

**REMOVAL OF PHARMACEUTICAL COMPOUNDS MEFENAMIC ACID AND  
NAPROXEN FROM AQUEOUS MEDIA BY USING GRANULAR ACTIVATED  
CARBON (GAC) IN A FIXED BED COLUMN SYSTEM**



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AND NAPROXEN FROM AQUEOUS MEDIA BY USING GRANULAR ACTIVATED  
CARBON (GAC) IN A FIXED BED COLUMN SYSTEM**



A thesis submitted to Bahria University, Islamabad in partial fulfillment of the requirement for  
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## ABSTRACT

Pharmaceutical compounds including non-steroidal anti-inflammatory drugs such as mefenamic acid and naproxen are increasingly detected in water bodies globally, raising serious concerns about their ecological and public health impacts. Conventional wastewater treatment systems are largely ineffective at removing these micropollutants from water sources.

This study designed and evaluated a simple gravity-driven granular activated carbon fixed-bed adsorption column for the removal of mefenamic acid and naproxen from synthetic aqueous solutions. The column was constructed from a plastic syringe packed with layers of cotton, gravel, silica sand, and GAC with dimensions of 1 cm gravel, 4 cm sand, and 4 cm GAC respectively.

Stock solutions of 1000 mg/L were prepared from pharmaceutical tablets and diluted to working concentrations of 5 mg/L, 25 mg/L, and 50 mg/L. The effects of initial concentration, solution pH (acidic and basic conditions), contact time, and water matrix type (distilled and tap water) on removal efficiency were systematically investigated. All samples were analyzed by UV-Visible spectrophotometry at 270 nm.

The column achieved a maximum removal efficiency of 97.1% for mefenamic acid at 50 mg/L in distilled water. Removal efficiency increased with higher initial concentration and longer contact time for both pharmaceutical compounds. Mefenamic acid showed preferential removal under acidic conditions while naproxen performed marginally better under basic conditions above its pKa charge state. Tap water experiments showed reduced removal efficiency compared to distilled water due to competitive adsorption by dissolved matrix substances.

Mefenamic acid consistently outperformed naproxen under all experimental conditions due to its higher hydrophobicity and stronger affinity for the activated carbon surface. The findings contribute to SDG 6 and SDG 3 by demonstrating the technical feasibility of a low-cost, electricity-free approach to pharmaceutical water treatment.

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## LIST OF ABBREVIATIONS

|       |                                                        |
|-------|--------------------------------------------------------|
| API   | Active Pharmaceutical Ingredient                       |
| AMR   | Antimicrobial Resistance                               |
| GAC   | Granular Activated Carbon                              |
| HPLC  | High-Performance Liquid Chromatography                 |
| MA    | Mefenamic Acid                                         |
| NPX   | Naproxen                                               |
| OECD  | Organization for Economic Co-operation and Development |
| pH    | Potential of Hydrogen                                  |
| PMC   | PubMed Central                                         |
| SDG   | Sustainable Development Goal                           |
| UV    | Ultraviolet                                            |
| WHO   | World Health Organization                              |
| WWTP  | Wastewater Treatment Plant                             |
| WWTPs | Wastewater Treatment Plants                            |

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# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Pharmaceutical Contaminants in Wastewater

Aquatic ecosystems have been exposed to a complex cocktail of synthetic chemical compounds in the context of global escalation in the production and consumption of pharmaceuticals in the 21st century, making it one of the most critical emerging environmental issues of the new century (Khalidi-Idrissi et al., 2023). In total, these substances are known as Active Pharmaceutical Ingredients (APIs) and cover a wide range of therapeutic classes such as Analgesics, Antibiotics, Hormones, Psychotropic drugs and much more, which are crucial for modern human and veterinary medicine (Moulika et al., 2026).

Pharmaceuticals once administered are not completely metabolized by the human body. Studies show that a large percentage of these compounds is eliminated in the parent form, or as active metabolites via municipal sewage systems (Thakur et al., 2023). Wastewater treatment plants (WWTPs) are thus becoming important point sources of these micropollutants, as the main part of the natural water cycle encounters human activity. Generally, the treatment plants are designed to remove bulk organic matter and nutrients from water with the activated sludge system and do not have any special mechanisms to neutralize these persistent synthetic molecules (Cacus-Makowska et al., 2023; Moulika et al., 2026; Singh et al., 2024).

