Proceeding of International Joint Conference on UNESA
Homepage: https://proceeding.unesa.ac.id/index.php/pijcu
e-ISSN: 3032-3762

PIJCU, Vol. 3, No. 1, December 2025
Page 1-11

Adsorption of Cr(VI) using Natural Magnetite (Fe_3O_4)

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Keywords:
Magnetite,
Natural,
Adsorption,
Heavy Metal Cr,
Water Remediation

ABSTRACT

Heavy metal contamination, particularly hexavalent chromium ions [Cr(VI)] in aquatic environments, has become a serious issue due to its toxic and carcinogenic nature. This study aims to synthesize a composite adsorbent material based on magnetite (Fe_3O_4) modified with polypyrrole (PPy) and evaluate its performance in the adsorption of Cr(VI) ions from aqueous solutions. The PPy–magnetite composite was synthesized via in-situ polymerization and characterized using FTIR, FESEM-EDX, and XRD to identify its morphology, functional groups, and crystallinity. Adsorption experiments were conducted by varying pH (1, 3, and 4), contact time (45, 60, and 90 minutes), initial Cr(VI) concentration (20, 40, and 60 ppm), and adsorbent dose (40, 60, and 100 mg). Characterization results indicated interactions between –NH and –N= groups of PPy and the Fe_3O_4 surface, as well as the formation of a porous structure. Optimal adsorption conditions were achieved at pH 4, 60 minutes of contact time, and an initial Cr(VI) concentration of 40 ppm. The Langmuir isotherm model showed the best fit (R^2 = 0.9979), with a maximum adsorption capacity (q_m) of 54.645 mg·g^{-1} and a removal efficiency of 99.92%. Adsorption kinetics followed the pseudo-second-order model (Ho–McKay) with an R^2 of 0.9827 and a rate constant (k_2) of 0.007 g·mg^{-1}·min^{-1}, indicating a chemisorption mechanism. AAS analysis confirmed the high effectiveness of the PPy–magnetite adsorbent in removing Cr(VI) ions from solution.

INTRODUCTION

Water pollution caused by rapid industrial development and urbanization has become a serious global issue. Among various contaminants, heavy metals are of particular concern due to their high toxicity, non-biodegradability, and tendency to bioaccumulate in living organisms, ultimately leading to long-term ecological imbalance and severe health impacts. Hexavalent chromium [Cr(VI)] is considered one of the most hazardous heavy metals, frequently released from industrial processes such as electroplating, leather tanning, pigment formulation, and stainless-steel production. Its high solubility and strong oxidative characteristics enable Cr(VI) to remain mobile in aquatic systems and penetrate biological membranes, thereby exerting mutagenic and carcinogenic effects on humans and other organisms (Abewaa et al., 2024; Valentín-Reyes et al., 2019).

Conventional treatment technologies—including chemical precipitation, ion exchange, membrane filtration, and electrochemical methods—present several operational drawbacks, such as high treatment cost, secondary waste generation, and complex maintenance procedures. In contrast, adsorption has emerged as a simple, cost-effective, and environmentally friendly approach capable of removing Cr(VI) even at low concentrations. The overall adsorption efficiency is highly influenced by physicochemical properties of the adsorbent, including surface area, porosity, functional groups, and redox reactivity (Juturu et al., 2024). Therefore, the development of efficient, regenerable,

and sustainable adsorbent materials remains a critical challenge in advanced wastewater purification.

Natural magnetite (Fe_3O_4) has received considerable attention due to its superparamagnetic behavior, relatively large surface area, and intrinsic redox properties that facilitate both adsorption and partial reduction of Cr(VI) to the less toxic Cr(III). However, magnetite nanoparticles tend to aggregate and undergo oxidation, which decreases their surface activity and overall stability during treatment processes. To address these limitations, surface modification and composite formation using conductive polymers has been widely explored. Among these materials, polypyrrole (PPy) is notable for its excellent chemical stability, porous structure, redox-active framework, and electrostatic affinity toward negatively charged Cr(VI) oxyanions, such as CrO_4^{2-} and $Cr_2O_7^{2-}$ (Setshedi et al., 2024; Nogueira et al., 2024).

