

REVIEW ARTICLE

A systematic literature review on the effects of synthesis conditions on the physicochemical properties of activated carbons and their performance in methylene blue adsorption

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Abstract

This systematic literature review (SLR) describes the trend of study in activated carbons (ACs) from various biomass sources, specifically coconut shells, rice husks, and bamboo, for the adsorption of methylene blue (MB). Data acquisition and extraction from online databases were performed to review and discuss the effects of the synthesis parameters (*i.e.*, carbonization temperature and holding time) on the physicochemical properties of the synthesized ACs (*i.e.*, surface area, pore volume) and the effects of physicochemical properties to the MB adsorption. The SLR shows that the carbonization temperatures significantly affect the surface area and pore volume of ACs synthesized from coconut shells and rice husks, while ACs synthesized from bamboo were significantly influenced by the holding time. MB adsorption by ACs from all three biomasses increased with the increasing surface area and pore volume. This SLR could be a guideline to the synthesis ACs from biomass for the removal of dye in wastewater.

Keywords: Activated Carbon; Coconut Shell, Rice Husk, Bamboo, Biomass Waste; MB Adsorption; Systematic Literature Review

1.0 Introduction

Studies on the production of activated carbons (ACs) from various biomass sources have been emerging more recently due to their high potential as adsorbents for removing water pollutants like heavy metals and dyes from various industries. Despite the emergence of new carbonaceous materials such as carbon nanotube and graphene, ACs still preferable for industrial and

commercialized adsorbents due to their low cost, large surface area, and high porous structure. They are also better in performance and stability during the adsorption process [1].

On the other hand, various industries have been partly responsible for water pollution through the inappropriate discharge of their production effluents. Among all industries, the textile finishing industry caused the highest water pollution due to the discharge of dyes in their effluents. About 10 to 15% of the dyes exist in the effluents and methylene blue, MB ($C_{16}H_{18}N_3S$) is the typical example [2]. The presence of dyes in water increases the water's turbidity, limiting the amount of sunlight that passes through the water for the photosynthesis process of aquatic plants. Therefore, the use of ACs synthesized from biomass sources would certainly be a favorable option to produce low-cost adsorbents for the adsorption of pollutants in the textile industry.

However, many biomass sources are being converted into ACs and the studies keep increasing. ACs synthesized from different biomass sources could have different physicochemical properties and adsorption performance. In addition, various factors affect the synthesis and performance of ACs. Different biomass sources could result in ACs with varying structures of pore and surface area distributions, reflecting the differences in adsorption capability towards MB removal. Hence, a comparative study is encouraging to show the correlations of the synthesis parameters to the physicochemical and adsorption properties of the ACs.

In this systematic literature review (SLR), we identified the gap of recent studies in online databases (i.e., ScienceDirect, Lens.org and Scopus) surrounding the potential ACs synthesized from biomass sources, which are abundantly available in Malaysia and specifically used for the dye removal application. Based on the research trend between 2015 and 2020, there is still a lack of comparative studies on the synthesis parameters, physicochemical properties, and MB adsorption of ACs from various biomass sources. Hence, this SLR was made to identify the top three biomass sources being used for its production. Then, comparative analyses were performed to compare and find the effects of the synthesis parameters on the physicochemical properties and the effects of the physicochemical properties on the adsorption properties of ACs in the removal of MB in wastewater.

2.0 Materials and Methods

2.1. Identification of research trend and biomass sources selection

In this study, two types of searches were performed, requiring multiple databases to be used: patent search and scholarly works search. Both were necessary to produce a thorough analysis of the research trend in ACs in Malaysia. The selection was made based on the number of existing patents and scholarly works on the ACs. The patent search was performed using the Lens.org database. The key features of the database that allow for a better trend analysis, such as mapping keywords by countries, were also applied for this study. Meanwhile, ScienceDirect and Scopus databases were selected for the scholarly works search due to their extensive databases of reliable literature and refinement features. The selection of three biomass sources with the highest potential for ACs synthesis for dye removal application was made using the search string: (“activated carbon” AND “bamboo” AND (“methylene blue” OR “dye”)) across these databases. The resulting trend in the number of published papers from recent studies (from 2015 to 2020) was analyzed for biomass selection.

2.2. Selection of research articles for analyses

The identification and selection of relevant literature for the final list of papers were made by three phases of the screening process such as follows:

- i. Initial phase: Advanced command search feature using the refined search string was applied.
- ii. First phase: The papers were filtered based on their title and abstract.
- iii. Second phase: The selection criteria were applied during full-text reading to get the final selection of literature.

After searching the list of potential papers based on the search string generated, all the documents obtained on the results page were exported as BibTex files to screen through the titles and abstracts. Only literature relevant to the current study was included for the second phase screening. The selected literature was then assessed whether they would be included or excluded based on the proposed inclusion and exclusion criteria needed to answer the research questions. The snowballing method was also applied to find additional documents relevant to the study among the resulting papers. Snowballing was done by skimming through the list of references cited in the articles, screening based on the title and abstract, and being assessed with the same inclusion and exclusion criteria. The proposed inclusion and exclusion criteria were as follows:

Inclusion Criteria:

- i. Studies described ACs derived from biomass sources that are available within Malaysia.
- ii. Studies described the preparation and activation methods used to produce the ACs.
- iii. Studies included the adsorption or physicochemical properties of the ACs.
- iv. Studies described the adsorption properties towards MB.
- v. Studies must be published from 2015 to 2020.

Exclusion Criteria:

- i. All systematic review, meta-analysis papers.
- ii. The study paper was not written in English.
- iii. Studies only described ACs derived from biomass sources that are unavailable in Malaysia.
- iv. Studies described ACs derived from other non-biomass sources.
- v. Studies described only ACs derived from non-abundant biomass sources in Malaysia.
- vi. Studies published before 2015.

2.3. Data extraction & analysis

All data extracted from selected literature were based on the formulated research questions (Table 1). The effects of synthesis conditions (carbonization temperature and holding time) on the physicochemical properties (surface area and total pore volume of ACs) derived from the selected three biomass sources were highlighted. Furthermore, the relation between the physicochemical properties and the adsorption performance of the ACs based on percentage dye removal (%) was also analyzed.

Table 1: Research questions formulated from PICOC strategy

Research Questions	
Main RQ	What are the differences in dye adsorption performance of the ACs synthesized from different biomass sources for wastewater treatment application in Malaysia?
Sub-RQ 1	What are the potential ACs which can be produced from biomass sources in Malaysia for dye removal in wastewater treatment?
Sub-RQ 2	What factors contribute to the adsorption and physicochemical properties of ACs for dye removal application?
Sub-RQ 3	How do the preparation conditions affect the physicochemical properties of the synthesized ACs from different biomass sources?

Sub-RQ 4	How do the physicochemical properties affect the adsorption properties of ACs produced from different biomass sources?
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2.4. Normalization of MB removal data

For the removal of methylene blue, it was realized that some research articles used different experimental conditions, especially on the mass of the AC, concentration of the MB and the reaction time. Therefore, the unit of the MB removal was normalized as follows:

$$\text{Normalized Removal of MB} = \frac{\text{removal of MB (\%)}}{\text{mass of AC (mg)} \times \text{concentration of MB} \left(\frac{\text{mg}}{\text{L}}\right) \times \text{contact time (h)}}$$

Therefore, the unit can be simplified as % removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹.

3. 0 Results and Discussion

Activated carbon (AC) is a versatile material composed of carbon with an amorphous solid structure with a high degree of porosity and a well-developed surface area with a wide range of functional groups on its surface [3]. ACs are widely used in many applications, but they are mainly known as adsorbents due to their highly porous structure. The most significant part of its structure is its pores, which are mainly categorized into three groups which are macropores, mesopores, and micropores [4]. With the exception of macropores that provide the least contribution, all other pores give rise to AC's preference as an adsorbent material as they increase the surface area. Their presence is the primary source of adsorption not only for water and gas but also for removing contaminants and certain chemicals from the gas and liquid substances [5]. Malaysia has contributed to many published studies on ACs synthesized from abundantly available local biomass sources. For example, based on the data collected from the Lens.org database (Fig. 1a), Malaysia has contributed up to 35.7%, 32%, and 27.9% of published studies on ACs synthesized from palm oil empty fruit bunches (EFB), palm kernel shell, and durian shell, respectively. Nonetheless, the current number of published research articles on ACs synthesized from these biomasses is still insufficient to be selected in this review which requires more extensive data on the physicochemical and adsorption properties.

As shown in Fig. 1b, the number of published papers in the databases is significantly low (< 500 papers) for ACs synthesized from palm oil EFB, palm kernel, and durian shell. In comparison to these, a higher number of papers (>1500 papers) were more focused on the coconut shell, rice husk, and bamboo, signifying the increasing interest by the science community in using these

biomasses to synthesize ACs, especially in Asian countries. Therefore, 43 research articles (were selected based on the three chosen biomass sources (coconut shell, rice husk, and bamboo) for further analysis in this review.

The initial screening resulted in a total of 190 articles. After removing 41 duplicate records, a total of 149 articles were accessed, out of which 56 articles were excluded based on their titles and abstracts. Next, 93 full-text articles were reviewed, out of which 44 studies were eligible for the qualitative analysis according to the established inclusion criteria. The 44 papers were composed of articles containing the physicochemical and adsorption properties of ACs from the three selected biomass sources with the respective distributions: coconut shell [6-22], bamboo [16, 23-34] and rice husk [35-48]. The analyses were divided into two categories: (a) synthesis conditions affecting the differences in physicochemical properties of ACs and (b) effects of physicochemical properties onto MB adsorption performance. A list of papers and tabulated information can be referred to Table S1-S3 in the electronic supplementary information.

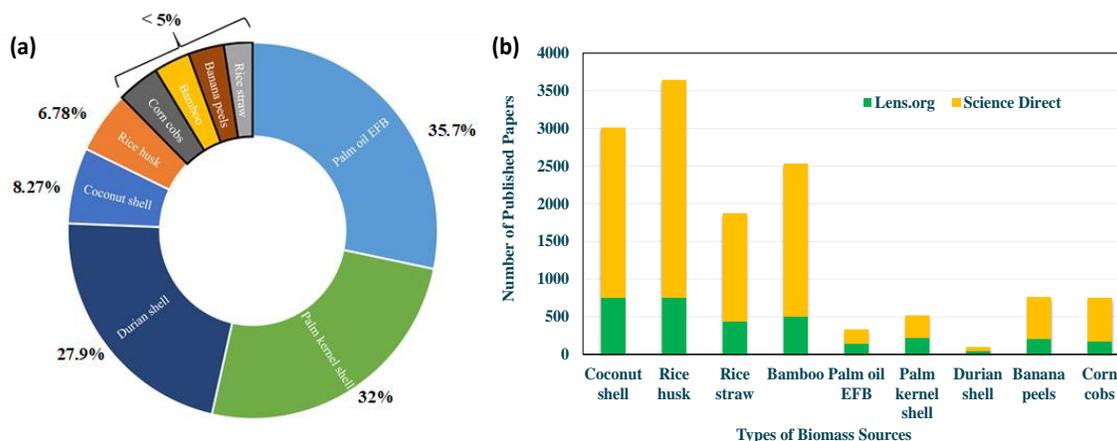


Fig. 1: (a) Percentage of published scholarly works on ACs synthesized from various local biomass sources contributed by research institutions within Malaysia based on the Lens.org database and (b) Cumulative number of published papers on ACs synthesized from different biomass sources globally from 2015 to 2020 based on the Lens.org and ScienceDirect databases.

The standard method used in the synthesis of AC comprises of 2 steps: (1) carbonization and (2) activation. Depending on the precursor type, some may require the sample's additional pre-treatment process before proceeding to the two steps mentioned above. Carbonization or pyrolysis is a thermal treatment process to decompose raw materials (in this case, biomass) at higher temperatures under inert gas purge and is usually performed in a furnace. This process is the crucial

step in the production of biochar from biomass. It is necessary to improve the carbon content of the solid and remove volatile matters and tars [49]. During the carbonization process, several factors may influence the quality of the final product (AC), including the temperature as a key factor, followed by the heating rate of reaction, the amount and flow rate of the inert gas supplied, and holding time. Higher temperatures will generally result in higher amounts of volatile species released that contribute to low biochar yields. However, studies have shown that better ACs qualities can be achieved despite the low yield. Finding the right temperature is crucial. If it is too high, undesirable substances (usually tar) appear to form as deposits due to the formation of narrow pore structures of precursors during this devolatilization process [50]. Therefore, although better biochar quality can be obtained with higher temperatures, applying higher temperatures beyond the necessary values will not be feasible since it may result in a lower yield of carbon content with lower available surface area for adsorption due to the presence of tars on the AC's surface that may occupy the pores.

The carbonization process often leads to low biochar adsorption capability. As a result, an activation step is introduced to increase the biochar's surface area and pore structures, including its pore sizes. The activation process first eliminates the disorganized carbons (tarry substances that block the pores) and exposes the lignin to the activating agents to develop the microporous structures. The heat treatment at high temperature of the pores' walls would then widen up the existing meso and macropores, thus, reducing the volume of micropores.

In this step, the biochar formed earlier will be transformed into ACs and activated using various activation methods such as physical, chemical, physicochemical, or microwave-assisted activation. In some cases, the activation process takes precedence over the carbonization process; however, it depends on the biomass sources used. Physical activation consists of heat and gas (*e.g.*, steam, CO₂, N₂, or a mixture of different oxidizing gases), chemical activation by chemical agents (*e.g.*, acid, base, metal oxide, alkaline metal), physicochemical activation by heat and chemical, and microwave-assisted activation by microwave radiation [51]. The activation process depends on different parameters such as particle size, retention time, impregnation ratio, process structure, activation time, precursor properties, and chemical substances [52].