Furthermore, these substances are released constantly, and the continuous release of these substances can also be termed as "pseudo-persistence", in which the substances are continually replenished without reaching ecologically relevant concentrations below the release level. (Kummerer, 2021) Pharmaceutical waste may be released into the environment as a result of mismanagement of unused drugs in domestic wastewater and in landfills, and agricultural runoff of wastewater contaminated by manure (Uddin et al., 2024).

A high concentration of these compounds in the water bodies can cause serious long-term health and environmental impacts ranging from nanograms to micrograms per liter (ng/L to µg/L) (Moulika et al., 2026; Patel et al., 2025). Chronic exposure to these low dose pharmaceutical mixtures is known to cause endocrine disruption, behavioral changes and reduced reproductive fitness in various aquatic organisms (MDPI, 2025; Singh et al., 2024). In addition, subtherapeutic

levels of antibiotics in wastewater are a major contributor to the global antimicrobial resistance (AMR) pandemic that helps develop and transfer AMR genes in microbial communities (Barkha Madhogaria et al., 2024; Singh et al.,). It was pointed out that the advanced tertiary treatment technologies such as adsorption on GAC were needed to effectively remove the impacts of pharmaceutical contaminations from the effluents before discharge to receiving water bodies (Ashraf et al., 2024; Moulika et al., 2026).

## **1.2 NSAIDs are Emerging Contaminants**

It is worth to note that non-steroidal anti-inflammatory drugs (NSAIDs) are an important group of emerging contaminants, as they are widely used globally, have almost no metabolism in humans and are stable in aquatic environment (MDPI, 2026; PMC, 2026). These are commonly used in veterinary medicine and human medicine for pain relief, to reduce fever, and as medicine for inflammatory disorders, and are readily available, either legally or illegally and often sold over the counter, with high levels of consumption.

The emerging designation is based on several important factors:

NSAIDs are continuously added to wastewater systems unlike traditional pollutants which can have a single point of discharge. If the half-life of the individual compounds is relatively short at the conditions, then their constant excretion and disposal will mean that they will be found in ecologically important concentrations, which are termed pseudo-persistence.

Currently, the conventional wastewater treatment plant (WWTP) is mostly activated sludge plants with BOD (biochemical oxygen demand) and TSS (total suspended solids) treatment. But, many NSAIDs have a complex chemical structure and most of these agents exist in the aqueous phase, which means that these drugs are not suitable for these biological treatment systems (He et al., 2017). Removal efficiencies of common NSAIDs can vary from very low to very high and high amounts of these drugs can persist in the surface waters of conventional plants (He et al., 2017).

NSAIDs are designed to have biological activity at low therapeutic levels. In aquatic ecosystems with concentrations of these compounds, they can cause chronic, non-lethal effects. Exposure to oxidative stress, endocrine disruption and reproductive effects have been associated with several aquatic organisms, such as fish and mollusks (He et al., 2017).

Some NSAIDs are structurally stable, and naturally resistant to degradation process such as microbial decomposition (MDI) and photo degradation (MDP), especially those with bicyclic aromatic rings. If they can pass through the treatment barriers, they can be deposited in the sediment and waters and can be passed up the food chain (He et al., 2017).

Monitoring and removal of NSAIDs have become increasingly a priority in the scientific world and in regulatory authorities to combat these problems. The adoption of the most up-to-date treatment technologies, such as the granular activated carbon (GAC) or the advanced oxidation processes (AOPs), is now considered crucial to mitigate the risks from these pollutants to biodiversity and public health (Mansouri et al., 2021).

### **1.3 Naproxen and Mefenamic Acid as Emerging Contaminants**

They are referred to as "pseudo-persistent" because they are continually released into aquatic systems at a rate greater than they are removed by natural processes. Pharmaceutical plant effluents, surface water, as well as ground water (He et al., 2017). often contain higher amounts of some non-steroidal anti-inflammatory drugs (NSAIDs).

#### **1.3.1 Global and Local Occurrence**

These compounds are ubiquitous, they are closely associated with human consumption and are associated with limitations in the conventional wastewater treatment plant (WWTP) treatment of wastewater because they are hard to remove from the wastewater (He et al., 2017).

