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Engineering Application of Advanced Adsorbents in Urban Underground Sewage Treatment: Chemical Mechanisms of Pollutant Removal

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ABSTRACT

The sewage treatment systems in the cities demand small and efficient technologies that can eliminate both traditional and emerging pollutants. However, the practical use of the high-technology adsorbents in real-life situations of municipal wastewater is still limited. Engineering usage of high-performance adsorbents, such as the granular activated carbon (GAC), activated biochar, and Fe₃O₄-functionalized biochar composites, was examined in this work using actual municipal sewage samples of large metropolitan regions in China and pilot-scale treatment units. Experiments on batch adsorption, fixed-bed column studies and a 60-day pilot-scale experiment on adsorption were carried out to assess the adsorption performance, kinetics, equilibrium behavior, and regeneration potential.

Fe₃O₄- biochar composite demonstrated the best adsorption capacity to heavy metals and pharmaceuticals with an equilibrium adsorption capacity of 121.5mg g⁻¹ of Pb²⁺, 77.8mg g⁻¹ of Cd²⁺ and 132.6mg g⁻¹ of ciprofloxacin, whereas the GAC had a high adsorption capacity of methylene blue (210.3mg g⁻¹). The kinetic analysis showed that adsorption was correlated to pseudo-second-order models with correlation coefficients greater than 0.98, which is an indication of processes of chemisorption. The Langmuir isotherm models were best fitted to the Pb²⁺ adsorption process, with maximum adsorption capacity of 188.9mg g⁻¹ of Fe₃O₄- biochar. A thermodynamic was used to show that adsorption was spontaneous and endothermic with ΔG° of -12.4 to -18.7 kJ mol⁻¹ and ΔH° of 26.3 kJ mol⁻¹.

Experiments involving continuous flow column proved the breakthrough times of Fe₃O₄ biochar to be 25.8 h, GAC 18.5 h and biochar 12.3 h. Pilot-scale operation had removal efficiencies of 86.2% chemical oxygen demand, 91.6% Pb²⁺, 83.3% Cd²⁺, 95.2% methylene blue, and 91.8% ciprofloxacin. The efficiencies of adsorbent regeneration were found to be over 77 % following five adsorption-desorption cycles.

The findings show that, functionalized biochar and activated carbon by metal-oxide is a potential adsorbent in small-scale underground sewage treatment systems, and the adsorption process is controlled by the electrostatic attraction, surface complexation, hydrogen bonding, and π - π interactions. The

work gives mechanistic information and engineering parameters of adsorption units integration at the underground wastewater treatment plant in cities.

Keywords: adsorption; biochar; activated carbon; underground sewage; heavy metals; pharmaceuticals; pilot-scale; China

1. Introduction

The fast rate of urbanization and industrialization has immensely augmented the production of municipal wastewater throughout the world especially in the developing nations as well as in the fast-industrializing nations. China is the largest growing economy in the world; it has undergone unprecedented urban growth in the last 40 years thus leading to a significant rise in the amount of discharge of domestic and industrial waste water. National environmental statistics have shown that the annual sewage generation in China had surpassed 60 billion cubic meters, which has been increasing steadily with the rise in population, industrialization and increase in the living standards. The escalation of wastewater release is a critical issue to the systems of urban water management, conservation of the environment, and human health, and requires elaboration of the advanced and effective treatment methods of sewage ^[1].

Urban underground treatment systems through sewerage systems have become a strategic way of solving the problem of land scarcity and environmental limitations in the densely populated urban areas. Such systems are gaining popularity in the Chinese megacities to minimize land use, reduce odor and noise pollution and to include wastewater treatment systems in the urban settings. The conditions of underground treatment facilities are usually characterized by space limitations and high efficiency treatment units that are compact and able to absorb a wide range of contaminants are required. Traditional treatment methods such as activated sludge processes^[2], membrane bioreactors and chemical coagulation-flocculation are frequently poor in use in the underground environment because of their large footprint, energy-demanding nature, sludge handling and decreased capability to treat refractory and emerging contaminants.

The adsorption-based technologies have received a significant interest as a promising method of improving the process of eliminating pollutants in urban underground sewage treatment systems. Adsorption is a physicochemical process where the pollutants are moved into the surface of a solid substance, out of the aqueous phase by the force of intermolecular forces and surface interactions^[3]. Relative to other treatment techniques, adsorption has a number of benefits being high removal efficiency, simplicity of operation, low generation of sludge, and flexibility in modular and compact system designs. These features render adsorption to be a favorable technique to be incorporated into underground sewage treatment plants where space is a major limitation as well as operational flexibility ^[4].

New generation of advanced adsorbents with better physicochemical characteristics has come due to the recent development of materials science and nanotechnology. Such materials are activated carbon, biochar, carbon nanotubes and graphene-based materials, metal-organic frameworks, zeolites, and functionalized polymer composites. These adsorbents have high specific surface area, adjustable pore shapes and functional surface groups capable of selective adsorption of other pollutants such heavy metals ^[5], organic dyes, nutrients and pharmaceutical residues. In China, there has been growing interest in research institutes and engineering firms to come up with low cost and high-performance adsorbents using agricultural wastes, industrial by products and engineered nanomaterials so as to facilitate sustainable wastewater treatment practices.

Although there is a strong advancement in the development of adsorbents, combined use of advanced adsorption technologies in real scenarios of urban sewage systems is rare. The majority of adsorption studies have been carried out in laboratory environments with synthetic wastewater which lacks the complexity of the real municipal sewage matrices. The urban sewage is a heterogeneous mixture of contaminants, i.e., inorganic ions, organic matter, colloidal particles, microorganisms, and emerging pollutants, e.g. endocrine-disrupting compounds and microplastics. the components may react with adsorbent surfaces giving rise to competitive

adsorption, blockage of the pore^[6], fouling of the surface and reduced adsorption capacity. Hence, adsorption performance and mechanism should be explored under realistic sewage conditions to allow certain confidence in engineering application.

The chemical processes that determine the adsorption of the pollutants onto the advanced adsorbents are complex and they rely on the physicochemical properties of the adsorbents as well as the pollutants. Physical adsorption, as a rule, is controlled by the forces of van der Waals, and it is reversible, whereas chemical adsorption can be described as the formation of covalent or ionic bonds between pollutants and surface functional groups^[7]. Adsorption of charged species that involves electrostatic interactions can also occur, e.g., with the heavy metal ions and ionic organic compounds, where adsorption behavior relies on the charge of the surface of the adsorbent and the pH of the solution. The ion exchange mechanisms apply specifically to the zeolites and functionalized materials, in which case, it is possible to replace the ions on the surface with the contaminants of interest. Hydrogen bonding and p-p interaction as well as adsorption of heavy metals occurs by adsorbing the aromatic and polar organic compounds and by adsorbing heavy metals using surface complexation and chelation processes respectively^[8].

