



Synthesis of Activated Carbon/Magnetite Nanocomposite Modified with Povidone for Adsorption Applications of Heavy Metal Ions

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Abstract

This study reports on activated carbon/magnetite nanocomposite modified with povidone for the adsorption applications of heavy metal (lead) ions. The synthesis routes used in this study were coprecipitation, activated carbon activation, and ex-situ methods. The data analysis results showed that the activated carbon/magnetite nanocomposite modified with povidone had a crystallite size of 9.45 nm with a single-phase inverse cubic spinel structure. The activated carbon/magnetite nanocomposite modified with povidone showed successful synthesis with the appearance of octahedral and tetrahedral Fe–O lattice vibrations (440 and 550 cm⁻¹), O–H (3200–3395 cm⁻¹), C=O (1655 cm⁻¹), and C–N (1182 cm⁻¹) functional groups. Furthermore, the activated carbon/magnetite nanocomposite modified with povidone exhibited a chunky and spherical morphology, with a particle size of 39.91 nm. The nanocomposite had a large specific surface area and pore volume, namely 129.237 m²/g and 0.198 cm³/g, respectively, thus providing many active sites for adsorption. Interestingly, the nanocomposite was superparamagnetic, facilitating its separation from the solution after adsorption. The adsorption efficiency of this nanocomposite was 71.01% with an adsorption capacity of 29.6415 mg/g in 180 minutes at a volume of heavy metal solution of 25 mL with a theoretical concentration of 100 ppm, adsorbent dose of 0.05 g, pH 5, and room temperature, and followed with pseudo-second order adsorption kinetics model. The resulting removal efficiency is better than magnetite/povidone, which has a removal efficiency of 70%.

1. Introduction

Wastewater generated from various industrial activities, including battery manufacturing, automobile production, electroplating, electronics, paint, steel, ceramics, dye, and fertilizer industries, is a major source of lead contamination in aquatic environments [1, 2]. Lead is one of the most hazardous heavy metals due to its non-biodegradable nature, persistence in the environment, and tendency to bioaccumulate throughout the food chain, resulting in severe toxic effects on living organisms [3, 4]. Human exposure to lead has been associated with numerous adverse health effects, including abdominal pain, neurological impairment, kidney dysfunction, cardiovascular disease, respiratory disorders, and, in severe cases, death [5, 6]. According to

the World Health Organization (WHO), lead exposure was responsible for more than 1.5 million deaths and approximately 33 million disability-adjusted life years (DALYs) worldwide in 2021 [6]. Furthermore, lead concentrations in many Indonesian water bodies have been reported to exceed the WHO permissible limit for drinking water of 0.01 mg/L [7]. Therefore, the development of effective technologies for removing lead from contaminated wastewater is of critical importance.

Various technologies have been developed for the removal of lead from industrial wastewater, including ion exchange, liquid membrane separation, membrane filtration, electrocoagulation, chemical precipitation, and adsorption [8, 9]. However, many of these techniques suffer from inherent limitations, such as secondary waste

generation, membrane fouling or operational instability, high chemical and energy consumption, and the strong dependence of ion-exchange performance on resin properties and solution chemistry [8, 10]. Among these technologies, adsorption is considered one of the most promising approaches because of its high removal efficiency, low operating cost, operational simplicity, and the potential for adsorbent regeneration and reuse [11, 12].

In adsorption applications, magnetite has received considerable attention owing to its excellent magnetic properties, low toxicity, and environmental compatibility [13]. Its superparamagnetic behavior enables rapid separation from aqueous solutions using an external magnetic field, facilitating adsorbent recovery while minimizing material loss and the generation of secondary pollution [14, 15]. Despite its advantages, magnetite still faces challenges: it is easily oxidized when exposed to air or acids, thereby reducing its magnetic properties. Furthermore, magnetite easily agglomerates, reducing its dispersibility [16, 17].

To overcome the oxidation and agglomeration of magnetite, surface modification with povidone (polyvinylpyrrolidone, PVP) has been widely employed. Povidone forms a protective coating on the magnetite surface through coordination between its carbonyl groups and surface Fe ions, providing steric stabilization that suppresses particle aggregation and limits crystal growth. Consequently, the modified particles exhibit improved oxidation resistance, enhanced dispersion stability, and more uniform crystal sizes [18].

