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Original Research Article

Utilization of Pyrolysed Tyres Scrap as Heavy Metal Adsorbent

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Abstract

Scrap tires continuously generated due to increasing motorized vehicle use. In Indonesia, tire waste generation reaches approximately 11 million tons annually. One processing method is pyrolysis, which converts tires into carbon, oil, and metals. However, the carbon, comprising 25–30% of tire conten, remains underutilized. This study aims to compare the performance of Tire-derived Activated Carbon (TAC) and Commercial Activated Carbon (CAC) in Pb(II) removal from wastewater. The carbon was activated using 98% H_2SO_4 (1:1 w/w) and heated in a fluidized bed reactor at 600, 650, and 700 °C for 1 hour. The optimal TAC was produced at 600 °C, with a specific surface area of 103.162 m^2/g. FTIR analysis confirmed the presence of -OH, C=C, and C=O functional groups, and SEM revealed a porous structure. Adsorption tests at varying pH and initial Pb(II) concentrations showed optimum performance at pH 5 and 30 mg/L. The Langmuir model fitted the data well, indicating monolayer adsorption on a homogeneous surface. TAC achieved 96.32% removal efficiency in synthetis medium and a maximum adsorption capacity of 240.80 mg/g, significantly higher than CAC (62.68%, 151.366 mg/g). These results demonstrate the potential of TAC as an effective low-cost alternative adsorbent for heavy metal removal.

Keywords: Activated carbon; adsorption; pyrolisis; scrap tire; sulfuric acid

1. Introduction

The rapid growth of the automotive industry has led to a significant increase in waste rubber tire generation. Globally, over 1 billion tires were produced in 2014 alone (Shahraki et al., 2021), and demand was projected to reach 3 billion units by 2019, with an annual growth rate of 4% and estimated sales of USD 258 billion (Shulman, 2019; Evans & Evans, 2006; Lindenmuth, 2006). In Indonesia, scrap tire waste is estimated at 11 million tons per year (Sumiyanto et al., 2022). This poses a major environmental challenge, as waste tires are non-biodegradable and accumulate in landfills, contributing to long-term pollution (Saleh & Gupta, 2014).

Recent advancements highlight pyrolysis as a state-of-the-art technology for handling scrap tires. This process converts waste tires into valuable by-products such as pyrolytic oil, volatile gases, and solid carbon-rich residues (Martinez et al., 2013). One of the products from tire pyrolysis is a carbon-rich material and potentially can be used as a precursor to make activated carbon for heavy metal adsorption (Saleh & Gupta, 2014). It is an economical way to reduce the direct disposal of waste tires (Shahraki et al., 2021). In recent studies, the adsorption capacity of carbon from scrap tires is low. This is due to the low surface area of the carbon. However, the surface area texture of the carbon can be improved (Acosta et al., 2016).

To address this limitation, activation processes are employed to enhance the physical and chemical characteristics of carbon. Activating agents such as KOH, NaOH, HCl, H_2SO_4 have been widely used for carbon activation process (Fung et al., 2012). Chemical activation, especially using strong acids like H_2SO_4, is known to enhance surface area and introduce oxygen-containing functional groups (Isahak et al., 2013), thereby improving adsorption performance (Doja et al., 2022; Acosta et al., 2016). The quality of activated carbon is important along with the increasing industrial activities that contribute to environmental pollution, the water pollution in particular. Adsorption is one of the most economical and effective methods for reducing the environmental impact of heavy metal pollution (Baunsele & Missa, 2020). Various materials have been used as adsorbents for removing heavy metals, with activated carbon being one of the most efficient options (El-Maadawy et al., 2024). Despite its potential, tire-derived carbon often suffers from low initial adsorption capacity due to limited surface area and pore development. While several studies have explored the activation process, few have provided a systematic comparison between tire-derived activated carbon (TAC) and commercial activated carbon (CAC), especially under varying pH and metal ion concentrations. Additionally, there is limited literature evaluating the adsorption mechanism using isotherm models to quantify the performance of TAC for heavy metal removal.

This study aims to evaluate the adsorption efficiency of Pb(II) using activated carbon derived from scrap tires, activated with H_2SO_4, and compare its performance with commercial activated carbon. The effects of pH and initial Pb(II) concentration on removal efficiency are analyzed, while the adsorption behavior is modeled using both Langmuir and Freundlich isotherms. Model of isotherm adsorption is able to give the information about adsorption maximum capacity to evaluate adsorbent performance (Wang & Guo, 2020). The findings are expected to contribute to the growing body of knowledge on sustainable wastewater treatment using waste-derived adsorbents.

2. Methods

2.1 Raw Material

The primary material used in this study was carbon obtained from the pyrolysis of waste tires from a scrap tire processing industry in Gresik, Indonesia. The scrap tires were pyrolyzed at 500 °C to produce carbon along with oil and gas by-products. The resulting carbon was ground using a laboratory blender and sieved through 200–325 mesh to obtain uniform particle sizes. To remove residual moisture that could affect adsorption performance, the carbon powder was oven-dried at 105 °C until constant weight, preparing it for further activation and adsorption experiments. In order to characterize the carbon from scrap tire, ASTM techniques were used to determine the proximate (moisture, volatile matter, fixed carbon and ash content) analyses of the samples. Results from proximate of the cerbon from scrap tire are listed in Table 1.