The studies carried out in other international urban areas have consistently found naproxen and mefenamic acid in surface water. Not quickly removed after release) as they are stable in nature (photochemical half-lives of naproxen in surface waters are around 10 to 27 days). (Tixier et al., 2003).

In these areas (fast-urbanizing metropolitan areas in Pakistan), these drugs are more likely to be discovered in municipal drains and river systems. Due to high population density and the widespread use of partially or untreated sewage treated local water bodies, concentration of these NSAIDs in local effluents can be similar or even higher than found in developed countries (Ali et al., 2019)

## **1.4 Ecotoxicological Significance**

These concentrations may appear low but NSAIDs are biologically potent and therefore present a high risk to the environment. They are formulated to target certain enzymatic pathways (such as cyclooxygenase inhibitors) common in many aquatic species (He et al., 2017).

Developmental and physiological effects have been reported in non-target species (fish, mollusks and crustaceans) exposed chronically to naproxen and mefenamic acid.

### **1.4.1 Documented Effects**

oxidative stress, histopathological changes in the liver and kidney tissues of fish, impaired reproductive success (decreased fecundity, growth rates) (He et al., 2017).

They are persistent, and they bioaccumulate in primary consumers (zooplankton, snails etc.), a risk that might result in triphasic transfer and influence higher triphasic consumers as well as food web interactions in the water column (He et al., 2017).

There was no significant trend detected for monitoring data and they are summarized below. The following summarizes monitoring results.

It is difficult to monitor these substances and advanced analytical methods such as UV spectrophotometry or liquid chromatography-mass spectrometry (LC-MS) are often necessary to accurately measure their presence. These residues have been detected continuously, and this means that there is an important water quality management challenge: these chemicals do not cause high short-term mortality, but they can cause long-term damage to aquatic ecosystems and human health and thus necessitate immediate actions of water treatment to minimize their impact on aquatic biodiversity and human health (MDPI, 2026; ResearchGate, 2026).

## **1.5 The conventional wastewater treatment plant (WWTP) can only process a small number of materials:**

The activated sludge process is the most frequently used process in traditional wastewater treatment plants (WWTPs) and wastewater treatment plants are traditionally designed for removal of bulk organic matter (biochemical oxygen demand or BOD), suspended solids (TSS) and nutrients (N and P) (Cacus-Makowska et al., 2023). These systems are highly effective in controlling common primary wastewater parameters but are not specifically engineered to treat

emerging contaminants in primary wastewater with unique physicochemical properties, like non-steroidal anti-inflammatory drugs (NSAIDs) such as naproxen and mefenamic acid.

These conventional systems have the following general problems in pharmaceutical residue reduction:

The molecular structure of many pharmaceutical compounds tends to be complex and stable, often with aromatic compounds that are not very susceptible to the microbial process in the activated sludge system (MDPI, 2026; Singh et al., 2024). Conventional reactors are home to bacteria that are used to digest easily biodegradable sources of carbon, and it often fails to break down synthetic pharmaceutical molecules and may even be inhibitory to these microorganisms at certain concentrations.

Removing organic contaminants in secondary treatment is often a combination of biodegradation and partitioning to sludge (Physicochemical Incompatibility). The other two drugs, naproxen and mefenamic acid, however, are relatively hydrophilic (He et al., 2017).as opposed to being more hydrophobic. This indicates that they do not get sufficiently removed during sludge washing but are discharged directly to receive water.

Based on empirical data, it is found that the removal efficiencies in the range of 10–30% are generally not high enough in conventional WWTPs to ensure environmental non-accumulation of these compounds (Cacus-Makowska et al., 2023; Singh et al., 2024). The contact time between the compounds and the biomass is too short, and therefore the degradation of those compounds will have little significance, which will further increase the bypass of those compounds in the system.

The effectiveness of the microbial community is highly susceptible to changes in the influent, pH changes and temperature changes. Further it is difficult for these plants to remove trace-level pharmaceutical contaminants, and sometimes these contaminants are even partially released to the effluent, when plants are already stressed from high inflows and/or complex industrial loads (Ashraf et al., 2024).