Previous studies have demonstrated that povidone modification reduced the average particle size of magnetite from 857 nm to 225 nm and produced smaller, more homogeneous crystals [18]. In addition, magnetite/povidone adsorbents have been reported to achieve approximately 70% removal efficiency for Cu(II) ions [19]. Although these findings demonstrate the potential of povidone-modified magnetite, its adsorption performance remains limited, possibly due to its relatively low specific surface area. To address this limitation, incorporating activated carbon is a promising strategy owing to its high porosity, large specific surface area, and abundant surface functional groups that provide additional adsorption sites for heavy metal ions [20, 21]. Therefore, this study developed a povidone-modified activated carbon/magnetite nanocomposite as an adsorbent for removing Pb(II) ions from aqueous solutions.

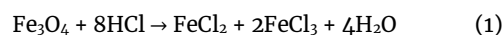
2. Experimental

2.1. Materials

The materials used in this study included dry iron sand, dried old coconut shells, Povidone K-30, hydrochloric acid (HCl, 2 M), ammonium hydroxide (NH₄OH, 6.5 M), nitric acid (HNO₃, 2 M), distilled water, deionized water, methanol, 70% ethanol, and lead nitrate (Pb(NO₃)₂) as the Pb(II) source.

2.2. Experimental

Cleaned and ground coconut shell charcoal was first carbonized at 600°C in a non-inert atmosphere (air) using a muffle furnace to remove volatile components. The resulting charcoal was then chemically activated by soaking in 2 M HNO₃ for 2 h, followed by washing with 70% ethanol until neutral and drying in an oven to obtain activated carbon powder [22]. Meanwhile, 20 g of separated iron sand was dissolved in 58 mL of 2 M HCl to produce FeCl₂ and FeCl₃ in a theoretical Fe²⁺/Fe³⁺ molar ratio of 1:2 based on the stoichiometric dissolution of magnetite, as described by Equation (1).



A 1.5% (w/v) Povidone K-30 solution was added to the FeCl₂/FeCl₃ solution, after which NH₄OH (6.5 M) was added dropwise until precipitation occurred. No additional oxidation or reduction treatment was applied during the dissolution process. The resulting precipitate was washed repeatedly with methanol and distilled water until a neutral pH was achieved, and then dried in an oven to obtain povidone-modified magnetite powder [23].

The activated carbon and povidone-modified magnetite were separately dispersed in deionized water, mixed, and sonicated until a homogeneous suspension was obtained. The suspension was subsequently dried in an oven to produce the activated carbon/magnetite nanocomposite modified with povidone, with a final product yield of approximately 90% [24].

Batch adsorption experiments were performed by mixing 0.05 g of adsorbent with 25 mL of a 100 ppm Pb(II) solution under continuous stirring at 720 rpm. After adsorption, the adsorbent was separated magnetically, sieved, and the residual Pb(II) concentration in the filtrate was analyzed. The adsorption capacity (q_e) and Pb(II) removal efficiency were calculated using Equations (2) and (3), respectively [25].

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

$$\% \text{Removal} = \frac{C_0 - C_e}{C_0} \times 100\% \quad (3)$$

2.3. Characterization

The synthesized materials were characterized using several analytical techniques. X-ray diffraction (XRD; X'Pert PRO) was employed to determine the crystal structure and crystallite size. The crystallite size was estimated by Rietveld refinement using Rietica software. Surface morphology and elemental composition were analyzed using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX; FEI Inspect S50). Functional groups were identified using Fourier transform infrared spectroscopy (FTIR; Shimadzu IRPrestige-21). Magnetic properties were evaluated using a vibrating sample magnetometer (VSM; Quantum Design PPMS® VersaLab™ Cryogen-Free 3 Tesla). The specific surface area, pore volume, and average pore diameter were determined using the Brunauer–Emmett–Teller (BET) method with a Micromeritics TriStar II Plus 3020 analyzer (P/N 302/00000/22). The adsorption performance toward

Pb(II) ions was evaluated by measuring the residual lead concentration using atomic absorption spectroscopy (AAS; Thermo Scientific iCE 3000).