2.2 Preparation of activated carbons

A total of 15 g of pyrolyzed carbon from waste tires was mixed with H_2SO_4 (Merck, 98%) solution for 3 hours at room temperature (approximately 30 °C), using a mass ratio of carbon to H_2SO_4 of 1:1 w/w. The impregnation process was stirred in a 250 mL beaker glass at 300 rpm. The mixture was poured into a porcelain dish and dried in an oven Thermo Scientific at 120 °C for 24 hours. The chemically activated carbon was placed in a fluidized bed reactor and purged with nitrogen gas (N_2) at a flow rate of 100 cm^3/min, then thermally activated at varied temperatures of 600 °C, 650 °C, and 700 °C for 1 hour. The resulting activated carbon was washed with 0.5 N NaOH solution, followed by repeated washing with hot distilled water until the pH of the rinse water reached neutral. It was then rinsed again with cold distilled water, dried at 120 °C for 24 hours, and stored in a desiccator.

2.3 Characterization of Activated Carbon

Proximate analysis were carried out by a thermogravimetric analyzer (Nabertherm LE 6). Surface texture characteristics of sample were determined by N2 adsorption at -196 °C with the accelerated surface area and porosimetry system (Quantachrome TouchWin). The specific surface area was calculated from the isotherm by Brunauer Emmett–Teller (B.E.T) equation. Surface analyses were performed to determine the physical and chemical characteristics, such as morphological and elemental content analysis was conducted by scanning electron misroscopy energy dispersive X-ray (SEM-EDX-Termo Fisher), and surface functional groups was determined by Fourrier Transform Infrared (FTIR-SHIMADZU 8400S).

2.4 Batch Sorption Experiment

Adsorption was carried out by adding 0.1 g of activated carbon to 25 mL of Pb(II) synthetic waste water containing 50 mg/L (Rahayu et al., 2023) with varying pH levels (3, 4, 5, 6, and 7). The adso (1) process was conducted for 60 minutes under magnetic stirring. After the contact time, the solution was filtered to separate the activated carbon, and the filtrate was analyzed for Pb(II) ion concentration using Inductively Coupled Plasma (ICP) Avio 550 max. The pH value that showed the highest adsorption efficiency was subsequently used in the following experiments. Pb(II) ion solutions were prepared at various concentrations: 5 mg/L, 10 mg/L, 15 mg/L, 20 mg/L, and 30 mg/L. Each 100 mL solution was adjusted to the optimum pH determined from the pH effect adsorption experiment. Each solution was analyzed using ICP to obtain the baseline concentration data. Subsequently, 0.05 g of TAC was added to each solution and stirred using a magnetic stirrer for 60 minutes (Annisa et al., 2024). After the adsorption process, the mixtures were filtered using Whatman 42, and the filtrates were analyzed for Pb(II) concentration using ICP Avio 550 max. The percentage of efficiency removal of heavy metal ions and the adsorption capacity were calculated using Equations (1) and (2) as follows:

$$\%\,Adsorption\ efficiency\ = \frac{(C_o - C_e)}{C_o} \times 100\% \tag{1}$$

$$q_e\ = \frac{(C_o - C_e)\,V}{W} \tag{2}$$

Where q_e is the amount of metal ion adsorbed per unit weight of activated carbon at equilibrium (mg/g), C_e is the equilibrium concentration of the adsorbate in the solution (mg/L), C_0 is the initial concentration of the adsorbate (mg/L), W is the mass of the adsorbent (g), and V is the volume of the solution (L).

2.5 Adsorption Isoterm Model Langmuir and Freundlich

The adsorption mechanism of TAC is analyzed using Langmuir and Freundlich isotherm models. The Langmuir isotherm model was associated with adsorption process through the single layer formation process (monolayer). This model is based on the assumption of homogeneous distribution of adsorption site, constant adsorption energy, neglectable interactions between adsorbate molecules [El-Sayed, 2011]. The Langmuir linear isotherm model can be expressed by Equation (3)

$$\frac{C_e}{q_e} = \frac{1}{Kl\,qm} + \frac{1}{qm}\ Ce \tag{3}$$

The Freundlich isotherm represents multilayer adsorption on the heterogeneous surface with the considerable interaction between the adsorbed molecule with the distribution of its non-uniform absorption energy between the surfaces [Sadaf & Bhatti, 2014]. The equation model can be defined by Equation (4).

$$log q_e = log K_F + \frac{1}{n} log C_e \tag{4}$$

Where Ce represents the equilibrium concentration of adsorbate (mg/L), qe is the amount of adsorbate which was absorbed by the adsorbent (mg/g), *Kl* is the ratio of adsorption and desorption rate (L/mg), *qm* is the maximum adsorption capacity (mg/g). Kf [mg/g (L/mg)n] is the Freundlich constant which is related to adsorption capacity (referring to the quantity of adsorbate in the adsorbent) and n is the Freundlich constant which indicates the intensity of adsorption. The value of n gives an indication of how well the adsorption process is

3. Results and Discussion

3.1. Proximate Analysis of Raw Material

The results of the proximate analysis of the carbon from scrap tire are listed in Table 1. A comparison with the results of several previous studies is also presented (Diez et al., 2004; Galvagno et al., 2002; Li et al., 2004). It illustrated that the carbon produced by pyrolysis of scrap tires at 500 °C in this study shows quite competitive quality.

