

Adsorption Removal Of Emerging Contaminants In Pharmaceutical Effluents Using Synthesized Composite Cu–Fe Nanoparticles From Copper Mine Tailings

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Abstract

The discharge of pharmaceutical wastewater containing active pharmaceutical ingredients (APIs) and other emerging contaminants poses significant environmental challenges due to their persistence, bioactivity, and resistance to conventional treatment technologies. This study investigated the adsorptive treatment removal of emerging contaminants in pharmaceutical wastewater using synthesized composite copper–iron (Cu–Fe) nanoparticles from mine tailings as a sustainable approach to wastewater remediation and mining waste valorization. Synthesized composite Cu–Fe nanoparticles from copper mine tailings was used for adsorption study. Batch adsorption experiments were conducted to evaluate the effects of adsorbent dosage, contact time, pH, and reusability. Physicochemical analysis was conducted on the pharmaceutical wastewater to determine the water quality. The physicochemical parameters determined are chemical oxygen demand (COD), biochemical oxygen demand (BOD), total dissolved solids (TDS), total suspended solids (TSS), turbidity, and selected heavy metals and selected emerging contaminants. The treated samples used analytical techniques such as inductively coupled plasma optical emission spectroscopy (ICP-OES) and Gas Chromatography Mass Spectrometry (GC-MS). The inorganic emerging contaminant maximum dosage removal revealed a range of 95.18 % – 99.82 % while the maximum contact time removal revealed a range of 94.69 % – 98.77 %. The organic emerging contaminants maximum dosage removal revealed a range 96.33 % – 98.75 % while maximum contact time revealed a range of 95.54 % – 99.38 %. The reusability of the Cu–Fe NPs showed that it can be used for adsorption treatment for six runs effectively before decline in efficiency. In conclusion, the Cu–Fe NPs is a very efficient and effective adsorbent for emerging contaminant wastewater treatment.

Keywords: Pharmaceutical wastewater; Emerging contaminants; Cu–Fe nanoparticles; Adsorption treatment, GC-MS.

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I. Introduction

The rapid growth of pharmaceutical industry has significantly improved global healthcare by providing essential medicines for the prevention and treatment of diseases. However, pharmaceutical manufacturing industries generate large volume of wastewater containing a complex mixture of organic and inorganic pollutants, most of which are biologically active and persistent in the environment [1-3]. Pharmaceutical wastewater is characterized by high chemical oxygen demand (COD), biochemical oxygen demand (BOD), total dissolved solids (TDS), suspended solids, residual solvents, heavy metals, active pharmaceutical ingredients (APIs), and different products of transformation [4-6]. The discharge of inadequately treated pharmaceutical effluents into aquatic environment has become a major environmental concern due to the potential ecological and public health risks associated with these contaminants [7-9]. The pollutants present in pharmaceutical wastewater such as emerging contaminants (ECs) have attracted increasing scientific and regulatory attention for more than two decades due to their widespread occurrence, ecological impacts, potential risks, and persistence to human health [9-11]. Emerging contaminants comprises of a wide range of substances which include antibiotics, hormones, endocrine-disrupting compounds (EDCs), pharmaceuticals, personal care products, disinfection by-products, metabolites, surfactants, and other biologically active chemicals that are frequently detected in wastewater and aquatic environments [1];[12-14]. Unlike conventional pollutants, emerging contaminants are typically detected at trace concentrations ranging from nanograms to micrograms per litre. However, the continuous release of EDCs into aquatic systems which results in pseudo-persistence that leads to chronic environmental exposure and adverse ecological effects. Numerous studies have demonstrated that these contaminants can disrupt endocrine functions, induce reproductive and developmental abnormalities in aquatic organisms, promotion of antimicrobial resistance, and contribution to chronic toxicity in both wildlife and human populations [12];[15-16]. Facilities for manufacturing pharmaceuticals are among the major point