These are inherent constraints that require the adoption of "polishing" tertiary treatment systems that can remove some of the more persistent micropollutants. Biological treatment (conventional) is inadequate in terms of the performance of the technology, and the use of adsorption technology,

specifically granular activated carbon (GAC) is emerging as the solution (Ashraf et al., 2024; Moulika et al., 2026).

## **1.6 Adsorption as a Treatment Method:**

Adsorption is recognized as one of the most effective tertiary treatment technologies for the removal of persistent organic micropollutants (OMPs) from wastewater, particularly when conventional biological processes have failed to meet regulatory requirements (Ashraf et al., 2024). In the context of wastewater engineering, adsorption is defined as the physical or chemical mass transfer of solute molecules (adsorbates) from the liquid phase onto the surface of a porous solid material (adsorbent) (Lach & Szymonik, 2019).

The mechanism of adsorption for pharmaceutical residues like naproxen and mefenamic acid is multifaceted, involving several types of interactions:

### **1.6.1 Van der Waals Forces**

Weak, long-range physical interactions that initiate the attraction between the pharmaceutical molecule and the adsorbent surface.

### **1.6.2 Hydrophobic Interactions**

Since many NSAIDs exhibit hydrophobic characteristics, they preferentially seek to leave the aqueous phase and attach to non-polar surfaces, such as the carbonaceous matrix of an adsorbent (Mansouri et al., 2021).

### **1.6.3 Electron Donor-Acceptor Interactions**

This is a crucial mechanism where the aromatic rings of the NSAIDs interact with the graphene layers of the adsorbent, significantly enhancing the uptake of the contaminant (Lach & Szymonik, 2019).

Unlike advanced oxidation processes (AOPs), which chemically transform pollutants sometimes creating toxic intermediate by-products adsorption provides a reliable method for the physical separation and containment of pollutants (Ashraf et al., 2024). This makes it a preferred "polishing" step, as it is relatively easy to operate, modular in design, and highly efficient at reducing contaminant concentrations to below the detection limit (Mansouri et al., 2021).

## **1.7 Granular Activated Carbon (GAC) and Fixed-Bed Column Systems**

Granular Activated Carbon (GAC) is the most widely utilized adsorbent in industrial and municipal wastewater treatment due to its exceptionally high internal surface area—often exceeding and its well-developed microporous structure (Lach & Szymonik, 2019).

### **1.7.1 Granular Activated Carbon GAC as an Adsorbent**

GAC is typically derived from carbonaceous precursors such as coconut shells, coal, or wood, which undergo high-temperature activation to develop a complex network of pores. For the removal of naproxen and mefenamic acid, the pore size distribution is critical; micropores are responsible for the bulk of the surface area and are the primary sites for the adsorption of small pharmaceutical molecules (Lach & Szymonik, 2019).

## **1.8 Fixed-Bed Column Systems**

In large-scale treatment applications, GAC is rarely used in batch systems. Instead, it is deployed in fixed-bed column (also known as packed-bed reactors), where the wastewater is passed continuously through a bed of GAC (Lach & Szymonik, 2019). This setup offers several engineering advantages

## **1.9 Continuous Operation**

Unlike batch processes that require downtime for filling and emptying, fixed-bed columns allow for uninterrupted treatment.

**Breakthrough Dynamics:** The performance of a column is characterized by a "breakthrough curve," which maps the concentration of the pharmaceutical in the effluent over time. The "breakthrough point" occurs when the concentration of the contaminant in the effluent rises to a predetermined limit, signaling that the GAC bed has reached its capacity and requires regeneration or replacement (Lach & Szymonik, 2019).

**Predictive Modeling:** Through the application of kinetic models such as the Thomas model or the Yoon-Nelson model engineers can mathematically predict the lifespan of the carbon bed based on flow rate, initial concentration, and the physical properties of the GAC (Lach & Szymonik, 2019).

The successful implementation of these systems depends on hydraulic control and the prevention of preferential flow paths (channeling) within the carbon bed, ensuring that the entire surface area of the GAC is utilized effectively (Imim, 2024).

#### **1.10 Role of Sand and Gravel in Multi-Layer Filtration**

The use of a multi-layer system (including a supporting gravel base and a sand filtration layer) is important for the operational life and efficiency of the subsequent Granular Activated Carbon (GAC) layer in the design of a fixed-bed column system. GAC has been used as the primary adsorbent to remove pharmaceutical molecules from water, but it is very sensitive to the presence of suspended solids (Imim, 2024).