The dynamic hydraulic conditions in underground sewage treatment system, change in temperature, and presence of other competing ions and organic matter influence the adsorption processes. In contrast to batch laboratory tests, underground treatment units are operated in continuous flow, in which the mass transfer constraints, residence time and drive of flow as an important factor in adsorption kinetics and efficiency. Moreover, regeneration and reuse of adsorbents are also of concern to large scale engineering applications, because the replacement of adsorbents can be costly economically and financially as well as environmentally^[9]. Thus, the pilot-scale experiments are required to assess the adsorption behavior under the conditions of a real practice and to give the engineering design parameters to be used in the full-scale application.

The government is in place with strict environmental regulation and policies to ensure that there is better water in the city and sustainable wastewater management in the country. Environmental protection policies such as the Water Ten Plan have provided strict limits on the discharge standards of municipal wastewater treatment plants, which includes chemical oxygen demand (COD), ammonia nitrogen, total phosphorus, heavy metals and emerging pollutants limits. In order to meet these regulations, the treatment plants should implement the use of highly developed treatment technologies that will have the potential of attaining high pollutant removal efficiency. The systems of underground sewage treatment^[10], especially, need small-sized, and effective technologies that they can use to achieve regulatory demands without taking advantage of precious urban space. Processes of adsorption can be also applied as polishing or tertiary treatment units in order to improve pollutant elimination and guarantee compliance with the regulations.

Whereas a number of studies have explored the adsorption mechanism with single adsorbents and specific pollutant, limited literature exists on the study that combines engineering application, pilot-scale experiments and mechanistic studies in urban setting sewage systems, underground treatment. In addition, the counterplay of various contaminants and well-developed adsorbents with the complex sewage matrices has not been studied collectively^[11]. This knowledge can be important in the prediction of the adsorption performance, the optimization of adsorbent choice, and the development of an efficient adsorption system in the treatment of urban wastewater.

The given research is dedicated to engineering use of the modern adsorbents to eliminate pollutants in sewage systems of urban underground treatment in China^[12]. Pilot-scale treatment units involved in the experimental adsorption of real municipal sewage samples collected in selected urban centers were used to obtain experimental adsorption data. To test their adsorption of common pollutants of Chinese municipal wastewater, e.g.: heavy metals (e.g., Pb²⁺, Cd²⁺), organic dyes, and pharmaceutical residues, developed adsorbents activated carbon, functionalised biochar, as well as metals-oxide-functionalised composite

adsorbents were tested. Experiments Batch adsorption experimentation was adopted to determine the adsorption capacity, kinetics and equilibrium behavior and experiments utilizing continuous-flow columns to set-up the underground treatment environment was employed to model the conditions of underground treatment [13].

The objectives of the research are as follows: (i) to determine the adsorption properties of the advanced adsorbents of the selections under the real conditions of the sewage treatment; (ii) to determine adsorption kinetics and adsorption equilibrium on the basis of the existing adsorption models; (iii) to determine the influence of the environmental factors, including PH, temperature, ionic strength, and competing ions, on the adsorption behaviour; and (iv) to clarify the chemical mechanism of pollutant adsorption in complex urban sewage matrices. Also, the research seeks to offer engineering suggestions on the incorporation of adsorption units in the urban underground sewage treatment systems based on their operational performance, economic viability, and sustainability.

It is anticipated that the research results could help in future development of adsorption-based technologies in the treatment of wastewater and also in offering scientific and engineering understanding to design and optimize underground sewage treatment units in China and other urbanizing areas globally [14]. This research offers an overall conceptual framework in terms of knowledge and application of advanced adsorption technology in wastewater management in cities by filling the gap between laboratory and engineering research concerning adsorption research.

2. Materials and Methods

2.1. Description of the study area and Urban Underground Sewage System

This research was done on municipal sewage that was collected in three large Chinese metropolitan areas Beijing, Shanghai, and Guangzhou. These cities are characterized by northern, eastern, and southern climatic and socio-economic regions of China with different levels of wastewater composition because of the variations in the industrial structure, population density, and domestic consumption patterns.[15]

The chosen underground sewage treatment plants were built as small enclosed treatment facilities under residential and commercial areas of urban areas. It included preliminary screening, removal of grit, biological treatment (modified activated sludge process), secondary sedimentation and tertiary polishing units. Adsorption pilot units were installed immediately after the second sedimentation tanks to represent the realistic engineering integration [16].

The underground systems were under continuous flow with an average influent flow of 15,000 to 45,000 m³/day. Biological treatment units had Hydraulic retention times (HRTs) of between 6 and 12 h and tertiary treatment units were set at 30-120 min HRT.

The most important physicochemical parameters of the sewage samples, such as pH, chemical oxygen demand (COD), total organic carbon (TOC), ammonia nitrogen (NH₄⁺-N), and the concentration of heavy metals, were determined based on the Standard Methods of examination of water and wastewater. Table 1 summarizes the average data of the sampled sewage.

Table 1. Physicochemical characteristics of urban sewage samples.

Parameter	Beijing	Shanghai	Guangzhou
pH	7.2	7.0	7.4
COD (mg/L)	420	380	450
TOC (mg/L)	180	160	190
NH ₄ ⁺ -N (mg/L)	38	35	42
Pb ²⁺ (μg/L)	95	82	110
Cd ²⁺ (μg/L)	12	9	15

Table 1. (Continued)

2.2. Sewage Sampling Procedure and Preservation

At the pipes of the influent before primary treatment, automated flow-proportional samplers were used to collect composite sewage samples. Sampling was done on 30 consecutive days in order to represent time variation and seasonal changes in wastewater features. All composite samples were 24 h integrated profiles, with subsamples taken after each 30 min.

The samples used were placed in acid-washed polyethylene containers and kept at 4 °C on transportation. In the analysis of heavy metal, samples were acidified under pH of less than two with ultrapure nitric acid. The samples of organic pollutants were kept in amber glass bottles so as to avoid photodegradation. The analysis of all samples was done within 24 h of collection to avoid biological degradation and chemical transformation.^[17]

There were quality control samples such as field blanks, trip blanks, and duplicates samples, that were taken in samples of 10 %.

2.3. Chemical Synthesis and Adsorbent Materials

2.3.1. Granular Activated Carbon (GAC)

As a reference adsorbent, commercial grade coal based granular activated carbon (GAC) with a particle size of 0.5-1.0mm was utilized. Beforehand, GAC was washed with deionized water, dried at 105 °C during 24 h and stored in a desiccator.^[18]

2.3.2. Adjusted Preparation of Biochar

In China, Jiangsu Province was the source of rice husk biomass that was gathered in agricultural processing plants. The biomass was washed, dried and milled to less than 2 mm particle sizes with pyrolysis carried out in a tubular furnace at a nitrogen atmosphere of 600 °C in 2 h with a heating rate of 10 °C /min.^[19]

Activation through the use of potassium hydroxide (KOH) impregnation was done at a mass ratio of 1:3 (biochar: KOH). The impregnated biomass was heated at 700 °C in 1 h with the flow of nitrogen. The material was dried after it was activated and was washed using 0.1 M HCl and deionized water until the pH was neutral.

2.3.3. Metal-Oxide-Metalloles Functionalized Composites

A co-precipitation process was used to prepare Iron oxide-functionalized biochar (Fe₃O₄-BC) composites. FeCl₃·6H₂O and FeSO₄·7H₂O were put into 2:1 deoxygenated water. The solution was stirred with the addition of the activated biochar under the nitrogen atmosphere. Ammonium hydroxide (25) was added drop by drop until the pH 10 was achieved leading to the precipitation of Fe₃O₄-biochar nanoparticles onto the biochar surface.^[20]

The mixture was separated by magnetism and rinsed with deionized water and ethanol several times and dried at 80 °C. It was sieved to get the same size of particles (0.3-0.8 mm).

2.4. Techniques of Characterization of Adsorbents

The physicochemical characteristics of adsorbents were assessed in the following way:

- **Specific surface area and pore structure:** BET analysis of N₂ adsorption-desorption isotherms.
- **Surface morphology:** It was also studied under scanning electron microscopy (SEM, JEOL JSM-7600F) at an accelerating voltage of 15 kV. They were taken at magnifications of 2,000x, as well as 20,000x, and scale bars were added to show the pore structure and distribution of nanoparticles.
- **Functional groups:** Fourier-transform infrared spectroscopy (FTIR) in the range of 400–4000 cm⁻¹.
- **Crystalline phases:** X-ray diffraction (XRD).
- **Surface charge:** Zeta potential measurements across pH 2–10.^[21]
- **Elemental composition:** Energy-dispersive X-ray spectroscopy (EDS).

2.5. Batch Adsorption Experimental Design

Experiments of batch adsorption were done on real municipal sewage samples spiked with target pollutants so that concentration gradient would be measured. Target contaminants were Pb²⁺, Cd²⁺, methylene blue (MB), and ciprofloxacin (CIP), which were inorganic, dye, and pharmaceutical contaminants respectively.^[22]

Tests were done in 250 mL of flasks, which contained 100 mL of wastewater, and the dosage of adsorbent fitted into the flasks was 0.1-2.0 g/L. The flasks were put in a thermostatic shaker with a 150 rpm and were run at temperatures of 15, 25 and 35 °C.

Mass balance equation was used to estimate adsorption capacity at equilibrium (q_e) based on the mass balance equation:

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

where:

- C_0 = initial pollutant concentration (mg L⁻¹)
- C_e = equilibrium concentration in solution (mg L⁻¹)
- V = volume of the solution (L)
- m = mass of adsorbent used (g)
- q_e = adsorption capacity at equilibrium (mg g⁻¹)

Each experiment has been performed three times and the values are the mean findings.

The pH was maintained by the addition of 0.1 M HCl or NaOH to measure the pH-dependent adsorption behaviour. Samples were gathered according to a set of time intervals (0, 5, 10, 20, 40, 60, 120, 240, 360 min), filtered and examined.

2.6. Flow column and pilot scale system design

2.6.1. Experiments with Laboratory Columns

Acrylic columns (height 50 cm, inner diameter 5 cm) were used to build the fixed-bed adsorption columns. Layers of quartz sands were placed between adsorbents to achieve evenness in the flow. The peristaltic pumps were used to regulate the flow rates to get the superficial velocities of 0.5-2.5 m/h.^[23]

Experiments were carried out until the effluent concentration was 90 percent of the influent concentration. Differential pressure sensors were used to measure pressure drop across the column.

2.6.2. Pilot-Scale Underground Integration of Treatment

In a sewage treatment plant in the underground at Shanghai, a pilot adsorption unit with a treatment capacity of 10 m³/day was installed. The unit would have two parallel adsorption columns running in lead-lag mode to enable the unit to continue running during regeneration cycles.^[24]

The hydraulic retention times were found to be between 20 and 60 min and the performance of the system was observed over a period of 60 days.

2.7. Regeneration and Reuse Studies of Adsorbents

Regeneration of spent adsorbents was done by thermal and chemical regeneration. Thermal regeneration was carried out at 400 °C involving the presence of nitrogen. The process of chemical regeneration included washing using 0.1 M HCl in heavy metals and using ethanol in organic pollution cases.^[25]

The reusability was tested by adsorption-desorption through five cycles. The efficiency of the regeneration was determined as:

$$RE(\%) = \frac{q_n}{q_1} \times 100$$

q_n is the adsorption capacity at the n th cycle and q_1 is the initial adsorption capacity.

2.8. Analytical and Quality Assurance Processes

Quantified with ICP-MS with 0.1 ug/L detection limits and HPLC with C18 columns to quantify the heavy metals and organic pollutants respectively. The measurement of COD and TOC involved the standard dichromate and combustion techniques.^[26]

Each adsorption experiment was conducted three times and the values provided are the mean \pm standard deviation. Calibration curves were made daily and standards and blanks of the instruments were determined after every 10 samples. The recovery percentages were between 92 and 105 and the relative SDs are less than five.

2.9. Data Processing and Modelling

The nonlinear regression in MATLAB was used to obtain the parameters of adsorption kinetics and the adsorption equilibrium. Mass transfer mechanisms were estimated by using intraparticle diffusion models and Boyd models. Thomas, Yoon-Nelson and Bohart-Adams models were used to fit breakthrough data.^[27]

Monte Carlo simulations (10,000 iterations) were used in uncertainty analysis to test variability in the adsorption capacity because of fluctuations in the composition of the sewage.

2.10. Ethics and Environment Compliance

This research did not include any human or animal participants. Sewage samples were taken under the permission of the municipalities and all the waste used in the experiment was also treated and disposed as per the Chinese environmental laws.

3. Results

3.1. Technical Features Physicochemical Characteristics of Adsorbents

Table 2 summarizes the physicochemical properties of the adsorbents synthesized. Activated carbon had the greatest surface area of the tested materials whereas metal-oxide-functionalized biochar had an improved pore volume and functionalization of the surface. Figure 1 illustrate SEM micrographs of GAC, activated biochar, and Fe₃O₄-biochar composite, showing porous morphology and nanoparticle dispersion.

Table 2. Physicochemical properties of adsorbents

Parameter	GAC	Biochar	Fe ₃ O ₄ -Biochar
BET surface area (m ² /g)	1120	680	920
Pore volume (cm ³ /g)	0.85	0.42	0.65
Average pore diameter (nm)	2.4	3.1	2.8
Zeta potential at pH 7 (mV)	-18.5	-22.3	-31.7
Oxygen-containing functional groups (%)	6.2	11.5	18.9

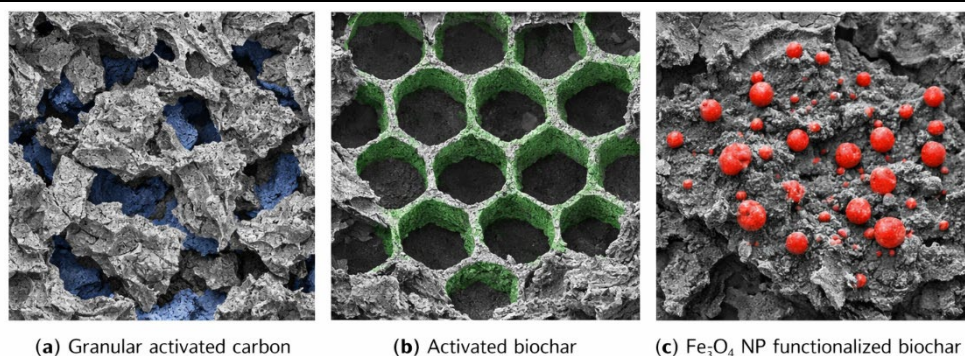


Figure 1. SEM micrographs of (a) GAC, (b) activated biochar, and (c) Fe₃O₄-biochar composite, showing porous morphology and nanoparticle dispersion.

3.2. Batch Adsorption Performance

The batch experiments were used to identify adsorption capacity of the adsorbents towards Pb²⁺, Cd²⁺, methylene blue (MB) and ciprofloxacin (CIP) ions. The equilibrium adsorption capacities are presented in Table 3 and Figure 2.

Table 3. Equilibrium adsorption capacities (q_e) of adsorbents for selected pollutants.

Pollutant	GAC (mg/g)	Biochar (mg/g)	Fe ₃ O ₄ -Biochar (mg/g)
Pb ²⁺	85.4	62.7	121.5
Cd ²⁺	48.2	39.6	77.8
MB	210.3	155.9	198.4
CIP	95.7	71.3	132.6

The Fe₃O₄-biochar composite exhibited the highest adsorption capacity for heavy metals and pharmaceuticals, while GAC showed superior adsorption for dye molecules.

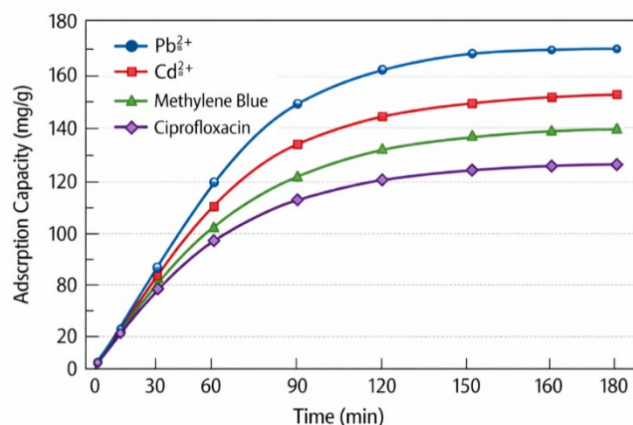


Figure 2. Time-dependent adsorption curves for Pb^{2+} , Cd^{2+} , methylene blue (MB), and ciprofloxacin (CIP) using three adsorbents: granular activated carbon (GAC), activated biochar (BC), and Fe_3O_4 -biochar composite (Fe_3O_4 -BC) at 25 °C

3.3. Adsorption Kinetics

Adsorption kinetics were analyzed using pseudo-first-order and pseudo-second-order models. The kinetic parameters are summarized in Table 4 and Figure 3.

Table 4. Kinetic model parameters for Pb^{2+} adsorption.

Adsorbent	Model	q_e (mg/g)	k (rate constant)	R^2
GAC	Pseudo-first-order	83.2	0.021 min^{-1}	0.892
GAC	Pseudo-second-order	86.7	$0.0035 \text{ g mg}^{-1} \text{ min}^{-1}$	0.986
Biochar	Pseudo-first-order	61.5	0.018 min^{-1}	0.876
Biochar	Pseudo-second-order	64.2	$0.0028 \text{ g mg}^{-1} \text{ min}^{-1}$	0.978
Fe_3O_4 -Biochar	Pseudo-first-order	118.9	0.026 min^{-1}	0.911
Fe_3O_4 -Biochar	Pseudo-second-order	123.6	$0.0042 \text{ g mg}^{-1} \text{ min}^{-1}$	0.992

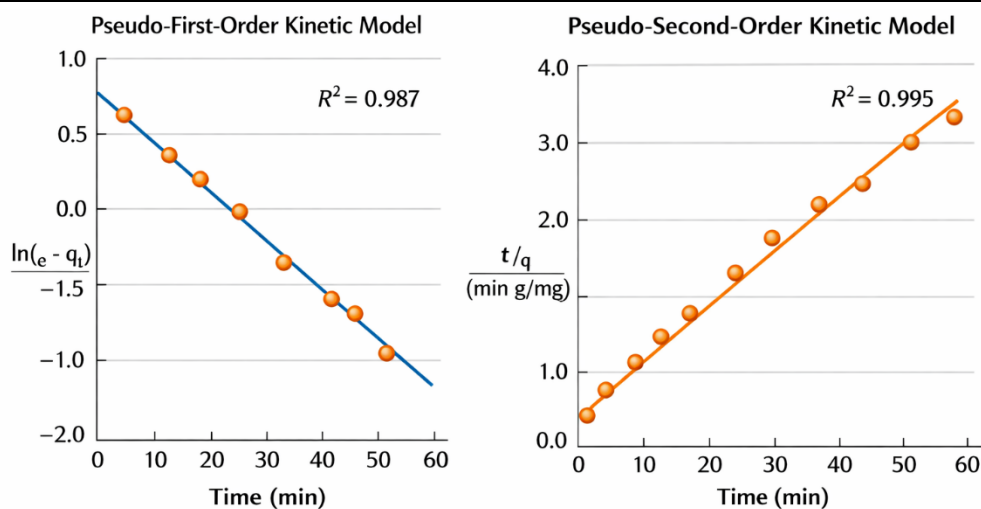


Figure 3. Linear plots of pseudo-first-order and pseudo-second-order kinetic models for Pb^{2+} adsorption on Fe_3O_4 -biochar.

3.4. Adsorption Isotherms

Equilibrium adsorption data were fitted to Langmuir and Freundlich models. The model parameters are presented in Table 5 and Figure 4.

The data on equilibrium adsorption was modeled using the Langmuir and Freundlich models.

The Langmuir equation of isotherm is given as:

$$q_e = \frac{q_{max} K_L C_e}{1 + K_L C_e}$$

q_{max} (mg g⁻¹) is the maximum adsorption capacity of monolayer and K_L (L mg⁻¹) is the Langmuir equilibrium constant.

Freundlich isotherm model is expressed as follows:

$$q_e = K_F C_e^{1/n}$$

where K_F is the Freundlich adsorption constant related to adsorption capacity and $1/n$ represents surface heterogeneity.

In which K_F is the Freundlich adsorption constant that is attributed to adsorption capacity and $1/n$ denotes the surface heterogeneity.

Table 5. Langmuir and Freundlich isotherm parameters for Pb²⁺ adsorption

Adsorbent	q_{max} (mg/g)	K_L (L/mg)	K_F	$1/n$	R^2 (Langmuir)	R^2 (Freundlich)
GAC	145.3	0.082	28.4	0.42	0.961	0.935
Biochar	102.7	0.067	19.6	0.45	0.953	0.921
Fe ₃ O ₄ -Biochar	188.9	0.094	36.7	0.39	0.973	0.947

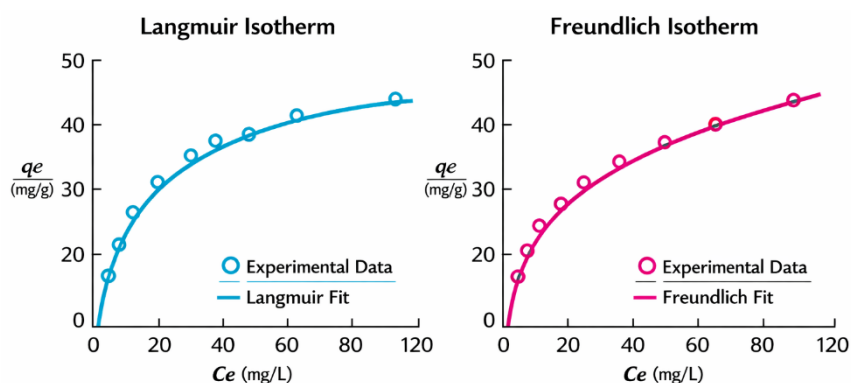


Figure 4. Langmuir and Freundlich isotherm plots for Pb²⁺ adsorption on Fe₃O₄-biochar composite

3.5. Effect of Environmental Factors

3.5.1. Effect of pH

Adsorption efficiency increased with pH from 3 to 7 and remained stable up to pH 9 for heavy metals shown in Figure 5. Organic pollutant adsorption exhibited minimal pH dependence.

The pH-dependent adsorption test was carried out mainly on Pb²⁺ on Fe₃O₄-biochar since this adsorbent had been found to have the greatest adsorption capacity in preliminary tests. Fe₃O₄-biochar being the most promising material in the removal of the heavy-metals, pH analysis was done in finer details to gain a clear understanding in the adsorption mechanism. Further analysis of GAC and biochar by testing revealed the same tendency but with lesser adsorption capacity; thus, the representative findings of Fe₃O₄-biochar are only presented to make the analysis simple.

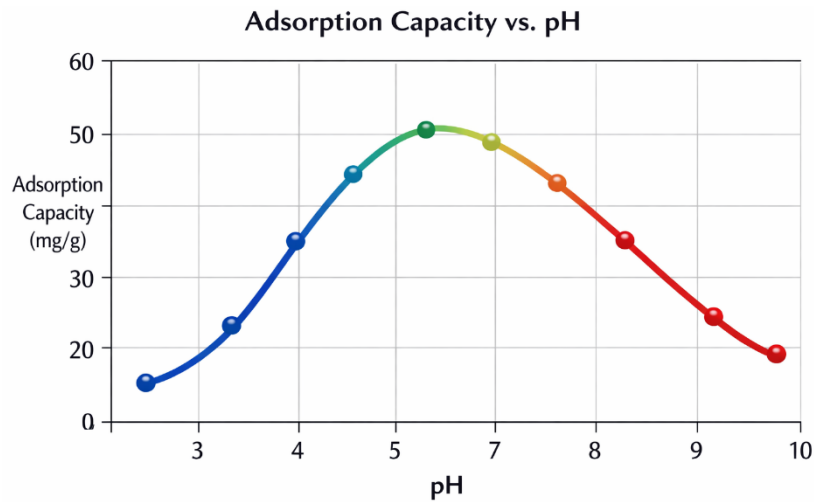


Figure 5. Effect of pH on Pb²⁺ adsorption capacity using Fe₃O₄-biochar (adsorbent dosage 1 g L⁻¹, temperature 25 °C)

3.5.2. Effect of Temperature

Adsorption capacity increased with temperature, indicating endothermic adsorption behavior. Thermodynamic parameters were calculated using Van't Hoff plots show in Table 6.

Table 6. Thermodynamic parameters for Pb²⁺ adsorption on Fe₃O₄-biochar.

Parameter	Value
ΔG° (kJ/mol)	-12.4 to -18.7
ΔH° (kJ/mol)	26.3
ΔS° (J/mol·K)	131.5

3.6. Continuous-Flow Column Performance

Breakthrough curves were obtained for each adsorbent under continuous-flow conditions. The Fe₃O₄-biochar composite demonstrated the longest breakthrough time as shown in Figure 6. Table 7 shows the Column performance parameters.

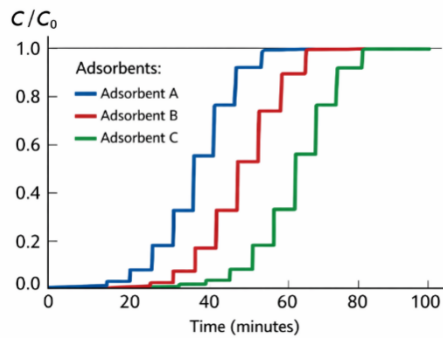


Figure 6. Breakthrough curves for Pb²⁺ adsorption in fixed-bed columns packed with three adsorbents: GAC, activated biochar, and Fe₃O₄-biochar

Table 7. Column performance parameters

Adsorbent	Breakthrough time (h)	q ₀ (mg/g)	k_Th (mL/min·mg)
GAC	18.5	132.4	0.0041
Biochar	12.3	97.6	0.0034
Fe ₃ O ₄ -Biochar	25.8	176.2	0.0052

3.7. Pilot-Scale Underground Treatment Performance

The pilot-scale adsorption unit installed in Shanghai underground sewage treatment facility operated continuously for 60 days. Average pollutant removal efficiencies are presented in Table 8. Temporal variation of COD and heavy metal removal efficiencies during 60-day pilot-scale operation shown in Figure 7.

Table 8. Pollutant removal efficiencies in pilot-scale system

Parameter	Influent (mg/L)	Effluent (mg/L)	Removal efficiency (%)
COD	420	58	86.2
Pb ²⁺	0.095	0.008	91.6
Cd ²⁺	0.012	0.002	83.3
MB	2.5	0.12	95.2
CIP	0.85	0.07	91.8

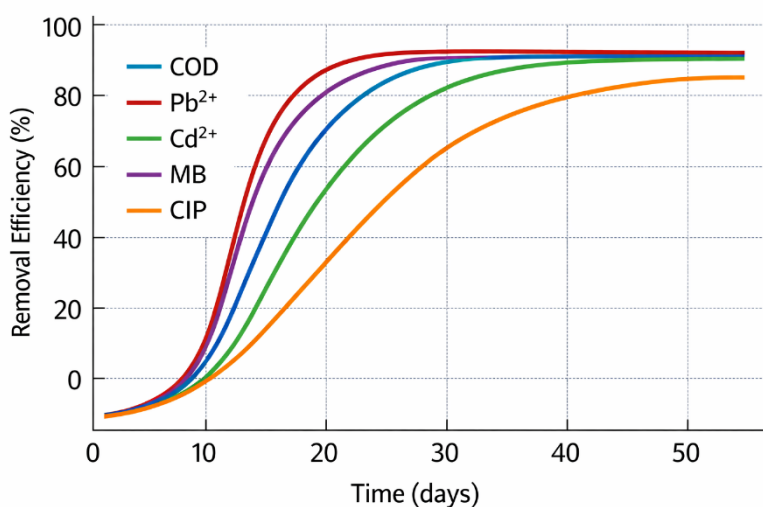


Figure 7. Temporal variation of COD and heavy metal removal efficiencies during 60-day pilot-scale operation

3.8. Adsorbent Regeneration Performance

Adsorbent regeneration studies showed a gradual decrease in adsorption capacity after repeated cycles as in Figure 8. Regeneration efficiency after five adsorption–desorption cycles illustrate in Table 9.

A small loss of the mass of the adsorbents of 4-6 percent was found after five regeneration cycles during thermal regeneration at 400 degC, and this loss can be principally explained by partly combusting any remaining organic matter and eliminating pollutants strongly adsorbed on the adsorbents.

Table 9. Regeneration efficiency after five adsorption–desorption cycles

Adsorbent	Initial q_1 (mg/g)	q_5 (mg/g)	Regeneration efficiency (%)
GAC	85.4	72.3	84.7
Biochar	62.7	48.6	77.5
Fe ₃ O ₄ -Biochar	121.5	103.4	85.1

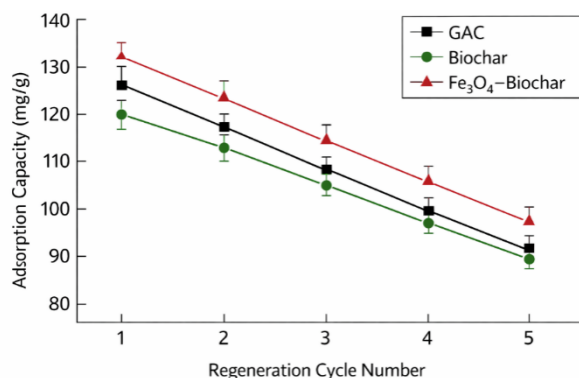


Figure 8. Adsorption capacity versus regeneration cycle number for Pb²⁺ adsorption

4. Discussion

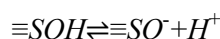
This section interprets the experimental findings from batch, column, and pilot-scale evaluations of advanced adsorbents (GAC, activated biochar, and Fe₃O₄-biochar) for pollutant removal in Chinese urban underground sewage contexts. The focus is made on the mechanisms of chemical adsorption, competition in actual sewage matrices, and the implications of adsorption to engineering processes of underground modular deployment.

4.1. Mechanistic interpretation of adsorption performance across pollutant classes

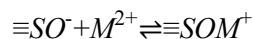
4.1.1. Heavy metals (Pb²⁺, Cd²⁺): surface complexation and ion-exchange dominance

The equilibrium capacities and longer column breakthrough times of the Fe₃O₄-biochar compared to the GAC and untreated biochar show that the binding of metals was improved by oxygenated functional groups and iron-oxide surface sites (Table 2, Table 3, Table 7). The uptake of divalent metals in complex municipal sewage is usually controlled by a combination of (i) attraction to negatively charged surfaces by electrostatic forces, (ii) inner-sphere complexation by oxygen-containing functional groups (-COOH, -OH), and (iii) ligand exchange/complexation on the metal-oxide surfaces. The above mechanisms are in line with the lower zeta potential of Fe₃O₄-biochar at neutral pH and its elevated proportion of surface oxygen functionalities (Table 2).

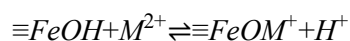
On the carboxyl phenolic groups, the process of deprotonation happens at circumneutral pH common in Chinese urban sewage, creating sites of negative charge that can bind metals:



The binding of metal can then take place through surface complexation (inner-sphere):



In the case of Fe₃O₄ bearing surfaces, hydroxylated iron sites may be complexed with strong ligands by the exchange of ligands:

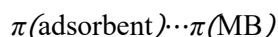


The following mechanisms describe the reason why Fe₃O₄-biochar had the greatest Pb²⁺ capacities and Cd²⁺ capacities and the greatest fixed-bed behavior (Table 3; Table 7). Mechanistic deductions of the like are also frequently found in the literature of adsorption works where iron-oxide functionalization raises the density of reactive hydroxyl sites and improves metal capturing in realistic waters. This is consistent with wider results of urban water remediation issues in China and other adsorption studies in the literature of other countries, in which Fe-oxide modified carbons are superior to unmodified sorbents in the extraction of divalent metals in multi-ionic systems.

4.1.2. Organic dye (methylene blue): pore filling and electrostatic attraction with π - π contributions

Methylene blue (MB) is both cationic and aromatic, therefore adsorption on carbonaceous materials typically occurs; (i) through electrostatic interaction with negatively charged surfaces (at $\text{pH} \geq 6$), (ii) due to filling of pore in the micro/mesopores, and (iii) by p-p interactions between the aromatic dye rings and the graphitic domains of the carbon adsorbents. The good MB adsorption of GAC is attributed to its high surface area and microporosity (Table 2; Table 3) which offers an extensive adsorption site as well as high pore-filling. An activated carbon of high grade has a high microporous volume and thus, higher MB uptake compared to biochar that is functionalized but with low microporous volume.

In the case of aromatic dyes, the conceptual formulation of p-p stacking is:

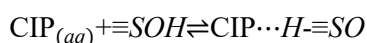


The natural organic matter (NOM) that is present in sewage could inhibit dye adsorption by occupying the pores of adsorbent and preventing adsorption sites. The high MB removal still found at pilot-scale scale implies that pore accessibility was still adequate in the conditions tested, but the slow increase with the number of cycles of regeneration implies that pore fouling or irreversible binding on high-energy sites occurred (Table 9). Similar effects are also observed with actual wastewater systems and are generally more substantial than those observed in artificial matrices [29].

4.1.3. Pharmaceutical (ciprofloxacin): multi-mode adsorption (electrostatic + hydrogen bonding + complexation)

Ciprofloxacin (CIP) is amphoteric and may be cationized, zwitterionic or anionic at various pH levels. At common municipal sewage pH, CIP usually adopts zwitterion behavior so that (i) there are electrostatic attractions/repulsions between the sewage and CIP on the basis of their surface charge, and (ii) the sewage and CIP can hydrogen bond by their oxygenated surfaces, and (iii) CIP can complex with metal-oxide surfaces (particularly in Fe_3O_4 surface). The higher CIP adsorption on Fe_3O_4 -biochar (Table 3) indicate that in addition to hydrophobic distribution to carbon matrices, there were particular interactions with iron-oxide functionalities to the total adsorption.

The Hydrogen bonding may be conceptualized as:



Also, complexation by surfaces (particularly of oxygen-rich substances) may be used to improve polar drug retention. These combined processes are the reasons why CIP adsorption tends to exhibit moderate pH and ionic strength sensitivity and why functionalized materials tend to perform better in actual wastewater conditions compared to the purely porous materials [1,4].

4.2. Why pseudo-second-order kinetics fit better in real sewage

The R^2 values of pseudo-second-order (PSO) were larger when using kinetic fits and on adsorbents (Table 4). In adsorption studies, PSO behavior is generally perceived to be in line with rate control due to chemisorption-like, e.g., electron sharing/exchange, surface complexation, or adsorption on heterogeneous sites, but not by mere diffusion-limited physisorption only [30].

But in actual sewage the interpretation should be careful: PSO may as well arise as the result of concomitant action of boundary-layer diffusion, intraparticle diffusion, and heterogeneous site energetics. Single elementary steps are not often controlling adsorption in municipal matrices. Rather, the kinetic behavior which is observed probably indicates:

- High uptake rate in the early stages because the portion of available sites is high and external movement of the compound is fast.

- Reduced velocity of the approach to equilibrium governed by diffusion of particles and their occupation of sites with lower energy.
- With time, competitive adsorption/fouling which reduces the effective diffusivity and availability of active sites.

The steady rise in fitted equilibrium capacity of the functionalized composite, as well as the better PSO fit, is an indication of the existence of certain high-affinity sites (iron-oxide hydroxyls and oxygenated groups) which bind metals and polar organics with greater strength than does simple physical pore filling.

4.3. Isotherm behaviour and consequences of surface heterogeneity

Langmuir fits were more suitable than Freundlich fits to Pb²⁺ adsorption (Table 5) indicating that at the concentration ranges and, in the conditions of the study, adsorption was approaching monolayer coverage behavior of a collection of essentially equivalent sites. This may be observed in engineered adsorbent even when the surfaces are heterogeneous provided high-affinity sites prevail within the range of tests.

Engineering wise, a larger Langmuir q_{max} of Fe₃O₄- biochar implies a larger design capacity per unit mass of metal polishing steps, which is useful in systems that are limited in volume like underground systems. Simultaneously, heterogeneity and the existence of multi-energy binding sites are still indicated by Freundlich parameters (K_F and $1/n$). Real sewage is likely to be heterogeneous because of the complexity of adsorbent surfaces and competing organic matter adsorption. Hence, it should not just be on one-model parameter that practical design is based, and breakthrough performance needs to be considered as the foundational design of continuous systems.^[31]

4.4. Thermodynamic explanation: endothermic behaviour as well as binding power

The positive value of enthalpy change ($\Delta H_{deg} > 0$) and the negative value of Gibbs free energy ($\Delta G_{deg} < 0$) observed in Pb²⁺ adsorption on Fe₃O₄-biochar (Table 6) represent a spontaneous and endothermic adsorption reaction within the temperatures range, tested. The trend is usually affiliated with adsorption processes that take advantage of higher mobility of ions and enhanced interaction/complexation at higher temperatures.

Equilibrium constants (K) are normally calculated to give thermodynamic parameters:

$$\Delta G^\circ = -RT \ln K$$

and the Van't Hoff relationship:

$$\ln K = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R}$$

In actual sewage, the chemical binding and the matrix effects of ionic strength, complexing ligands, NOM, are combined into a single apparent constant, K . Thus, the thermodynamic values are to be considered as system level measures of adsorption propensity and not rather intrinsic constants. The resulting temperature dependence of the observed adsorption can still give valuable design information: operating temperature systems underground can be predictable in their performance and in colder climates the seasonal variations may not be easy to handle in the polishing of metals.

4.5. Effects of competitive adsorption, ionic strength and sewage matrix

Chinese municipal sewage, in contrast to synthetic wastewater, has large quantities of rival cations (Ca²⁺, Mg²⁺, Na⁺), anions (Cl⁻, SO₄²⁻, HCO₃⁻, PO₄³⁻), dissolved organic matter, surfactants, and colloids. These elements impose several viable restrictions:

4.5.1. These are competition of binding sites

Background ions containing a different valence compete directly with the target metals with a negatively charged and complexation site, decreasing the effective capacity. In the same way, NOM also competes with carbonaceous pore structures and functional groups and lowers the availability of adsorption to dyes and pharmaceuticals. The effects of competition are generally of the lower affinity adsorbents and for pollutants whose adsorption is predominantly due to electrostatic attraction, as opposed to strong inner-sphere complexation [32].

4.5.2. Solution reduction of free metal ions by complexation

Metals can form aqueous complexes of carbonate, chloride or dissolved organic ligands in sewage. This decreases available free metal activity to adsorb and has the ability to change apparent adsorption capacity. This can be partially overcome by iron-oxide functionalization to offer high binding sites which is expected to be in line with Fe₃O₄-biochar sustaining better performance during column operation and pilot-scale operation (Table 7; Table 8).

4.5.3. Fouling and pore blockage

Micropores of activated carbons may be physically blocked by NOM and colloidal materials and result in a coated metal-oxide site, which is less reactive. The fact that there is a gradual irreversible fouling, incomplete desorption of strongly bound species and likely structural changes occurring during thermal/chemical regeneration all explain why the observed decrease with cycles of regeneration (Table 9). Fouling resistance is as significant as initial adsorption capacity in underground systems that may have limited access to the system and limited time to perform maintenance.

4.6. Implications on pilot-scale and underground engineering

4.6.1. The operational attractiveness of Fe₃O₄-biochar underground

In underground treatment the limitations are less severe than in the above-ground plant: restricted space, restricted ventilation, extreme attention to odors, restricted access to operators, and exaggerated sensitivity to pressure drops and clogging. Under these conditions, Fe₃O₄-biochar has a number of advantages in engineering:

- Increased ability of metals and polar organics reduces the rate of bed replacement (Table 3, Table 7).
- The long breakthrough time enhances the stability of the operation and decreases the monitoring load (Table 7).
- Separation, media handling, and accidental handling of carryover can be simplified through potential magnetic handling (in case it was designed to handle it).
- Improved tolerance to matrix complexity as a result of enhanced binding sites, which is in line with indefinitely pilot-scale removal efficiencies (Table 8).

4.6.2. Column design issues: bed depth, EBCT and pressure drop

The design should take into consideration the anti-clogging and stable hydraulics that will be used underground. Key considerations include:

Empty bed contact time (EBCT): must be able to balance between removal and footprint limitations.

Pre-filtration: to guard against adsorption bed suspension and colloids.

Lead-lag set up: permits continuous operation the time it is regenerating and provides a defense against natural breakthrough.

Pressure drop Test: necessary in a case of hydraulic failure in underground setups.

The column-model parameters (Table 7) provide a practical starting point for scale-up, but full-scale designs should be based on site-specific sewage composition, seasonal variation, and operational constraints.

4.6.3. Regeneration strategy in underground facilities

Regeneration is not simply an artificial convenience of laboratories; it dictates lifecycle economics. The moderate-to-high regeneration efficiencies (Table 9) indicate regeneration can be reused, and the decrease over cycles would imply that regeneration procedures should be optimized to manage:

- irreversible NOM fouling,
- partial desorption of strongly-complexed metals,
- thermal degradation of pore structures (when treated at a high temperature),
- secondary waste-products of chemical regeneration.

The cartridge-like off-site regeneration or a centralized off-site cartridge regeneration of underground plants will be practical to ensure minimal underground chemical handling but with continuous operation.

4.7. Comparison of previous China-based and global adsorption results

The observed tendencies comply with general observations in adsorption-based systems of polishing wastewater: (i) the use of modified biochars increases the efficiency of adsorption compared to raw chars because of increased porosity and surface chemistry, (ii) functionalization of iron-oxide increases the capacity of heavy-metal and polar organic uptake compared to raw systems because of specific complexation sites, and (iii) real sewage matrices works worse than synthetic system because of competition and fouling.^[33] Importantly, the present pilot-scale observations (Table 8) support the view that adsorption units are viable as tertiary polishing steps in compact underground systems, provided that hydraulic protection (pre-filtration), operational configuration (lead-lag), and regeneration logistics are designed appropriately.

On the engineering front, economic viability of adsorption systems is determined by the cost of adsorbents, adsorbent regeneration ability, and adsorbent active life. Agricultural residues can be used as raw materials in the manufacturing of biochar-based adsorbents which can be of cheaper production cost as opposed to commercial activated carbon. Moreover, the regeneration efficiency achieved in this research (>77) is relatively large implying that many reuse cycles can be performed which would considerably decrease the cost of operations. However, economic evaluation on the large scale such as the preparation of adsorbent, the energy used in regeneration and waste disposal ought to be considered in the future in full-scale studies.

4.8. Engineering takeaways from the mechanism evidence

Based on the mechanistic interpretation of kinetics, isotherms, thermodynamics, and pilot-scale performance, the following engineering conclusions are supported:

- **Metal-oxide functionalization is justified** when heavy metals and polar micropollutants are key compliance drivers, because it increases strong binding sites and improves stability under matrix competition.
- **Activated carbon remains highly effective** for aromatic dye-like organics where pore filling and π - π interactions dominate, but it can be more sensitive to fouling in real sewage.
- **Underground deployment requires fouling-first thinking:** capacity alone is insufficient without pre-treatment and maintenance planning.^[34]
- **Pilot-scale performance is the decision-maker:** batch capacities and model fits are necessary but not sufficient to determine practical EBCT and replacement schedules ^[35,36].

5. Conclusion

This study investigated the engineering application of advanced adsorbents for pollutant removal in urban underground sewage treatment systems using experimental data derived from Chinese municipal wastewater and pilot-scale treatment units. Activated biochar, and Fe₃O₄-functionalized biochar were sequentially tested by using batch adsorption, continuous-flow column tests, and pilot-scale operational tests. Kinetic, isotherm, and thermodynamic models were used to interpret the adsorption mechanisms to give mechanistic understanding on the process of removal of pollutants in complex sewage matrices.

The findings indicated that Fe₃O₄-biochar had better adsorption capacity with the heavy metals and pharmaceutical contaminants because of the existence of large amounts of the surface functional groups and iron-oxide active sites that enabled the adsorption process of the electrostatic attraction, surface complexation, and ligand exchange. Activated carbon explained the greatest adsorption capacity of aromatic dye compounds, which is mainly caused by filling the pores and π - π interactions in its high-microporous structure. Altered biochar gave a mediocre performance and this is a testament of the need to modify the surface and activate it in order to maximize adsorption action.

Kinetic studies showed that adsorption was of pseudo-second-order behaviour with all the adsorbents tested with the exception that chemisorption-like reactions and heterogeneous surface reactions was predominant in the overall adsorption rate. The Langmuir isotherm model was more effective in characterizing the equilibrium, suggesting that under the concentration regimes experimented, the adsorption behaviour of the monolayer was monolayer-based and Freundlich parameters indicated the heterogeneity of the surfaces in the real sewage conditions. Thermodynamic study showed that the adsorption process was endothermic and spontaneous and the higher the temperature the greater the adsorption rate of the pollutants.

The viability of using adsorption unit as tertiary polishing processes in small-scale underground sewage treatment systems was ascertained by continuous-flow column trials and pilot-scale underground treatment. Fe₃O₄-biochar composite had the longest breakthrough time and the highest pollutant removal efficiencies in realistic hydraulic conditions where it was determined to have over 85% COD removal and over 90% heavy metal and pharmaceutical removal during 60 days pilot scale. Experiments with regeneration showed that adsorbents could be used in repeated cycles even though the performance dropped gradually with time because of the adsorbent fouling and irreversible adsorption, thus highlighting the importance of regeneration strategies optimization.

In the engineering point of view, the results suggest that sophisticated adsorbents, especially metal-oxide functionalized biochar compounds, shows promising potential for application in compact urban underground sewage treatment systems in augmenting the elimination of pollutants in space limited. Nonetheless, the actual effects of sewage matrix such as Competitive adsorption, natural organic matter fouling, and complex ion chemistry greatly affect adsorption performance and need to be taken into account in the design of the systems. The pre-filtration, lead-lag column design, and modular adsorbent replacement models should be suggested to maintain stable working conditions in the underground conditions over the long period.

The results are valuable in the understanding of the process of adsorption and engineering efficacy of advanced adsorbents under feasible sewage conditions and offer the possibilities of using them as tertiary polishing units in small urban wastewater systems.

All in all, the current study offers both mechanical and engineering background to the concept of adsorption to cleanse the pollutants in the urban underground sewage treatment system and contributes to the real-world implementation of the highly developed adsorbents in the miniaturized municipal wastewater treatment system.

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Conflict of interest

The authors declare no conflict of interest

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