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Rubber Seed Shell Based Activated Carbon as Potential Biosorbent for the Removal of Heavy Metals from Aqueous Solution

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Abstract

Heavy metals contamination in water body and aquatic ecosystems has significantly affects and posing serious threats to the environment, aquatic life, and public health. Due to its widespread occurence and harmful effects, adressing this issues remains a critical challege. This study explores the utilization of a widely available agricultural by-product in Malaysia such as rubber seed shells (RSS), as a raw material for producing low-cost activated carbon (AC) for heavy metal removal applications. To enhance the surface porosity and adsorbent characteristics, the RSS was chemically activated using sodium hydroxide (NaOH) and carbonized. Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD) analyses was carried out to characterized the rubber seed shell ras sample (raw-RSS) and rubber seed shell activated carbon (RSS-AC), respectively. The removal efficiency of lead (Pb) and copper (Cu) were evaluted via batch biosorption test under varying conditions of initial concentration (200 - 800 ppm), contact time (15 - 60 min), and biosorbent dosage (0.05 - 0.20 g). Results shows that under optimum conditions of 200 ppm initial concentration, 60 minutes contact time, and 0.20 gram biosorbent dosage yielded removal efficiencies of 77.45% for Cu and 99.20% for Pb. These results highlight that rubber seed shell based activated carbon as an effective, eco-friendly biosorbent for wastewater treatment.

1. Introduction

The significant increase of heavy metals in water body and aquatic ecosystems due to rapid industrialization has become major concern. Even at low concentrations, heavy metals such as cadmium (Cd), zinc (Zn), copper (Cu), lead (Pb), and mercury (Hg) are considered hazardous due to their toxicity and non-biodegradability [1]. The release of these heavy metal pollutants from industrial effluents poses severe risks to the environment, aquatic life, and public health if not properly treated. Due to this reason, it is important to treat metal-contaminated wastewater before being discharge into the water body. However, treating contaminants from industrial discharge is a costly and complex process. Therefore, finding the alternatives for a cost-effective and environmentally friendly treatment or process is crucial for industries generating heavy metal wastewater.

In recent years, biosorption has emerged as a promising alternative, offering a sustainable, cost-effective, and highly efficient method for removing heavy metals compared to conventional methods [2]. Biosorption involves the use of a biosorbent to bind specific ions or molecules at the sorbate-biosorbent interface, leading to a reduction in sorbate concentration in the solution [3]. In simpler terms, biosorption refers to the tendency of biological materials to adsorb specific ions or molecules to its body. Various biosorbents including living or dead microorganisms (e.g., algae, fungi, and bacteria), plant-based materials, agricultural biomass, and biopolymers have been studied for their application in water and wastewater remediation. Among these, biosorbents derived from agricultural waste have been reported as a promising alternative for heavy metal removal [4].

Agricultural waste structural composition, which includes cellulose, hemicellulose, lignin, and functional groups such as carbonyl, phenolic, and alcoholic groups has demonstrated high potential biosorption capacity [5-6]. These functional groups exhibit high affinity for metal ions by donating electron pairs to form complexes with the ions in solution [7]. Furthermore, agricultural waste is cost-effective, biodegradable, and efficient for water purification [8]. As a result, agricultural waste has been extensively investigated as a precursor for activated carbon (AC) preparation.

Rubber seed shells (RSS) are an agricultural by-product regarded as waste and have been identified as a low-cost precursor for AC production. Their high carbon content, abundant availability, and volatile characteristics make RSS an excellent replacement for conventional high-cost activated carbon adsorbents [9]. While RSS has applications in manure production, animal feedstock, and fuel substitutes [10], its disposal as waste remains prevalent, leading to environmental problems such as land pollution, odor issues, and contamination in rubber plantation areas. According to the Malaysian Rubber Board, approximately 1.1 million hectares of land in Malaysia are dedicated to rubber plantations, with each hectare producing around 150 kg of RSS per season, which is often considered waste due to its inedibility [11].