The Filtration Column is composed of multiple functional layers that serve to filter out particles of various sizes.

The coarse gravel layer is the foundation layer of the column and is a drainage structure. It keeps the fine filtration material (sand and GAC) from escaping through the column effluent port and provides a uniform hydraulic distribution of the wastewater, avoiding preferential flow paths or "channeling" that could lead to areas of the wastewater treatment plant where the wastewater is not being treated.

The sand layer serves as a mechanical pre-filter and is located between the gravel and the GAC. It is mainly used to remove coliform and Total Suspended Solids (TSS) from the influent. The sand layer keeps these particles from causing blinding or pore clogging of the GAC (Imim, 2024). As particulate matter fills GAC pores, the inside surface area is unavailable for pharmaceutical molecule adsorption, and the amount of adsorption may decrease significantly. As such, the sand layer can greatly increase the operational service life of the GAC media before it needs to be regenerated.

A multi-layer treatment method makes a strong and integrated treatment unit that is able to treat real wastewater conditions in which the suspended solids would otherwise affect the tertiary adsorption step (Imim, 2024).

## **1.11 Operational Factors**

For a GAC-based fixed-bed system, the efficiency is dependent on a number of critical operational parameters, which control mass transfer kinetic with target pollutants such as naproxen and mefenamic acid and breakthrough behavior.

The pH of the wastewater solution is probably most important because it determines the degree of ionization of both the NSAIDs and the GAC surface. Being acidic substances, naproxen and mefenamic acid have different solubility and charge in relation to the pH. If the pH is lower than the of the pharmaceutical, it is likely to be present in the neutral form, which usually has a greater affinity to the hydrophobic surface of the GAC (Lach & Szymonik, 2019).

### **1.11.1 Contact time**

Time of interaction of the pharmaceutical molecule and the adsorbent. The necessary contact time needs to be provided to allow the solutes to be transferred from the bulk liquid phase into the micropores of GAC, where most of the adsorption sites are (Lach & Szymonik, 2019).

## **1.12 To monitor the removal of organic pollutants (OCPs), using UV Spectrophotometry**

The UV-Vis spectrophotometry is a non-destructive method which allows the quantification of pharmaceuticals in a rapid manner. To monitor the absorbance of the compounds of interest (i.e. naproxen and mefenamic acid) in real-time across the entire column, the absorbance at specific wavelengths ( $\lambda$ ) can be measured (Lach & Szymonik, 2019)

Although the literature is replete with data on the effectiveness of Granular Activated Carbon (GAC) in pharmaceutical micropollutant removal, there are considerable research gaps on the use of these materials in the field scale or semi-field scale, especially in the development of urban centers such as Pakistan. (Mansouri et al., 2021).

Most of the published literature is concerned with batch adsorption which does not reflect the hydraulic complexity and mass transfer limitations associated with fixed-bed column systems in continuous flow.

Integrated multi-layer filtration (IMF) (which is the combination of sand/gravel pre-filters and GAC) for the simultaneous removal of multiple NSAIDs has received little consideration in the

research literature. There is not a good understanding of the synergistic effects it has on operational life and regeneration needs of GAC.

Most of the literature on which the model is founded is based on ultrapure water or synthetic matrices of wastewater. The effect of local, high-sediment wastewater matrices, which typically include competing organic matter, dissolved solids and different ionic strengths, on competitive adsorption and breakthrough of GAC for naproxen and mefenamic acid is not known (Mansouri et al., 2021)

### **1.13 Research Gap**

The presence of pharmaceutical drugs in water bodies has become a major environmental issue, as they are continuously discharged, persist in the water, and can have an impact on the environment (Kummerer, 2021; Patel et al., 2025). There are many studies exploring the use of adsorption to remove pharmaceutical compounds, but most of the published studies have been conducted in batch adsorption systems in highly controlled laboratory conditions (Kummerer, 2021; Mansouri et al., 2021). These studies offer useful insights into adsorption mechanisms and adsorption equilibrium but are not likely to represent the hydraulic conditions found in actual water treatment applications.