3. Results and Discussion

The XRD pattern of the activated carbon/magnetite nanocomposite modified with povidone exhibits the characteristic inverse cubic spinel structure of magnetite, consistent with AMCSD model No. 0007423, as shown in Figure 1. The most intense diffraction peak appears at $2\theta = 35.59^\circ$, corresponding to the (311) crystal plane. Additional diffraction peaks are observed at $2\theta = 30.20^\circ$, 43.33° , 53.76° , 57.30° , and 62.90° , which are indexed to the (220), (400), (422), (511), and (440) crystal planes, respectively. These diffraction peaks agree well with those reported for magnetite in previous studies [26]. The addition of povidone does not change the magnetite diffraction pattern [27]. The absence of activated carbon peaks is due to its amorphous nature; these results are consistent with previously reported studies [28, 29].

The results of quantitative analysis indicate that the size of magnetite crystallites is 9.45 nm, with a lattice parameter of 8.350 Å. This crystallite size is considerably smaller than the previously reported value of 17.69 nm for unmodified magnetite [30], suggesting that povidone effectively suppresses crystal growth during nanoparticle formation. The polymer likely acts as a capping or stabilizing agent, limiting crystal growth while promoting nucleation, thereby producing smaller crystallites [31]. The measured lattice parameter is slightly lower than the standard value for bulk magnetite (approximately 8.39–8.40 Å). This slight contraction may be attributed to nanoscale size effects and an increased density of lattice defects, both of which can influence the crystal lattice parameters [32].

The FTIR results are shown in Figure 2 and Table 1. The strong peaks at 3200–3395 cm^{-1} can be attributed to the stretching vibrations of the hydroxyl (OH) groups in alcohols and phenols, which are part of organic bonds. These results are consistent with previous research reports, which assign functional groups at wavenumbers between 3200–3645 cm^{-1} to O–H bond vibrations [33]. At wavenumbers of 440 cm^{-1} and 550 cm^{-1} , Fe–O lattice vibrations associated with tetrahedral and octahedral sites in the spinel structure of magnetite were identified, respectively [34]. Meanwhile, the aromatic C–H ring group and the out-of-plane bend aromatic C–H in activated carbon are detected at wavenumbers of 814 cm^{-1} and 881 cm^{-1} , respectively [35, 36].

The stretching of single C–O bonds in activated carbon is formed at wavenumbers of 918–1146 cm^{-1} [36, 37]. In addition, aromatic activated carbon compounds contain a C=C double bond at a wavenumber of 1607 cm^{-1} [35]. The C–N stretching band of povidone is located at a wave number of 1182 cm^{-1} [38]. Meanwhile, other identified povidone functional groups are –C=O primary amide stretching and asymmetric CH_2 stretching at a wavenumber of 1655 cm^{-1} , respectively. Furthermore, there are vibrations at 2839 and 2878 cm^{-1} which are associated with the C–H stretching mode in povidone [39].

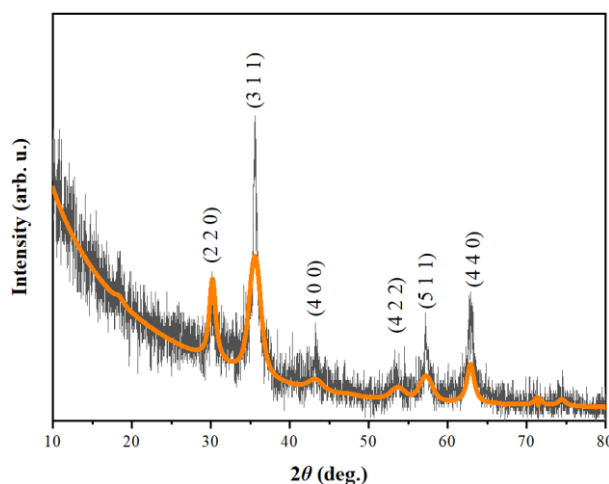


Figure 1. XRD pattern of activated carbon/magnetite nanocomposite modified with povidone

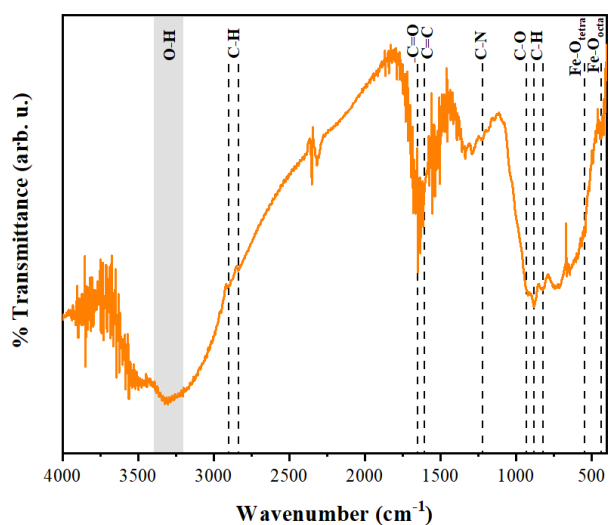


Figure 2. FTIR spectrum of activated carbon/magnetite nanocomposite modified with povidone

Table 1. FTIR results of activated carbon/magnetite nanocomposite modified with povidone

Peak position (cm^{-1})	Assignment	Component
440	Fe–O octahedral	Fe_3O_4
550	Fe–O tetrahedral	Fe_3O_4
814	Aromatic ring C–H	Activated carbon
881	Aromatic C–H out-of-plane bend	Activated carbon
918–1146	stretching of single C–O bonds	Activated carbon
1182	C–N stretching	PVP
1607	C=C double bond	Activated carbon
1655	–C=O stretching	PVP
2839	C–H stretching	PVP
2878	C–H stretching	PVP
3200–3395	O–H bond	Activated carbon

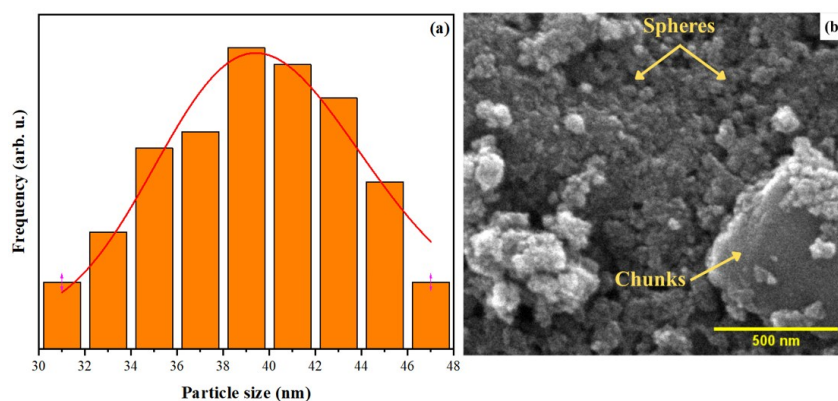


Figure 3. (a) Particle size and (b) morphology of activated carbon/magnetite modified with povidone

Figure 3(a) shows the surface morphology of activated carbon/magnetite nanocomposite modified with povidone from SEM image results, where this nanocomposite has a morphology in the form of chunks (asymmetric or irregular) of activated carbon and spheres of magnetite modified with povidone. The analysis result in Figure 3(b) shows that the size of spherical magnetite particles is 39.91 nm. In previous research, the size of magnetite particles was 63.19 nm [30]. This result indicates that povidone successfully forms a protective layer on magnetite, thereby reducing agglomeration and particle size [18].

There is a discrepancy between the sizes obtained from the XRD and SEM results. This difference is caused by the size obtained from XRD (9.45 nm) representing the size of a single crystallite from a coherent diffraction domain, while the particle size measured from SEM (39.91 nm) corresponds to the physical dimensions of the particles, which may consist of several crystallites and agglomerated structures [40]. The EDX result shows carbon (C) as the most abundant element at 52.36%. This is due to the high composition of activated carbon. Meanwhile, the oxygen (O) composition is 28.14%, and iron (Fe) is 18.14%. Apart from that, there are impurities in the form of silicon (Si) and titanium (Ti), which most likely originate from the natural precursor material, magnetite, namely iron sand.