Table. 1 Chemical characteristics of scrap tire waste used as raw material

Parameter	This study	Diez et al., 2004	Galvagno et al., 2002	Li et al., 2004
Temperature of pyrolisis (°C)	500	550	550	550
Moisture content (%)	2,0	1.00	3.57	1.28
Ash content (%)	17.3	16.50	15.33	14.58
Volatile Matter (%)	8	1.20	12.78	6.92
Fixed Carbon (%)	72.7	81.30	66.19	77.22
Total Sulfur (%)	2.82	1.81	2.13	1.40

The characterization results of the carbon obtained in this study and a comparison with several previous studies (Diez et al., 2004; Galvagno et al., 2002; Li et al., 2004), it can be concluded that the pyrolyzed carbon produced from waste tires at 500 °C in this research exhibits a reasonably competitive quality. The moisture content of 2.0% is relatively low and comparable to the value reported by Galvagno (3.57%), although slightly higher than those reported by Diez et al. (1.00%) and Li et al. (1.28%). The ash content, at 17.3%, is higher than in all referenced studies (ranging from 14.58–16.5%), indicating the presence of inorganic residues from the raw material, such as metals or silica from the waste tires, which were not fully decomposed or eliminated during pyrolysis (Botahala, 2002). The volatile matter content in this carbon was 8.0%, higher than that reported by Diez et al. (1.20%) and Li et al. (6.92%), but lower than Galvagno's result (12.78%). This relatively low volatile content reflects a successful pyrolysis process in removing volatile components, although not as efficient as in the study by Diez. Meanwhile, the fixed carbon content reached 72.7%, which is higher than Galvagno's (66.19%) but slightly lower than the values reported by Diez et al. (81.30%) and Li et al. (77.22%). These results suggest that the carbon produced in this study has a high fixed carbon content, indicating good potential as an adsorbent or energy material, although further optimization of the pyrolysis process could enhance its quality.

3.2. Activation

The thermal activation process on carbon results in mass loss as shown in Table 2. Carbon activated at activation temperature 600°C, 650°C, and 700°C. Carbon activation is a process aimed at increasing the surface area and porosity of carbon materials through physical or chemical treatments. This process is intended to develop an optimal pore structure for adsorption applications (Kolur et al., 2019). In this study, chemical activation using sulfuric acid (H_2SO_4) was selected due to its ability to oxidize organic compounds and open carbon pores through dehydration reactions, which remove volatile components and leave behind a porous carbon framework. Isahak et al. (2013) reported that carbon dehydrated with sulfuric acid exhibits greater surface area and porosity compared to other activation methods. After activation with sulfuric acid, the carbon surface typically contains sulfonic and oxygen-containing functional groups, which enhance its affinity toward adsorbates, although they may slightly reduce pore volume. This highlights that adsorption activity is influenced not only by surface structure but also by the presence of functional groups. The activation temperature range of 600–700 °C was chosen based on literature indicating it as optimal for sulfur compound decomposition while minimizing the degradation of the carbon structure (Sirimuangjinda et al., 2019; Prasetyo et al., 2011). The high yield of activated carbon (>90%) observed across all temperature variations demonstrates the effectiveness of this method in preserving carbon mass while developing porosity. However, a slight reduction in mass was noted with increasing temperature due to the release of surface-bound functional groups (Kayiwa et al., 2021)

Table 2. Mass before and after carbon activation

Carbon	Activation Temperature	Mass before activation (g)	Mass after activation (g)
TAC-1	600 °C	10	9.920
TAC-2	650 °C	10	9.702
TAC-3	700 °C	10	9.412

The carbon mass decreased from 10 g to 9.920 g (600°C), 9.702 g (650°C), and 9.412 g (700°C), indicating that increasing the activation temperature increases the mass loss. This mass loss is caused by the release of volatile compounds and decomposition of organic materials during the pyrolysis process, as well as selective oxidation that forms pores (Yahya et al., 2015). At higher temperatures (700°C), the mass removal is greater (5.88%) than at 600°C (0.08%), as the higher thermal energy accelerates the gasification reaction between the carbon and the activation agent (N2), thereby increasing material degradation (Lozano-Castelló et al., 2001). Activation efficiency can be attributed to the formation of an optimal pore structure, where temperatures of 650-700°C are generally considered effective for producing highly porous carbons with large surface areas (Jagtoyen & Derbyshire, 1998).

In addition, the reduction in mass can also be attributed to the chemical activation process using sulfuric acid (H_2SO_4) conducted under a nitrogen atmosphere. Sulfuric acid acts as a strong dehydrating agent (Amirudin et al., 2020), especially at elevated temperatures. The higher the activation temperature, the greater the degradation and release of volatile compounds in the form of gases such as CO, CO_2, and water vapor, thereby decreasing the final mass of the resulting activated carbon. Increasing the temperature accelerates the development of pore structures on the carbon surface. The formation of these pores through chemical activation leads to partial loss of carbon mass. However, despite the reduction in final mass, the resulting activated carbon tends to exhibit a higher surface area and improved adsorption properties (Pasaribu et al., 2024). This process is facilitated by the use of an inert atmosphere (N_2), which prevents complete combustion. High-temperature heating helps remove residual non-carbon compounds, producing a more ordered carbon structure, though some carbon mass is inevitably lost. Continuous nitrogen flow into the reactor during heating has been shown to enhance the fixed carbon content (Hasan et al., 2020).

3.3. Surface area and porosity

The BET test in this study resulted in the surface area of the Tire Carbon without Activation (TC) and Tyre Active Carbon (TAC) which can be seen in Table 3.

Table 3. Carbon pore characteristics

Parameter	Unit	TC	TAC-1 (600 °C)	TAC-2 (650 °C)	TAC-3 (700 °C)
Surface area	m^2/g	86.980	103.162	96.491	100,944
Total pore volume	cm^3/g	0.242	0.250	0.303	0.291
Mean pore diameter	nm	8.302	4.847	6.280	5.778

Before activation, the carbon (TC) had a surface area of 86.980 m^2/g, which increased to 103.162 m^2/g after activation at 600 °C (TAC-1). The pyrolyzed tire-derived carbon activated with sulfuric acid (1:1 w/w ratio) at 600 °C showed a notable improvement in surface area. Further activation at higher temperatures, 650 °C and 700 °C (TAC-2 and TAC-3)—resulted in surface areas of 96.491 m^2/g and 100.944 m^2/g, respectively. The increase in surface area of the activated carbon was attributed to the removal of ash and other impurities during the activation and combustion processes, which helped open up the carbon pores (Slamet, 2006). However, the surface area enhancement was not particularly significant. A study by Sirimuangjinda et al. (2013) reported that activation with sulfuric acid at 600 °C produced an extremely high surface area of 1037.10 m²/g, while increasing the temperature to 700 °C actually reduced the surface area to 876.60 m²/g. This decrease was likely caused by pore blockage or re-closing due to redeposition of ash or inorganic impurities that were not fully decomposed during activation. Residual or newly formed ash at high temperatures may settle back into the pores, narrowing or even sealing previously open pores (Ioannidou & Zabaniotou, 2007).

Moreover, excessive heat can also lead to graphitic restructuring that causes pore shrinkage or collapse. Ioannidou & Zabaniotou (2007) noted that lignocellulosic materials tend to form pores more easily compared to carbon-black-based materials such as rubber (waste tires) or plastic. The impregnation ratio (i.e., the mass ratio of activating agent to raw material) is another key factor in determining activation success. Common ratios range from 1:1 to 3:1, depending on the type of activator and precursor. Jagtoyen & Derbyshire (1998) reported that insufficient acid results in incomplete carbonization and limited pore formation. Increasing the ratio of activating agent (from 1:1 to 3:1) can significantly enhance surface area from around 400 m²/g to over 1500 m²/g (Lua & Yang, 2004).

The total pore volume also increased following activation. Before activation, the pore volume was 0.242 cm³/g. After activation, it increased to 0.250 cm³/g (TAC-1), 0.303 cm³/g (TAC-2), and 0.291 cm³/g (TAC-3). The greatest increase occurred at 650 °C (TAC-2), suggesting this temperature is optimal for pore development, particularly mesopores. The average pore diameter decreased after activation, from 8.302 nm to 4.847 nm (TAC-1), 6.280 nm (TAC-2), and 5.778 nm (TAC-3). This reduction indicates that activation led to the transformation of larger pores (macropores) into smaller ones—namely micropores or mesopores—which are more effective for adsorbing heavy metals or small molecules. Overall, carbon activation at 600–700 °C proved effective in enhancing the physical properties of activated carbon. The temperature of 600 °C appears most optimal for increasing surface area and forming micropores, while 650 °C is more favorable for developing pore volume. Although 700 °C remains effective, there are indications that excessively high temperatures may damage the pore structure.

3.4. Surface Functional Group

The functional groups present in the activated carbon derived from waste tires show FTIR spectrum ranging from 4,000 to 450 cm⁻¹. Figure 1 shows the infrared spectrum from the FTIR spectroscope test of the active carbon sample. All three activated carbon samples exhibited strong peaks around 1100 cm⁻¹, corresponding to Si–O, S=O, and C–O functional groups. A peak at approximately 1600 cm⁻¹ indicates the presence of a carbonyl (C=O) group, while the peak near 2900 cm⁻¹ is associated with C–H (alkyl) stretching. The broad peak observed around 3400 cm⁻¹ typically signifies the presence of hydroxyl (–OH) groups. These peaks suggest that the activated carbon contains hydrogen bonds originating from alcohol and silicate groups. Among the three samples, the lowest transmittance was observed in the spectrum of Carbon C (TAC-3), indicating a higher degree of degradation or removal of functional groups such as C=O, C–H, and –OH due to the higher activation temperature of 700 °C. The functional groups identified in the waste-tire-derived activated carbon show similarities to those reported by Dimpe et al. (2017), whose study analyzed spectra in the range of 4000–450 cm⁻¹. Peaks centered around 3400, 1700, and 1627 cm⁻¹ were attributed to the presence of –OH groups from phenols and C–O bonding. The peak at 3400 cm⁻¹ was associated with O–H stretching vibrations of hydroxyl groups (Al-Saadi et al., 2013), while the 1700 cm⁻¹ peak indicated stretching vibrations characteristic of carboxyl groups, reinforcing the presence of C=O bonding. Aromatic skeletal stretching vibrations were observed at approximately 1627 cm⁻¹ (Dimpe et al., 2017).

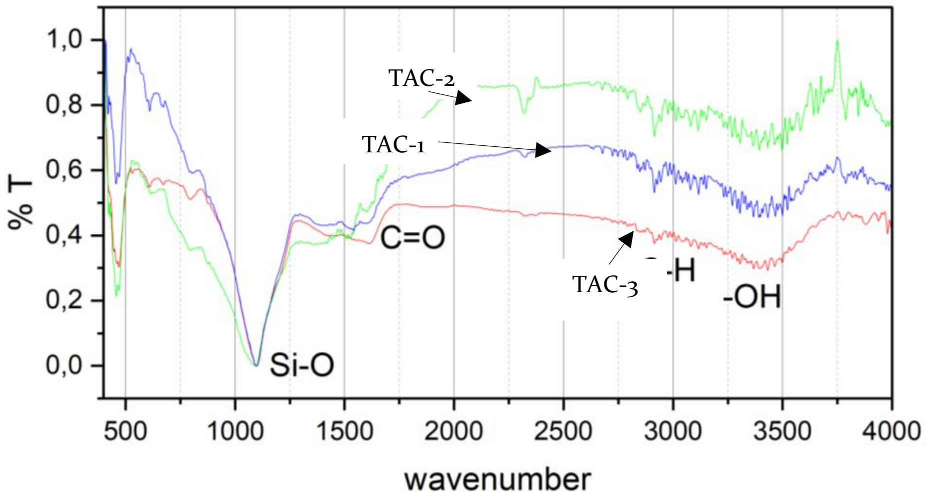


Figure. 1 FTIR test result of active carbon from scrap tyre

3.5. Moisture and Ash Content

Mosture and ash moisture content tests were conducted on TAC-1. This is based on the results of specific surface area testing using BET analysis which shows the most optimal number with a specific surface area value of 103.162 m²/g. The moisture content of the activated carbon derived from waste tires at an activation temperature of 600 °C was 0.69%. Moisture content is an important parameter for activated carbon, as it can influence its adsorption capacity. Activated carbon is hygroscopic, especially when it contains silica compounds or polar functional groups such as –OH, which tend to absorb water vapor from the atmosphere. The higher the water content, the active pores on the carbon can be filled by water molecules, reducing the ability of carbon to adsorb other substances, such as heavy metals (Elijah et al., 2024). Therefore, good activated carbon generally has the lowest possible moisture content.

Table. 4 The ash content value of activated carbon derived from waste tires

Activation	Ash Content (%)	References
KOH impregnation with a 4:1 ratio (activating agent to precursor) followed by thermal treatment at 750 °C.	28	Acosta et al. 2016
Physical activation using CO_2 gas followed by thermal treatment at 550 °C	10,1	Hofman et al. 2011
KOH impregnation at a 1:1 weight ratio followed by thermal activation at 700 °C.	4,72	Ozbas et al. 2019
H_2SO_4 impregnation at a 1:1 weight ratio followed by thermal activation at 600 °C.	35,60	This study

The ash content of TAC-1 is 35.60 %. High ash content usually comes from a non-optimal carbonization process, such as a carbonization temperature that is too high or a long carbonization time, which causes the formation of mineral salts and fine particles that increase ash content. Ash refers to the metal oxides present in activated carbon, consisting of non-volatile minerals that do not evaporate during ashing (Botahala, 2022). Ash content significantly affects the quality of activated carbon. Excessive ash can clog the pores of activated carbon, thereby reducing its surface area (Ikawati & Melati, 2009). Reducing ash content can be achieved by increasing the amount of activating agent used (Fiqriawan et al., 2023). According to the Indonesian National Standard (SNI), the maximum allowable ash content for powdered activated carbon is 10%. High ash content in activated carbon may result not only from the characteristics of the raw material but also from technical factors during measurement. Common sources of error include deviations from standard ashing temperature and duration—e.g., heating at ≤ 750 °C until constant weight is achieved. Additionally, improper heating procedures may cause fixed carbon to continue burning, leading to its inclusion in the ash content. These findings highlight the importance of rigorous control over the ashing procedure, including proper sample preparation, instrument calibration, and adherence to standard laboratory protocols such as those set by ASTM or SNI. Previous studies that measured ash content in activated carbon derived from waste tires are presented in Table 4.

3.6. Morfology of Activated Carbon

The results of SEM micrograph analysis of carbon samples before and after activation are shown in Figure 2. SEM images before and after activation visually show similarities in terms of rough and dense surface morphology.

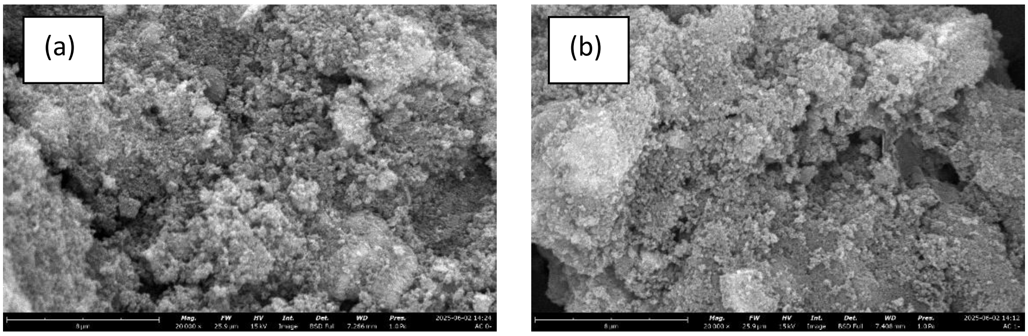


Figure 2. SEM test results of carbon (a) before activation (b) after activation

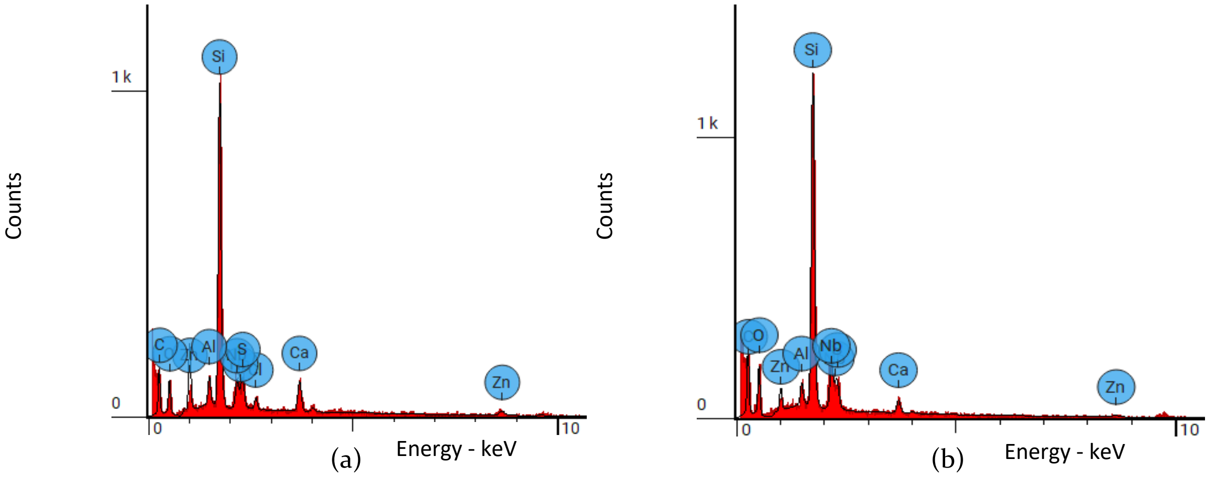
SEM images before and after activation visually showed similar surface morphology characterized by a rough and dense texture. However, quantitative porosity analysis revealed changes in the amount of porosity formed after activation. Following activation, the surface structure exhibited signs of micropore formation and fine cracks, indicating an increase in the material's porosity. The cavities on the surface of the activated carbon are caused by the evaporation of chemical reagents during heating, which leaves behind spaces previously occupied by the reagents (Sirimuangjinda et al., 2013). Fig. 2 (a) shows the surface of the carbon before activation with a texture that tends to be dense and relatively dense particle agglomeration. Meanwhile, Fig. 2 (b) shows the morphological changes after activation, where the surface appears smoother and more open. This is in line with the increase in specific surface area value from 86.98 m²/g to 103.162 m²/g after the activation process, which indicates the formation of new pores or enlargement of existing pores. This increase indicates that activation is capable of expanding the adsorptive surface area of carbon by removing volatile substances and opening the pore structure. Both chemical and physical activation are known to enhance the surface area and pore volume of activated carbon, making them essential processes in adsorption applications, including the removal of heavy metals from wastewater (Sudaryanto et al., 2022; Taki & Salman, 2023).

Table 5. EDX results of Carbon from scrap tires

Element	Before acttivation	After activation	Before activation	After activation
	% Mass		% Atom	
C	29.800	35.764	52.295	57.766

Element	**Before acttivation**	**After activation**	**Before activation**	**After activation**
	% Mass		**% Atom**	
O	10.600	14.186	13.963	17.199
Al	2.900	2.098	2.266	1.509
Si	25.500	24.176	19.133	16.695
S	10.800	16.284	2.450	3.400
Cl	2.400	-	1.426	-
Ca	7.500	2.997	3.944	1.451
Zn	7.100	2.398	2.289	0.711
Nb	3.400	2.098	2.234	1.269

Table 5. showed a relative increase of C (from 52.3% to 57.8%) and O (from 13.96% to 17.20%) atoms after activation, indicating the opening of the carbon structure and the addition of oxygen functional groups on the surface (Acosta et al, 2016). A decrease in metallic and inorganic elements is also evident in the data (mass %): Al dropped from 2.9 to 2.10 %, Si from 25.5 to 24.2 %, Ca from 7.5 to 3.0 %, Zn from 7.1 to 2.40 %, and Cl disappeared completely, due to demineralization by sulfuric acid of the pyrolyzed carbon residue (Gonzales-Gonzales et al., 2020). The increase in sulfur from 10.8% to 16.3% could be related to the increase in inorganic S concentration due to the formation of sulfonate groups on the carbon surface (Low & Yee, 2021). These results suggest the activation process with H_2SO_4 is effective in enriching carbon and oxygen content, increasing surface area and reducing metal contaminants to improve the quality of activated carbon as an adsorbent (Gonzales-Gonzales et al., 2020). Figure 4.5 is the EDX spectrum of carbon from used tires (a) before activation and (b) after activation.



(a) (b)

Figure 3. EDX spectra of carbon from scrap tires (a) before activation and (b) after activation

Based on the study by Lopez et al. (2019), the elemental composition of activated carbon derived from waste tires is dominated by carbon (C), oxygen (O), nitrogen (N), phosphorus (P), and sulfur (S). The highest concentrations are carbon and oxygen. The activated carbon produced primarily consists of C and O, which are the two main elements commonly found in activated carbon. The presence of sulfur is attributed to the interaction between H_2SO_4 and the tire-derived carbon during the activation process. According to Ozbas et al. (2019), the carbon content in activated carbon from waste tires was reported to be 66.93%, while the oxygen content was 33.07%.

3.7. Adsorption Capacity and Isotherms

The adsorption process was conducted on Pb(II) solution with pH variations of 3, 4, 5, 6 and 7 with 0.1 g activated carbon. Figure 4 is graph of Pb(II) adsorption effiecieny against pH.

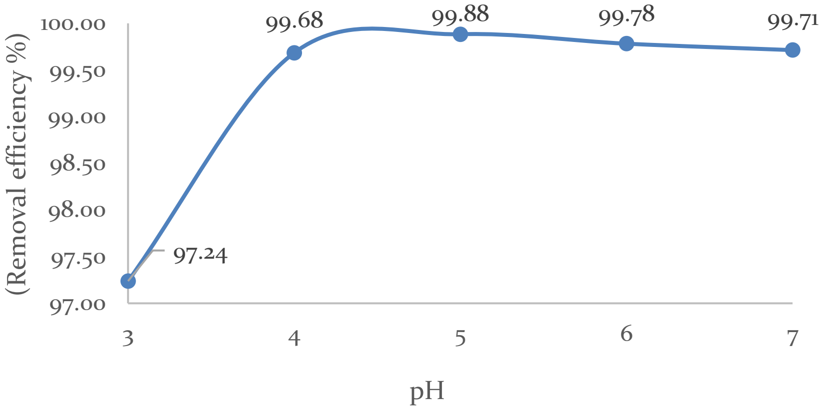


Figure 4. The effect of pH on the adsorption of Pb(II)

The optimum pH for Pb(II) adsorption occurred at pH 5, with an adsorption efficiency of 99.88%. This pH was subsequently used in experiments involving varying adsorbate concentrations. According to Xu et al. (2007), at low pH levels (<4), Pb^{2+} ions dominate because acidic conditions inhibit hydrolysis. As the pH increases (4–8), hydroxide species such as $Pb(OH)^+$ and $Pb(OH)_2$ begin to form. At pH levels above 10, the predominant species shifts to $Pb(OH)_3^-$ due to strongly basic conditions. The adsorption of Pb(II) onto activated carbon is most effective at around pH 5, where positively charged Pb^{2+} species are still dominant and can interact with the negatively charged surface of the activated carbon. At pH values below 3, the presence of excess H^+ ions competes with Pb^{2+} for adsorption sites, reducing efficiency. Conversely, at pH levels above 7, the formation of neutral or negatively charged species decreases adsorption effectiveness. These findings are consistent with Babel & Kurniawan (2003) and Mohan & Pittman (2006), who reported that the optimal pH range for heavy metal adsorption is between 4 and 6, and that precipitation at higher pH levels can hinder the adsorption process. The results of Pb(II) adsorption under varying Pb(II) concentrations can be seen in Figure 5.

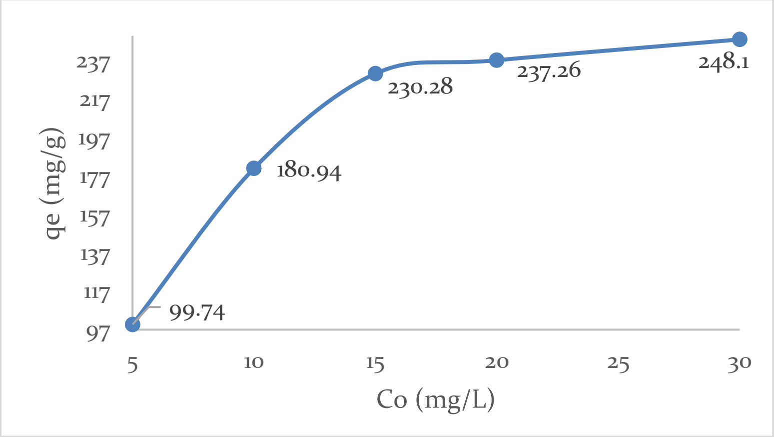
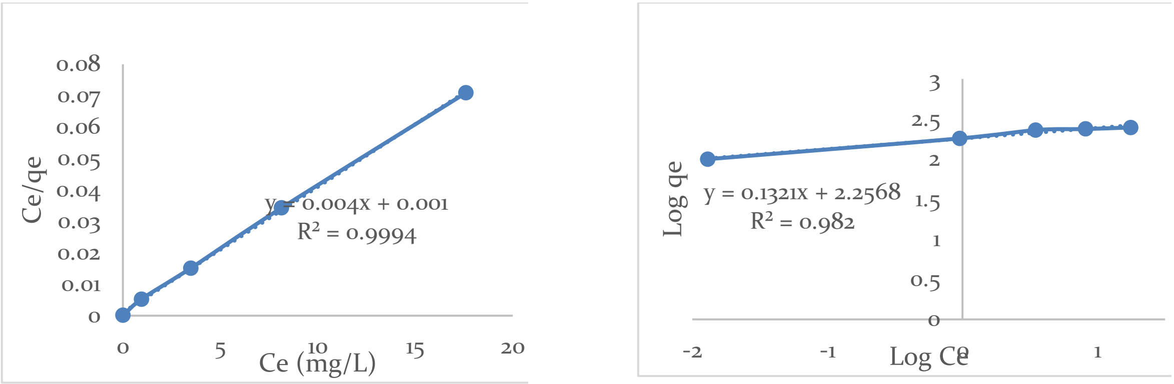


Figure 5. The effect of Pb(II) concentration

At a low initial concentration (C_0) of 5 mg/L, nearly all adsorbate was adsorbed with an efficiency of 99.74% and an adsorption capacity (q) of 99.74 mg/g. This indicates that the adsorbent possessed many available active sites, allowing it to bind almost all adsorbate molecules from the solution. This finding aligns with Al-Ghouti & Da'ana (2020), who explained that at low adsorbate concentrations, nearly all molecules can be adsorbed due to the high ratio of active sites to adsorbate molecules. As the initial concentration (C_0) increased from 10 to 30 mg/L, the adsorption capacity (q) continued to rise from 180.94 to a peak of 248.10 mg/g (at C_0 = 30 mg/L), although the adsorption efficiency decreased from 90.47% to 41.35%. This decline is attributed to the saturation of active sites on the adsorbent. When C_0 increases, the mass transfer driving force accelerates initial adsorption, but the limited number of active sites leads to a reduction in efficiency (Foo & Hameed, 2010). Higher C_0 also enhances the diffusion rate of adsorbate molecules to the adsorbent surface; however, once saturation is reached, unadsorbed molecules remain in the solution (Weber & Morris, 1963). Figure 6 shows the Langmuir and Freundlich isotherm models as the calculation result of Pb(II) adsorption. The mathematic equation model in Figure 6 shows that adsorption Pb(II) was better fitted to the Langmuir model at Figure 6a compared to the Freundlich model at Figure 6b. The same thing happened to the mathematical equation.



(a) (b)

Figure 6. Pb(II) adsorption by TAC-1 by the Langmuir and Freundlich isotherm

In this study, Langmuir and Freundlich models were applied to model the adsorption isotherm data. The results of adsorption isotherm analysis showed that Langmuir and Freundlich models provided important information about the adsorption mechanism of activated carbon in Table 6. The Langmuir model showed values of q_m = 250 mg/g and K_L = 4 L/mg, with a coefficient of determination R^2 = 0.9994, which indicates that the adsorption process follows a monolayer pattern on a homogeneous surface with very high affinity (Weber & Chakraborty, 2022).

Table 6. Adsorption Isotherm Constant of TAC

Isotherm	Parameter	Unit	Value
Langmuir	qm	mg/g	250
	KL	L/mg	4
	R^2	-	0.9994
Freundlich	n	-	7.57
	KF	mg/g (L/mg)n	9.55
	R^2	-	0.982

Previous studies have documented the modification of waste tires as adsorbent materials. For example, in a study by Nieto-Marquez et al. (2017), adsorbent were prepared from waste tires by different activating agent/waste tire ratios (1:2 w/w) for the removal of ions Pb(II). Based on their results, the maximum adsorption capacities of the adsorbent were 49.7, mg g^{-1} for Pb(II). respectively. Mustafa et al. (2003) produced a tire activated carbon for the removal of Pb, and reported a maximum Pb adsorption capacity of 19.96 mg/g. The maximum adsorption capacities for ions Pb(II) of TAC from previous studies were lower than those achieved by the present work (250 mg/g). Moreover, compared to other adsorbent materials reported in the literature (Table 7).

Table 7. Adsorption capacities for different types of adsorbents reported in literature.

Adsorbent	Maximum Adsorption Capacity (mg.g$^{-1)}$	References
TAC	250	This study
Cauliflower leaves biochar	177.82	Ahmad et al. (2018)
Beta-cyclodextrin polymers	196.40	He et al. (2017)
Carbon nanotube sheets	117.64	Tofighy & Mohammadi (2011)
Coconut derived activated carbon	92.72	Anirudhan & Sreekumari (2011)

3.8. Comparison with Comercial Activated Carbon

The adsorption performance of three types of carbon adsorbents—Commercial Activated Carbon (CAC), Tire Carbon without activation (TC), and Tire-derived Activated Carbon (TAC)—was evaluated using an initial Pb(II) concentration of 30 mg/L. The adsorption result can be seen in Table 8.

Table 8. Activated carbon adsorption comparison test results

Carbon	Co (mg/L)	Ce (mg/L)	Removal Efficiency (%)	qe (mg/g)
CAC	30	11.836	60.540	151.366
TC	30	15.043	49.850	124.641
TAC	30	1.104	96.320	240.800

The TAC showed the lowest equilibrium concentration (C_e) of 1.104 mg/L, demonstrating maximum capability in removing Pb(II). The adsorption efficiency achieved was 96.32%, with an adsorption capacity (q_e) of 240.800 mg/g. The activated carbon contains functional groups (such as –OH, –COOH, or sulfur groups) that interact with Pb(II) via electrostatic forces or hydrogen bonding, thereby enhancing adsorption performance (Foo & Hameed, 2010). The adsorption capacity of the activated tire-derived carbon is attributed to the activation process, which increases the surface area and pore volume. According to Doja et al. (2022), chemical activation enhances carbon's adsorption ability by producing microporous structures and surface functional groups that are more reactive toward heavy metal ions. A high ash content (around 30%) does not necessarily reduce adsorption if the ash particles do not block active pores. In fact, some minerals in the ash (such as silica or alkali metals) may act as additional active sites for adsorption. However, if the ash clogs the pores, adsorption performance will decline (Yahya et al., 2015).

Tire-derived carbon inherently exhibits graphitization properties due to the rubber and carbon black components used in tires, resulting in a more ordered and conductive carbon structure (Mui et al., 2010). This can enhance affinity for certain pollutants compared to commercial activated carbon derived from wood or coal. In contrast, pyrolyzed carbon without activation demonstrated a lower adsorption efficiency of 49.85%, adsorption capacity of 124.641 mg/g, and a C_e value of 15.043 mg/L. Commercial activated carbon showed an adsorption efficiency of 60.54% and adsorption capacity of 151.366 mg/g. his is in line with the study by Shahraki et al. (2021), which reported that activated carbon derived from waste tires has an adsorption capacity for Pb(II) of 322.5 mg/g, whereas commercial activated carbon (CAC) exhibited a capacity of only 42.5 mg/g. These results indicate that activated carbon produced from waste tires has the potential to serve as an alternative adsorbent due to its superior adsorption efficiency compared to commercial activated carbon.

4. Conclusions

Carbon derived from the pyrolysis of used tires and activated with sulfuric acid exhibits promising characteristics as an adsorbent. Activated carbon treated at 600 °C demonstrated the highest specific surface area, reaching 103.162 m^2/g, with a total pore volume of 0.25 cm^3/g and an average pore diameter of 4.847 nm—indicating optimal porosity. FTIR analysis confirmed the presence of functional groups such as -OH (3,400 cm^{-1}), C=C (1,500 cm^{-1}), and C=O (1,700 cm^{-1}), which contribute significantly to Pb(II) adsorption. The activated carbon also exhibited a moisture content of 0.69% and an ash content of 35.60%. SEM-EDX analysis showed increased pore size and quantity after activation, while the carbon content also increased to 35.764%, further supporting its enhanced structural and chemical properties. Adsorption experiments revealed that Pb(II) removal was most effective at pH 5, and the data fit well with the Langmuir isotherm model, suggesting monolayer adsorption on a homogeneous surface. The Tire-derived Activated Carbon (TAC) achieved an adsorption efficiency of 96.32% and a capacity of 240.80 mg/g, outperforming Commercial Activated Carbon (CAC), which showed only 60.54% efficiency and 151.366 mg/g capacity. These findings indicate that TAC is a highly effective and promising alternative adsorbent for removing heavy metals from aqueous solutions.

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