Granular Activated Carbon (GAC) has been widely reported to be one of the most effective adsorptive materials for removing pharmaceutical residues from contaminated water because of its high surface area, porous structure and high affinity towards organic micropollutants (OMPs) (Lach & Szymonik, 2019). An evaluation of the performance of the low-cost gravity-driven fixed-bed column filtration systems containing multiple filtration layers like gravel, sand and GAC for simultaneous removal of naproxen and mefenamic acid from aqueous solutions has not been mostly studied.

Moreover, there is still limited literature available in developing countries as the consumption of pharmaceuticals, rapid urbanization, and poor wastewater treatment practices are on the rise, especially in Pakistan (Ashraf et al., 2024; Singh et al., 2024). The effect of the operational parameters (initial concentration, pH, contact time and water matrix characteristic) of the locally used multi-layer filtration systems on the adsorption performance has not been studied in detail. Hence, this study aimed to fill in these research gaps by testing the efficiency of GAC based fixed-

bed column system to remove naproxen and mefenamic acid using various experimental conditions.

#### **1.14 Problem Statement**

Pharmaceutical products are used extensively, leading to the persistent discharge of pharmaceutical residues in wastewater, surface water and groundwater worldwide. Non-steroidal anti-inflammatory drugs (NSAIDs) like naproxen and mefenamic acid are commonly found, as they are very popular medicines that are not fully metabolized by the human body and are not effectively removed from wastewater during standard treatment processes (Thakur et al., 2023; Moulika et al., 2026).

Traditional wastewater treatment works (WWWs) are mainly aimed at removal of suspended solids, biodegradable organic matter and nutrients, but not at persistent pharmaceutical micro-pollutants (Cacus-Makowska et al., 2023). As a result, significant amounts of pharmaceutical waste can pass through treatment systems and eventually contaminate natural waters, which can have harmful impacts on aquatic life, harm ecological processes and become a long-term public health issue (Singh et al., 2024; Patel et al., 2025).

All these are further complicated in Pakistan by the population growth, rising demand of pharmaceuticals, limited treatment capacity and improper treatment of wastewater which is being discharged into receiving water bodies. Although the impact of pharmaceutical contamination on the environment is increasing, few studies have tested practical and cost-effective treatment technologies for local applications. Hence, effective, affordable and sustainable treatment methods to reduce pharmaceutical contamination in water sources is urgently needed and are a research priority. This study tackles this challenge by assessing the effectiveness of a multi-layer fixed-bed GAC system to remove the pharmaceuticals naproxen and mefenamic acid from water contaminated with these compounds

#### **1.15 Objectives**

- To evaluate the removal efficiency of mefenamic acid and naproxen using a Granular activated carbon (GAC) fixed-bed column.
- To investigate the effect of concentration, pH and contact time and water source on removal efficiency.

### **1.16 Significance of the Study**

The present study is important from an ecological, scientific and practical viewpoint as it deals with a new problem in aquatic environments, which is pharmaceutical pollution. Pharmaceutical residues are now a worldwide concern because of their ubiquity, bioactivity and potential effects on water ecosystems and human health (Kummerer, 2021; Patel et al., 2025).

The study is environmentally significant because it helps develop efficient measures for minimizing the amount of pharmaceutical waste entering natural waters prior to its release. Enhanced elimination of naproxen and mefenamic acid may reduce ecological hazards like oxidative stress, reproductive dysfunction, endocrine-disruption, and biodiversity loss in aquatic organisms (Singh et al., 2024; PMC, 2026).

The benefit of decreasing contamination of water resources by pharmaceutical residues can be improved water quality and decreased human exposure to emerging contaminants. Additionally, by controlling pharmaceutical pollution, the environmental factors of antimicrobial resistance and ecosystem degradation can be decreased in the long-term (Barkha Madhogaria et al., 2024).

The proposed multi-layer filtration system involves the use of materials such as gravel, sand, and granular activated carbon, which are readily available and at a low cost, potentially making it a viable treatment method in developing nations and resource-poor communities. This study could give useful information for the design and optimization of low-cost tertiary treatment systems.