sources of emerging contaminants in the environment. During production processes, substantial quantities of active pharmaceutical ingredients (APIs), intermediates, solvents, and transformation products may enter wastewater streams through washing of equipment, formulation operations, cleaning procedures, production losses, and accidental discharges, which are contributors to environmental contamination and ecological risks [17-19]. The occurrence of antibiotics in pharmaceutical wastewater is alarming because their continuous release into the environment can exert selective pressure on microbial communities, thereby facilitating the emergence and dissemination of antibiotic-resistant bacteria and antibiotic resistance genes. Antimicrobial resistance (AMR) has been identified by the World Health Organization as one of the most pressing global health challenges of the twenty-first century while wastewater effluents are increasingly recognized as important reservoirs and transmission routes for resistance determinants in aquatic ecosystems [20-23]. The treatment of pharmaceutical wastewater is very challenging because several pharmaceutical compounds are designed to resist biological degradation and maintain chemical stability, which result in their persistence in conventional wastewater treatment systems and the environment [24-25]. Several investigations have confirmed the occurrence of pharmaceuticals and their transformation products in treated wastewater effluents, surface water, groundwater, and even drinking water supplies, which indicate their incomplete removal during conventional treatment processes. The persistence and widespread distribution of these contaminants have prompted intensive research into advanced treatment technologies such as adsorption, advanced oxidation processes, membrane filtration, photocatalysis, and nanotechnology-based remediation systems to achieve more effective contaminant removal and environmental protection [26-29]. Different advanced treatment technologies have been developed for the removal of emerging contaminants from wastewater such as membrane separation, advanced oxidation processes, photocatalysis, electrochemical oxidation, ozonation, enhanced biological treatment, and adsorption. Among these approaches, adsorption treatment has gained considerable attention due to its operational simplicity, high removal efficiency, economic feasibility, low energy requirements, and effectiveness in treating contaminants present at trace concentrations. Furthermore, adsorption minimizes the formation of potentially harmful oxidation by-products which can be easily incorporated into conventional wastewater treatment infrastructures to make it one of the most attractive technologies for the remediation of pharmaceutical wastewater and emerging contaminants [30-33]. The efficiency of adsorption-based wastewater treatment systems is largely governed by the physicochemical properties of the adsorbent which include specific surface area, pore structure, pore volume, surface functional groups, surface charge characteristics, and the intrinsic affinity of the adsorbent towards target contaminants. These properties determine the availability of active adsorption sites, mass transfer rates, adsorption mechanisms, and ultimately the overall contaminant removal performance [33-35]. In recent years, nanotechnology has revolutionized wastewater treatment through the development of advanced nanomaterials possessing exceptional adsorption, catalytic, magnetic, and redox properties. Due to their high surface area-to-volume ratios, abundant active sites, tunable surface chemistry, and enhanced reactivity. Nanomaterials have demonstrated remarkable efficiency in the removal of heavy metals, emerging contaminants, dyes, and pharmaceutical residues from complex wastewater matrices [36-39]. Among the various nanomaterials investigated, copper nanoparticles exhibit high catalytic efficiency, antimicrobial activity, and strong affinity toward a variety of organic contaminants due to their reactive surface chemistry. Iron nanoparticles in contrast, possess excellent adsorption characteristics, magnetic and redox properties that facilitate the removal of contaminants through the adsorption and reduction mechanisms. The integration of copper and iron into bimetallic Cu-Fe nano composites can generate synergistic effects that enhance adsorption performance, catalytic activity, stability, and recovery efficiency in wastewater treatment systems [40-43]. The synergistic interaction between copper and iron within bimetallic Cu-Fe nano composites enhances contaminant removal performance through a combination of adsorption and redox mechanisms. These processes include electrostatic attraction, surface complexation, ion exchange, pore diffusion, π - π interactions, and catalytic reduction processes. Consequently, Cu-Fe nano composites have demonstrated high removal efficiencies toward pharmaceuticals, dyes, heavy metals, and other emerging contaminants while offering advantages such as magnetic recoverability, improved stability, and reusability, which make them promising materials for advanced wastewater treatment applications [43-45]. Despite the remarkable performance of engineered nanoparticles in environmental remediation, their widespread commercial application remains constrained by high costs of synthesis, dependence on high-purity precursors chemical or reagent, significant energy requirements, and concerns regarding environmental impacts associated with conventional production methods. These limitations have stimulated growing interest in sustainable nanoparticle synthesis strategies that utilizes waste-derived resources such as industrial by-products, agricultural residues, and mine tailings, thereby reducing production costs while promoting circular economy principles and resource recovery [40][46-47]. Mine tailings are among the largest solid wastes generated by the mining industry and they are produced in enormous quantities during ore extraction and mineral processing operations [48-49]. Although, they are traditionally regarded as waste, tailings which frequently contain appreciable amounts of residual metals such as copper, iron, zinc, cobalt, and

manganese that can be recovered and utilized as secondary raw materials. Poorly managed tailings storage facilities may lead to acid mine drainage generation, heavy metal mobilization, soil degradation, and contamination of surrounding water resources [50-51]. The extraction of valuable metals from mine tailings for synthesis the nanoparticle offers an environmentally sustainable approach that simultaneously mitigates environmental risks, promotes resource recovery, reduces waste disposal burdens, and supports circular economy initiatives [52-55]. The synthesis of composite Cu-Fe nanoparticles from mine tailings aligns with the principles of the circular economy and sustainable materials management by transforming mining waste into value-added products for environmental remediation [50];[56]. The utilization of mine tailings as precursor materials for the synthesis of Cu-Fe nanoparticle reduces the dependence on commercially available metal salts, lowers production costs, minimizes waste generation, and promotes sustainable mining practices [57-58]. Furthermore, mine-tailing-derived nanoparticles exhibit physicochemical properties comparable to those synthesized from conventional reagents grade salts while offering significant economic and environmental advantages [59-60]. In addition, Fe-based and bimetallic nanoparticles have been widely reported to demonstrate strong potential for environmental remediation due to their high reactivity and surface properties, further and to support their application in wastewater treatment systems [61-62]. Recent studies have demonstrated the effectiveness of waste-derived or synthesized metal nanoparticles for the removal of various contaminants from aqueous systems, particularly through adsorption and catalytic degradation [63-64]. However, research specifically focused on the application of mine-tailing-derived composite Cu-Fe nanoparticles for the treatment of real pharmaceutical wastewater remains limited, as most available studies have concentrated on synthetic or single-component contaminant systems rather than complex real effluents [65-66]. In particular, there is insufficient information regarding their adsorption behaviour toward complex mixtures of emerging contaminants typically present in pharmaceutical wastewater which include antibiotics, endocrine-disrupting compounds, and other micropollutants [18];[67]. Furthermore, adsorption mechanism studies such as surface interaction pathways, redox contributions, and synergistic effects in Cu-Fe bimetallic systems remain underexplored in mine-tailing-derived materials [61];[68]. Understanding these aspects is essential for evaluating the feasibility of implementing composite Cu-Fe nanoparticles derived from mine tailings in large-scale pharmaceutical wastewater treatment systems. Therefore, this present study investigates the adsorptive removal of emerging contaminants from pharmaceutical wastewater using composite Cu-Fe nanoparticles synthesized from Cu mine tailings. The study aims to use synthesized composite Cu-Fe nanoparticles, to evaluate their adsorption performance under varying operational conditions, to determine the effects of parameters such as dosage, contact time, adsorbent and pH, In addition, the reusability and sustainability of the synthesized nano composites are assessed to determine their practical applicability in wastewater treatment. By integrating waste valorization with advanced adsorption technology, this study contributes to the development of environmentally friendly and cost-effective solutions for the remediation of pharmaceutical wastewater and the mitigation of emerging contaminant pollution in aquatic environments. The regeneration process of spent adsorbents can be repeated several times, however, the regenerated adsorbent exhibits reduced adsorption capacity in comparison to fresh adsorbents [69]. Choosing the right regeneration technique is vital for improving the desorption efficiency of the contaminant. Factors such as type of adsorbent, contaminant, stability of the adsorbent, toxicity of the spent adsorbents, the cost and energy requirement of the regeneration process are important for the feasibility of the industrial-scale application. There are several approaches applied to recover and regenerate spent adsorbents, such as magnetic separation [70-72], thermal desorption [73], solvent regeneration [74], microwave irradiation [75], supercritical fluid regeneration [76] advanced oxidation process [77] and microbial-assisted adsorbent regeneration [78].

II. Materials And Methods

Sample and sampling

Pharmaceutical wastewater samples were collected from the effluent discharge point of a pharmaceutical manufacturing industry in Ikeja, Lagos state, Nigeria using two different pre-cleaned containers. The first sample was collected in an amber glass bottle for organic emerging contaminant to prevent photodegradation of light sensitive pharmaceutical compounds. The second sample was collected with a plastic container for physicochemical analysis and the third sample was collected with plastic container with the addition of 5 mL concentration nitric acid to prevent metals from undergoing oxidation. All the samples were stored in a refrigerator regulated at 4 °C.

Chemicals and reagents Used

Chemicals and reagents used in this research include NaOH, NaBH₄, HNO₃, FeCl₃, H₂SO₄, MnSO₄ were purchased from local chemical stores and used without further purification. Synthesized composite Cu-Fe NPs and raw pharmaceutical wastewater containing some emerging contaminants such as dichlofenac, paracetamol, ibuprofen, ciprofloxacin, tetracycline, propyl paraben, ethyl paraben, methyl paraben, ampicillin, etc.

Preparation of Solutions

Real-time pharmaceutical wastewater was collected directly from the effluent discharge point of a pharmaceutical manufacturing facility in Ikeja, Lagos State and transported to the laboratory in clean amber bottles and plastic containers. The samples were stored at 4 °C and analyzed within 24 hours to preserve their physicochemical characteristics and contaminant composition.

No synthetic stock solutions of emerging contaminants were prepared, as the pharmaceutical wastewater naturally contained a mixture of emerging contaminants, including pharmaceutical residues and other organic pollutants. Prior to adsorption experiments, the wastewater was filtered through Whatman No. 1 filter paper to remove suspended solids and large particulate matter.

The synthesized copper–iron (Cu–Fe) composite nanoparticles obtained from mine tailings were dried, ground into fine powder, and stored in airtight containers before use. 0.2g, 0.4g, 0.6g, 0.8g and 1.0g of the nanoparticles were added directly to 50 ml of the pharmaceutical wastewater in five different 100 ml volumetric flasks during batch adsorption treatments. The mixtures were agitated under controlled conditions of pH, contact time, adsorbent dosage, and temperature to evaluate the removal efficiency of emerging contaminants from the wastewater.

For adsorption-desorption studies, 0.2 M sodium hydroxide (NaOH) solution was prepared using analytical-grade reagent and distilled water. This solution was used to regenerate the spent nanoparticles by removing adsorbed contaminants from its surface.

All glassware used in the preparation and treatment processes was washed thoroughly, rinsed with distilled water, and dried before use to avoid contamination.

Adsorption Treatment

The raw pharmaceutical wastewater was subjected to adsorption treatment by optimizing the dosage, contact time and pH. Adsorption treatment was carried out using 0.2g, 0.4g, 0.6g, and 0.8g of the dried synthesized composite Cu-Fe nanoparticles that were weighed into different 250 ml conical flask containing 50 ml of raw wastewater each. The conical flasks were covered with foil paper and placed on an orbital shaker for 120 mins. for the optimization studies. After agitation, the nanoparticles were separated from the mixture by filtration process and the filtrate solution concentrations were determined using ICP-OES and GC-MS for inorganic and organic emerging contaminants respectively. During adsorption, the adsorbates attached to the surface of the adsorbent through electrostatic attraction, Contact time treatment was carried out at 30 mins, 60 mins, 90 mins, and 120 mins using optimized dosage of the dried synthesized composite Cu-Fe NPs into four different 250 mL conical flasks containing 50 mL of raw wastewater, and as received pH of the raw wastewater before subjecting to shaking. The adsorption treatment was carried out by varying the pH of the raw wastewater at 3, 6, 9, and 12 using the optimized contact time, and dosage of the dried synthesized composite Cu-Fe NPs.

Analytical Techniques

Physicochemical parameters of the pharmaceutical wastewater were analyzed using standard analytical instruments. The pH was measured using a Hanna HI2211 digital pH meter, while conductivity and total dissolved solid were determined using Jenway 4510 conductivity meter and Hanna HI98301 TDS meter respectively. COD analysis was carried out using a HACH DRB200 COD reactor and DR3900 spectrophotometer. BOD and DO analysis were determined using Velp Scientifica FTC 90E BOD Incubator and Hanna HI9146 Dissolved Oxygen Meter respectively. Heavy metal concentrations were analyzed using a Buck Scientific 210 VGP Atomic Absorption Spectrophotometer. Emerging contaminants were quantified using Agilent 7890B GC coupled with 5977A MSD.

III. Results And Discussion

Physicochemical Analysis of Pharmaceutical wastewater

The raw pharmaceutical wastewater was analyzed for pH, COD, BOD, TSS, TDS, DO, EC, salinity, turbidity as presented in Figures 1 and 2. The results of Turbidity, DO, BOD, COD, Salinity and TDS are within the WHO standard permissible limits while pH, TS, TSS and EC are not within the WHO permissible limits. The concentrations of all the anions are within the WHO permissible limits except choride ion [79].

Table 1: WHO drinking water permissible limit

PARAMETER	RESULTS	NIS -WHO STANDARD
pH	2.73	6.5-8.5
Turbidity	11.95	NTU 30 max
Dissolved Oxygen (DO)	6.80	15 mg/L max
Biochemical Oxygen Demand (BOD)	14.24	30 mg/L max
Chemical Oxygen Demand (COD)	12.41	80 mg/L max
Salinity	2.68	1000 mg/L (max)

Total Solid (TS)	4334.0	1500 mg/mL (max)
Total Suspended Solid (TSS)	3133.5	2000 mg/L (max)
Total Dissolved Solids (TDS)	1200.00	2000 mg/L (max)
Conductivity	4115.00	2500 μ S/cm (max)
Chloride ion (Cl^-)	882.28	250 mg/L (max)
Nitrate (NO_3^-)	13.11	45 mg/L (max)
Phosphate (PO_4^{3-})	1.44	4.5 mg/L (max)
Sulphate (SO_4^{2-})	37.14	250 mg/L (max)

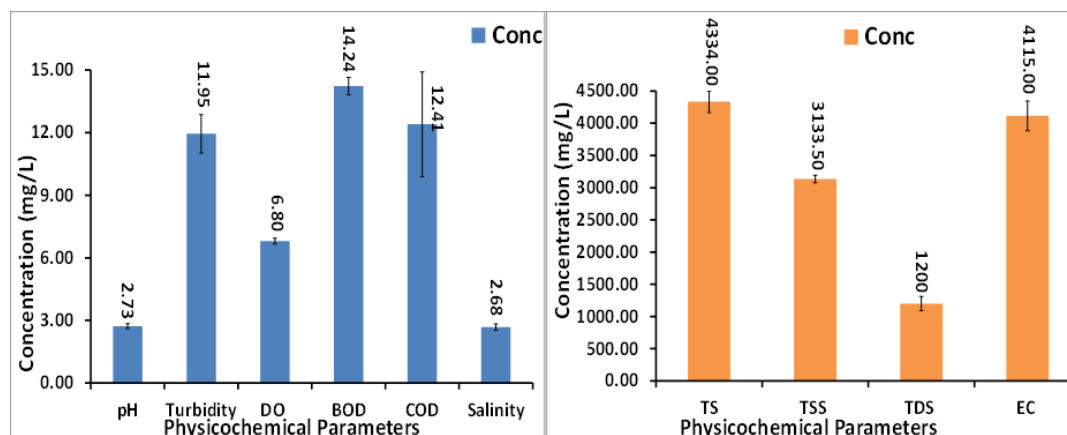


Figure 1: Physicochemical Analysis of Pharmaceutical Wastewater

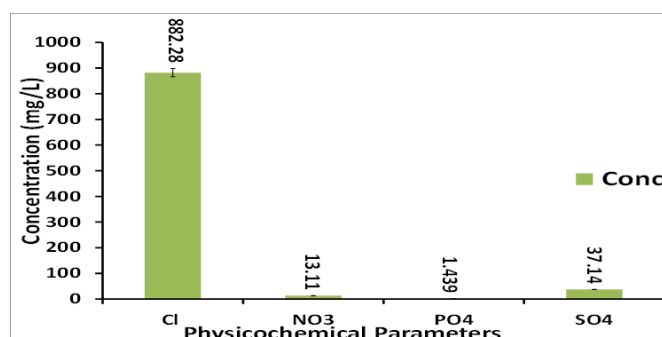


Figure 2: Physicochemical Analysis of Anions in Pharmaceutical Wastewater

Inorganic Emerging Contaminants

Inorganic of dosage treated pharmaceutical wastewater using synthesized Cu-Fe NPs presented in Figs. 3-5 are As, Ba, Cd, Cr, Hg and Pb. The inorganic emerging contaminant treatment results revealed an average dosage of 0.6 g. The treatment showed that as the uptake capacity decrease, the percent removal of the inorganic emerging contaminant increased with the maximum range of 95.18 % - 99.82 %.

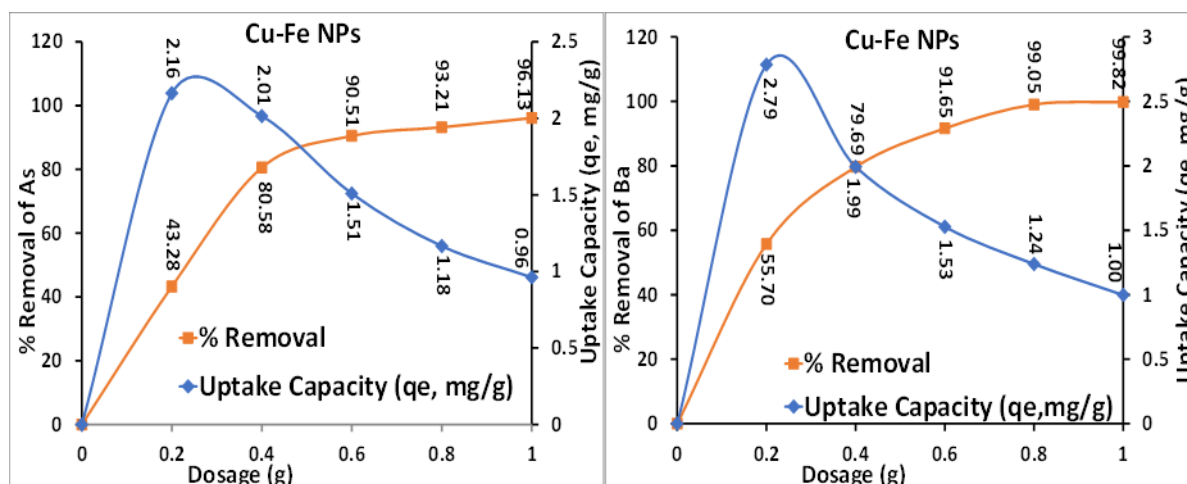


Figure 3: Dosage adsorption of As (A) and Ba (B) in Pharmaceutical Wastewater

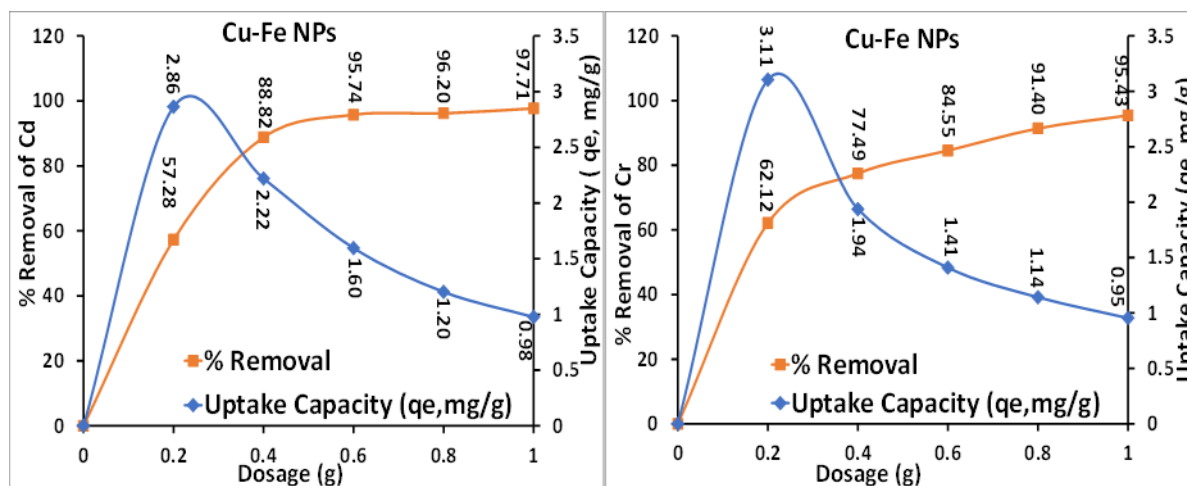


Figure 4: Dosage adsorption of Cd (A) and Cr (B) in Pharmaceutical Wastewater

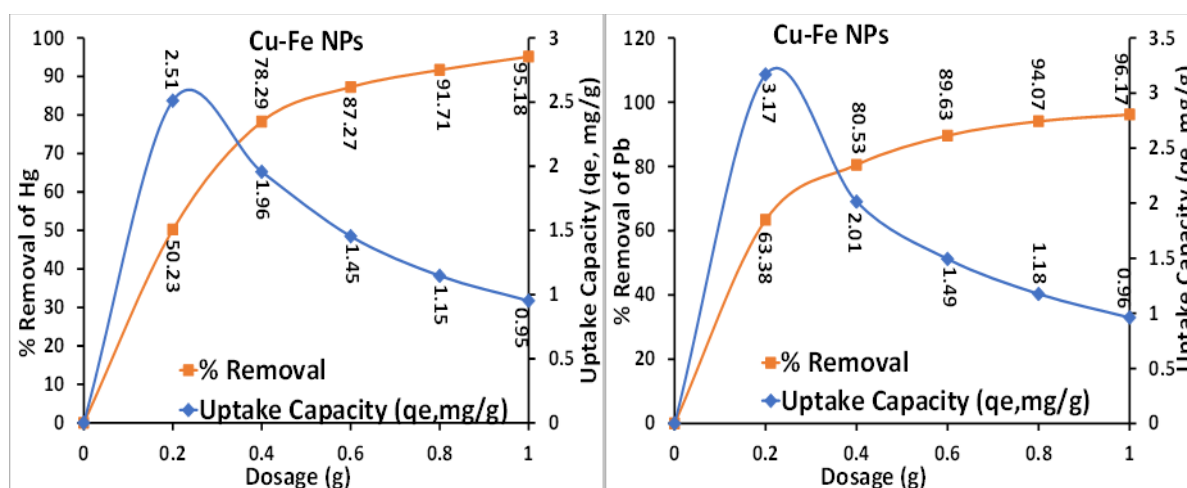


Figure 5: Dosage adsorption of Hg (A) and Pb (B) in Pharmaceutical Wastewater

Contact Time

Inorganic (As, Ba, Cd, Cr, Hg and Pb) emerging contaminant contact time treatment of pharmaceutical wastewater using synthesized Cu-Fe NPs is presented in Figs. 6-8. The results of the contact time treatment revealed average contact time of 60 minutes. The treatment results showed that as the contact time increase, the percent removal of the emerging contaminant increased with maximum range of 94.69 % - 98.77 %.

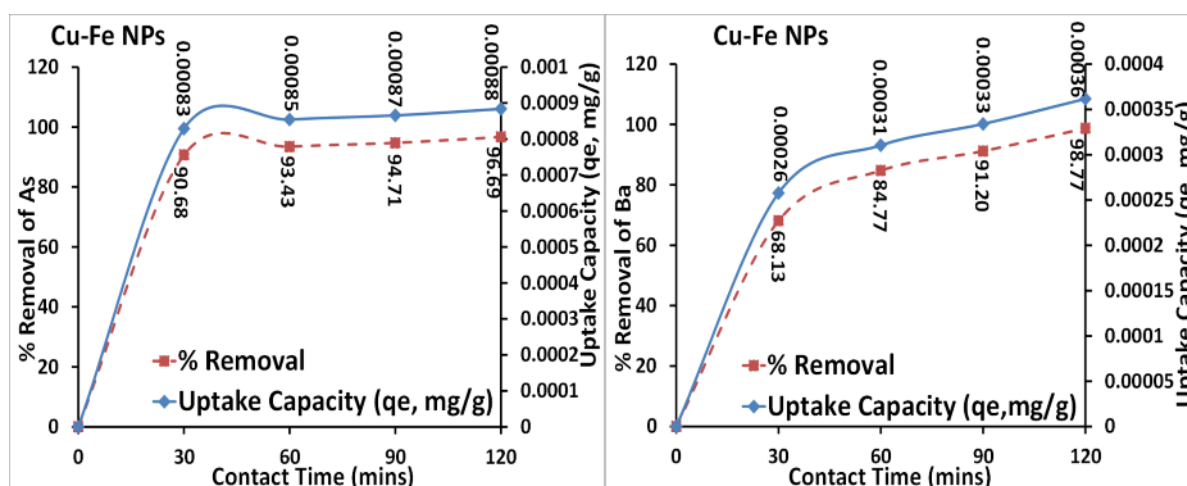


Figure 6: Dosage adsorption of As (A) and Ba (B) in Pharmaceutical Wastewater

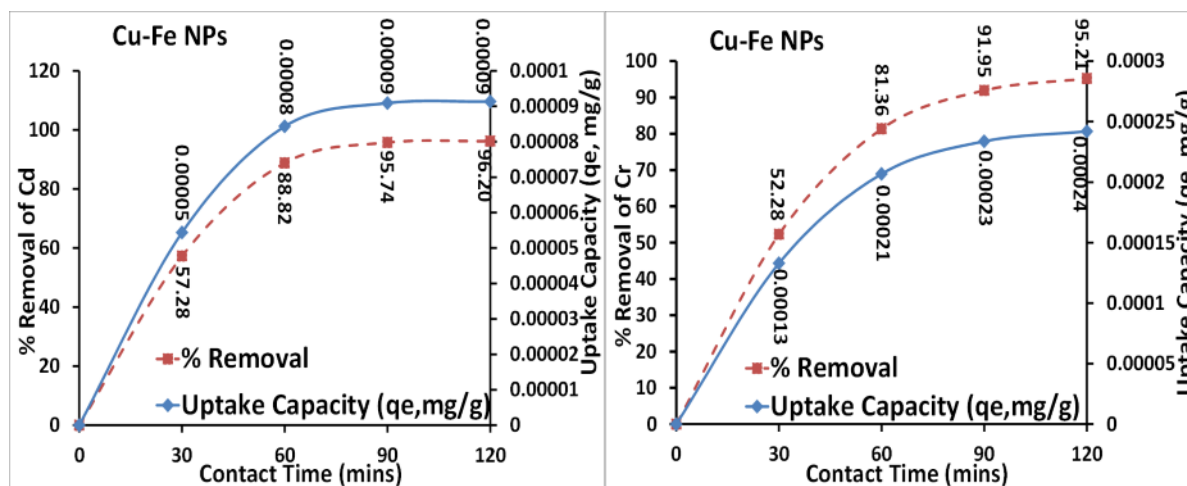


Figure 7: Dosage adsorption of Cd (A) and Cr (B) in Pharmaceutical Wastewater

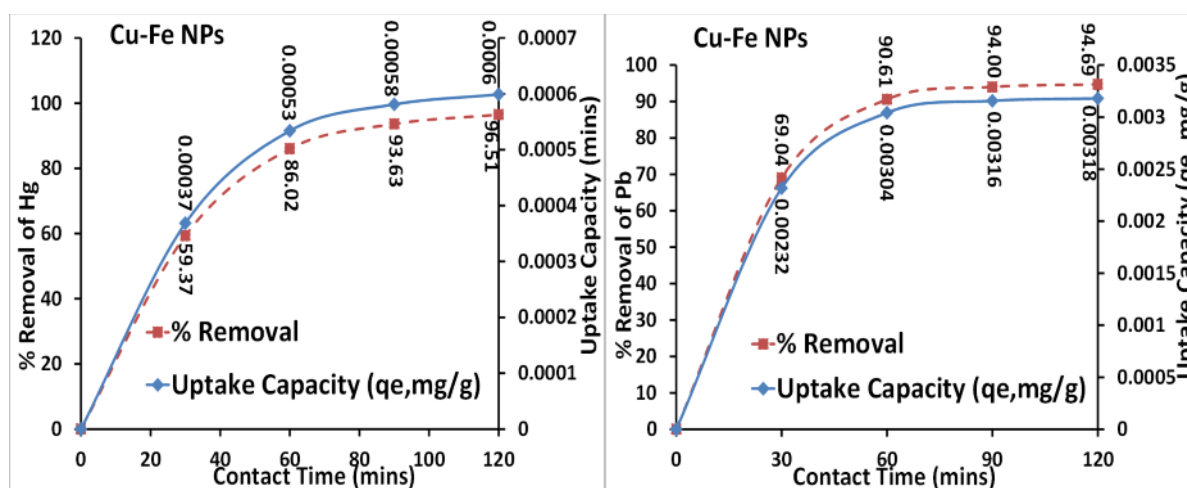


Figure 8: Dosage adsorption of Hg (A) and Pb (B) in Pharmaceutical Wastewater

Removal of Organic Emerging Contaminants

Dosage Adsorption

The dosage treatment removal results of some selected organic emerging contaminant (Dichlofenac, Paracetamol, Ibuprofen and Amplicus) presented in Figures 9 – 10 revealed an average optimum dosage of 0.6 g. The trend of the dosage treatment showed that as the uptake capacity decrease, the percent removal of the organic emerging contaminants increased with the maximum range of 95.18 % - 98.75 %.

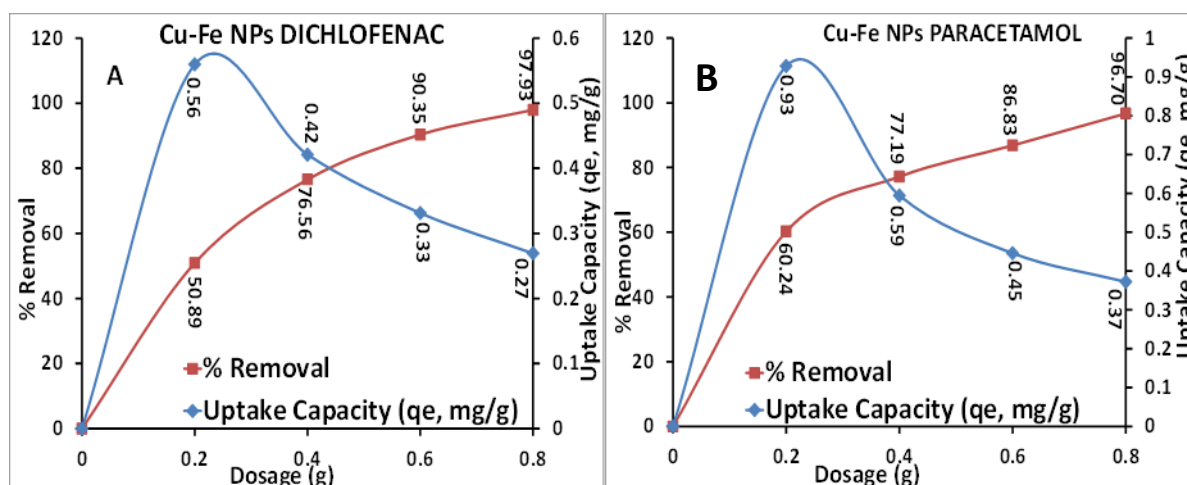


Figure 9: Dosage adsorption of Dichlofenac (A) and Paracetamol (B) in Pharmaceutical Wastewater

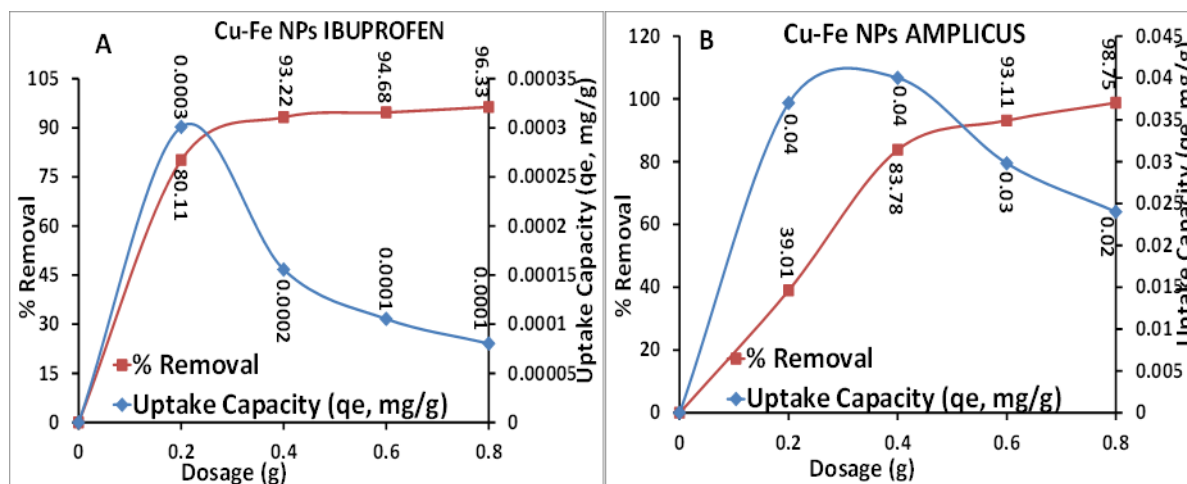


Figure 10: Dosage adsorption of Ibuprofen (A) and Amplicus (B) in Pharmaceutical Wastewater

Contact Time

The adsorption contact time removal of selected organic (Dichlofenac, Paracetamol, Ibuprofen and Amplicus) endocrine disruptors from pharmaceutical wastewater presented in Figs. 11-12, revealed the removal of an optimum contact time to be 60 minutes. The trend of removal revealed that as the time increase, the percent removal also increase with maximum range of 94.54 % - 99.38 %.

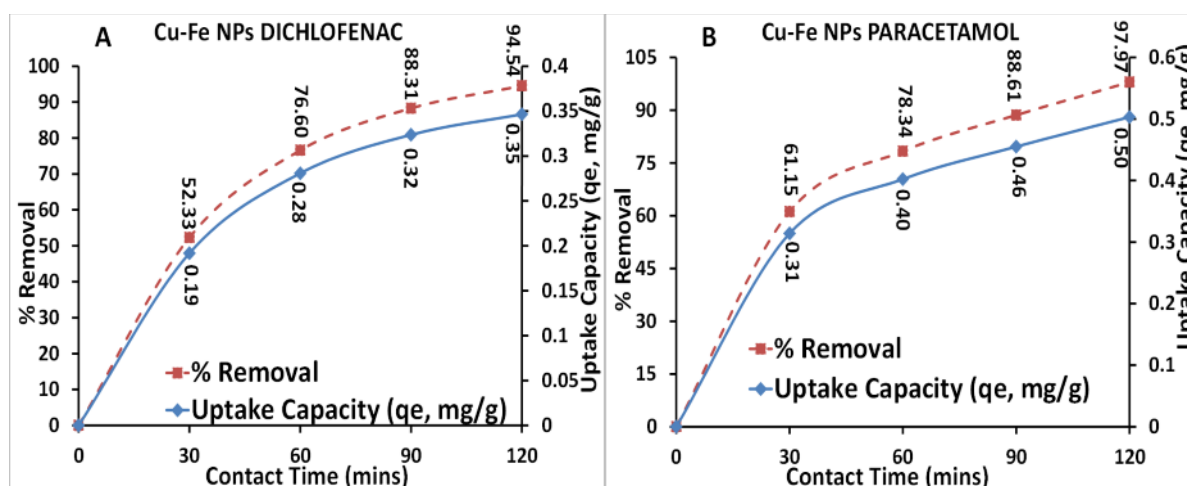


Figure 11: Contact Time adsorption of Dichlofenac (A) and Paracetamol (B) in Pharmaceutical Wastewater