In this SLR, the comparison analysis was only done for the carbonization process's synthesis conditions: carbonization temperature and holding time. According to data extracted in Fig. 2(a-

b), Bamboo ACs have wider ranges of carbonization temperature that can be applied during the synthesis process to obtain significant surface areas and pore volumes than that of ACs from rice husk and coconut shells. On average, bamboo ACs have the surface area and pore volume between 400 to 1500 m²g⁻¹ and 0.2 to 1.0 cm³g⁻¹, respectively. The carbonization temperature ranged from 40 to 800°C during the carbonization process. The minimum temperatures required for carbonization of bamboo into biochar are lower than the average needed for ACs, which usually kept more than 300°C to achieve a high surface area (>1000 m²g⁻¹) and porosity.

In addition, based on the results in Fig. 2(a), the temperature maintained at around 105°C is possible for bamboo ACs to obtain a high surface area up to the range of 1456 to 2348 m²g⁻¹. Even at this low temperature, a higher surface area and pore volume can be achieved by carbonizing the bamboo in an almost vacuum atmosphere (96.6% vacuum) and a longer holding time. The vacuum condition significantly reduces the pressure inside the heating chamber that can achieve a similar extent of carbonization process as when carbonized at high temperatures (700 to 800°C) [23].

On the other hand, without vacuum, the surface area and the pore volume increased with the increase in carbonization temperature when performed within the range of 600 to 800°C. The higher temperature resulted in mesopores and macropores' formation, thus resulting in the increased surface area of the ACs that would be useful in the adsorption for MB [27]. The presence of mesopores and macropores is encouraged for the adsorption of MB since the size of the dye particle is 1.5 nm, which would not be sufficient with the presence of only micropores (<2 nm). Therefore, larger pore sizes are necessary to allow more dye particles to diffuse into the pores and be adsorbed onto their surfaces.

Results in Fig. 2 (a-b) also show that the carbonization temperature for coconut shell ACs needed to achieve similar surface area and pore volume as bamboo ACs (500 to 1500 m²g⁻¹ and 0.2 to 1.0 cm³g⁻¹) was relatively limited to the range between 600 to 700°C. Besides using high carbonization temperature, the high degree of the surface area of ACs can also be obtained when the ACs are made into nanofibers using the electrospinning method [6]. The advantage of using coconut shell AC nanofibers is their reusability which can maintain up to 96% at the third cycle of reusing the AC nanofibers. In another reported study, iodine-treated AC nanofibers (abbreviated as I-ACNF) resulted in the highest surface area and higher reusability than AC nanofibres without the iodine treatment (abbreviated as ACNF) and AC powder (ACP) [6]. Incorporating the iodine treatment helped retain the fiber structures. On the other hand, ACP removed a higher amount of MB relative

to the I-ACNF and ACNF. However, ACF was hard to be recycled and separated from the bulk solution due to its powder form [6].

Meanwhile, the results of the rice husk ACs in Fig. 2(a-b) show that the range of carbonization temperatures required is within the high capacity (450 to 700°C). However, the average surface area (500 to 1000 m²g⁻¹) and pore volume (0.2 to 0.6 cm³g⁻¹) were slightly lower than bamboo and coconut shell ACs. Rice husk ACs resulted in a higher surface area (>1000 m²g⁻¹) in the lower temperature ranges (450 to 500°C). However, at higher temperatures beyond 500°C, the resulting surface area significantly dropped to only 68 to 645 m²g⁻¹. Higher temperatures destroyed most of the regular microporous structure during violent gasification reactions, leaving behind large holes in place [39]. Therefore, the distribution of microporous structure decreases significantly and reduces the surface area available for adsorption of the dye that would otherwise have developed at lower temperatures. When the destruction of the microporous structure has occurred, additional chemical activation that should have improved the porosity was seen not significantly to affect the already damaged structure. In addition, at temperature of 500°C, the surface area of the sample rice husk ACs was considerably higher than the rest of the rice husk ACs (Fig. 2a). This was due to the high pore volume that reached as high as 1.8 cm³g⁻¹ due to H₃PO₄ activation, which reacted with the silicon element naturally present in rice husk ash (rich in silica, up to 90 to 98%), resulting in the formation of phosphates that could easily be removed by washing [35].

On the other hand, graphs in Fig. 3(a-b) describe the effects of carbonization holding time on the ACs. For bamboo ACs, the holding time of 1 hour was sufficient to yield ACs with a high surface area in the range of 1000 to 2200 m²g⁻¹ (Fig. 3a). When the holding time was increased from 2.5 hours to 10 hours, the surface area and pore volume could be increased from 1456 to 2348 m²g⁻¹ and 0.94 to 2.3 cm³g⁻¹, respectively. A significant difference was observed on the surface area of the ACs at different holding times where a wider pore distribution and significantly more pores, especially the mesopores (> 2 nm) were produced with longer holding time [23]. The higher pores and wider distribution resulted in a more porous structure, thus increasing the overall AC's surface area available for the dye adsorption.

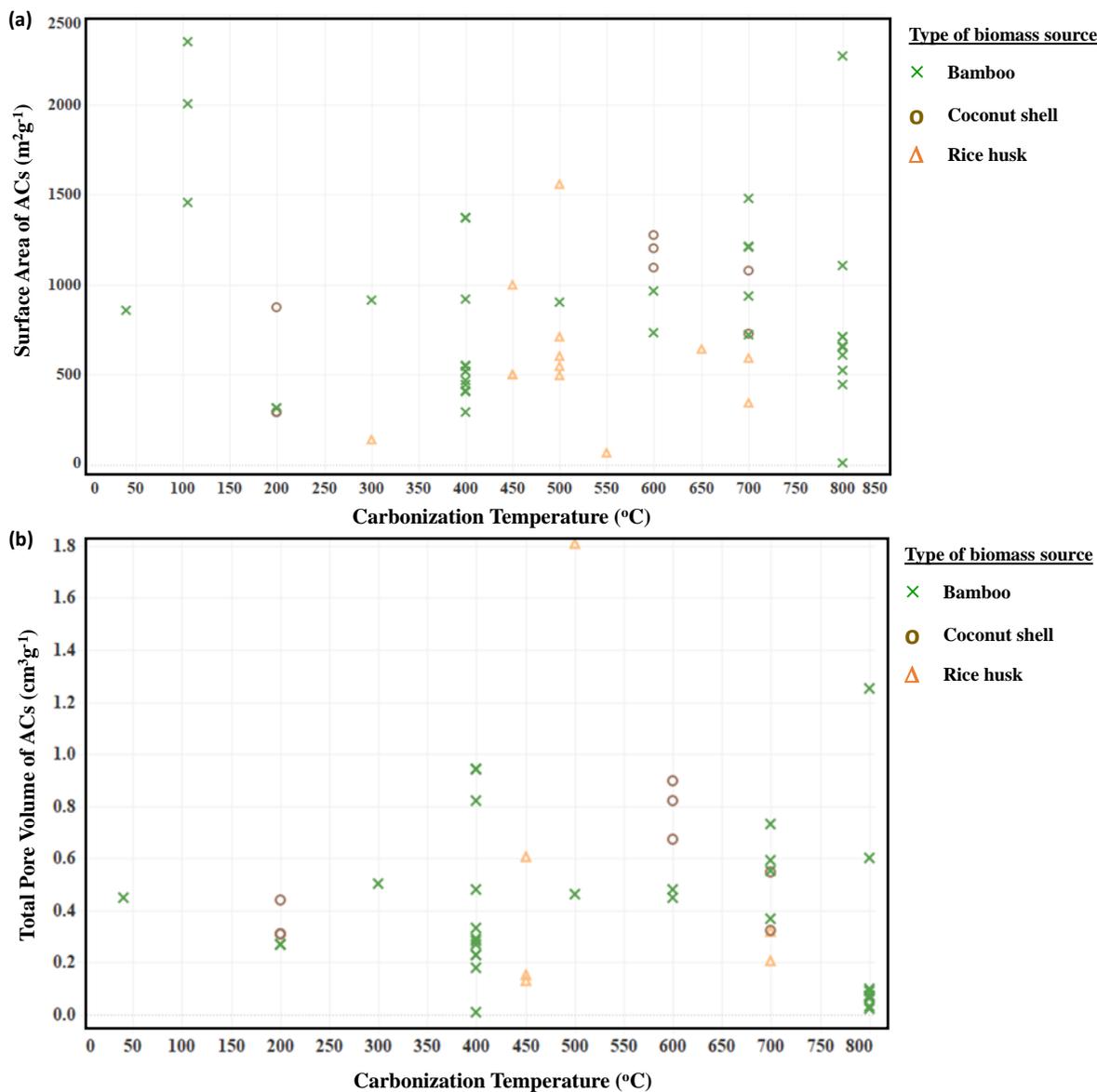


Fig. 2: Graph of (a) surface area of ACs versus carbonization temperature and (b) total pore volume of ACs versus carbonization temperature.

Results in Fig. 3(a-b) also show no significant changes in the surface area of rice husk AC when the carbonization process performed for 1 hour and 5 hours. Both holding times resulted in surface areas ranging from $300 m^2g^{-1}$ to $700 m^2g^{-1}$. However, the holding time could be kept as low as 0.5 hours yet still yielding twice as higher surface area and pore volume beyond $1500 m^2g^{-1}$ and $1.8 cm^3g^{-1}$, respectively, when the rice husk was impregnated with H_3PO_4 during chemical activation. The surface area and pore volume of the rice husk ACs also increase with the increase in the impregnation ratio (H_3PO_4 : Rice Husk) from 3:1 to 5:1, highlighting the chemical agent impregnation ratio as a potential external factor that improved rice husk's physicochemical

properties [35,37]. The holding time of 1 hour has yielded coconut shell ACs with surface areas of 700 to 1500 m^2g^{-1} and pore volume of 0.5 to 1.00 cm^3g^{-1} , which is relatively higher than the average rice husk and bamboo values ACs. In general, higher holding time increases the surface area and volume ratio due to more extended carbonization reactions occurring to the biomass. However, a longer reaction time at a high-temperature carbonization process will increase the synthesis cost and operational hazards.