In the academic field, this study is useful for advancing current knowledge on the adsorption of pharmaceuticals in a fixed-bed system and would be useful for future study into the adsorption kinetics, breakthrough characteristics, regeneration of the adsorbent, pilot-scale implementation, and optimization of wastewater treatment parameters for pharmaceutical adsorption. This study's results could be used as a base for future research to enhance sustainable water treatment methods for emerging contaminants

***Table Ecotoxicological Significance.1 Comparison of GAC filter with world-class water treatment technologies***

| <b>Feature</b>                 | <b>This GAC Filter</b> | <b>Commercial GAC Systems</b> | <b>Industrial Membrane Filtration</b> | <b>Reverse Osmosis Plants</b> |
|--------------------------------|------------------------|-------------------------------|---------------------------------------|-------------------------------|
| Cost                           | Rs 1,000–2,000         | Rs 50,000–500,000             | Millions of rupees                    | Millions of rupees            |
| Electricity needed             | No                     | Yes                           | Yes                                   | Yes                           |
| Technical expertise needed     | No                     | Yes                           | Yes                                   | Yes                           |
| Pharmaceutical Removal         | Up to 97.1%            | High                          | High                                  | Very High                     |
| Suitable for rural areas       | Yes                    | Limited                       | No                                    | No                            |
| Eco-friendly Materials         | Yes                    | Partial                       | No                                    | No                            |
| Accessible to poor communities | Yes                    | No                            | No                                    | No                            |

### **1.17 Relevance to Global Water Quality Challenges**

According to the United Nations, a significant proportion of the global population still lacks access to safely managed drinking water, particularly in low- and middle-income countries (OECD, 2025). Pharmaceutical contamination of water sources is an emerging and growing concern in both urban and rural areas, driven by increased global pharmaceutical consumption, inadequate wastewater treatment, and improper pharmaceutical disposal practices. NSAIDs including mefenamic acid and naproxen have been detected in surface water, groundwater, and treated

drinking water at concentrations ranging from nanograms to micrograms per liter in numerous countries worldwide, raising concerns about long-term ecological and human health impacts (Lach and Szymonik, 2019).

The findings of this study contribute to the growing body of evidence supporting GAC adsorption as a technically viable approach for pharmaceutical micropollutant removal from water. The low-cost design evaluated here may offer a useful foundation for developing affordable treatment solutions appropriate for resource-limited settings, though further research including field testing and scale-up studies would be necessary before practical deployment could be recommended.

## **CHAPTER 2**

### **MATERIALS AND METHODS**

#### **2.1 Research Methodology for removal of Mefenamic Acid and Naproxen**

This study was carried out to test whether a simple, low-cost GAC column could effectively remove mefenamic acid and naproxen from water. The experiments were designed to check how different factors including concentration, pH, contact time, and water type affected how well the column worked. All experiments were done at room temperature in the laboratory. A UV-Visible spectrophotometer was used to measure drug concentrations in the samples before and after column treatment. A fixed-bed column setup was chosen for this study because it is closer to how real water treatment systems work in practice compared to simple batch experiments (Sotelo et al., 2012).

#### **2.2 Materials and Chemicals**

For the pharmaceutical compounds, mefenamic acid tablets (500 mg) and naproxen tablets (500 mg) were obtained from a local pharmacy. Tablets were used instead of pure analytical standards because they are more accessible and represent a realistic drug source.

For the column, Granular Activated Carbon (GAC) was used as the main adsorbent material. GAC was chosen because of its large surface area, high adsorption capacity for organic pollutants, and its wide use in water treatment studies (Rakić et al., 2015). Silica sand and gravel were used as supporting layers inside the column. Cotton was placed at the bottom outlet of the syringe to hold all materials in place.

For chemicals, methanol was used to dissolve the drug tablets, hydrochloric acid (HCl 0.1N) was used to lower pH, and sodium hydroxide (NaOH 0.1N) was used to raise pH. Distilled water was used for preparing all solutions and for rinsing equipment.

The equipment included a 50 mL plastic syringe as the column, a graduated measuring cylinder, a UV-Visible spectrophotometer, a digital pH meter, and a timer.

**Table 2.1 List of chemicals and materials used in the study**

|     |                                 |                                                                                     |                  |
|-----|---------------------------------|-------------------------------------------------------------------------------------|------------------|
| 1.  | Mefenamic Acid (500mg)          |    | Ponston          |
| 2.  | Naproxen Acid (500mg)           |    | Aleve            |
| 3.  | Methanol                        |    | Merck            |
| 4.  | