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The impact of Activation processes on Paracetamol Removal by Sesbania Activation Carbon

Basem Mohammed Al-howri[12], Muna Awadh Alrebaki[12], Ahmed Mubarak Alsobaai[2], Noorashrina A. Hamid[1], and Suzylawati Ismail[1*]

[1] *School of Chemical Engineering, Universiti Sains Malaysia, Engineering Campus, 14300 Nibong Tebal, Penang, Malaysia*
[2] *Chemical Engineering Department, College of Engineering and petroleum, Hadhramout University, Mukalla, Yemen*

[*] chsuzy@usm.my *Email of the corresponding author*

Abstract **–** The adsorption of paracetamol from water utilising a few different activation processes for sesbania-derived activated carbon was examined in the current work. The adsorbent was prepared by cutting, washing, and drying at 90 °C, then crushing and purification with acetone and activated with phosphoric acid and aluminum chloride. The carbonization of those sesbania occurs at a high temperature of 450 °C with N_2 gas acting inert until the temperature reaches 65 °C. Each process's effects were then examined using different methodologies after the activation phase. For the paracetamol adsorption investigation, the technique with the highest efficiency and most cost-effective processing will be chosen. Using the thermal activation method at 90 °C for 24 hours, the highest elimination percentage of paracetamol was approximately 89.3 %, with an equilibrium period of around 30 min. Under defined parameters, the adsorption performance was compared in terms of maximum removal percentage and equilibrium time. The raw material used to prepare activated carbon, the activated carbon prepared by sesbania, and the used adsorbent after adsorb paracetamol were analyzed by SEM and EDX analysis in order to identify eventual structural and adsorptive characteristics. The findings showed that prepared sesbania-activated carbon could be used as a low-priced adsorbent to remove paracetamol from water.

Keywords – Activated Carbon, Sesbania Trees, Paracetamol, Adsorption, Activation Process

1.0 Introduction

The United Nations (UN) established the 17 Sustainable Development Goals (SDGs) in 2015 to address social, economic, and environmental issues [1]. The 17 Sustainable Development Goals are displayed in Figure 1. (SDGs). The current study aims to investigate four main SDGs (SDGs 6 clean water and sanitation, SDGs 8 decent work and economic growth, SDGs 9 industry, innovation, and infrastructure, and SDGs 12 responsible consumption and production) by producing adsorbent from undesirable sources (waste sources) (SDGs 9, and SDGs 12), purifying and improving water quality (SDGs 6) by using affordable methods (SDGs 8).

Human life relies on water to survive, which also contains various nutrients and minerals. Strict regulations and limits have rapidly increased in monitoring surface water bodies over the last few years due to deteriorating environmental concerns. The quality of the water is impacted by the geological composition of the earth's surface and manufactured activities like agriculture, sewage discharge, construction, and other associated activities that constrain water use. The interplay between physical, chemical, and biological factors significantly impacts water quality [3].

Figure 1: The 17 Sustainable Development Goals (SDGs) [2]

The term Contaminants of Emerging Concern refers to a large class of natural and man-made compounds that have either just entered the environment or have existed for a very long time but have only recently been recognised as being extensively dispersed and possibly dangerous. Not all pollutants of increasing concern are regulated or covered by standard monitoring systems, and it is not yet completely known how much risk they cause to the environment and public health. Examples include prescription drugs, personal care goods, industrial and household chemicals, pesticides, and items manufactured from man-made nanomaterials [4].

The level of aquatic pollution, which is directly tied to the health and welfare of society, significantly influences the quality of the world's water. The problem of pollution brought on by emerging contaminants has grown considerably in importance in the twenty-first century. Emerging contaminants are a broad category of products with various properties that may persist in the environment, accumulate in animal and human tissues, and perhaps be dangerous even in minute quantities. Some of these developing pollutants include pharmaceuticals, personal care items, plasticizers, pesticides, flame retardants, industrial additives, surfactants, nanomaterials, mycotoxins, and phytotoxins [5].

For instance, paracetamol is frequently found in groundwater, surface water, drinking water, municipal wastewater, and sewage treatment due to its high stability, solubility, and hydrophobicity [6]. In light of this, it is necessary to eliminate paracetamol from aquatic ecosystems [7]. Paracetamol is a common analgesic and antipyretic. For COVID-19 symptoms, paracetamol is the first-line antipyretic and analgesic medication. Furthermore, paracetamol consumption is expected to soar because it was recommended to treat the symptoms of the COVID-19 epidemic [8].

The global paracetamol market has been significantly impacted by the COVID-19 (coronavirus disease) pandemic, according to the research and markets analysis "Paracetamol Market-Growth, Trends, COVID-19 Impact, and Forecasts (2021-2026)". This is due to the widespread use of paracetamol, particularly at home, in the early treatment of COVID-19-infected patients. The compound annual growth rate for industrial growth is 4.3 percent (for the study period 2018-2020) and 0.7 percent (2020-2026). The paracetamol market is predicted to rise from USD 750.5 million in 2020 to 788.5 million USD by 2026 [9]. It is essential to find a solution to the paracetamol and other toxins of rising concern that cause aquatic contamination.

Sesbania (Prosopis spp. Mimosaceae, Leguminosae), commonly called mesquite, has been described as a plant with quick growth and spreading, especially in dry and semi-arid conditions, and as thorny shrubs or small trees (3 to 15 meters) [10]. Sesbania trees are drought-resistant, generate many seeds, and have a long-life cycle. In addition, their taproot is growing deeply, which lowers the water table at a depth of more than 30 m and causes other trees to dry up from a lack of water [11]. It spreads and grows rapidly because the seeds are hard for animals to digest, and the leaves are bitter. It is considered a dangerous weed in many countries. Due to its competition with grasses and ability to dry up the soil, this tree is seen by some cultures as an invasive weed [12]. The International Union for Conservation of Nature (IUCN) has listed Sesbania as one of the top 100 invasive species because it frequently encroaches on agricultural land, depletes rangelands, and harms biodiversity in many countries. As one of the total 100 invasive species, Sesbania poses a severe irritant to ecosystems [11].

Sesbania is prevalent in 129 nations throughout the world [13]. Texas, the second-largest state in the USA, has over 70 million ha of land, yet the value of 22 million ha of that land has reduced due to the sesbania invasion [10]. In South Africa, Sesbania trees covered 1.47 million acres in 2009 [14]. The sesbania invasion in the United Arab Emirates reached its peak rate of expansion in 2019 and took up an area of around 16 km^2, as opposed to 0.2 km^2 in 1990 [15]. Sesbania has damaged an estimated 1.5 million hectares of irrigated land in Sudan [12]. Although sesbania trees and their products have practically endless uses, researchers and governmental bodies are still baffled as to how to stop the plants from encroaching in new places [10]. As a result, this study aims to explore the potential

of producing activated carbon from this unwanted plant. Commercial activated carbon can easily be applied to remove paracetamol from water [7],[6]. However, due to the high cost of commercial activated carbon, it is necessary to find ways to produce activated carbon from inexpensive sources to lower the cost and create a more economical process. As a result, scientists are working on low-cost adsorbent that is made from a variety of wastes and by-products, such as using Cannabis sativum Hemp [16], coffee grounds biomaterial [17], Brazil nutshells [18], rice husk [19], biochar precursor [20], and from orange peel [21].

In this study, activated carbon made from sesbania is tested for its ability to extract paracetamol from water. The effectiveness of the adsorption was assessed using several activation procedures, including varied hydrochloric acid concentrations as an activation agent in chemical activation and various investigated temperatures in thermal activation methods.

2.0 Experiment

2.1 Materials and Method

Sesbania woods were procured in Hadramout, the Republic of Yemen, and then cleaned, crushed, ground, and mill-sieved into an appropriate size before being cleaned and let to air dry. Paracetamol and acetone were supplied by Sigma Aldrich, aluminium chloride and hydrochloric acid were supplied by R&M Chemicals, and EMSURE ACS delivered phosphoric acid. All other compounds are of the analytical variety and can be used directly. All aqueous solutions were prepared using distilled water.