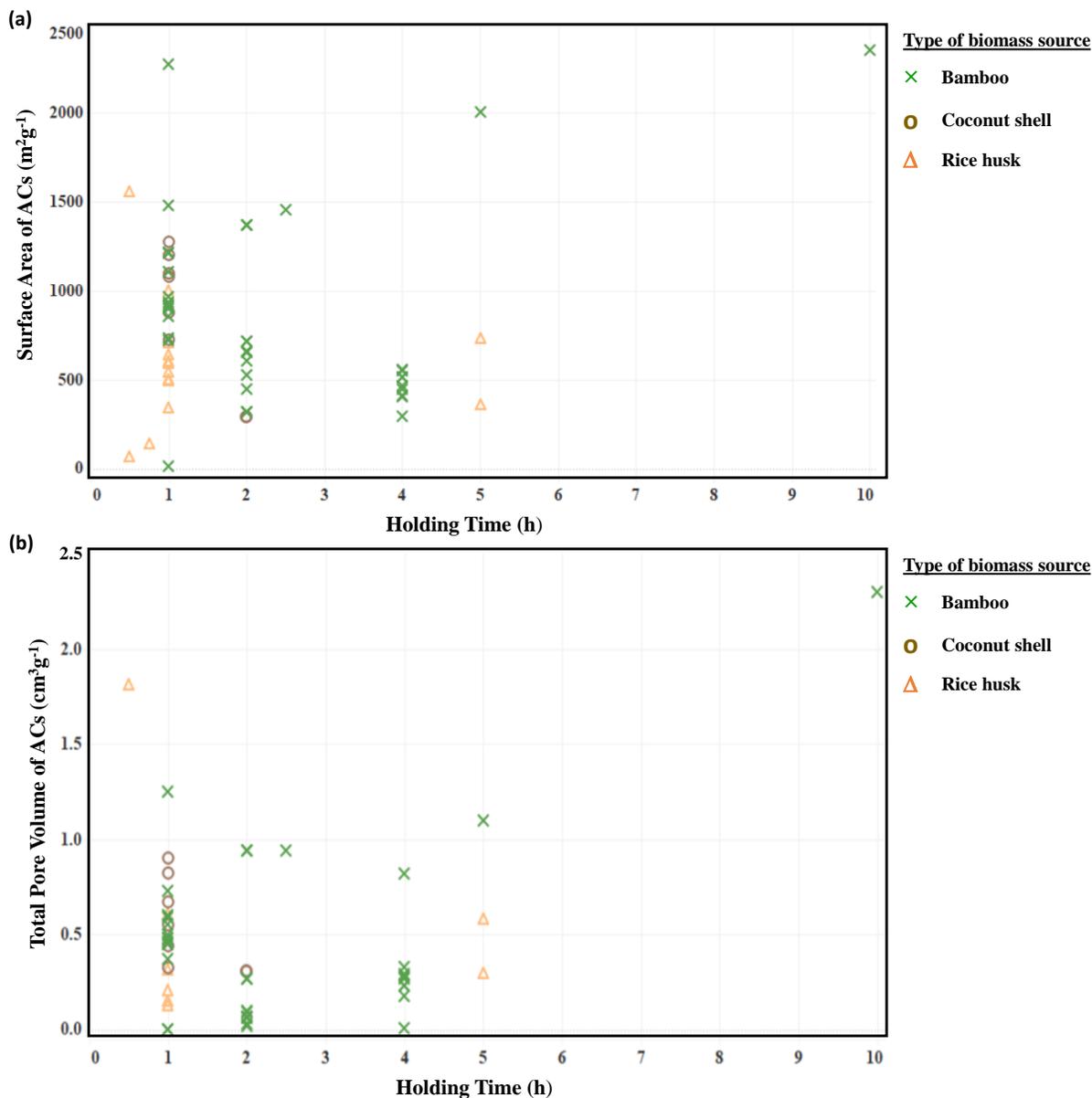


Fig. 3: Graphs of (a) surface area of ACs versus carbonization holding time and (b) total pore volume of ACs versus carbonization holding time.

