniques. The analysis was performed using an Agilent 1260 Infinity II HPLC system (Agilent Technologies), equipped with a C18 column (Agilent ZORBAX Eclipse Plus, 4.6 \times 250 mm, 5 μ m particle size). The mobile phase consisted of 65% acetonitrile and 35% DI water, and the flow rate was set at 0.5 mL/min. Detection was performed at a wavelength of 234 nm. [15]. The experimental data were analyzed using

Microsoft Excel. All experiments were conducted in triplicates, and the results were expressed as mean values with standard deviations. In the adsorption experiments, the effect of pH (ranging from 3 to 11) on methomyl removal efficiency was analyzed. Statistical significance was assessed using one-way ANOVA at a significance level of $p < 0.05$ to determine the differences in adsorption efficiencies across the different experimental conditions.

Results and Discussion

Characterization of biochars

Table 1 shows that the physical characteristics of biochar produced at 300°C are somewhat brownish, resembling wood, which suggests incomplete combustion. In contrast, biochar produced at 400°C and 500°C is entirely black, indicating complete combustion. Therefore, only biochar produced at 400°C and 500°C was selected for CHN/O analysis to determine its composition.

The Biochar synthesized at a temperature of 500 °C for 2.5 hours exhibited the highest carbon content at 78.149%, with an H/C ratio of 0.026, consistent with other studies that indicate the significant influence of pyrolysis temperature on biochar's elemental composition. Higher pyrolysis temperatures increase the carbon concentration in biochar while reducing hydrogen and nitrogen levels, enhancing the chemical stability of the biochar. This makes it well-suited for agricultural and environmental applications, such as soil improvement and pollutant adsorption. Furthermore, as the H/C ratio decreases, the aromatic structure of the biochar increases, leading to greater stability and improved pollutant adsorption capacity [16].

Then, the selected biochar was treated with phosphoric acid. From Table 2, the modification of phosphoric acid could increase the specific surface area of biochar produced from cassava rhizome, which was proven by BET analysis. Although the change in specific surface area was small, the biochar modified with phosphoric acid showed a higher average pore diameter, similar to Peng's study [13].

Table 1 Elemental compositions of Biochar obtained from the different temperatures




Photo/ Sample Biochar		% C	% H	% N	Ratio H/C
	300 °C / 2.5 hr.	N/A			
	400 °C / 2.5 hr.	75.132	3.7878	1.7910	0.0504
	500 °C / 2.5 hr.	78.149	2.0398	0.6919	0.0261
Abbreviation: NA = not available.					

Table 2 Physiochemical characteristics of biochars from BET analysis

Sample	Surface Area (m ² /g)	Average pore diameter (Å)	Total pore volume (cc /g)
Biochar CA	2.29	157.40	0.0176
Biochar+ H ₃ PO ₄	3.39	654.4	0.0133

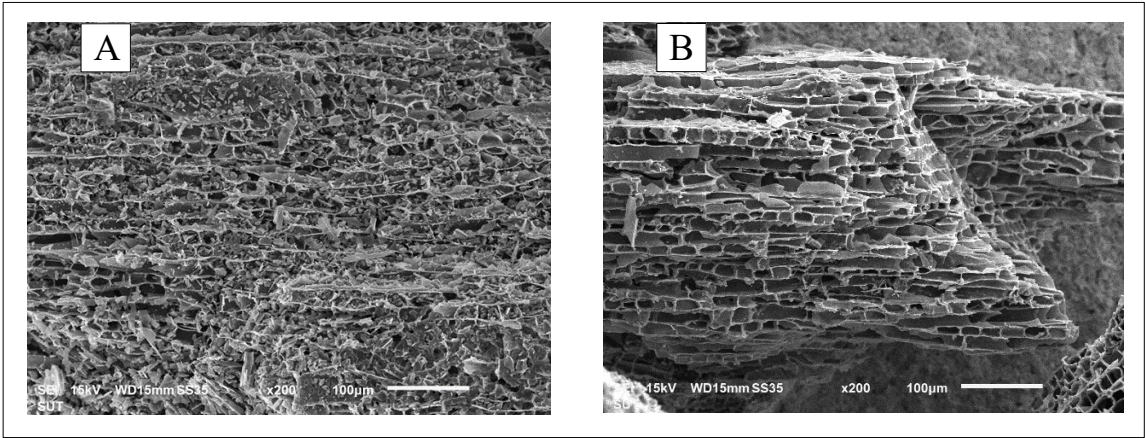


Figure 1 SEM images of (A) Biochars CA.
(B) BiocharCA + H₃PO₄.

