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Optimization of Contact Time and Stirring Speed in The Adsorption of Cd(II) Using *Acacia crassicarpa* Bark Powder as Adsorbent

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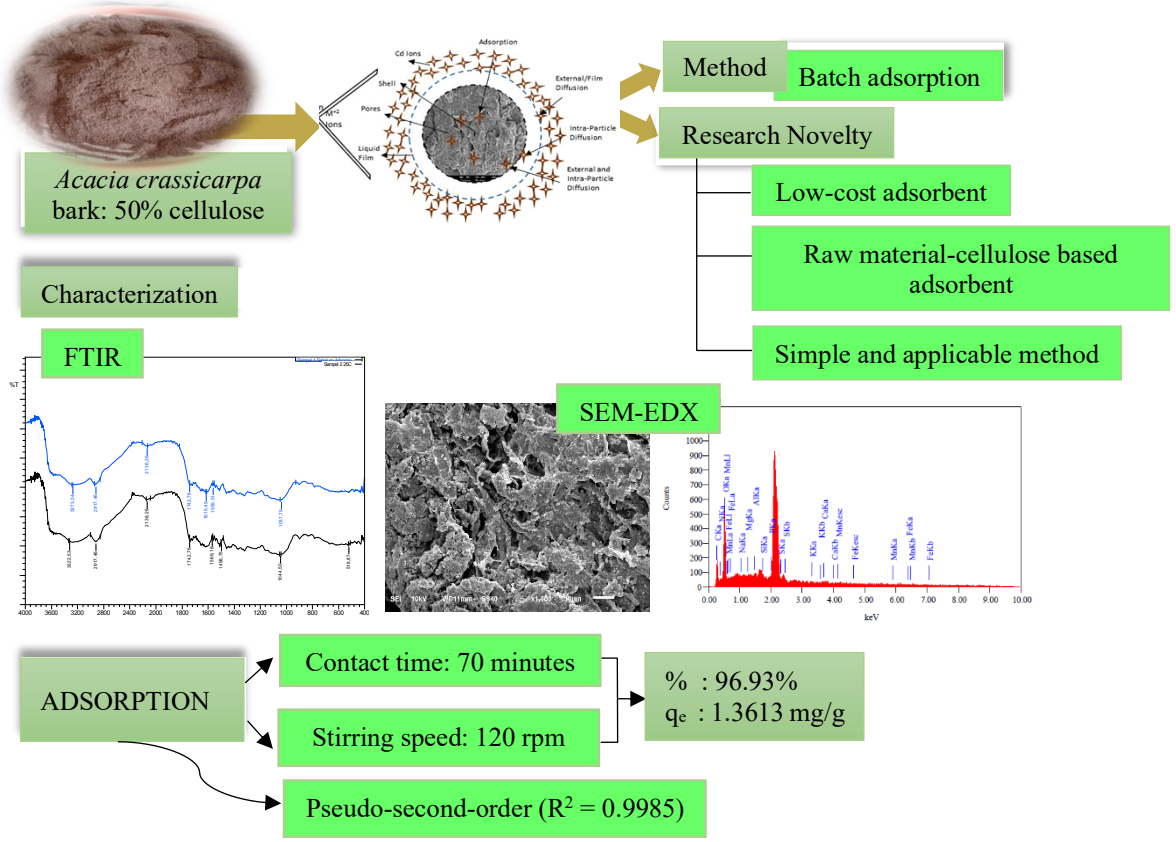
GRAPHICAL ABSTRACT

The aim of this research was to determine the optimum contact time, stirring speed, and adsorption kinetic minutes of *Acacia crassicarpa* bark powder for Cd(II) removal.