The adsorption of MB is influenced by the adsorbent's properties and the adsorption conditions used during the adsorption process. Among the adsorption parameters, the dose of AC, initial concentration of the MB, and contact time are the parameters that were commonly varied and studied. They significantly influenced the adsorption properties of ACs. Therefore, the collected maximum dye removal (%) data were normalized in this study to ensure they are comparable across different published studies. The normalization was done by representing the percentage of MB removal data as percentage dye removal per dose of AC per dye concentration per contact time (% removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹). After data normalization, a more significant trend about the physicochemical properties to the performance of ACs could be observed.

Based on the graphs in Fig. 4(a-b), the average range of maximum MB removal could be achieved by ACs produced from all three biomass sources falls within 0.2 to 2 % removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹ when the range of surface area and pore volume of the ACs obtained are between 12 to 2200 m²g⁻¹ and 0.07 to 1.25 cm³g⁻¹ respectively. Within the average range, the dye adsorption capability towards MB increased with the increase of surface area and pore volume, indicating the favorability towards developing these two properties (surface area and pore volume) for improved MB adsorption. Higher surface area increases the adsorbents' available adsorption sites, which would allow more dye particles to adsorb onto its surface, thus increasing the dye removal capability from the wastewater.

Furthermore, according to Fig. 4a for bamboo ACs, the percentage removal could be drastically increased up to 10.0 % removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹ even at a relatively lower surface area of 734 m²g⁻¹ when the bamboo was steam activated. This is because steam activation slightly decreased the content of acidic groups and slightly increased the range of primary groups, which later increased the pH. When steam-activated, the essential nature of the ACs indicates its potential application when the primary groups are desired, such as in the adsorption of cationic dyes like MB [53]. This observation agreed with the information reported by Zhang et al. (2014) where higher pH is preferable during MB adsorption [54]. More cationic dyes could be adsorbed onto the surface of ACs due to less competition with the H⁺ ions at higher pH.