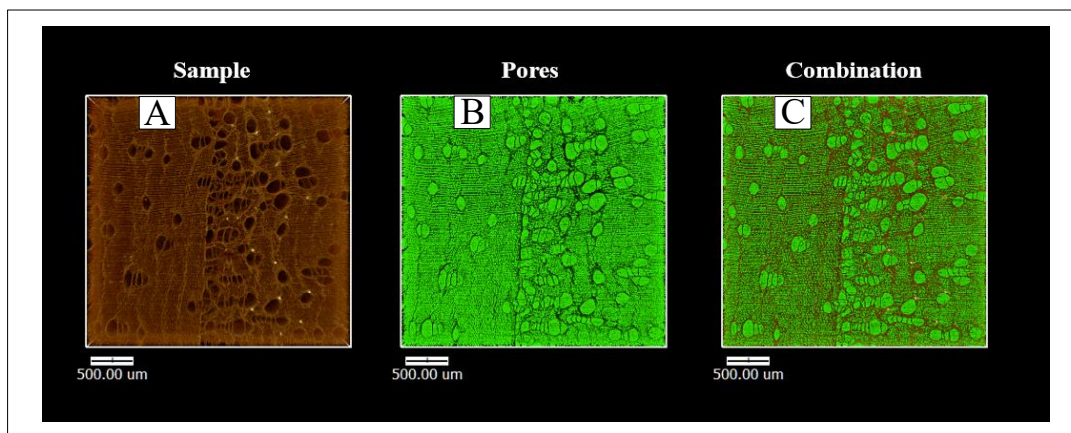


Figure 2 Results of the X-ray Tomographic Microscopy (XTM) technique of the synthesized biochar cassava rhizomes 500°C/2.5 hr.

From Figure 1, This SEM image reveals significant changes in the structure after treatment with phosphoric acid (H_3PO_4). The porous structure becomes apparent, and the surface appears more collapsed or compacted, which may be due to the corrosive effect of phosphoric acid, which may cause some of the mesopores to collapse. The structure in Image B shows that although the micropores are increased (resulting in increased surface area), the larger pores may be partially blocked or their volume reduced. The 3D X-ray characteristics and porosity (%) of biochar were studied using the X-ray Tomographic Microscopy (XTM) technique from Synchrotron Light Research Institute (SLRI). XTM results in Figure 2 show a 3D X-ray image in which Figure 2A shows only biochar solid structure (brown color), Figure 2B shows only the pore in the biochar sample, and Figure 2C shows both biochar solid structure combined with pore in structure. From XTM measurement, a porosity of 28% was obtained, which means that the remaining 72% was solid biochar.

From Table 2, The BET analysis supports these visual observations, showing that the surface area increased after modification (from 2.29 m^2/g to 3.39 m^2/g), while the total pore volume decreased (from 0.0176 cc/g to 0.0133 cc/g). This increase in surface area is likely due to the creation of more micropores, while the

reduction in pore volume and the larger pore diameter (from 157.4 Å to 654.4 Å) suggest that some of the mesopores were collapsed or blocked during the treatment. The chemical nature of H_3PO_4 treatment can explain this phenomenon. The acid not only reaches the surface of the biochar but also introduces new functional groups, especially in micropores. While these micropores increase the surface area, larger mesopores' collapse or partial blockage reduces the overall pore volume.

Additionally, the increase in average pore diameter may result from structural changes like the selective expansion or merging of particular pores during the acid treatment. Other studies report similar effects. For example, Peng et al. (2017) found that H_3PO_4 modification of biochar increased surface area by promoting microporosity while reducing pore volume due to structural collapse of mesopores [13]. Similarly, Chen et al. (2018) observed a significant increase in surface area after H_3PO_4 treatment, despite lower pore volume, due to the creation of additional functional groups that enhance adsorption properties [17]. In conclusion, H_3PO_4 treatment increases the surface area by enhancing micropore development, even though mesopores may collapse or become blocked, resulting in reduced pore volume but improved adsorption efficiency.

