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*Research Paper*

# Pharmaceutical Wastewater Treatment Using Activated Carbon in a Fixed-Bed Column

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## ABSTRACT

Though beneficial for health, pharmaceuticals have emerged as persistent environmental contaminants over recent decades. Discharges from wastewater treatment plants are a primary source of these pharmaceuticals in natural water bodies, as conventional treatment methods often fail to remove them effectively. Fixed-bed columns with GRANULATED ACTIVATED CARBON (GAC) are widely used in water treatment facilities to eliminate organic micropollutants; however, their efficiency in systematically removing pharmaceutical waste from wastewater still needs to be explored. This study evaluates the performance of a GAC fixed-bed adsorption column for removing ibuprofen, paracetamol, and ciprofloxacin from wastewater, focusing on contact time as a critical parameter.

**Key Findings:** Initial concentrations of ibuprofen, paracetamol, and ciprofloxacin in the wastewater were 0.068 mg/L, 0.08486 mg/L, and 0.068 mg/L, respectively. After 45 minutes, the column achieved removal efficiencies of 100% for ibuprofen, 92.8% for paracetamol, and 67.6% for ciprofloxacin, with no significant changes in concentrations observed at 90 minutes. These results suggest that GAC fixed-bed adsorption columns can rapidly and effectively reduce pharmaceutical concentrations in wastewater, with ibuprofen showing complete removal in under an hour. This study contributes to understanding GAC's effectiveness in treating pharmaceutical wastewater. It highlights the potential of optimised contact time for efficient removal, underscoring its viability as an industrial treatment option.

***Keywords:*** Pharmaceutical wastewater; emerging contaminants, adsorption

## 1. INTRODUCTION

Emerging contaminants, including pharmaceutical compounds, represent a growing environmental and public health challenge due to their persistence and bioaccumulation potential. Pharmaceuticals are especially concerning, because of their poor biodegradability, stability, and diverse chemical structures, enabling them to accumulate in water bodies, where they impact both aquatic ecosystems and human health (de Andrade et al., 2020; Feizi et al., 2021). Although typically present at trace levels, often in the parts-per-billion (ppb) to parts-per-million (ppm) range, these contaminants can adversely affect ecosystems by disrupting endocrine function in wildlife and promoting antibiotic resistance (de Andrade et al., 2018; Manisalidis et al., 2020).

Conventional wastewater treatment plants often struggle to remove these persistent compounds effectively resulting in detectable levels of pharmaceuticals in effluent wastewater and, subsequently, in surface and even drinking water (Bijlsma et al., 2021; Peng et al., 2018). Research indicates that conventional treatment methods typically reduce pharmaceutical contaminants to levels only as low as 1 µg/L, which may still pose risks to environmental and human health (Eniola et al., 2022; Marson et al., 2022).

Among various treatment techniques, adsorption-based methods offer a promising solution due to their simplicity, high efficiency, and ability to prevent hazardous byproduct formation. Activated carbon is widely used in adsorption processes, with granular activated carbon favoured in industrial applications for its reusability and effectiveness in continuous flow systems like fixed-bed columns (Rathi & Kumar, 2021; Vinayagam et al., 2022). Fixed-bed GAC columns are already widely employed in drinking water facilities to eliminate organic micropollutants, and their scalable design makes them suitable for removing contaminants in industrial wastewater applications (de Franco et al., 2017; Patel, 2020).

However, while granulated activated carbon has been studied extensively for removing various organic micropollutants, limited research exists on its specific effectiveness in removing pharmaceutical contaminants in fixed-bed systems, especially under continuous flow conditions that mimic real-world applications. Key parameters affecting granulated activated carbon performance in fixed-bed systems include contact time, flow rate, bed depth, and initial contaminant concentration, which can influence adsorption efficiency (Bijlsma et al., 2021; Sylvia et al., 2018).

## 2. MATERIALS AND METHODS

### 2.1 Reagents

The pharmaceuticals used for adsorption experiments were ibuprofen (99.92% purity, ATABAY-TURKEY), paracetamol (99.3% purity, BASF CORPORATION), and ciprofloxacin (99.3% purity, Chemo Pharmaceuticals). To ensure high purity, all solutions were prepared using deionized water from a Milli-Q Millipore system (Milli-Q plus 185). Granular activated carbon, used as the adsorbent material in the fixed-bed column, was procured from PT. Indo Carbon Primajaya.

### 2.2 Effluent Characteristics

Pharmaceutical wastewater was collected from a manufacturing facility in Riyadh Industrial City and characterized before treatment, Initial pharmaceutical concentrations were measured as ibuprofen at 0.068 mg/L, paracetamol at 0.08486 mg/L, and ciprofloxacin at 0.068 mg/L. These characteristics established a baseline for evaluating the GAC-based fixed-bed column's efficiency in removing pharmaceutical contaminants.

### 2.3 Fixed-Bed Column Setup and Experimental Conditions

The experimental setup consisted of a PVC-based fixed-bed adsorption column with an internal diameter of 10 cm and a height of 80 cm. The Fixed-Bed column was packed with Granular Activated Carbon up to 60% of its volume, creating a densely packed layer designed to maximize contact between the pharmaceutical-laden influent and the adsorbent surface area.

A 12 V, 60 W high-pressure pump was used to maintain a continuous flow rate of 1 GPM (gallon per minute), regulated by a flow meter and control valve placed externally along the influent line. The influent solution was transferred through the setup by a pump to ensure a steady, controlled flow. Samples were collected from the effluent at intervals of 0, 45, and 90 minutes to assess the effect of contact time on the adsorption efficiency of the granulated activated carbon.

Figure 1 illustrates the fixed-bed column setup. It shows the influent entering the top of the column, passing through a dense, granular GAC layer, and exiting as effluent. Each component, including the flow meter, valve, and pump, is clearly marked along the line to depict the arrangement used to achieve consistent flow and maximize adsorption interaction.

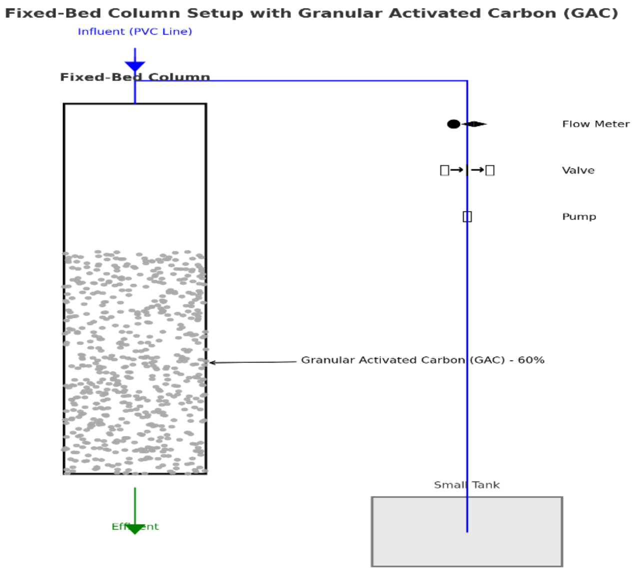

**Figure 1: Fixed-Bed Column Setup with Granular Activated Carbon (GAC))**

## 2.4 Sampling and Analytical Procedures

The concentration of each pharmaceutical compound was determined using high-performance liquid chromatography (HPLC) following the guidelines outlined in the British and United States Pharmacopoeia:

1. **Ibuprofen Analysis**
   Following British Pharmacopoeia guidelines, 20 mg of ibuprofen was dissolved in 2 mL of acetonitrile and diluted to 10 mL with mobile phase A (0.5 mL phosphoric acid, 340 mL acetonitrile, and 600 mL deionized water, diluted to 1000 mL). Detection was set at 214 nm.
2. **Paracetamol Analysis**
   Using British Pharmacopoeia procedures, 50 mg of paracetamol was dissolved in 100 mL water and further diluted with methanol for a final concentration of 0.1 mg/mL. The detection wavelength was 254 nm, and the flow rate was 0.9 mL/min.
3. **Ciprofloxacin Analysis**
4. Based on United States Pharmacopoeia, 10 mg of ciprofloxacin was dissolved in 100 mL of water with ammonium hydroxide and diluted for analysis. After further dilutions, the final solution was analyzed at a wavelength of 278 nm.

All samples were filtered before HPLC analysis to ensure accuracy and precision.

## 2.5 Data Analysis

The removal efficiency (RE) for each pharmaceutical compound was calculated to evaluate the performance of the GAC fixed-bed column over time. Removal efficiency was determined using the following equation:

$$RE(\%) = \left( \frac{C_{initial} - C_{final}}{C_{initial}} \right) \times 100$$

Where:

(C initial) is the initial concentration of the pharmaceutical at time 0.
(C final) is the concentration of the pharmaceutical at each sampling time point (45 or 90 minutes).

## 3.1.1 Behavior of Ibuprofen at Different Times

The initial concentration of ibuprofen at 0 minutes was 0.068 mg/L, which dropped to zero within 45 minutes, as shown in Table 1. This complete removal demonstrates high adsorption efficiency and rapid kinetics on GAC, likely due to strong hydrophobic interactions between ibuprofen molecules and the GAC surface. The rapid removal rate indicates that ibuprofen’s molecular structure is highly compatible with GAC adsorption, making it particularly effective in systems utilizing GAC as an adsorbent.

**Table 1 Concentration of Ibuprofen at different times:**

| Time | At 0 min | After 45 min | After 90 min |
|---|---|---|---|
| Concentration of ibuprofen | 0.068 | 0.00 | 0.00 |

### 3.1.2 Behavior of Paracetamol at Different Times

The paracetamol concentration decreased from 0.08486 mg/L to 0.00613 mg/L within the first 45 minutes, stabilizing without further reduction at 90 minutes, as shown in Table 2. This rapid decrease, followed by an equilibrium phase, suggests that paracetamol initially binds quickly to GAC but does not fully saturate due to moderately weaker adsorptive interactions compared to ibuprofen.

**Table 2 Concentration of Paracetamol at different times:**

| Time | At 0 min | After 45 min | After 90 min |
|---|---|---|---|
| Concentration of paracetamol mg/L | 0.08486 | 0.00613 | 0.00613 |

### 3.1.3 Behavior of Ciprofloxacin at Different Times

The concentration of ciprofloxacin dropped from 0.068 mg/L to 0.022 mg/L within the first 45 minutes and remained at this level for up to 90 minutes (Table 3). This slower, partial removal suggests that ciprofloxacin’s larger, polar molecular structure interacts less effectively with GAC’s mostly nonpolar surface, leading to lower adsorption efficiency than ibuprofen and paracetamol.

**Table 3 Concentration of Ciprofloxacin at different times:**

| Time | At 0 min | After 45 min | After 90 min |
|---|---|---|---|
| Concentration of ciprofloxacin mg/L | 0.08486 | 0.022 | 0.022 |

## 3.2. DISCUSSION

### Adsorption Efficiency and Kinetics

The GAC fixed-bed column showed strong adsorption efficiency, especially for ibuprofen, which was entirely removed within 45 minutes. This quick removal likely comes from ibuprofen's hydrophobic nature and smaller molecular size, which interact well with the nonpolar surface of GAC. Smaller, hydrophobic molecules often experience faster adsorption on activated carbon due to van der Waals forces and hydrophobic interactions. These results align with Hamed et al. (2020), supporting GAC's practical effectiveness for pharmaceuticals like ibuprofen.

Differentiated Adsorption Among Pharmaceuticals

The differences in removal efficiency for ibuprofen, paracetamol, and ciprofloxacin highlight how molecular structure affects adsorption performance. Ciprofloxacin's lower adsorption efficiency may stem from its larger size and polar functional groups, which create steric hindrance and lead to weaker interactions with GAC. Studies by Marzbali and Esmaieli (2017) also observed that polar pharmaceuticals often need longer contact times or modified adsorbents for optimal adsorption. Paracetamol's moderate removal efficiency indicates that it initially interacts well with GAC but may not bind as strongly as ibuprofen. These results suggest that adsorbent modifications might enhance performance for more complex compounds, while GAC is effective for specific structures.