The results of the VSM characterization are in the form of a hysteresis loop curve of the activated carbon/magnetite nanocomposite modified with povidone, as shown in Figure 4. The analysis results show that the nanocomposite has a saturation magnetization (M_s) value of 14.64 emu/g. Meanwhile, the remanent magnetization (M_r), coercivity field (H_c), and susceptibility (χ) values are 0.01 emu/g, 23.32 Oe, and 6.87×10^{-5} , respectively. Based on the results of this analysis, it can be seen that the nanocomposite exhibits near-superparamagnetic/soft magnetic properties with negligible remanence as indicated by an S-shaped curve, a high M_s value with a small magnetic field, a M_r value close to 0 emu/g, and a low H_c [41].

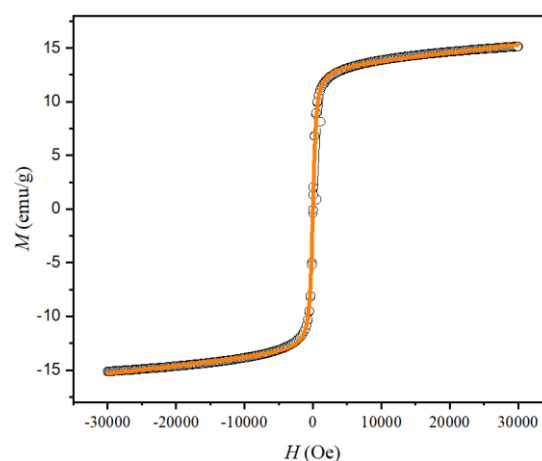


Figure 4. Hysteresis curve of activated carbon/magnetite nanocomposite modified with povidone

The pore structure of activated carbon/magnetite nanocomposites modified with povidone was characterized using BET with N_2 adsorption. It can be seen from Figure 5(a) that the N_2 adsorption-desorption isotherm of activated carbon/magnetite modified with povidone indicates that the material is mesoporous. Mesoporous materials are characterized by having a typical type IV shape curve, and in the range of $P/P_0 = 0.2-0.9$, a clear H3 hysteresis loop is formed. This H3 hysteresis loop is generally caused by the accumulation of sheet-shaped particles that form slit pores [42]. Referring to the pore size classification according to IUPAC, Figure 5(a) shows that activated carbon/magnetite modified with povidone is mostly mesoporous (2–50 nm) [43]. Furthermore, the measured volume of the nanocomposite is 0.19 cm³/g. Meanwhile, the nanocomposite has a specific surface area of 129.24 m²/g as measured by BET and a pore diameter of 5.78 nm, as shown in Figure 5(b). This nanocomposite has a high specific surface area, large pore volume, and small pore diameter. The attachment of povidone-modified magnetite to the surface of activated carbon causes blockage of the macropores, which leads to the loss of macropores in the composite material [44].

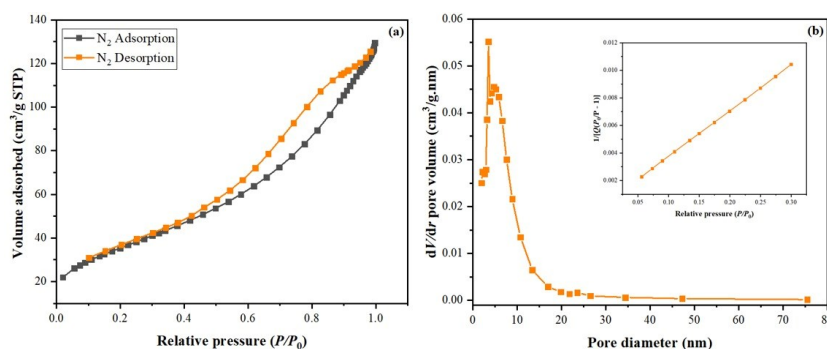


Figure 5. (a) N_2 adsorption–desorption isotherm plot and (b) surface area and pore size distribution of activated carbon/magnetite nanocomposite modified with povidone