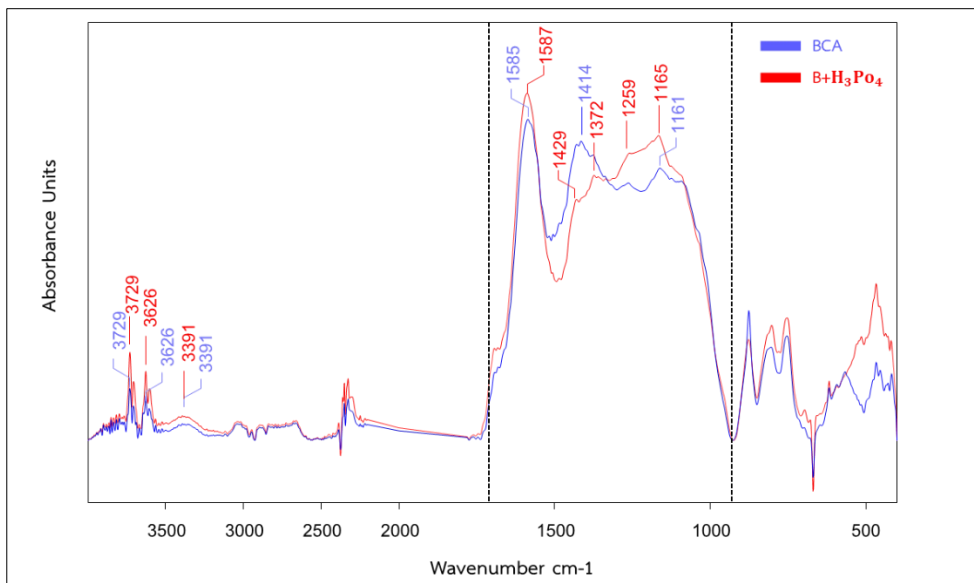


Figure 3 FTIR spectra of BiocharCA. and BiocharCA + H₃PO₄.

The results of the Fourier transform infrared spectrophotometer (FTIR) are shown in Figure 3. A peak at 3729–3391 cm⁻¹ This range corresponds to the stretching vibrations of -OH groups, indicating the presence of hydroxyl groups due to moisture or water content in the samples. The higher intensity in the B-H₃PO₄ sample suggests a more significant presence of hydroxyl groups compared to the untreated BCA sample [18]. A peak at 1587–1429 cm⁻¹ This region is associated with C=C stretching vibrations in aromatic rings characteristic of carbonized structures. The B-H₃PO₄ sample shows a more vigorous intensity, indicating enhanced aromatic carbon structures due to H₃PO₄ treatment [19]. A peak at 1372–1269 cm⁻¹ The peaks in this range can be attributed to C-H bending vibrations in methylene groups and C-O stretching vibrations in esters. The stronger absorption in the B-H₃PO₄ sample reflects the formation of new chemical bonds in the modified biochar structure. A peak at 1165–1101 cm⁻¹ This region corresponds to P=O stretching vibrations, which indicate the presence of phosphate groups introduced by the H₃PO₄ treatment. The distinct absorption in B-H₃PO₄ highlights the incorporation of phosphorus into the biochar structure [13]. The FTIR analysis shows significant structural changes in the biochar after treatment with phosphoric acid. The B-H₃PO₄ sample exhibits increased phosphate (-P=O) groups, enhanced aromatic

carbon structures (C=C), and more pronounced hydroxyl (-OH) groups. These changes result from crosslinking and new bond formation facilitated by the H₃PO₄ modification process, which strengthens the carbon skeleton and alters the biochar's functional groups [20]. Phosphoric acid treatment shows an increase in the formation of P=O, P=OOH groups, similar to Peng's studies [13], where P=O, P=OOH groups were detected after treatment with phosphoric acid. Research has studied the size of biochar for adsorption. It was found that small biochar had increased adsorption. Therefore, biochar is crushed before use [21]. The size and distribution were then measured using a laser scattering particle size distribution analyzer. For the particle size distribution analysis using laser scattering, it was found that the particle size distribution of biochar was in the range of 10–300 μm, and the average particle size was 54 μm, as shown in Figure 4.

By studying the pH value at zero surface charge, points of zero charge of the adsorbent are shown in Figure 5, where the value refers to the pH at which the sum of the surface charges of the adsorbent is equal to zero. When the pH value of the solution is lower than the value, the surface of the adsorbent will display a positive charge. When the pH value of the solution is higher than the value, it will cause the surface of the adsorbent to display a

negative charge [22]. The results of this study found that the point of zero charge of biochar from cassava rhizomes is 6.05, meaning that at pH of 6.05, the surface charge of cassava

rhizome biochar is zero, and the of cassava rhizome biochar modified with phosphoric acid is 5.15 shown in Figure 6B.

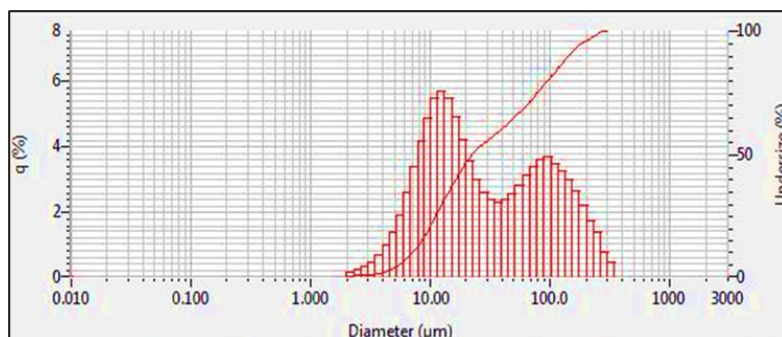


Figure 4 Measurement of size and distribution of biochar particles

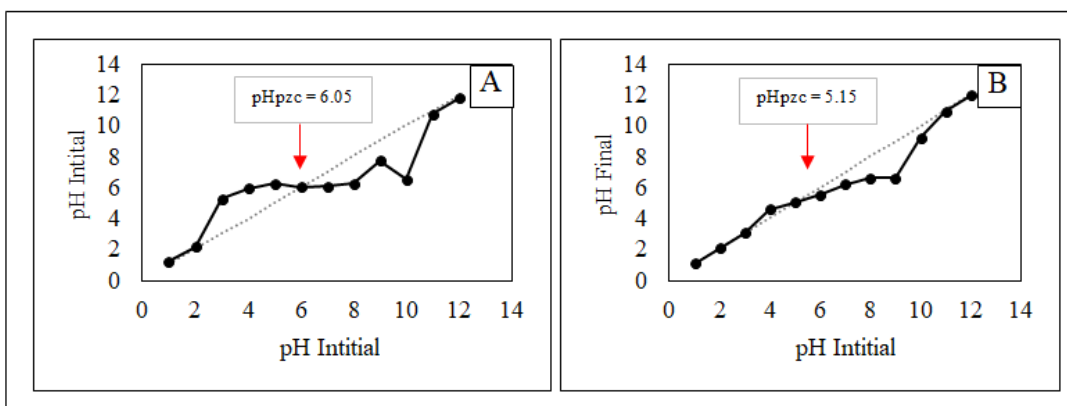


Figure 5 Points of Zero Charge (pH_{pzc}) of biochar.

(A) biochar from cassava rhizomes (B) biochar from cassava rhizomes modified with phosphoric acid

Adsorption Experiment

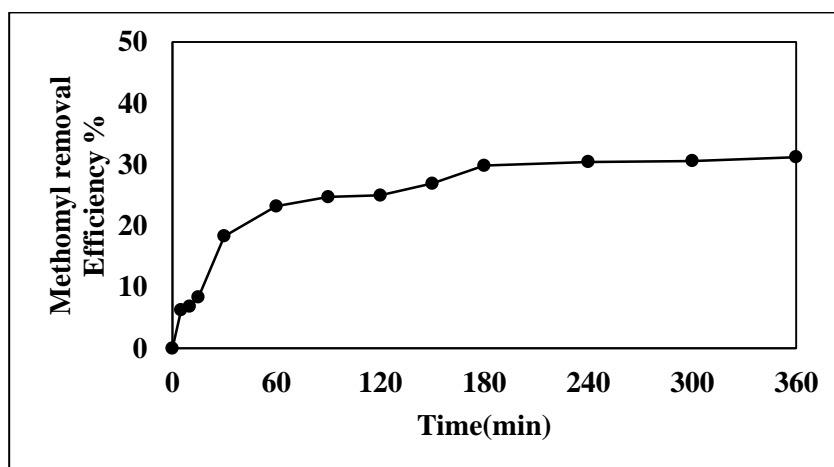


Figure 6 Effect of the contact time Initial concentration methomyl 10 mg/l, biochar from cassava rhizomes modified with phosphoric acid 1 g, Initial pH 6.20, agitation speed 200 rpm.

The ability to adsorb methomyl by biochar from cassava rhizomes modified with phosphoric acid increases with increasing contact time, and the adsorption rate increases rapidly during the first 180 minutes, as shown in Figure 6. However, over time, the adsorption rate caused by the movement of methomyl molecules in the adsorbent particles begins to

slow until equilibrium is reached at a contact time of 180 minutes. The adsorption capacity is most remarkable from 180 to 360 minutes, during which the adsorption rate equals the desorption speed. This study found that during the contact period of 180 minutes, the efficiency was 29.84%.

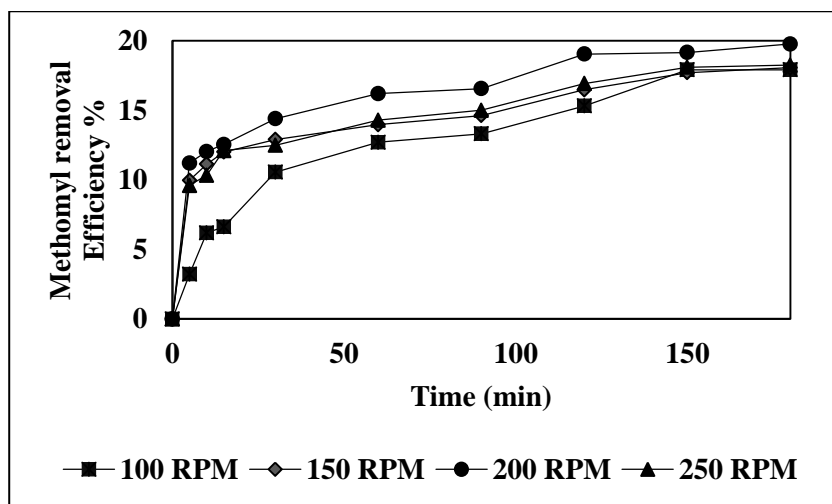


Figure 7 Effect of the agitation speed Initial concentration methomyl 10 mg/l, biochar from cassava rhizomes modified with phosphoric acid 1 g, Initial pH 6.00, agitation speed 200 rpm.

Figure 7 shows that when the agitation speed was increased, the methomyl adsorption efficiency by biochar was increased. However, when the speed is increased to more than 200 rpm, the adsorption efficiency decreases. The effect of turbulence is one of the critical factors that are important in controlling the solid-liquid mass transfer mechanism [23]. Figure 8 shows At pH 3, the biochar demonstrates the highest adsorption capacity (q_e) at 2.79 mg/g, indicating that in acidic conditions, biochar is highly effective at adsorbing methomyl. As pH increases, q_e decreases. At pH 5, it drops to 2.46 mg/g, and by pH 11, it reaches 2.05 mg/g. This decline in q_e is consistent with the fact that methomyl molecules become less available for adsorption due to increased hydrolysis in basic conditions. In addition, pH significantly affected methomyl removal efficiency, with the highest removal at pH 11 (50.81%). This was significantly different from the removal rates at pH 3–9, which ranged from 20.55% to 27.90% ($p < 0.05$). The higher efficiency at pH 11 is attributed to base-

catalyzed hydrolysis, where methomyl breaks down into less toxic by-products, rather than just adsorbing onto the biochar. This aligns with previous studies on methomyl degradation in alkaline conditions [22]. At pH 3, the adsorption efficiency was the highest (27.90%), likely due to stronger electrostatic interactions between the biochar and methomyl under acidic conditions. In contrast, neutral and slightly basic pH levels (7–9) showed lower efficiency due to reduced electrostatic attraction as methomyl becomes less charged. Post-hoc tests confirmed a significant difference between pH 3 and pH 11 ($p < 0.05$), supporting the idea that both adsorption and hydrolysis play key roles in methomyl removal at different pH levels. In summary, lower pH favors adsorption, while higher pH promotes degradation, consistent with the behavior of similar compounds [24]. Optimizing pH can therefore maximize methomyl removal depending on the desired mechanism. At pH 3, the biochar demonstrates the highest adsorption capacity (q_e) at 2.79 mg/g, indicating that in acidic conditions, biochar is

highly effective at adsorbing methomyl. As pH increases, q_e decreases. At pH 5, it drops to 2.46 mg/g, and by pH 11, it reaches 2.05 mg/g. In the research by Fathy, N. A., Attia, A. A., & Hegazi, B. (2016), the adsorption capacity of carbon xerogel was found to be 15.52 mg/g. Generally, carbon xerogel has a higher surface area and a more developed porous structure, which is the reason for its higher adsorption capacity [25]. This decline in q_e is consistent with the fact that methomyl molecules become less available for adsorption due to increased hydrolysis in basic conditions. Methomyl, a carbamate pesticide, undergoes rapid decomposition in alkaline conditions due to base-catalyzed hydrolysis. When the pH exceeds its pKa of 9.7, hydroxide ions (OH^-) attack the carbamate group in methomyl, accelerating its