Comparison with Similar Studies

The complete removal of ibuprofen within 45 minutes aligns with findings by Cyr et al. (2002), which showed that GAC effectively adsorbs small, hydrophobic contaminants in fixed-bed columns. Studies have shown that parameters like flow rate and influent concentration can impact efficiency, especially for larger, polar pharmaceuticals. This study's configuration achieved high removal rates without major changes, which confirms GAC's usefulness for treating hydrophobic pharmaceuticals in wastewater. For more polar compounds, like ciprofloxacin, other modifications or alternative adsorbents may be needed, as Patel (2019) and Igwegbe et al. (2020) suggested.

Mass Transfer Zone and Column Efficiency

The fixed-bed column includes a MASS TRANSFER ZONE (MTZ) where adsorption interactions are most active before saturation occurs. In this study, the quick adsorption of ibuprofen and paracetamol within the MTZ suggests effective adsorption before the bed reaches saturation. Ciprofloxacin's partial removal, however, shows that longer contact time or additional column stages might improve adsorption for larger or more polar molecules. Ekpete et al. (2011) noted that MTZ length and efficiency depend on flow rate, bed depth, and adsorbate properties.

Practical Implications and Optimization for Full-Scale Applications

The rising use of pharmaceuticals worldwide underscores the need for efficient, affordable wastewater treatment. While advanced tertiary treatments are costly, GAC offers a more accessible solution. This study shows that GAC is inexpensive and effective for many pharmaceutical compounds. Although the GAC column efficiently removed hydrophobic pharmaceuticals like ibuprofen, optimising the system could allow it to target a broader range of contaminants.

To achieve better results, we need to concede that some changes, such as using sequential column configurations, altering influent concentrations, or changing bed depth, could help improve adsorption for compounds with less affinity for GAC. Regular column regeneration could sustain high adsorption rates, preventing GAC saturation and maximising efficiency. Cyr et al. (2002) found that GAC regeneration extends column life and maintains performance, making it an economically viable choice for long-term wastewater treatment.

## 4. CONCLUSION

The global and rapid rise in pharmaceutical use has increased environmental and health risks, especially due to emerging contaminants in wastewater. Advanced tertiary treatments are often expensive, but activated carbon offers a practical, affordable solution for removing contaminants. This study tested (GAC) in a fixed-bed column to remove ibuprofen, paracetamol, and ciprofloxacin from pharmaceutical wastewater.

The results show that ibuprofen, a smaller and more hydrophobic pharmaceutical, was entirely removed within 45 minutes. Paracetamol also adsorbed well, decreasing from 0.08486 mg/L to 0.00613 mg/L in 45 minutes and then stabilising. Ciprofloxacin was only partially absorbed, dropping from 0.068 mg/L to 0.022 mg/L. This lower efficiency is likely due to ciprofloxacin's larger, polar structure, which interacts less favourably with GAC's nonpolar surface. These findings suggest that GAC is highly effective for hydrophobic pharmaceuticals but may need modifications or alternative adsorbents for more complex compounds.

The study supports using GAC as a practical and cost-effective option for removing specific pharmaceutical contaminants from wastewater. Future research should explore adjustments to flow rates, influent concentrations, and bed depths to broaden GAC's application to other pharmaceutical compounds. Modified or composite adsorbents and multistage column setups could further improve treatment for pharmaceutical wastewater.

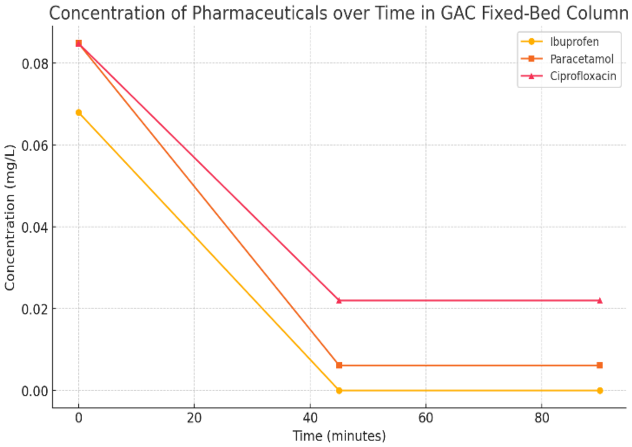

**Figure 2: Concentration of Pharmaceuticals over time.**

## Disclosure

I have nothing to disclose and no conflict of interest.

## Data Availability

The date will be available through the journal, but it is not available through other repositories.

## *Declaration by Authors*

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