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ABSTRACT

Acacia crassicarpa bark powder showed potential as an adsorbent due to its high cellulose content of approximately 50%. This research aimed to determine the optimum contact time, stirring speed, and adsorption kinetic models of *Acacia crassicarpa* bark powder for Cd(II) removal. Adsorbent characterization was conducted using a Surface Area and

Pore Size Analyzer, FTIR, and SEM-EDX, while batch adsorption experiments were conducted to evaluate performance. The adsorbent exhibited a specific surface area of 0.460 m^2/g and pore diameters ranging from 32.707 to 45.426 Å, indicating mesoporous characteristics. FTIR analysis identified functional groups such as O–H, C=O, and O–Cd. SEM-EDX analysis before adsorption revealed a rough surface with open, irregular pores and dominant elements including C, N, and O. After adsorption, the surface appeared smoother, with pores filled by Cd(II), as confirmed by Cd peaks in the EDX spectrum. Optimum conditions were obtained at a contact time of 70 minutes and a stirring speed of 120 rpm, resulting in an adsorption efficiency of 96.93% and a 1.3613 mg/g capacity. The adsorption kinetics followed a pseudo-second-order model (R^2 = 0.9985), indicating that the adsorption mechanism occurred via chemisorption.

1. INTRODUCTION

Acacia crassicarpa was widely cultivated in Industrial Plantation Forests (HTI) in Indonesia, with an average yield of 27 m^3/ha/year [1]. As the third-largest pulp and paper producer in Asia, Indonesia generated large quantities of *Acacia crassicarpa* bark as a by-product [2]. This bark had the potential to be utilized, as wood processing often left bark unused, even though bark accounted for approximately 10-15% of a tree's total volume [3]. *Acacia crassicarpa* bark was rich in lignin and cellulose, with a cellulose content of around 50%, making it a promising adsorbent for reducing heavy metal pollution through adsorption methods [4].

Adsorption was chosen due to its advantages: low cost, simple operation, and the ability to utilize natural biomass-based adsorbents [5]. Several parameters influenced the adsorption process, including contact time and stirring speed. Contact time was a key factor, as longer contact provided more opportunity for adsorbate molecules to interact with the adsorbent, thereby increasing adsorption capacity. However, adsorption capacity eventually reached a saturation point when equilibrium was achieved; therefore, determining the optimum contact time was essential to reach maximum adsorption [6]. The optimization of stirring speed aimed to identify the rate at which adsorption was most efficient [7]. Stirring that was too slow caused slower adsorption, while excessive stirring could damage the adsorbent structure and reduce efficiency. Thus, determining the optimum stirring speed was necessary for effective adsorption.

One commonly targeted heavy metal in adsorption studies was cadmium (Cd). Cadmium contaminated water from natural sources, such as rock weathering, and industrial sources, including metal, chemical, mining, and battery industries [8]. The maximum allowable cadmium concentration in water was 0.1 ppm [9]. Cadmium exposure negatively affected human health, especially the liver and kidneys [10], and could cause Itai-itai disease [11]. The permissible limits for cadmium were 0.003 ppm by the World Health Organization (WHO) and 0.005 ppm by the United States Environmental Protection Agency (EPA) [12].

Several studies have explored the use of bark as an adsorbent. The activated *Acacia crassicarpa* bark carbon was applied to remove Methyl Orange and Methylene Blue, obtaining adsorption capacities of 47.39 mg/g and 60.96 mg/g, respectively [13]. The powdered *Schleichera oleosa* bark was used to remove Pb(II), with an efficiency of 93% [14]. *Eucalyptus* bark powder was used to remove Cd(II), reaching 80.85% efficiency [15]. *Eucalyptus* sawdust was applied to adsorb Cd(II) from solution and achieved an adsorption efficiency of 89.3% [16]. This research reports the adsorption of Cd(II) using *Acacia crassicarpa* bark powder as a cellulose-based adsorbent.

2. EXPERIMENTAL METHODS

2.1. Materials and Apparatus

The apparatus used in this research included an analytical balance (Ohaus PR Series), hotplate (Thermo Scientific Cimarec), magnetic stirrer (IKA C-MAG MS10), oven (Stericell), desiccator (BRANDR desiccator BR65815-1EA), 100 and 200 mesh sieves (ASTM 11 Cat. 60 150 000150), porcelain crucibles, pH meter (Hanna), chopper, grinder, spray bottle, stirring rod, spatula, Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES; APHA 3120B), Scanning

Electron Microscope-Energy Dispersive X-ray Spectroscopy (SEM-EDX; JED-2300 Analysis Station–JEOL), Fourier Transform Infrared Spectroscopy (FTIR; IR Prestige Spectrophotometer Shimadzu), Surface Area and Pore Size Analyzer (Quantachrome Quadrasorb-Evo), and standard laboratory glassware.

The materials used in this research included *Acacia crassicarpa* bark, cadmium(II) sulfate monohydrate ($CdSO_4 \cdot H_2O$), 0.1 M sodium hydroxide (NaOH), 0.1 M hydrochloric acid (HCl), demineralized water (H_2O), distilled water (H_2O), Whatman No. 42 filter paper, aluminum foil, and ziplock plastic bags. A 1000 ppm Cd(II) stock solution was prepared by dissolving $CdSO_4.H_2O$ in demineralized water and diluting to volume. Standard Cd(II) solutions with concentrations ranging from 0.0 to 10.0 ppm were used to construct a calibration curve using ICP-OES at a wavelength of 214.439 nm.

2.2. Preparation of *Acacia crassicarpa* Bark Samples

Approximately 10 kg of *Acacia crassicarpa* bark was collected from an Industrial Plantation Forest (HTI) in Langgam District, Pelalawan Regency, Riau Province. The bark was washed with clean water and rinsed with distilled water. It was then chopped into small pieces, sun-dried for ± 5 days, and oven-dried at 100 ± 5°C for ± 2 days. The dried bark was ground using a grinder into powder form and sieved to obtain particles passing through a 100 mesh but retained on a 200 mesh. The resulting fraction (100 mesh < x < 200 mesh) was dried at 100 ± 5°C for 24 hours and stored in a ziplock bag.

2.3. Characterization of *Acacia crassicarpa* Bark Powder

Surface area, pore volume, and pore diameter were measured using a Surface Area and Pore Size Analyzer. FTIR spectroscopy was employed to identify functional groups before and after adsorption. SEM-EDX was used to observe the surface morphology and determine the elemental composition of the bark powder.

2.4. Adsorption Experiment

3.5 g of *Acacia crassicarpa* bark powder was added to 50 mL of 100 ppm $CdSO_4 \cdot H_2O$ solution in 100 mL beakers to determine the optimum contact time and stirring speed. The pH of the solution was adjusted to 8 using 0.1 M HCl and 0.1 M NaOH. For the contact time optimization, the mixtures were stirred at a constant speed of 120 rpm for 30, 50, 70, 90, and 100 minutes at room temperature [15]. For the stirring speed optimization, the contact time was fixed at 70 minutes while the stirring speeds were varied at 90, 100, 110, 120, and 130 rpm [14]. After each treatment, the mixtures were filtered using Whatman No. 42 filter paper, and the filtrates were analyzed using ICP-OES.

2.5. Calculation of adsorption efficiency and capacity

The adsorption efficiency and capacity were calculated using the following equations:

$$\% \quad = \frac{C_0 - C_e}{C_0} \times 100\% \tag{1}$$

$$q_e \quad = \frac{(C_0 - C_e)}{m} \times V \tag{2}$$

where C_0 and C_e are the initial and final concentrations of Cd(II) in solution (ppm), respectively; V is the volume of the solution in liters (L); and m is the mass of the adsorbent in grams (g).

3. RESULTS AND DISCUSSIONS

3.1. Characterization of *Acacia crassicarpa* Bark Powder

3.1.1 Surface area, pore volume, and pore diameter analysis

The porosity characteristics of *Acacia crassicarpa* bark powder before adsorption were analyzed using the Brunauer-Emmett-Teller (BET) method to determine specific surface area, pore size distribution, total pore volume, and average pore diameter. The Barrett-Joyner-Halenda (BJH) adsorption and desorption methods were used to determine mesopore diameters. In contrast, the Density Functional Theory (DFT) method was employed to identify the presence of micropores and macropores. The results are presented in Table 1.

TABLE I. Surface area, pore volume, and pore diameter analysis results of *Acacia crassicarpa* bark powder before Cd(II) adsorption.

Specific surface area (m^2/g)	Mesopore diameter (Å)	Total pore volume (cc/g)	Average pore diameter (Å)
0.460	32.707 (BJH adsorption method) 31.561 (BJH desorption method) 33.152-45.426 (DFT method)	4.361×10^{-3} for pores < 396.6 Å	3.79007×10^{2}

Based on the results, the adsorbent was categorized as mesoporous, with pore diameters ranging from 32.707 to 45.426 Å. Although the specific surface area was relatively low (0.460 m^2/g), it still achieved high Cd(II) removal adsorption efficiency (96.93%) and capacity (1.3613 mg/g) at a contact time of 70 minutes and stirring speed of 120 rpm. This result is attributed to the dominance of mesopores, whose sizes matched the ionic radius of Cd(II) (0.97 Å), allowing effective diffusion and interaction between adsorbent and adsorbate.

3.1.2 Functional group analysis

FTIR analysis of *Acacia crassicarpa* bark powder showed significant spectral changes before and after the adsorption of Cd(II), indicating the involvement of functional groups in the adsorption process.

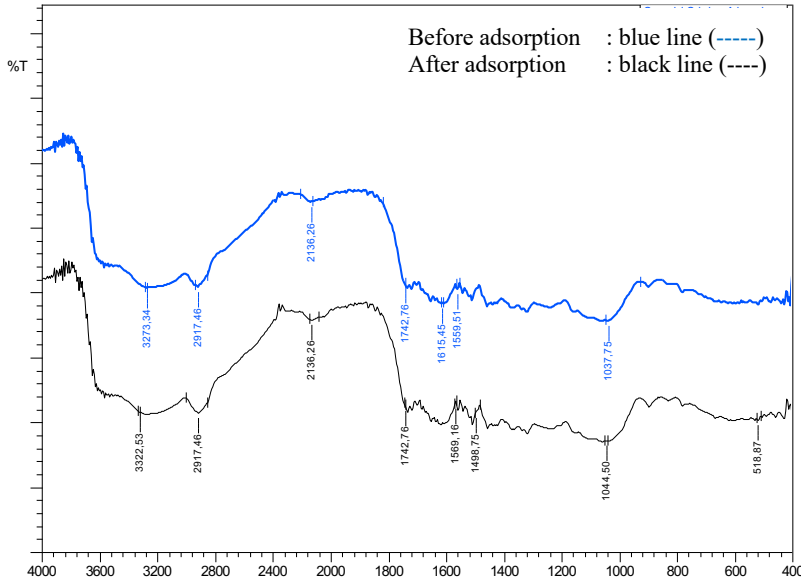

Figure 1. FTIR spectra of *Acacia crassicarpa* bark powder before and after Cd(II) adsorption.

The FTIR spectrum of *Acacia crassicarpa* bark powder before Cd(II) adsorption (Figure 1) showed a peak at 3273.34 cm^{-1}, indicating O–H vibrations from hydroxyl groups in cellulose, hemicellulose, and lignin. These groups are abundant and capable of binding metal cations. Peaks at 2917.46 cm^{-1} and 1037.75 cm^{-1} correspond to aliphatic C–H and ether C–O groups, respectively, integral to lignocellulosic structure [17]. The 2136.26 cm^{-1} peak indicates C–C bonds, while the 1742.76 cm^{-1} peak represents carbonyl (C=O) groups in hemicellulose [18]. Aromatic C=C groups, characteristic of lignin and hemicellulose, were observed at 1615.45–1559.51 cm^{-1}. After Cd(II) adsorption, several shifts in FTIR peaks were observed, indicating interaction between functional groups and Cd(II). A new peak appeared at 518.87 cm^{-1}, corresponding to O–Cd bonding [19], confirming the formation of chemical bonds (chemisorption) between Cd(II) and the functional groups, particularly O–H and C–O.

3.1.3 Surface morphology and elemental composition analysis of *Acacia crassicarpa* bark powder

The surface morphology of *Acacia crassicarpa* bark powder before and after adsorption was analyzed using a Scanning Electron Microscope (SEM) at magnifications of 10,000×, as shown in Figure 2.

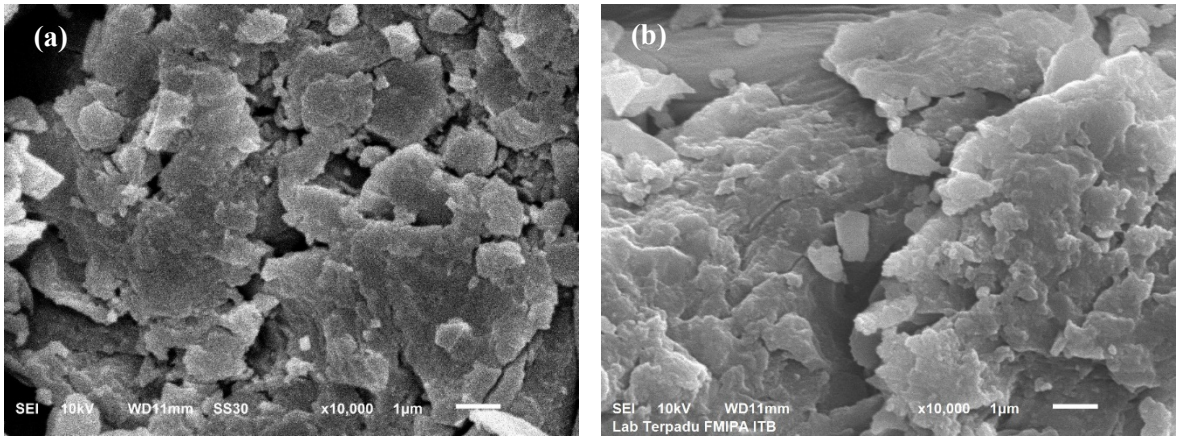

Figure 2. SEM images of Acacia crassicarpa bark powder at 10,000× magnifications: a) before and b) after Cd(II) adsorption.

SEM analysis revealed that, before adsorption, the surface of the *Acacia crassicarpa* bark powder appeared rough with open and irregularly distributed pores, indicating the presence of active functional groups available for Cd(II) binding. After adsorption, the surface appeared smoother and the pores were visibly filled, indicating that Cd(II) was successfully adsorbed onto the adsorbent surface. The elemental composition of the powder was identified using Energy Dispersive X-ray Spectroscopy (EDX), as presented in Figure 3 and Table 2.

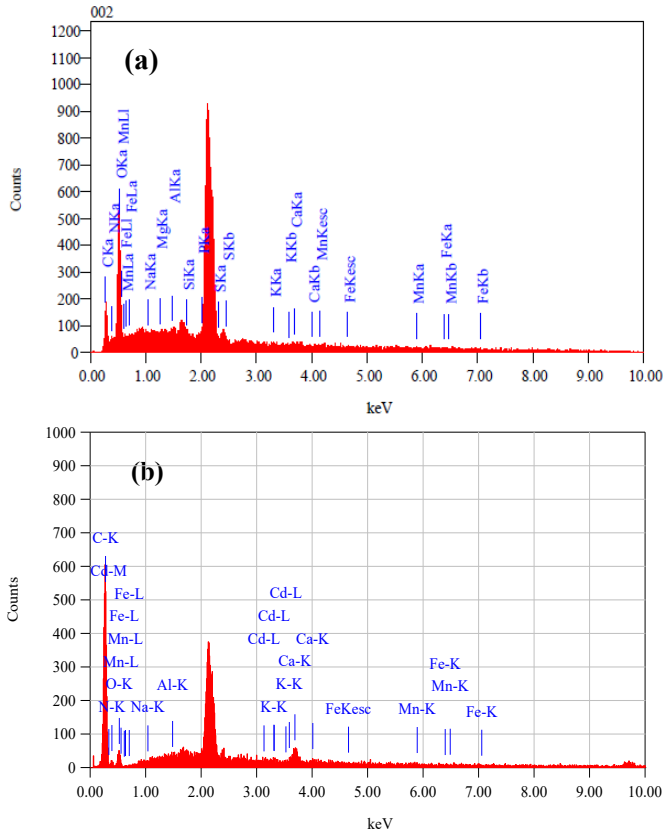

Figure 3. EDX spectra of Acacia crassicarpa bark powder: a) before and b) after Cd(II) adsorption.

TABLE II. Elemental composition of *Acacia crassicarpa* bark powder before and after Cd(II) adsorption.

Sample	Elemental composition (%wt)								
	C	N	O	Al	K	Ca	Mn	Fe	Cd
Before adsorption	16.88	7.49	67.54	1.88	2.34	1.47	0.65	1.75	-
After adsorption	57.67	24.69	12.88	0.24	0.26	3.15	0.43	-	0.67

The EDX analysis of *Acacia crassicarpa* bark powder before adsorption revealed major elements such as carbon (16.88%), nitrogen (7.49%), and oxygen (67.54%), along with minor elements like Al, K, Ca, Mn, and Fe. After Cd(II) adsorption, the appearance of Cd at 0.67% confirmed successful metal uptake. A significant increase in carbon (to 57.67%) and nitrogen (to 24.69%), along with a marked decrease in oxygen (to 12.88%), suggests active interaction between Cd(II) and O- or N-containing functional groups on the adsorbent surface. The reduction of minor metals may also indicate competitive binding or surface restructuring. These findings are supported by FTIR results, which showed peak shifts in O–H, C=C, and C–O regions, along with the appearance of a new peak at 518.87 cm^{-1} attributed to O–Cd bonding [19], confirming chemisorption as the dominant adsorption mechanism.

3.2. Adsorption Results of Cd(II) Using *Acacia crassicarpa* Bark Powder

3.2.1. Optimization of contact time

Adsorption was carried out at varying contact times of 30, 50, 70, 90, and 100 minutes, while other parameters were kept constant (adsorbent dose of 3.5 g, pH 8, 120 rpm, 100 ppm, and room temperature). As shown in Figure 4, the optimum contact time was 70 minutes, with an adsorption efficiency of 96.94% and a capacity of 1.3616 mg/g.

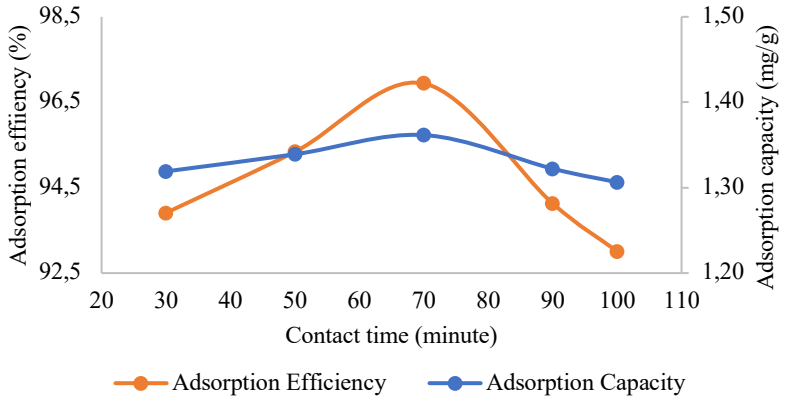

Figure 4. Effect of contact time on Cd(II) adsorption efficiency and capacity.

The efficiency and capacity increased over time due to the availability of active sites on the adsorbent surface, reaching equilibrium at 70 minutes. At this point, the adsorption and desorption rates became balanced, resulting in no significant further changes [20]. The initially high adsorption rate indicated strong interactions between Cd(II) ions and functional groups on the adsorbent surface. After 70 minutes, a slight decrease in efficiency and capacity was observed at 90 and 100 minutes, likely due to site saturation and partial desorption under dynamic conditions, rather than weak interactions [21]. This aligns with the chemisorption mechanism, where strong chemical bonding occurs, but desorption may still happen once equilibrium is surpassed. Therefore, identifying the optimum contact time is crucial to maintain maximum adsorption performance.