breakdown into simpler compounds and leading to its degradation into products like methomyl oxime and methylcarbamic acid [24]. This is consistent with findings from research of Wang, Z. et al. (2022), which show accelerated decomposition in basic environments due to nucleophilic attacks on carbamate structures, and Akl, M. A. et al. (2016), who demonstrated that in the pH range of 2-8, methomyl is relatively stable. In contrast, when the pH increased to 10, the residual methomyl content was only 12% of the original value. Moreover, at pH values as high as 12, methomyl was completely degraded into other compounds, which means that methomyl is relatively degraded in the alkaline solution. Therefore, the effect of pH on the adsorption capacity was investigated in the range of pH 2-8 [26].

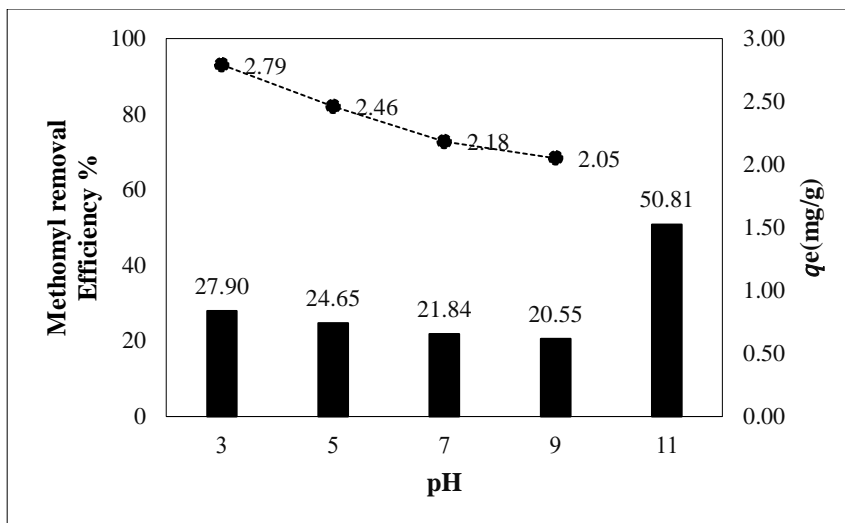


Figure 8 Effect of pH on methomyl removal. Initial concentration methomyl 10 mg/l, biochar from cassava rhizomes modified with phosphoric acid 1 g, agitation speed 200 rpm.

Conclusions

In this study, biochar was successfully synthesized from cassava rhizomes, with the optimal conditions being pyrolysis at 500°C for 2.5 hours. This condition produced biochar with a high carbon content of 78.149% and a low H/C ratio of 0.026, indicating its high stability. Biochar's stability is primarily attributed to its high carbon content and low H/C ratio, essential for applications requiring long-term carbon sequestration and enhanced material properties. Biochar was modified using phosphoric acid to

improve its physical and chemical characteristics. The modification increased the specific surface area from 2.29 m²/g to 3.39 m²/g and the average pore diameter from 1.57 Å to 6.54 Å, enhancing its porosity and adsorption capacity. The XTM measurements revealed a porosity of 28%, with the remaining 72% as solid biochar. The phosphoric acid treatment also introduced new functional groups, including P=O and P-OOH, as confirmed by FTIR analysis. These chemical modifications are critical for enhancing the biochar's reactivity and adsorption performance. The adsorption experiments

demonstrated that the biochar modified with phosphoric acid exhibited a significant adsorption capacity, particularly between 180 and 360 minutes, where equilibrium between adsorption and desorption was observed. Key factors influencing adsorption efficiency were agitation speed and pH, with optimal adsorption occurring at a stirring speed of 200 rpm. In acidic conditions (pH 3), the removal was 27.90%, attributed to strong electrostatic interactions between the biochar and methomyl. Neutral to slightly basic pH levels (7–9) showed lower efficiency due to reduced electrostatic attraction. This study demonstrates the potential of cassava rhizome biochar, particularly when modified with phosphoric acid, as an efficient and eco-friendly adsorbent for pesticide removal from aqueous solutions. Its high stability, porosity, and surface functionality make it a promising candidate for environmental remediation. Future research should explore the scalability of this method for industrial applications and further investigate the biochar's performance in actual environmental conditions. Additionally, the study could be expanded to include adsorption isotherms and kinetic models to better understand the adsorption mechanisms at varying pollutant concentrations.

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