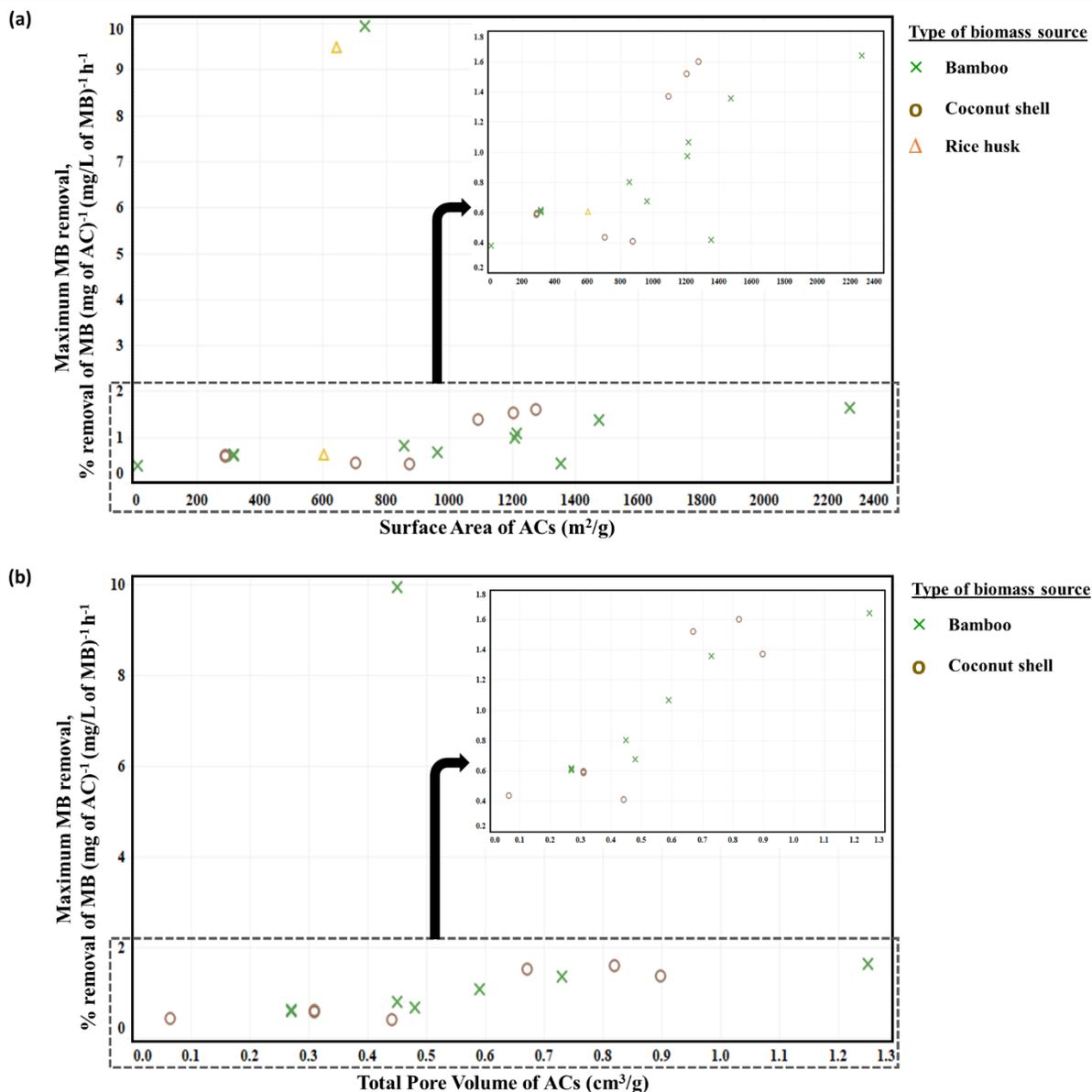


Fig 4. (a) Graph of maximum MB removal per dose of ACs per dye concentration per contact time versus the surface area of AC with an inset for the range of removal 0.2 to 1.8 % removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹, and (b) graph of maximum MB removal per dose of ACs per dye concentration per contact time versus the pore volume of ACs with an inset for the range of removal 0.2 to 1.8 % removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹.

Within the same range of surface area and pore volume, a similar trend in the percentage dye removal could be observed on coconut shell ACs. As mentioned previously, the surface area and the pore volume of coconut shells have been shown to be similar to that of bamboo ACs, although restricted to the carbonization temperatures of 600 - 700 °C. Furthermore, higher percentage dye removal may be achieved beyond 10.0 % removal of MB (mg of AC)⁻¹ (mg/L of MB)⁻¹ h⁻¹ at higher

surface area and pore volume using different activating agents during the chemical activation step, such as monoethanolamine [12]. Monoethanolamine increased the AC's surface alkalinity when nitrogen functional groups in the amine solution were introduced [12]. The surface treatment using a basic chemical agent is necessary to significantly enhance the MB adsorption onto the surface of coconut ACs.

According to Fig. 4a, for the rice husk ACs, the percentage of MB removal significantly increased from 0.6 to 9.5 % removal of MB $(\text{mg of AC})^{-1} (\text{mg/L of MB})^{-1} \text{ h}^{-1}$ with a slight difference in surface area $(603 - 645 \text{ m}^2\text{g}^{-1})$. The significant improvement in its adsorption capability, even with a slight increase in surface area, was achievable when the pH of the solution was kept between pH 8 to 11. At higher pH, the surface of the rice husk ACs became negatively charged, which increases the adsorption of the positively charged MB through electrostatic force attraction [55]. Furthermore, the higher surface area of rice husk up to $645 \text{ m}^2\text{g}^{-1}$ ACs resulted from ZnCl_2 activation. Although rice husks carbonized at higher temperatures $(>500^\circ\text{C})$ could destroy its pore structure, incorporating ZnCl_2 could overcome this problem. Instead, well-developed pores with a good porous structure were formed, thus increasing the surface area that provided higher adsorption sites for more dye adsorption [43].

4. Conclusions and Future Outlook

The surface area and the pore volume of the ACs were observed to be among the critical physicochemical properties that majorly influence the ACs' adsorption performance towards MB. With the increase in surface area and pore volume, a higher percentage of MB could be removed from the wastewater. These properties could be manipulated by applying different carbonization temperatures and holding times during the AC synthesis. It was observed that both bamboo and coconut shells could achieve similar ranges of surface area and pore volume of 500 to $1500 \text{ m}^2\text{g}^{-1}$ and 0.2 to $1.0 \text{ cm}^3\text{g}^{-1}$, respectively. However, the coconut shell's carbonization temperature is more limited in the range of 600 - 700°C than the bamboo, with a broader range of 40 - 800°C . When the two were compared, the percentage removal achieved by both ACs showed similar distribution across the surface area and pore volume. However, lower temperatures less than 500°C should be maintained for rice husks due to its AC structure that tends to be destroyed at higher temperatures. The holding time did not significantly impact rice husk and coconut shell ACs and was commonly sufficient to be kept at 1 hour. Meanwhile, bamboo ACs had higher surface area and pore volume

with a longer holding time. Furthermore, the three biomasses were seen to have significant improvements in dye removal through surface modification through steam activation, chemical activation, and alkaline pH.

Some potential areas surrounding ACs could be highlighted for future research, among the information gathered during the systematic literature review. For example, the regeneration of AC is one of the most critical aspects of its usage in adsorptive applications. The current industrial enterprises and municipalities using commercialized ACs require renewal and reactivation as feasible solutions to decrease carbon footprints and production costs. However, the studies from the research papers lack the information on the reusability for ACs synthesized from these biomasses, which could be further explored and beneficial to be included as part of the ACs' development for contaminant removal and commercialization potential.

Conflicts of interest

There are no conflicts to declare

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