The adsorption performance of *Acacia crassicarpa* bark powder shows superior results compared to other bark-derived adsorbents. In a research by [15], *Eucalyptus* bark powder achieved an adsorption efficiency of 80.85% for Cd(II) with an optimum contact time of 70 minutes. In contrast, this research obtained a higher adsorption efficiency of 96.93% under similar conditions, despite using untreated natural bark powder without any chemical or thermal activation. This demonstrates that *Acacia crassicarpa* bark powder is not only highly effective in removing Cd(II) from aqueous solutions but also offers advantages in terms of cost-effectiveness and environmental

sustainability due to the absence of energy-intensive preparation steps. These findings highlight the potential of this material as a competitive and eco-friendly alternative to other biosorbents.

3.2.2. Optimization of stirring speed

Adsorption was carried out at varying stirring speeds of 90, 100, 110, 120, and 130 rpm, while other parameters were kept constant (adsorbent dose of 3.5 g, pH 8, contact time of 70 minutes, 100 ppm, and room temperature). The data in Figure 5 show that the optimum stirring speed for Cd(II) adsorption using *Acacia crassicarpa* bark powder was 120 rpm, achieving an adsorption efficiency of 96.93% and a 1.3613 mg/g capacity.

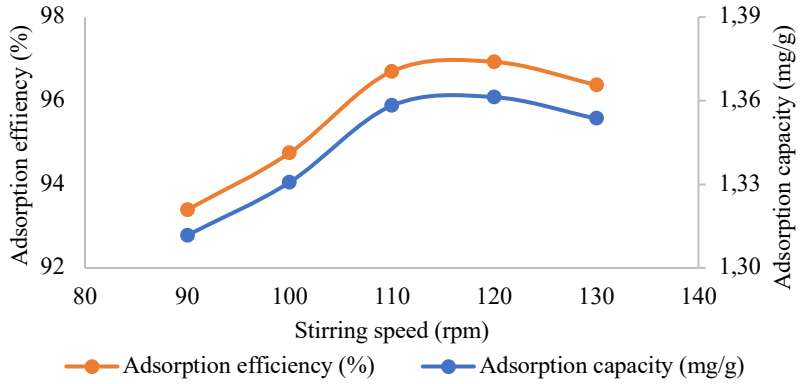

Figure 5. Effect of stirring speed on Cd(II) adsorption efficiency and capacity.

The increased stirring speed enhanced adsorption efficiency and capacity due to higher kinetic energy, which increased the collision frequency between adsorbent and adsorbate and reduced boundary layer resistance. It also accelerated mass transfer from the solution to the adsorbent surface [22]. At low stirring speeds, collisions are less effective, reducing adsorption performance. Conversely, excessively high stirring speeds (e.g., 130 rpm) may damage the adsorbent structure and cause desorption of Cd(II) due to the weakened interaction between the adsorbate and active sites [23]. Thus, 120 rpm is the optimal stirring speed to maximize adsorption performance before a decline occurs due to excessive agitation.

This research demonstrates that *Acacia crassicarpa* bark powder exhibits highly competitive adsorption performance compared to other bark-based adsorbents. [14] reported a Pb(II) removal efficiency of 93% using *Schleichera oleosa* bark powder at an optimum stirring speed of 120 rpm. In this research, *Acacia crassicarpa* bark powder achieved an even higher Cd(II) removal efficiency of 96.93% under the same stirring speed, without requiring any chemical or thermal activation. This highlights the advantages of *Acacia crassicarpa* bark as a natural adsorbent that is effective, simpler, more economical, and environmentally friendly. The successful adsorption is supported by its mesoporous structure and active functional groups such as O–H and C–O, which facilitate strong interactions with Cd(II) ions during adsorption.

3.3. Adsorption Kinetic Models

The adsorption kinetic Cd(II) models onto *Acacia crassicarpa* bark powder were analyzed using the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models based on contact time data (Table 3 and Figure 6).

Among these, the pseudo-second-order model provided the best fit, with a rate constant of 0.6615 g/mgmin and an R^2 value of 0.9985. This result suggests that the adsorption process followed a chemisorption mechanism, involving the formation of chemical bonds between the metal ions and the active sites on the adsorbent surface [24], which are typically strong and not easily reversible [25].

TABLE III. Kinetic models parameters for the adsorption of Cd(II) using *Acacia crassicarpa* bark powder.