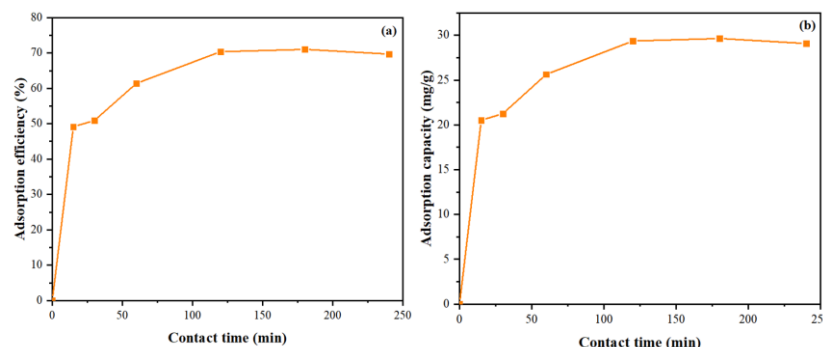


Figure 6. Effect of contact time on (a) removal efficiency and (b) adsorption capacity of the activated carbon/magnetite nanocomposite modified with povidone

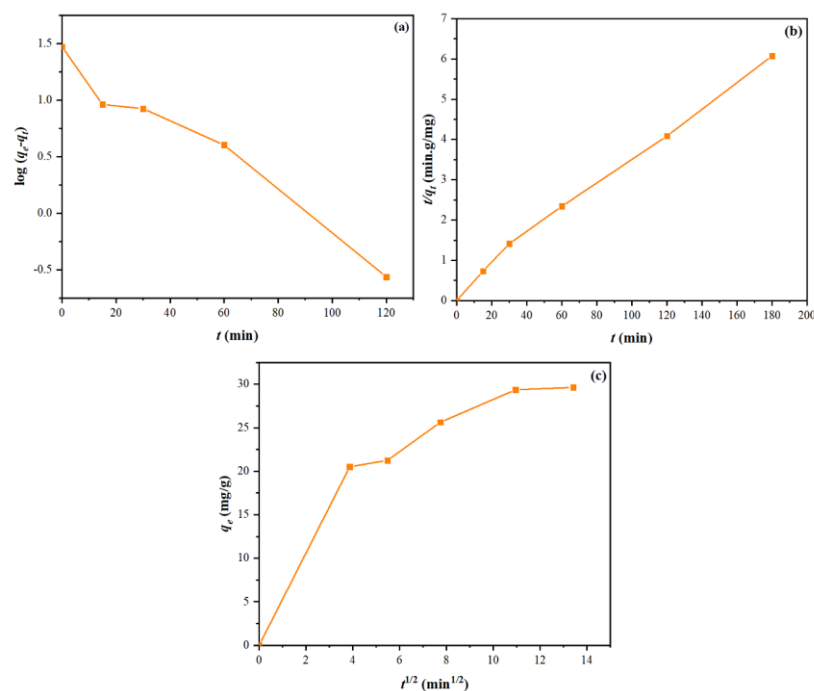


Figure 7. Graph of adsorption kinetics modeling of activated carbon/magnetite nanocomposite modified with povidone (a) Pseudo-first-order, (b) Pseudo-second-order, and (c) Intra-particle diffusion

Figures 6(a) and 6(b) illustrate the removal efficiency and adsorption capacity of the activated carbon/magnetite nanocomposite modified with povidone at different contact times. The adsorption experiments were performed at pH 5, using 25 mL of a 100 ppm heavy-metal solution, an adsorbent dosage of 0.05 g, and at room temperature. The adsorption equilibrium was reached after 180 minutes,

corresponding to a removal efficiency of 71.01% and an adsorption capacity of 29.64 mg/g.

The adsorption capacity increased rapidly during the first 15 minutes and then gradually approached equilibrium at 30, 60, 120, and 180 minutes. The rapid initial adsorption is attributed to the abundance of available active sites on the adsorbent surface, allowing efficient adsorption through surface interactions,

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