The Fe_3O_4@PPy composite integrates the magnetic separability and redox functionality of magnetite with the ion-exchange and electron-transfer capabilities of PPy, resulting in improved adsorption kinetics and enabling effortless magnetic recovery. Previous studies have reported that Fe_3O_4–PPy nanocomposites exhibit removal efficiencies exceeding 95% within short adsorption periods and remain effective after repeated regeneration cycles with minimal performance loss (Ali & Ismail, 2023; Wang et al., 2024). Additionally, the presence of redox-active nitrogen sites in PPy promotes the reduction of Cr(VI) to Cr(III), further enhancing the overall detoxification mechanism.

Despite these promising characteristics, the adsorption performance of Fe_3O_4@PPy systems remains highly dependent on process variables such as pH, initial metal concentration, adsorbent dosage, and contact time. A systematic optimization approach is required to evaluate the effect and interaction of these parameters using minimal experimental runs while maintaining high model accuracy (Ni'mah et al., 2022; Rahbar et al., 2016).

In this context, the present study aims to synthesize and optimize a natural magnetite–polypyrrole (Fe_3O_4@PPy) composite for Cr(VI) adsorption in aqueous systems. The magnetite precursor was obtained from natural deposits in Lumajang, East Java, and subsequently coated with PPy through in-situ oxidative polymerization. The resulting composite was characterized using FTIR, XRD, FESEM-EDX, and BET analyses to elucidate its structural, morphological, and surface properties. Furthermore, adsorption experiments were conducted and interpreted using equilibrium isotherm and kinetic models to determine optimal operational parameters, including pH, initial Cr(VI) concentration, contact time, and adsorbent dosage.

RESEARCH METHOD

This chapter presents the research methodology employed in this study, which includes the conceptual framework, materials, procedures, experimental design, characterization techniques, and data analysis methods. The purpose of this chapter is to ensure clarity, reproducibility, and scientific validity of the research process.

General Background of the Research

This study was designed to develop and optimize a magnetic composite adsorbent based on natural magnetite (Fe_3O_4) modified with polypyrrole (PPy) for the removal of hexavalent chromium [Cr(VI)] from aqueous solutions. The motivation behind this research stems from the demand for an efficient, low-cost, and magnetically separable adsorbent that can effectively mitigate environmental pollution caused by toxic heavy metals. The integration of Fe_3O_4 and PPy is expected to produce a synergistic composite material exhibiting improved adsorption capacity, structural stability, and regeneration potential, thereby supporting the advancement of sustainable and environmentally friendly wastewater treatment technologies.

Sample of Research

The primary raw material used in this research was natural magnetite (Fe_3O_4) obtained from Pasirian, Lumajang Regency, East Java, Indonesia. The material was washed, dried, and separated magnetically to obtain fine magnetite powder. Pyrrole monomer (C_4H_5N) was used as the organic precursor for PPy formation. Other chemicals included ferric chloride ($FeCl_3$) as an oxidizing agent, polyvinylpyrrolidone (PVP) as a stabilizer, ammonium persulfate (APS), hydrochloric acid (HCl), and potassium dichromate ($K_2Cr_2O_7$) as the Cr(VI) source for adsorption studies. All reagents were of analytical grade and used without further purification. Deionized water was used in all experiments to prevent contamination and ensure reproducibility.

Instrument and Procedures

The Fe_3O_4@PPy composite was synthesized through an in-situ chemical oxidative polymerization process. In this procedure, a predetermined amount of natural magnetite powder was first dispersed in an aqueous polyvinylpyrrolidone (PVP) solution under continuous mechanical stirring and ultrasonication to ensure homogeneous dispersion and prevent particle agglomeration. Subsequently, the pyrrole monomer was added dropwise into the suspension, followed by ferric chloride ($FeCl_3$) which served as the oxidizing agent to initiate polymerization. The polymerization reaction was carried out under controlled temperature and continuous agitation to facilitate the uniform deposition of PPy on the Fe_3O_4 particle surface. Upon completion, the resulting black precipitate was repeatedly washed with distilled water and ethanol to remove unreacted monomers and residual by-products, and then dried at 60 °C to obtain the final Fe_3O_4@PPy composite powder.

The prepared Fe_3O_4@PPy composite was characterized to determine its physicochemical properties using various analytical instruments. Fourier Transform Infrared Spectroscopy (FTIR, Bruker Tensor II) was employed to identify the surface functional groups. X-ray Diffraction (XRD, Rigaku SmartLab 3) was used to determine the crystal structure and phase composition. Field Emission Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (FESEM–EDX, Hitachi Regulus 8220) was performed to examine the surface morphology and elemental distribution.