Therefore, this study aims to utilize RSS agricultural waste, abundant in Malaysia, as a low-cost precursor for activated carbon production. Previous studies have demonstrated the potential of RSS-based activated carbon for the adsorption of various pollutants, including methylene blue, congo red, malachite green, crystal violet, phenol, chromium, copper, and zinc [12-17]. These findings highlight the potential of RSS to remove a wide range of pollutants from wastewater. Efficient utilization of RSS waste such as activated carbon will significantly reduce environmental pollution from agricultural waste as well as solving the problem of wastewater effluents.

The objectives of this study include the characterization of the prepared RSS-AC and evaluation of its biosorption capacity. The batch biosorption tests were conducted to evaluate the performance of RSS-AC as a potential biosorbent for heavy metal removal. Several parameters such as heavy metals initial concentration, contact time, and biosorbent dosage were investigated in the biosorption process. The study aims to provide insights into the efficient utilization of RSS waste for heavy metal wastewater treatment applications.

2. Materials and Methods

2.1 Materials

Analytical reagent grade of Copper Sulphate ($CuSO_4$), Lead Nitrate ($PbNO_3$) and Sodium Hydroxide (NaOH) were procured from R&M Chemicals, Malaysia. Raw material of RSS was collected from local rubber plantation at Malaysian Rubber Board, Sungai Buloh. All aqueous solution were prepared using distilled water.

2.2 Methods

2.2.1 Preparation of Sample

The collected RSS material was thoroughly washed, first with tap water and then several times with distilled water, to remove impurities and unwanted materials such as sand and dirt. The cleaned RSS was then dried in an oven at 110°C for 24 hours to eliminate any trapped moisture. After drying, the RSS was crushed into small pieces, ground, and sieved to achieve a particle size of 500 µm. The resulting powdered sample was stored in an airtight plastic container for further use.

2.2.2 Chemical Activation and Carbonization

The carbonization and activation of RSS was prepared according to the method described by Azry & Ahmad [18]. For activation, the RSS sample were activated by chemical activation method using NaOH as the activating agent.

Firstly, about 50 g of NaOH was dissolved with 250 mL distilled water. Then, 25 g of RSS was weighted and then soaked overnight in the freshly prepared concentrated NaOH solution for 24 hr. The sample were prepared with a constant impregnation ratio (IR) of 1:2 using Equation 1.

$$Impregnation\ ratio = \frac{W_{RSS}}{W_{NaOH}} \tag{1}$$

where W indicates the weight of RSS and NaOH used during the activation process, respectively. Next, the impregnated RSS solution was filtered and then carbonized at 600°C for 3 hr under a steady nitrogen (N_2) gas stream at heating rate of 10°C/min and a flow rate of 1 L/min in a tubular furnace. After carbonization, the activated carbon produced were let ventilated at room temperature until 60°C and then washed repeatedly with hot distilled water to eliminate any residual of NaOH. The washed activated carbon was then dried in the oven for 12 hr at 80°C. All produced activated carbon were placed in airtight plastic containers and stored in a desiccator.

2.3 Sample Characterization

Three characterization methods were employed for the RSS samples which were Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD). The analyses were performed on three types of samples which are raw RSS (before activation), RSS-AC before the biosorption process, and RSS-AC after the biosorption process.

2.3.1 Scanning Electron Microscopy (SEM) Analysis

The surface morphology of raw RSS and RSS-AC was analyzed using SEM (JEOL-7800F PRIME). Observations were conducted at an accelerated voltage of 0.50 kV under magnifications ranging from x5,000 to x10,000 to examine the textural changes and pore structure developed during activation and after biosorption.

2.3.2 Fourier Transform Infra-Red (FTIR) Analysis

The functional groups and chemical bonds present on the surface of the samples were analyzed using FTIR Spectrophotometer (Shimadzu IRTracer-100). The analysis employed the Attenuated Total Reflection (ATR) method, with spectra recorded in the range of 400–4000 cm^{-1} at a resolution of 4 cm^{-1}.

2.3.3 X-ray Diffraction (XRD) Analysis

XRD analysis was conducted to determine the crystalline or amorphous nature and phase composition of the samples. The diffraction patterns of RSS-AC were analyzed using an XRD Empyrean, model DSC-60 Plus Series, with Cu Kα irradiation (λ = 1.54056 Å) at 45 kV and 40 mA.

2.4 Batch Biosorption

The removal of heavy metals by RSS-AC was evaluated using a batch biosorption test. Initially, stock solutions of $CuSO_4$ and $Pb(NO_3)_2$ were prepared by dissolving specific amounts (200–800 mg) of each compound in 1 L of distilled water. Subsequently, 100 mL of each solution was transferred into 100 mL conical flasks to serve as standard solutions for the experiments.

The effects of initial heavy metal concentration (200 ppm, 400 ppm, 600 ppm, and 800 ppm), contact time (15 min, 30 min, 45 min, and 60 min), and biosorbent dosage (0.05 g, 0.10 g, 0.15 g, and 0.20 g) were analyzed to determine the optimal conditions for the prepared RSS-AC performance. All experiments were conducted at room temperature of 30°C, with the solutions stirred using a rotary shaker (IKA KS-3000i).

The initial (C_o) and final (C_i) concentrations of metal ions were measured using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES, PerkinElmer AVIO-500). The percentage removal of heavy metals was calculated using Equation 2:

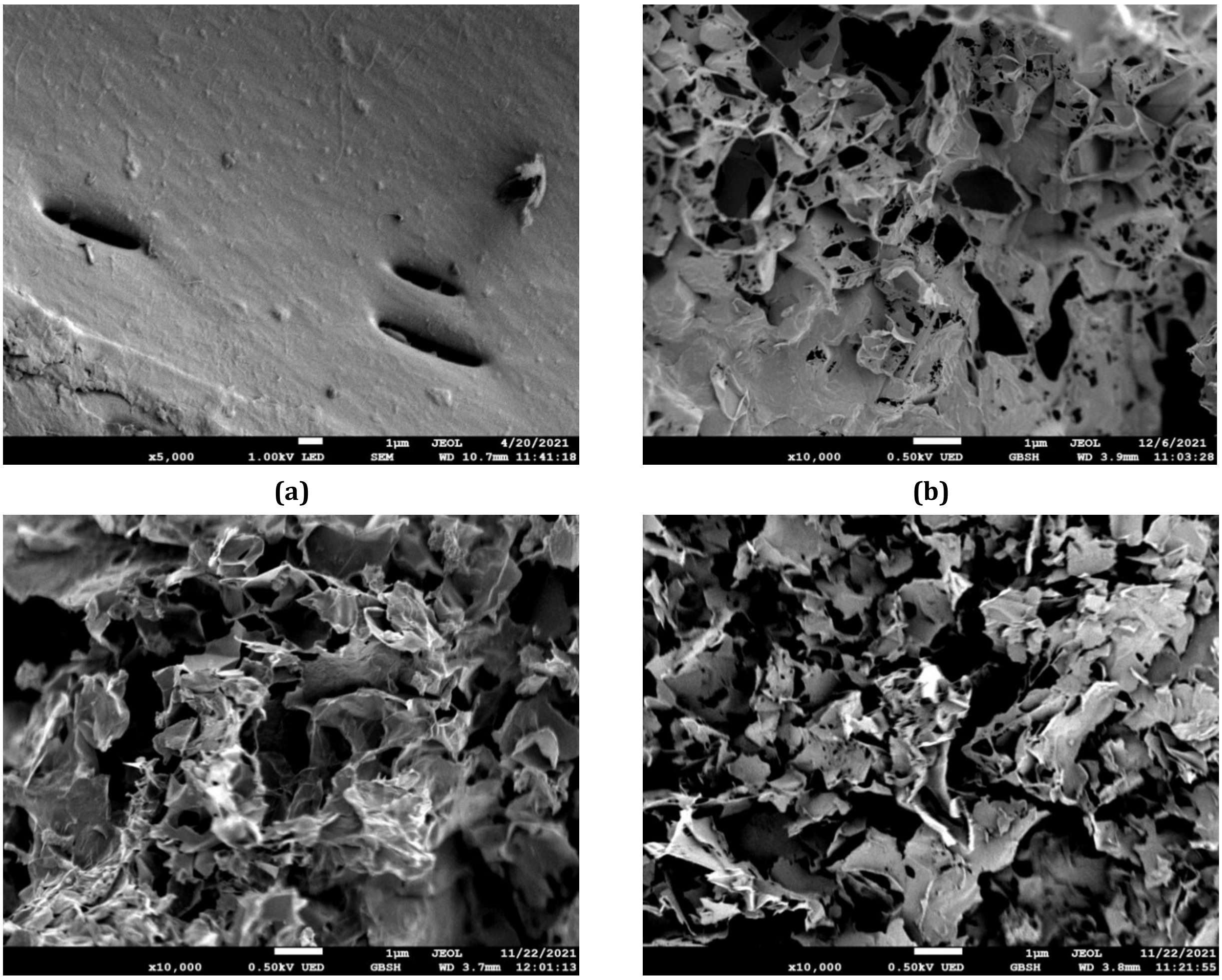
$$\%\ Removal = \frac{(C_o - C_i)}{C_o} \times 100\% \tag{2}$$

3. Results and Discussion

3.1 Scanning Electron Microscopy (SEM) Analysis

Fig. 1 illustrates the surface morphology of raw and activated rubber seed shell before and after biosorption of heavy metals. Fig. 1(a) depicts the surface morphology of the raw RSS material before chemical activation. The dense canal-like structure with minimal visible pore formation observed and relatively smooth surface, indicating a lack of significant porosity and adsorption sites, making it less effective for biosorption in its untreated state [17]. In contrast, the RSS-AC in Fig. 1(b) exhibit a deformed, porous structure of varying shapes and sizes. The changes of the RSS structure may be attributed to the NaOH activation and carbonization, enhancing the RSS porosity by removal of volatile components and non-carbonaceous components [19-20]. The development of the porous structure significantly increases the RSS surface area, which is essential for increasing the biosorption capacity of RSS-AC.

The SEM images in Fig. 1(c) and Fig. 1(d) further illustrates the RSS-AC biosorption performance for Cu and Pb ions, respectively. The reduction in visible pore structures for Cu-loaded RSS-AC image indicates that Cu ions have occupied the available pores and surface binding sites confirming the successful of the biosorption process. On the other hand, the Pb-loaded RSS-AC image reveals a more compact and layered surface, which probably due to stronger interaction between Pb ions and the functional groups on RSS-AC, resulting in more extensive coverage of the adsorption sites. This suggests that Pb ions have higher affinity compared to Cu ions. The result is in line with Melliti et al. [21], reported that the surface complexation and electrostatic attraction are the primary mechanism for heavy metal adsorption by activated carbon. The SEM images provided in this study proved that chemical activation of RSS using NaOH used has successfully enriched the porosity of the produced activated carbon.



(a) (b) (c) (d)

Fig. 1 *SEM Images of: (a) RSS raw sample; (b) RSS AC; (c) RSS AC loaded with Cu; (d) RSS AC loaded with Pb*

3.2 Fourier Transform Infra-Red (FTIR) Analysis

The changes in functional groups before and after the activation process were investigated by comparing the FTIR spectra of raw-RSS and RSS-AC as shown in Fig. 2. The spectra reveal several absorption peaks, each corresponding to specific functional groups, such as hydroxyl, carboxyl, and carbonyl groups.

In the raw RSS sample, the absorption peak observed between 1539–1872 cm^{-1} is primarily attributed to the presence of C=O stretching vibrations, indicates the conjugated and unconjugated carbonyl groups, such as carboxylic acids or ketones. These functional groups are formed through the breakdown of hemicellulose and cellulose [22]. However, the FTIR spectrum of RSS AC exhibits notable differences, including weaker intensity and the disappearance or weakening of many absorption bands. These changes occurred due to the processes involved in the preparation of RSS AC, including chemical impregnation, activation, and washing, as reported by Pagketanang *et al.* [23].

A significant difference is observed in the broad peak between 2139–3720 cm^{-1}, with specific peaks at 3028 cm^{-1} and 3705 cm^{-1} corresponding to the O–H stretching vibration of hydroxyl groups [16]. These peaks are more prominent in the raw RSS sample but weaken considerably in RSS AC, indicating the successful removal of moisture content during the activation process. Additionally, peaks between 732–983 cm^{-1} in the RSS AC spectrum can be attributed to the presence of alcohols (primary, secondary, and tertiary), phenols, ethers, and esters, which result in C–O stretching and O–H deformation vibrations on the activated carbon surface [24]. The result suggests that the use of NaOH as an activating agent effectively enhanced the removal of hydroxyl groups (O–H) during the activation process, leading to an increase in carbon content in the prepared RSS AC.

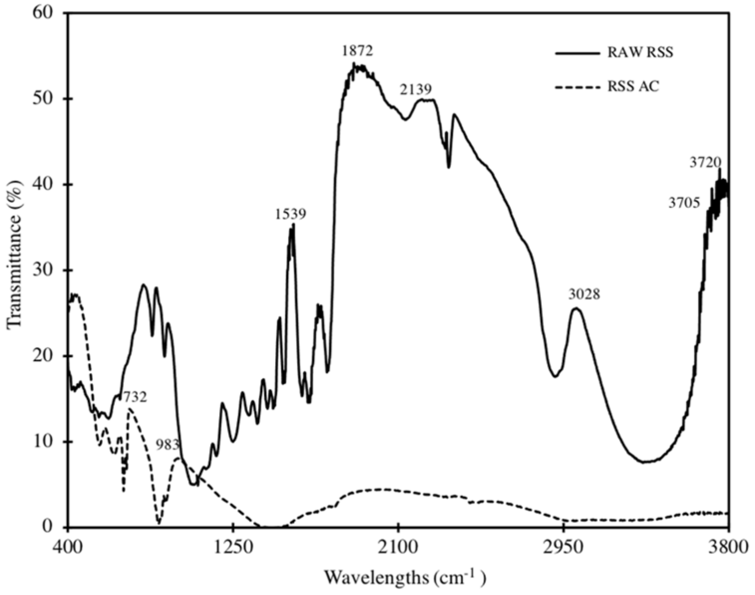


Fig. 2 *FTIR spectrum of raw RSS and RSS AC*

3.3 X-ray Diffraction (XRD) Analysis

The chemical composition of the activated carbon surfaces before and after the biosorption test was analysed using XRD to assess changes in the material's crystalline structure as shown in Fig. 3. The XRD patterns for RSS AC samples loaded with Cu and Pb after the biosorption test are shown in Fig. 3(a) and 3(b), respectively. These were compared with the XRD pattern of RSS AC before the biosorption process to identify new phase peaks arising from heavy metal adsorption.

The results confirm the uptake of Cu and Pb metal ions by RSS AC after the biosorption process. In Fig. 3(a), a diffraction peak at 12.74° is observed, indicating the presence of Cu with a relative intensity of 100%. Similarly, in Fig. 3(b), the RSS AC loaded with Pb exhibits new diffraction peaks at 24.73°, 27.23°, and 34.18°, corresponding to the presence of Pb metal ions with relative intensities of 100%, 72.32%, and 42.99%, respectively.

In addition, the XRD patterns also reveal the disappearance of some peaks present in the raw RSS-AC before the biosorption test, which at 21.18° and 29.54°. Shoba and Jeyanthi [25] reported that the absence of sharp diffraction peaks may be suggested that the materials exhibit an amorphous structure, which is consistent with current findings. The successful biosorption of Cu and Pb ions is indicated by the emergence of distinct new peaks in the XRD patterns, which also validates the interaction between the metal ions and the surface of the activated carbon.

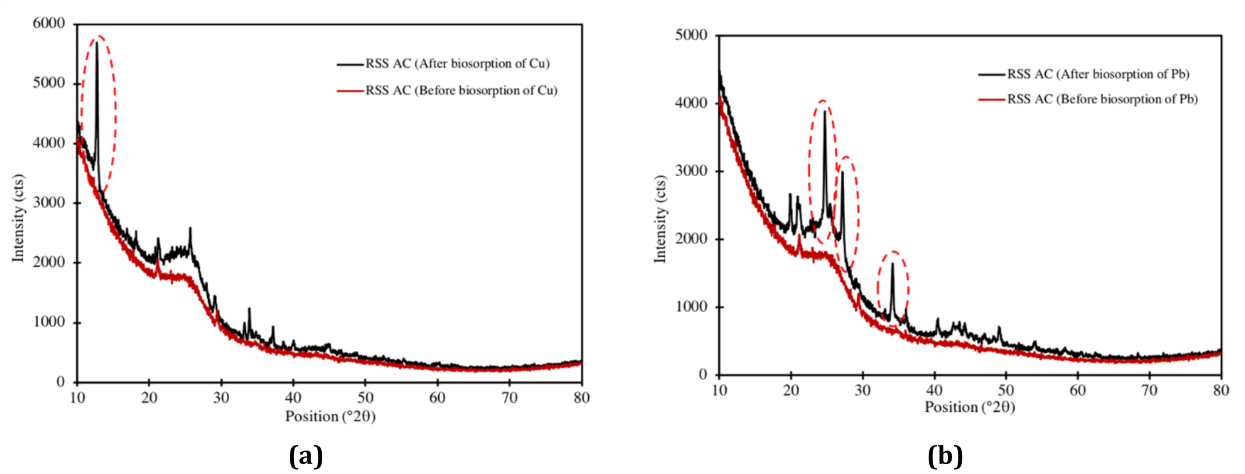


Fig. 3 *XRD analysis of RSS-AC before and after biosorption test of (a) Cu; and (b) Pb*

3.4 Batch Biosorption Test

3.4.1 Effect of Initial Concentration

The effect of different initial metal concentrations of Cu and Pb on the removal efficiency of heavy metals by RSS-AC is shown in Fig. 4. The results reveal that the Cu and Pb removal percentage decrease as the heavy metal concentration increased. The highest removal percentages were 15.6% for Cu and 26.8% for Pb observed at the lowest initial concentration of 200 ppm, indicating the effectiveness of the biosorption process at lower metal-ion concentrations [17]. This result can be attributed to the higher collision efficiency between biosorbent and heavy metal ions at lower concentrations, which facilitates faster biosorption [26].

Conversely, increasing the initial heavy metal concentrations significantly reduced the removal efficiency. The result shows that about 3% removal was observed for both Cu and Pb at 600 ppm and 800 ppm. This reduction could be attributed to overcrowding and competition among metal ions at the saturation of biosorption sites [27]. The observed plateau in removal efficiency may be contributed by overlapping of adsorption sites and the limited availability of active binding sites on RSS-AC [28]. This trend indicates that the adsorption capacity of RSS-AC had reached its maximum limit under these conditions. Therefore, further increases in the initial concentration did not lead to any significant improvement in heavy metal removal. Based on these findings, 200 ppm was selected as the initial concentration for subsequent experiments.

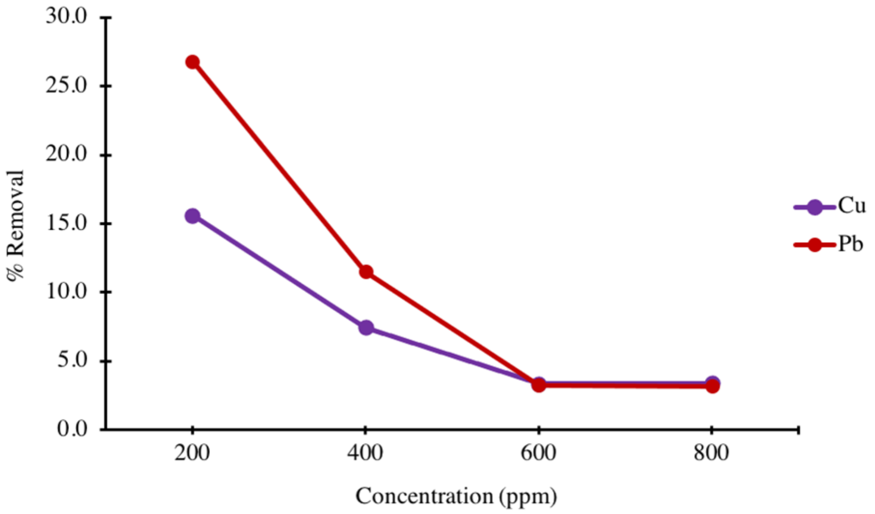


Fig 4 *Effect of initial concentration of heavy metals on the percentage removal of heavy metals (Contact time = 15 min; Biosorbent dosage = 0.05g; Stirring rate = 100 rpm)*

3.4.2 Effect of Contact Time

The effect of contact time on the biosorption efficiency of RSS-AC in removing heavy metals is shown in Fig. 5. The results revealed that the removal efficiency of Cu and Pb increased with longer contact times, until it reached a plateau. At 45 min contact time, the constant removal rate observed for Pb ions indicates the equilibrium had been achieved. This saturation effect can be attributed to the limited biosorbent dosage (0.05 g) used in the experiment, which suggests that only a finite number of binding sites were available for biosorption [29]. Consequently, extending the contact time beyond this point did not improve the removal efficiency, as the active sites on the biosorbent were fully occupied [30].

Furthermore, the result show that Pb achieved equilibrium slightly faster than Cu under the same conditions. This difference reveals the influence of specific properties of the metal ions on the biosorption process [17]. The rate of metal ions uptake or adsorption may influence by the factors such as ionic radius, valence, affinity towards functional groups (e.g., carboxyl groups), and the presence of cations [31]. Meanwhile, additional time was needed for Cu removal to reach equilibrium. According to Kumar *et al.* [32], higher removal efficiency at longer contact time was attributed to a reduction in the boundary layer resistance to mass transfer around the sorbent particles. These findings suggest that the optimal contact time for the RSS-AC in removing heavy metals is 60 minutes, where the highest removal efficiencies of 24.48% for Cu and 29.95% for Pb were observed, respectively.

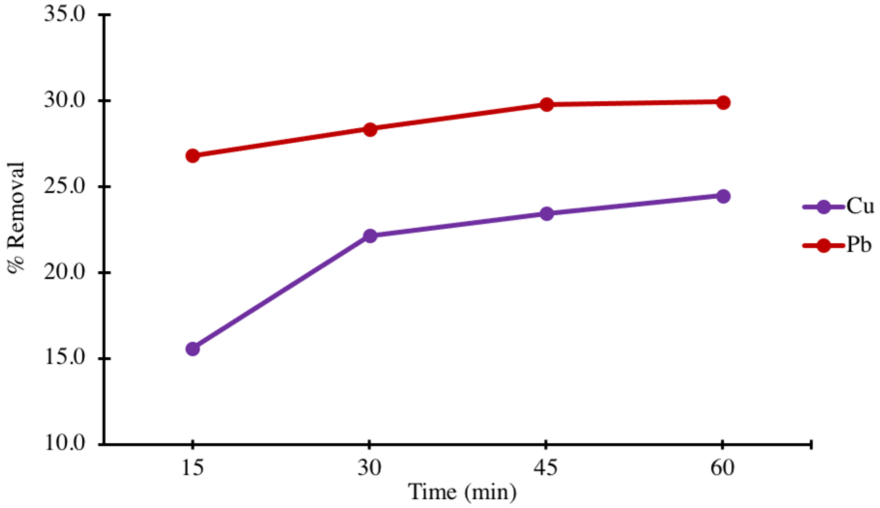


Fig. 5 *Effect of contact time on the percentage removal of heavy metals (Concentration = 200 ppm; Biosorbent dosage = 0.05 g; Stirring rate = 100 rpm)*

3.4.3 Effect of Biosorbent Dosage

The effect of biosorbent dosage on the removal of heavy metals by RSS AC produced is shown in Fig. 6. The results show that the removal efficiency of Cu and Pb were significantly enhance by the increase of biosorbent dosage. The removal rate for Cu increased from 35.54% at 0.05 g to 77.45% at 0.20 g, while Pb showed a more pronounced improvement, rising from 44.61% to 99.20%. This significant increment of the removal rate can be attributed to the higher availability of biosorption sites with increased biosorbent dosage, hence increase the surface area. The additional surface area and porosity of the activated carbon facilitate better access for heavy metal ions to the binding sites, enhancing biosorption capacity [20]. The observed trend can be explained by the presence of a greater number of binding sites or active biosorption sites that enables more metal ions to be captured effectively [33-34].

Interestingly, the results reveal that the RSS-AC exhibited a higher affinity toward Pb compared to Cu. The maximum biosorption of Pb was significantly higher than Cu under the same conditions indicating a more effective removal mechanism for Pb ions. This difference in removal efficiency can be attributed to the distinct physicochemical properties of the two metal ions, such as ionic radius, valence, and their interaction with the functional groups on the biosorbent surface [35]. Pb ions may exhibit stronger interactions with the RSS AC's surface due to their higher affinity for specific functional groups, as supported by the characterization analysis. The numerous O and N groups present upon the surface of RSS-AC promote complexation with Pb(II), which may have contributed to higher removal capacity [36]. These results demonstrate the higher capability of RSS-AC for removing Pb compared to Cu which align with Melliti *et al.* [21], who reported that Pb exhibited a higher affinity for activated carbon derived from date palm fiber compared to Cu.

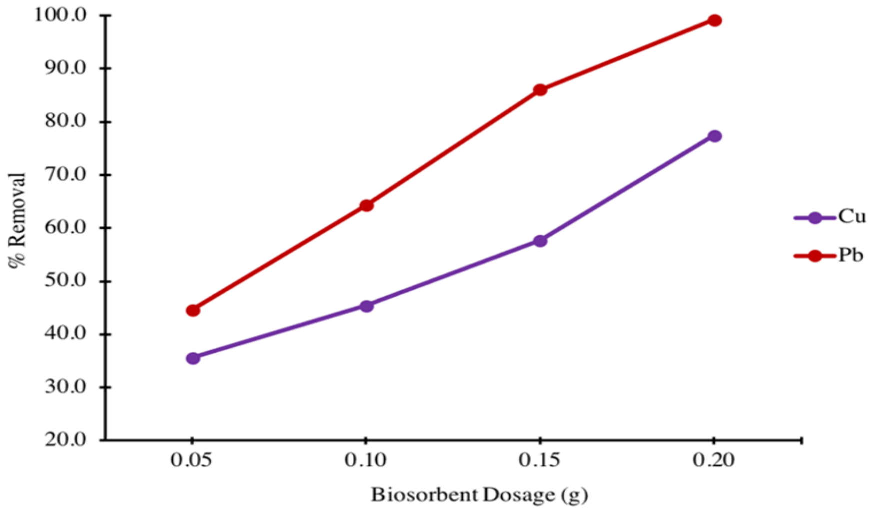


Fig. 6 *Effect of biosorbent dosage on the percentage removal of heavy metals (Concentration = 200 ppm; Contact time = 60 min; Stirring rate = 100 rpm)*

4. Conclusion

The findings of this research demonstrate that rubber seed shell (RSS), an agricultural waste, is suitable raw material for producing activated carbon (AC). Characterization analysis revealed that chemical activation of RSS using NAOH promotes high surface area and well-developed porous structure of RSS-AC. The highest removal rates of 77.45% for copper (Cu) and 99.20% for lead (Pb) were achieved at optimal conditions of 200 ppm initial heavy metal concentration, 60 minutes contact time, and a 0.20-gram biosorbent dosage. The result also indicates that RSS-AC has a higher affinity for Pb compared to Cu ions. These results highlight the potential of RSS AC as a cost-effective and eco-friendly biosorbent for heavy metal removal especially Pb.

The high Pb removal efficiency makes RSS-AC particularly suitable for industrial wastewater treatment in sectors such as battery manufacturing, metal plating, and paint production, where lead contamination is prevalent. Future studies should investigate the effects of varying activation conditions, such as acid concentration, activation temperature, and duration, to further enhance the adsorbent's efficiency and tailor it for different pollutants. Detailed kinetic and isotherm studies for various pollutants should also be conducted to better understand the adsorption mechanisms and optimize operational parameters for industrial-scale applications. Studies on the regeneration and reusability of RSS AC, using methods like thermal, chemical, or microwave-assisted regeneration, could improve its economic viability for long-term applications.

In conclusion, this research establishes RSS AC as a promising, low-cost, and environmentally sustainable biosorbent, particularly for lead removal. By expanding its application in various industries and optimizing its properties and regeneration methods, RSS AC has the potential to contribute significantly to sustainable wastewater treatment solutions.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Abd Halim Md Ali, Muhammad Hazwan Hamzah, Mohamad Akmal Abdul Rahim;* ***data collection:*** *Ahmad Hazwan Azhari, Shahirah Shamsulbahrin;* ***analysis and interpretation of results:*** *Muhammad Bukhari Rosly, Nomie Marini Aminallah;* ***draft manuscript preparation:*** *Muhammad Bukhari Rosly, Abd Halim Md Ali. All authors reviewed the results and approved the final version of the manuscript*

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