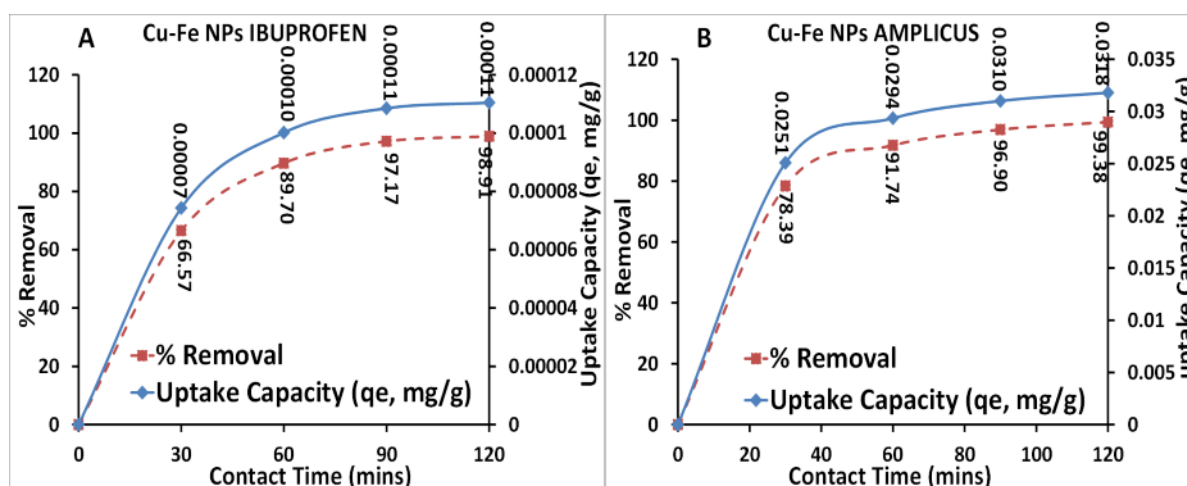


Figure 12: Contact Time adsorption of Ibuprofen (A) and Amplicus (B) in Pharmaceutical Wastewater

IV. Reusability

To assess practical applicability and economic feasibility, adsorption-desorption studies was conducted using sodium hydroxide (NaOH) as the desorbing agent is presented in Figure 13. The regenerated nanoparticles were reused over ten successive adsorption cycles. Their performance was monitored after each cycle and the results showed that the nanoparticles retained a high proportion of their initial adsorption capacity throughout the regeneration process by demonstrating good structural stability, regeneration potential, and reusability. The slight decline in adsorption efficiency observed after repeated cycles was attributed to partial blockage of active sites with minor material losses during regeneration. Consequently, composite Cu-Fe nanoparticles have demonstrated high removal efficiencies toward pharmaceuticals, heavy metals, and other emerging contaminants while offering advantages such as magnetic recoverability, improved stability, and reusability which makes it a promising material for advanced wastewater treatment applications [43-45]. The regeneration after desorption can be completed within a period of 60 min. The results confirmed that the cycle of adsorption, desorption, and regeneration can be reused for six (6) times without loss and degradation which started occurring slightly at the seventh (7) runs until tenth (10). However, limited data exist on the regeneration and reusability potential of such nanoparticles which is critical for evaluating their economic feasibility and operational sustainability in wastewater treatment applications [80-81]. The long-term environmental sustainability and potential secondary contamination risks associated with the deployment of mine-waste-derived nanomaterials have not been sufficiently addressed in the literature [82-83]. The regeneration after desorption can be completed within 60 min. These results confirm that the cycle of adsorption, desorption, and regeneration can be reused for six (6) times without loss and degradation started occurring slightly at the seventh (7) runs until tenth (10) runs.

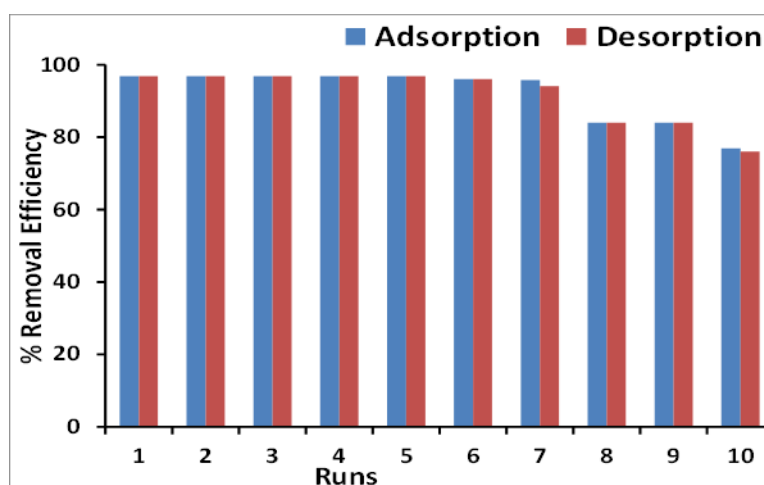


Figure 12: Reusability of synthesized Cu-Fe NPs for adsorption-desorption of Pb in Pharmaceutical Wastewater

V. Conclusion

From this study, the treatment of inorganic and organic emerging contaminants revealed the following:

- The synthesized Cu-Fe NPs showed effective removal of the emerging contaminants in the pharmaceutical wastewater.
- The adsorption treatment was effective because the maximum removal was over 90 %.
- Dosage adsorption treatment trend revealed that uptake capacity decrease while the percentage removal increases.
- The trend of contact time treatment revealed that the uptake capacity increase as the percent removal increases
- The reusability of the synthesized Cu-Fe NPs revealed that the cycles of adsorption, desorption and regeneration can be successfully reused for at least six times without any loss of adsorption efficiency and the degradation started from the seventh run to the tenth run.

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Conflicts of Interest

The authors declare no conflict of interest in this research work

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