In the isotherm and kinetic adsorption experiments, the pH range was restricted to 1, 3, and 4, while pH 2 was intentionally excluded. This decision was made to avoid experimental instability and physicochemical interferences commonly observed near this intermediate acidic condition. At pH 2, the protonation–deprotonation equilibrium on the Fe_3O_4@PPy surface becomes unstable. The partial deprotonation of polypyrrole (PPy) nitrogen sites and the competitive protonation of hydroxyl groups on Fe_3O_4 can result in inconsistent surface charge distribution, leading to non-reproducible adsorption data. Moreover, at this pH, chromium(VI) species ($HCrO_4^- \leftrightarrow Cr_2O_7^{2-}$ equilibrium) coexist in comparable proportions, causing variations in speciation that complicate kinetic and isotherm modeling (Zhang et al., 2021; Wang et al., 2022).

Additionally, prolonged exposure of Fe_3O_4-based materials to solution at pH 2 can promote partial dissolution of magnetite, surface oxidation to maghemite (γ-Fe_2O_3), and leaching of Fe^{2+} ions—all of which alter the surface chemistry and magnetism of the adsorbent (Abd El-Monaem et al., 2023; Abdel Maksoud et al., 2022). Such effects can distort the true adsorption behavior and hinder model fitting for Langmuir, Freundlich, pseudo-first-order, or pseudo-second-order kinetics.

Therefore, pH 2 was not included in the isotherm and kinetic tests to ensure experimental stability, reproducibility, and reliable modeling of adsorption mechanisms across the acidic range, with pH 1 providing a strongly protonated condition and pH 3–4 representing the transitional regime relevant to practical water-treatment systems (Eltaweil et al., 2024; Li et al., 2023).

Each experiment was carried out in a 250 mL Erlenmeyer flask containing 50 mL of Cr(VI) solution, shaken at constant temperature to reach equilibrium. After adsorption, the mixture was separated magnetically, and the residual Cr(VI) concentration was measured using Atomic Absorption Spectrophotometry (AAS). The adsorption efficiency (%) and adsorption capacity (q_e) were calculated based on the concentration difference before and after adsorption.

Data Analysis

Data analysis was performed using adsorption isotherm and kinetic models to evaluate the adsorption behavior of Cr(VI) ions onto the Fe_3O_4@PPy composite. The equilibrium data were fitted to the Langmuir and Freundlich isotherm models to determine the adsorption capacity, surface heterogeneity, and adsorption mechanism (monolayer or multilayer characteristics). The kinetic data were analyzed using the pseudo-first-order (Lagergren) and pseudo-second-order (Ho–McKay) kinetic models, as well as the intraparticle diffusion model, to identify the adsorption rate, rate-limiting step, and dominant adsorption mechanism. The adequacy and significance of each model were verified based on the correlation coefficient (R^2), error analysis, and the agreement between experimental and calculated adsorption capacities. Characterization results before and after adsorption were also compared to evaluate surface and chemical changes, confirming the mechanism of Cr(VI) adsorption and reduction on the Fe_3O_4@PPy composite surface.

Summary

This methodological framework ensures that the Fe_3O_4@PPy synthesis, characterization, and adsorption experiments can be accurately reproduced and statistically validated. The integration of systematic material synthesis with controlled adsorption studies strengthens the reliability of the findings and contributes to the development of efficient magnetic nanocomposites for heavy metal removal.

RESULTS AND DISCUSSION

Structural and Morphological Characterization

The successful modification of natural magnetite (Fe_3O_4) with polypyrrole (PPy) was confirmed through FTIR, XRD, and SEM–EDX analyses. The FTIR spectra demonstrated the characteristic Fe–O stretching vibration at approximately 574 cm^{-1}, confirming the presence of magnetite. Following polymerization, additional absorption bands appeared at around 1540 cm^{-1} and 1460 cm^{-1}, corresponding to the C–N and C=C stretching vibrations of pyrrole rings (Fig. 1(A)). The emergence of these new functional groups indicates the formation of chemical interactions between Fe_3O_4 and PPy molecular chains, suggesting that PPy was successfully coated onto the magnetite surface through in-situ oxidative polymerization rather than by simple physical adsorption.

XRD diffraction patterns of Fe_3O_4 and Fe_3O_4–PPy composites exhibited six major diffraction peaks at 2θ values of 30.1°, 35.4°, 43.1°, 53.5°, 57.0°, and 62.6°, corresponding to the (220), (311), (400), (422), (511), and (440) planes of magnetite (JCPDS 18-3974) (Fig. 1(B)). After modification, all major peaks remained, indicating that the spinel structure of magnetite was preserved. However, peak intensities, particularly at (311) and (511), slightly decreased and broadened, suggesting reduced crystallinity and the presence of an amorphous PPy coating (Fig. 1(C&D)). This finding demonstrates that the polymerization process introduced structural disorder without altering the magnetite phase stability.

FESEM images showed a clear transformation in surface morphology after modification (Fig. 2). Pristine Fe_3O_4 particles exhibited irregular, agglomerated granules, whereas Fe_3O_4–PPy displayed smoother, more uniformly distributed particles with visible polymer coverage. The polymer layer effectively prevented aggregation and enhanced surface roughness, which is beneficial for adsorption.

EDX elemental mapping confirmed the incorporation of carbon and nitrogen elements after modification. The unmodified Fe_3O_4 contained Fe (49.6 wt%) and O (38.4 wt%), while the composite showed Fe (33.4 wt%), O (34.1 wt%), and newly emerged C (26.5 wt%). The appearance of C and reduction of Fe signals validate the surface encapsulation of Fe_3O_4 by the PPy layer (Fig. 2(f)). Moreover, trace impurities such as Ti, Al, and Mg were significantly reduced, indicating purification during polymer formation.

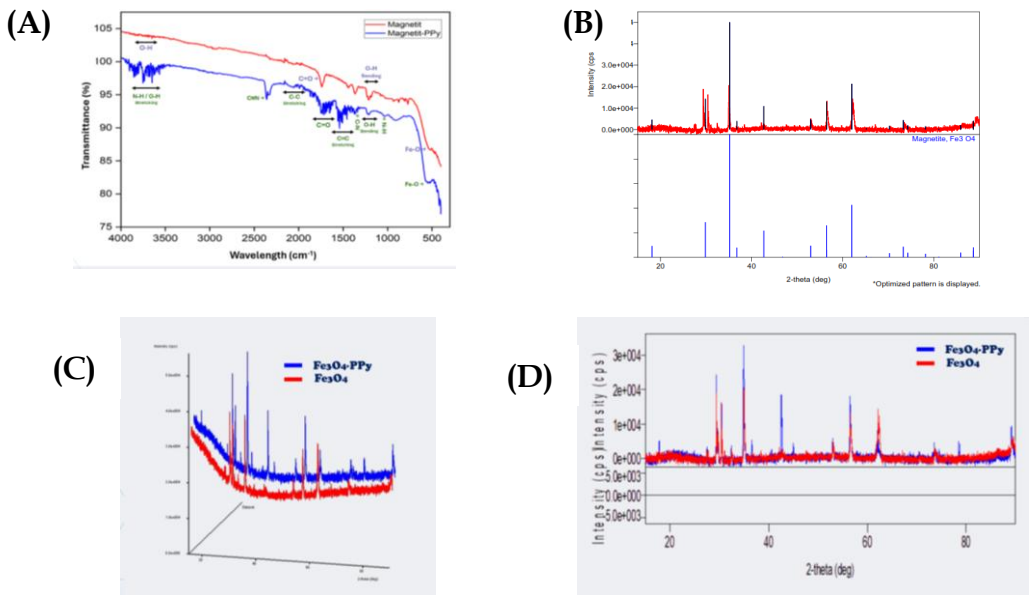

Figure 1. FTIR spectra (A); XRD characterization of Fe_3O_4 (B); XRD Plots of Materials (C) & (D).

Adsorption Behavior and Isotherm Modelling

The adsorption of Cr(VI) ions onto Fe_3O_4–PPy was investigated under various operating conditions of pH, initial concentration, contact time, and adsorbent dosage. The equilibrium data were analyzed using the Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich (D–R) isotherm models (Table 1). The Langmuir model exhibited the best fit with the experimental data (R^2 = 0.9979), indicating that Cr(VI) adsorption occurs on a homogeneous monolayer surface with identical active sites. The maximum adsorption capacity (q_m) obtained was 54.645 mg g^{-1}, reflecting the strong affinity between Cr(VI) ions and the active nitrogen groups on PPy (Zhang et al., 2021; Eltaweil et al., 2024).

The Freundlich constant (n = 1.68) suggests a favorable adsorption process, while the Temkin model showed a heat of adsorption (b = 215.3 J mol^{-1}) indicative of chemisorption. The D–R model yielded a mean free energy (E = 12.8 kJ mol^{-1}), confirming that the adsorption mechanism involves both electrostatic attraction and ion-exchange processes rather than mere physical adsorption (Abd El-Monaem et al., 2023; Li et al., 2023).

Adsorption kinetics were examined using pseudo-first-order, pseudo-second-order, and intraparticle diffusion models (Table 1). The results show that the pseudo-second-order model provided the best fit (R^2 = 0.9827), signifying that chemisorption dominates the process. The equilibrium adsorption capacity calculated from this model (q_e,calc = 51.42 mg g^{-1}) closely matches the experimental value (q_e,exp = 50.86 mg g^{-1}), supporting the reliability of the model (Wang et al., 2022; Liu et al., 2023).

The kinetic results, coupled with the isotherm data, confirm that the adsorption mechanism follows a chemisorption–diffusion hybrid model. Initially, Cr(VI) ions are attracted electrostatically to the protonated $-NH^+$ groups of PPy under acidic conditions (pH ≈ 3), followed by reduction to Cr(III) through electron transfer from Fe^{2+} in Fe_3O_4

and nitrogen moieties in PPy, consistent with previously reported mechanisms for Fe_3O_4-based magnetic composites (Abdel Maksoud et al., 2022; Song et al., 2024).

Isotherm Parameters		
Langmuir Isotherm	R^2	0,9979
	q_m ($mg.g^{-1}$)	54,645
	KL ($L.mg^{-1}$)	5464,481
Freundlich Isotherm	R^2	0,5548
	N	9,784
	K_f ($mg.g^{-1}$)	36,881
Temkin Isotherm	R^2	0,6632
	B_T ($J.mol^{-1}$)	702,199
	K_T($L.mg^{-1}$)	0,124
Dubinin-Radushkevich (D-R) Isotherm	R^2	0,9755
	q_m ($mg.g^{-1}$)	57,351
	β ($mol^2.J^{-2}$)	$6x10^{-5}$
	E($kJ.mol^{-1}$)	91,287

Kinetic Parameters		
Pseudo-1st Order Kinetics (Lagergren)	R^2	0,5856
	k_1 ($10^{-3}.min^{-1}$)	-0,016
Pseudo-Second-Order Kinetics (Ho and McKay)	R^2	0,9827
	k_2 ($10^{-4}.g.mg^{-1}.min^{-1}$)	0,007
Intraparticle Kinetics (Weber–Morris)	R^2	0,7766
	k_i ($mg.g^{-1}.min^{0,5}$)	2,833
	q_t ($mg.g^{-1}$)	34,992

Table 1. Isotherm and Kinetics Parameters for Cr(VI) Adsorption on Fe_3O_4–PPy Composite

Figure 2 shows the FE-SEM micrographs and corresponding EDX spectra of Fe_3O_4 and Fe_3O_4–PPy composites. The pristine Fe_3O_4 nanoparticles (Fig. 2a,b) display an aggregated quasi-spherical morphology with smooth surfaces, typical of magnetite particles formed by co-precipitation. After polymer coating, the Fe_3O_4–PPy composite (Fig. 2c,d) exhibits a rougher and more porous surface texture, indicating successful deposition of the PPy layer through in-situ oxidative polymerization. The granular and network-like morphology enhances the number of active sites for ion exchange and electrostatic adsorption of Cr(VI) ions.