2.2 Preparation of activated carbon and the characterization

As illustrated in Figure 2, Sesbania wood was chopped into small pieces and thoroughly cleaned with distilled water to remove contaminants. Next, the wood was dried at 90 ^{o} C for 24 hours to remove moisture until the weight remained constant, after which it was ground to a small size. Following grinding, the sesbania particle was repeatedly rinsed with distilled water, as depicted in figure 3. As seen in figure 4, the sample was then purified by being impregnated for 24 hours in acetone and distilled water before the activation ingredient was added.

Figure 2: Washing of sesbania

a b c

Figure 3: Rinsing process of sesbania after grinding
a: first rinsing after grinding, b: After rinsing for 5 times, c: after remove the dust and impurities

Figure 4: Acetone and distilled water impregnation for 24 hours.

As shown in figure 5, the sample was thoroughly cleaned with distilled water before being activated with phosphoric acid at a concentration of 1 M. Aluminum chloride, 0.5 gm, was also added to give chloride ions prior to the carbonation process. To remove the effects of the acid until it achieved a natural pH, the sample was rinsed several times with distilled water. It was then dried for 24 hours at room temperature.

Figure 5: Impregnation process in H_3PO_4 and 0.5 gm of $AlCl_3$ for 24 hr

For the carbonation process, the sample was placed in a vertical furnace with a constant flow of N_2 gas acting as an inert gas for one hour. To prevent the sample from combusting at high temperatures after the temperature reached 450 ° C, a continuous flow of N_2 was delivered as a cooling mechanism until the temperature reached 65 ° C. To choose the most effective and cost-effective technique, various carbonation methods for activation were compared for elimination % and time of equilibrium.

The morphologies of the pure sesbania, activated carbon prepared from it, and the used activated carbon after adsorb paracetamol were studied art with a scanning electron microscope SEM and energy dispersive X-Ray (EDX) composition analysis was conducted by using SEM-EDX, Quanta-450 FEG, FEI, Netherlands which gave us an idea of their porous structure and to identify the elemental composition of each sample.

2.3 Adsorption process

First, 0.01 g of paracetamol was added to 1 L of distilled water and mixed for an hour to create a paracetamol solution with a concentration of 10 ppm. Then 200 ml of this solution was obtained as a sample. The experiment's parameters were adjusted to a lab temperature of about 27 ° C and a pH of 7.8 after paracetamol was added to distilled water. The initial concentration was determined by UV-VIS measurement and recorded after stirring the solution for 30 minutes at a rotation speed of 200 rpm. 0.5 grams of produced activated carbon was added to adsorb paracetamol from water. Every 10 minutes, samples were taken to be measured and monitored the paracetamol concentration in the solution was.

All the experiments were conducted under fixed conditions:

- Initial concentration is 10 ppm
- volume of the solution is 200 ml.
- The temperature is around 27 ° C as lab temperature.
- The time of the experiment is 2 hr.
- The mass of the adsorbent is 0.5 gm.
- The speed of rotation is 200 rpm.

3.0 Results and discussion

3.1 Acid activation

The effect of applying various concentrations of hydrochloric acid (HCl) to activate the produced activated carbon on paracetamol removal percentage and equilibrium time was investigated. The experiments were assessed in the concentration range of 1 M to 2 M., as seen in Figure 6. The adsorption findings exhibited a favourable implication on the removal percentage, which increased from 75.8 to 83.1 percent with increased HCl concentration from 1 M to 2 M, respectively. However, the equilibrium time remained constant at 50 min at all concentrations. In general, the characteristics of adsorbents like carbon materials are improved when HCl activates certain materials [22],[23], bentonite [24], and biochar [25].

For activated carbon, hydrochloric acid during the treatment process improves the material's pore properties and speeds up the subsequent activation process [25]. The volume and size of the micropores both marginally increased [22], [25]. Numerous materials disappear after processing with HCl solution. This most likely resulted from the various inorganic minerals being washed away by the HCl solution after they had first gathered on the surface or in the pores of the activated carbon. The exposed pore structures and lamellar structures on the surface of activated carbon were significantly altered [25]. Washing with an acidic or alkaline solution may make the carbon surface more porous and further leach any residual metal. On the other hand, hazardous substances like NaOH and HF do not meet the criteria of green development. The intense oxidation of H_2SO_4 will promote the synthesis of oxygen-containing functional groups on the surface of carbon materials and lower their electrical conductivity. Therefore, the activation process is more beneficial and ecologically acceptable when employing an HCl solution [22].

After being activated with hydrochloric acid, the surface area and total volume of the activated carbon's pores increase, and they increase even more as the HCl concentration increases [25] ,[22].

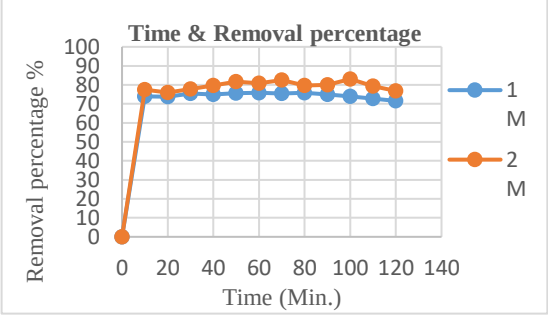

Figure 6: The performance results of Sesbania-made activated carbon prepared by using 1 and 2 M HCl for 24 hr.

3.2 Thermal activation

3.2.1 Thermal activation test at 300 ° C in furnace for different study intervals

The percentage of paracetamol removed was assessed about the impact of utilising a high temperature to activate the produced activated carbon by sesbania. To determine the effect of temperature and heating duration on the effectiveness of the produced activated carbon, the activated carbon was evaluated by heating to 300 ° C in the furnace at various time intervals (1 hour, 2 hours, and 4 hours). The results demonstrated that heating to 300 ° C has a beneficial effect on the elimination percentage, yielding 83.6 %, 86.1 %, and 85.1 % in 1 hour, 2 hours, and 4 hours, respectively. Time observed that the equilibrium was attained after 60 minutes of heating for 1 hour. However, after 2 hours to 4 hours of heating intervals are tested, more extended periods are required 80 min to reach equilibrium respectively. In order of equilibrium, time noticed that when heating for 1 hr., the equilibrium reached 60 min, but when heating for 2hr. and 4 hr., it reached 80 min. The experiment results also demonstrate that heating cannot continue for more than 4 hours, as the activated carbon turns to ash after around 6 hours. The results of the experiments for heat activation at various times are displayed in Figure 7.

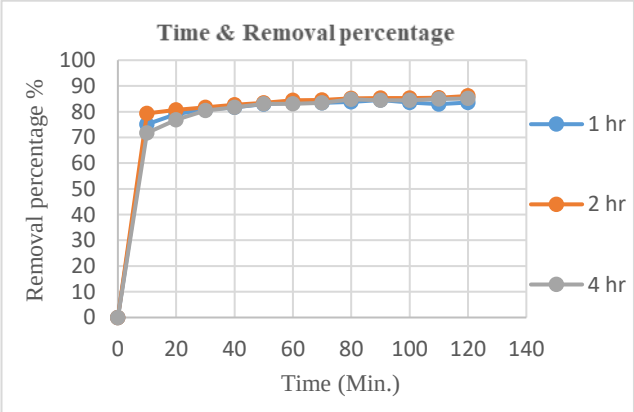

Figure 7: The performance results of Sesbania-made activated carbon prepared by heating in furnace to 300 ° C for 1, 2, and 4 hr.

3.2.2 Thermal activation test at 90 ° C for 24 hr. in autoclave

In this experiment, the effect of heating activated carbon produced by Sesbania over a prolonged period at low temperatures on the percentage of paracetamol removal was investigated. The prepared activated carbon was heated in an autoclave for 24 hours at 90 ° C to activate it. Compared to the results obtained with another activation approach, this method generated the best results since it achieves the elimination percentage of 89.3 percent in a relatively short equilibrium period of around 30 minutes. The findings demonstrated that this method is superior to previous activation techniques in many ways, as evidenced by the fact that it achieved the highest removal percentage in the shortest equilibrium time. Another benefit is that this activation technique does not require the addition of any chemicals, which lowers the cost of the activation process and makes it safer. Additionally, the activation temperature employed is low compared to the high temperatures used by activation in the furnace. The outcomes of activation in the autoclave at 90 ° C for 24 hours are illustrated in Figure 8. The temperature generally has a beneficial impact on adsorption characteristics; however, this effect is typically quite limited [25]. Thermal activation increases the porosity of the activated carbon but has little effect on the surface functional groups [26].

When utilising thermal activation, the heating can impact the final products' yields, morphologies, and microstructures. A moderate activation temperature helps produce activated carbons with a high surface area and a large pore volume [27]. Therefore, using 90 ° C instead of high temperatures like 300 ° C for thermal activation produces good performance.

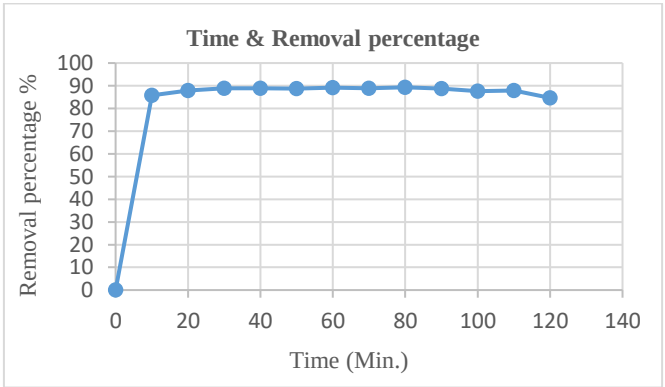

Figure 8: The performance results of Sesbania-made activated carbon prepared by heating in autoclave to 90 ° C for 24 hr.

3.3 Combine the acid and thermal treatments

To improve the characteristics of the activated carbon, increase the removal percentage, and

shorten the time of the adsorption process, thermal activation was utilised after the acid treatment to activate the prepared activated carbon made by Sesbania. To accomplish these goals, the sesbania was activated with 1 and 2 M HCl and later heated to 90 º C for 24 hours in an autoclave to activate the activated carbon. The outcomes demonstrated an improvement in paracetamol removal percentage and time to equilibrium. It is observed that the removal percentage of paracetamol by activated carbon treated with 1 M HCl increases from 75.8% to 79.8% while the equilibrium time decreases from 50 minutes to 30 minutes. The same beneficial impact was seen in activated carbon treated with 2 M HCl, increasing the percentage from 83.1 % to 84.6 % and decreasing the equilibrium time from 50 to 20 minutes. The dehydration process was extremely exothermic and formed hydro char when acid solution and carbon material were combined with heating [28]. The chemical oxidation of activated carbon by acids results in smaller surface area sizes; however, further thermal treatments cause these values to increase gradually by a few percent with rising heating temperature. Additionally, the overall pore volume decreases during chemical activation, and during thermal modification, it gradually increases with temperature. A decrease in these values may also be linked to an increase in the number of oxygen surface groups that can be expected to be immersed inside pores or block the entrance of pores, reducing the amount of carbon material that is accessible. This is because oxygen surface groups can be considered active centres at the opening of pores. Following thermal heating, an increase in surface areas results from the removal of oxygen surface groups that may be within or obstruct specific pores [29]. Figures 9 and 10 below demonstrate the outcome of combining the acid and thermal treatments and compare it with the result of employing only the acid treatment:

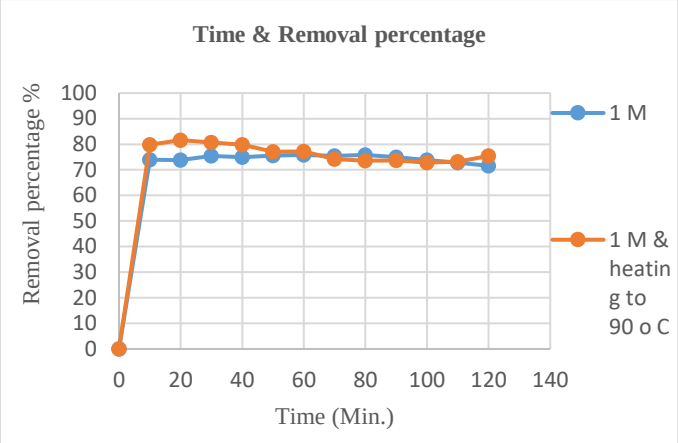

Figure 9: Activated by 1 M HCl Vs. activated by 1 M HCl then heating to 90 º C for 24 hr.

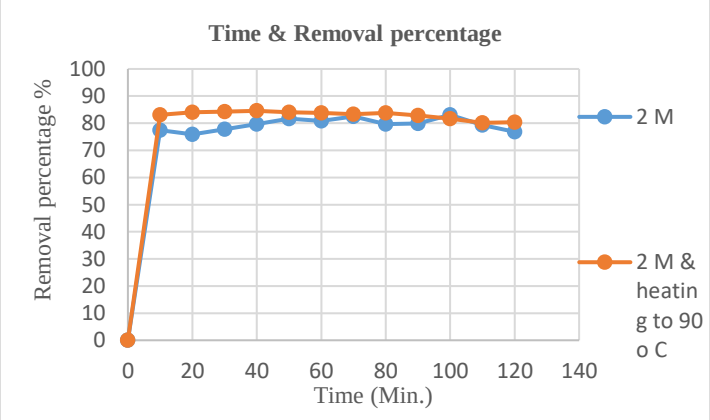

Figure 10: Activated by 2 M HCl Vs. activated by 2 M HCl then heating to 90 º C for 24 hr.

4.0 Summary and the discussion of the experiments

Overall, based on the results, all activation methods are beneficial and improve the prepared adsorbent because, when used directly after calcination, the prepared adsorbent had a lower efficiency than after activation by the various three methods, which gave a removal percentage of 76.6 % in an equilibrium time of 80 min. The results showed that all activation techniques could enhance the structure and characteristics of the obtained activated carbon to achieve improved performance. The findings of all activation techniques combined are summarised in Table 1:

Table 1: The summary of the results obtained by the whole activation methods

No.	Methods of activation	Removal %	Time of equilibrium (min.)
1	Used after calcination without activation	76.6	80
2	Activated by 1M HCl for 24 hr.	75.8	50
3	Activated by 1M HCl + heating to 90 ° C for 24 hr.	79.8	30
4	Activated by 2M HCl for 24 hr.	83.1	50
5	Activated by 2M HCl + heating to 90 ° C for 24 hr.	84.6	20
6	Activated by heating to 90 ° C for 24 hr.	89.3	30
7	Activated by heating to 300 ° C for 1 hr.	83.6	60
8	Activated by heating to 300 ° C for 2 hr.	86.1	80
9	Activated by heating to 300 ° C for 4 hr.	85.1	80

When comparing the results of the prepared activated carbon by sesbania and the commercial granular activated carbon with particle size 1.5-10 mm in terms of the removal percent, it is seen that the prepared adsorbent gave better efficiency in the removal of paracetamol before and after the activation against 60.98% obtained by the commercial granular activated carbon.

4.1 Material analysis

After choosing the thermal activation method at 90 ° C for 24 hr. in an autoclave as a suitable method to activate the sesbania-derived activated carbon, the scanning electron microscopy (SEM) images and Energy dispersive X-Ray (EDX) composition analysis was conducted by using SEM-EDX, Quanta-450 FEG, FEI, Czech Republic of pure sesbania, activated carbon prepared from it, and the activated carbon after adsorb paracetamol are shown in Figure 11 and table 2 a, b and c, respectively. SEM images provided useful information concerning the surface pore structure, and proved about the morphologies of each sample were completely different. Before calcination, the sesbania showed a bulk structure with a rough solid surface that still existed, as shown in Figure 11a. After thermal treatment, the structure of activated carbon was significantly different. The activated carbon produced by sesbania shows a porous

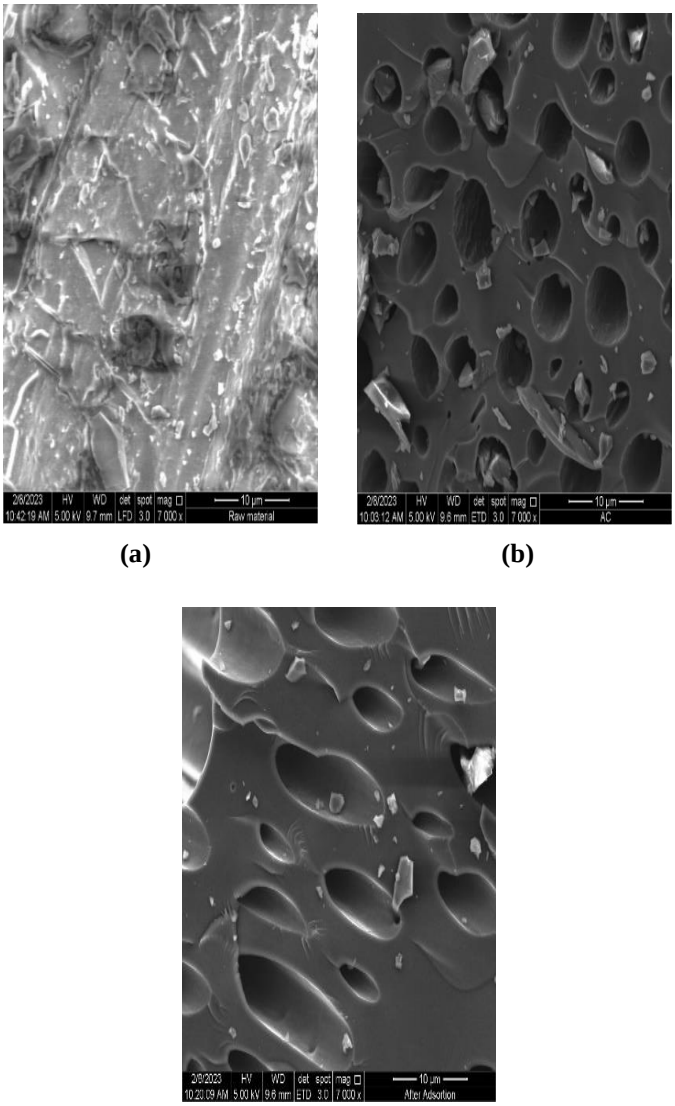

Figure 11: SEM micrograph.

a. SEM micrographs of the raw material
b. SEM micrographs of sesbania-derived activated carbon
c. SEM micrographs of activated carbon after adsorption

structure with plentiful pores and tremendous holes formed in the sesbania-derived activated carbon with enhanced surface area Figure 11b. This pore on the activated carbon material has been reported in other studies as well when using natural source material to prepare activated carbon and add chloride ions as an activation agent [30],[31],[32]. Therefore, the developed structure is favorable for adsorption, which may increase the adsorption capacity of activated carbon [30],[33],[17]. After adsorption, the depth of the pores is reduced due to adsorb paracetamol, and the total number of pores also reduces, as shown in Figure 11c. the edges of the pores structure on the surface were etched by the activation agents, leaving only pits and small holes

in the depths. The SEM images provide an intuitive understanding of the adsorption of activated carbon. Energy Dispersive X-Ray Analysis (EDX) are used for the same samples to identify the elemental composition of samples. The results obtained shown in table 2

Table 2: EDX analysis results

Elements	Atomic %
a- Raw material	
C	63.89
O	34.87
Cl	0.16
K	0.17
Ca	0.91
b- Sesbania-derived activated carbon	
C	81.38
O	13.39
P	5.24
c- activated carbon after adsorption	
C	85.31
O	13.07
P	1.62

5.0 Conclusion

This study successfully created a low-cost, effective sesbania-based adsorbent. The three distinct activation processes that were evaluated to create sesbania-based activated carbon showed that they can remove paracetamol from water with excellent removal percentages. According to this study, the highest amount of paracetamol removed is around 89.3 percent, and the equilibrium time is attained after 30 minutes when a thermal activation of 90 ^{o} C for 24 hours is utilised. The results also demonstrated that using a higher concentration of HCl, around 2M, as an activated agent is preferable. The SEM images and the EDX analysis of the elemental composition of each sample show the significant effect of the preparation and activation process in changing the structure of sesbania and the content of elements in it. Sesbania-based activated carbon is a new prospect for enterprises to consider, and it has the potential to indirectly address the significant biodiversity problem that this plant has caused in many countries. The study's findings suggest thermal activation because it produces safer and more cost-effective adsorbent while achieving excellent removal efficiency. The authors advise researchers to concentrate on enhancing the manufactured adsorbent by applying additional activation methods in future investigations to attain higher removal percentages.

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