Kinetic models	Parameter	Value
Pseudo-first-order	q_e experimental	1.3616 mg/g
	q_e calculated	0.0440 mg/g
	k_1	-0.0032 min^{-1}
	R^2	0.0203
Pseudo-second-order	**q_e experimental**	**1.3616 mg/g**
	q_e calculated	**1.3050 mg/g**
	k_2	**0.6615 g/mgmin**
	R^2	**0,9985**
Intraparticle diffusion	K	-0.0021 mg/g/$t^{1/2}$
	C	1.3464 mg/g
	R^2	0.0319

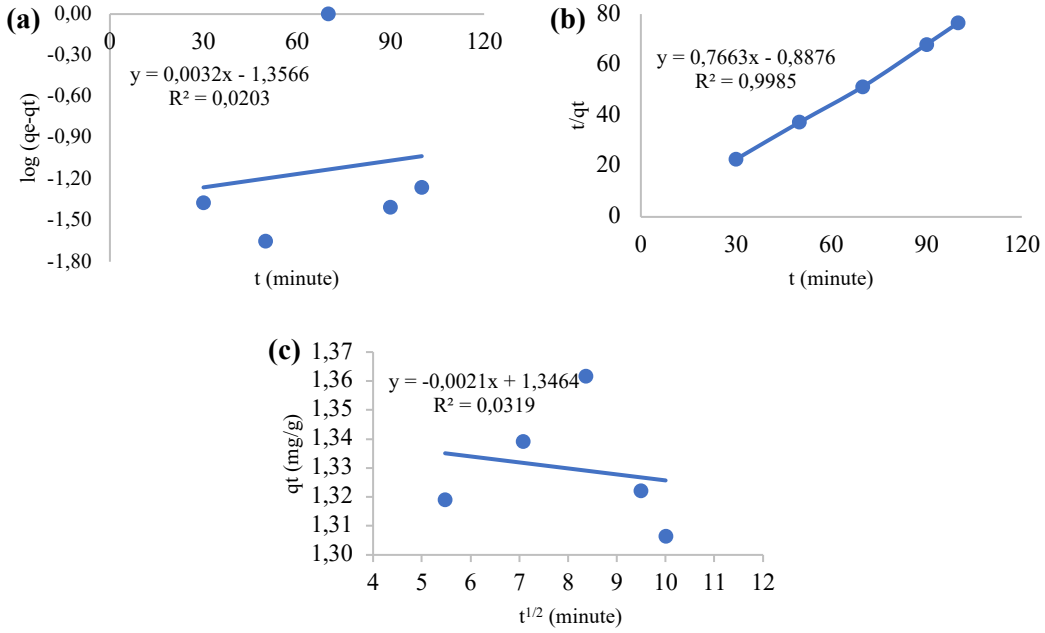

Figure 6. Kinetic models of Cd(II) adsorption on *Acacia crassicarpa* bark powder: a) pseudo-first-order, b) pseudo-second-order, c) intraparticle diffusion.

4. CONCLUSIONS

Based on the results of this research, *Acacia crassicarpa* bark powder was effective as an adsorbent for removing Cd(II). Surface area analysis showed that the adsorbent had a specific surface area of 0.460 m^2/g and a pore diameter ranging from 32.707 to 45.426 Å, classifying it as mesoporous. FTIR analysis confirmed the presence of functional groups like O–H, C=O, and O–Cd, which play important roles in the adsorption process. Furthermore, SEM-EDX characterization revealed noticeable morphological changes before and after adsorption and confirmed the presence of Cd elements on the adsorbent surface after the adsorption process. The adsorption of Cd(II) using *Acacia crassicarpa* bark powder achieved optimum performance at a contact time of 70 minutes and a stirring speed of 120 rpm, resulting in a maximum adsorption efficiency of 96.93% and an adsorption capacity of 1.3613 mg/g. The kinetic data were best described by the pseudo-second-order model, with a high correlation coefficient (R^2 = 0.9985) and a rate constant (k_2) of 0.6615 g/mgmin,

indicating that the adsorption mechanism was primarily governed by chemisorption involving strong interactions between Cd(II) and the active binding sites on the adsorbent.

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