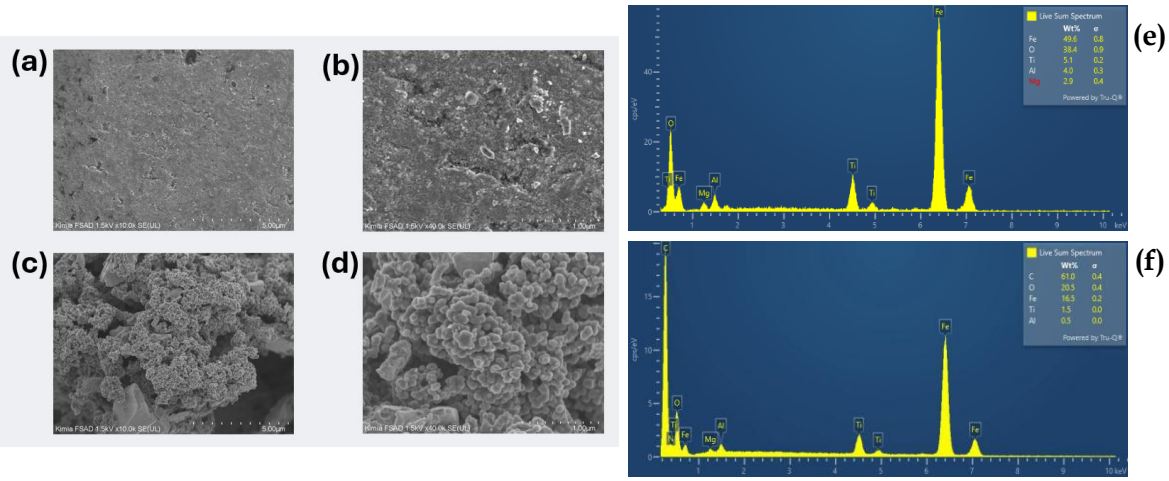

Figure 2. FE-SEM images of (a) Fe3O4 10k; (b)) Fe3O4 40k; (c) Fe3O4-PPy 10k; (d) Fe3O4-PPy 40k; (f) EDX of Fe_3O_4 and (d) EDX of Fe_3O_4-PPy

The EDX spectra (Fig. 2e,f) confirm the presence of Fe and O in the bare Fe_3O_4 sample, while additional peaks corresponding to C and N appear in the Fe_3O_4–PPy composite, verifying the successful incorporation of polypyrrole. The reduction in the Fe peak intensity in the composite spectrum suggests partial surface coverage of Fe_3O_4 by PPy, which effectively protects the magnetic core from oxidation and dissolution under acidic adsorption conditions.

These morphological and compositional transformations support the proposed adsorption mechanism in which protonated $–NH^+$ and –N= groups in PPy provide active binding sites for Cr(VI) through electrostatic attraction and subsequent redox conversion to Cr(III). Similar structural evolution and adsorption enhancement have been reported for Fe_3O_4–PPy and Fe_3O_4–PANI composites synthesized by in-situ polymerization, where the conductive polymer coating increases porosity, prevents nanoparticle aggregation, and strengthens chemisorption interactions (Wang et al., 2022; Eltaweil et al., 2024).

Furthermore, the observed particle morphology and elemental composition agree with earlier studies on hybrid magnetic adsorbents such as Fe_3O_4@PPy/graphene and Fe_3O_4@PANI, which also showed roughened surfaces and the appearance of N peaks in EDX spectra after polymer modification, corresponding to enhanced Cr(VI) removal efficiency above 95% (Liu et al., 2023; Song et al., 2024). The present findings therefore confirm that the successful PPy coating observed in Figure 2 plays a crucial role in improving both the stability and adsorption capacity of the Fe_3O_4–based composite.

CONCLUSION

This study successfully synthesized and modified a natural Fe_3O_4-based adsorbent composite modified with polypyrrole (PPy) through an in-situ polymerization method. The characterization results showed that the composite had a porous structure with chemical interactions between the PPy functional groups and the Fe_3O_4 surface, which increased the adsorption capacity. The adsorption test against Cr(VI) ions showed that the optimal conditions were achieved at pH 4, contact time of 60 minutes, and initial concentration of 40 ppm, with a removal efficiency reaching 99.92% and a maximum adsorption capacity of 54.645 mg $\cdot g^{-1}$. The Langmuir isotherm model and pseudo-second-order kinetics provided an excellent fit, indicating that the adsorption process took place in a monolayer with a chemisorption mechanism. Overall, the PPy–magnetite composite proved to be an effective and potential adsorbent for the remediation of Cr(VI) ions in waters.

ACKNOWLEDGEMENTS

The authors would like to express their sincere gratitude to Institut Teknologi Sepuluh Nopember (ITS) for providing research facilities and academic support throughout this study. Financial support from the Ministry of Finance of the Republic of Indonesia (Kementerian Keuangan RI) through the Indonesia Endowment Fund for Education (LPDP) is gratefully acknowledged.

The authors also extend their appreciation to all colleagues and laboratory staff who contributed technical assistance, valuable discussions, and constructive feedback during

the research process. Special thanks are conveyed to all related parties who generously dedicated their time and effort to ensure the completion of this research in a proper and timely manner.

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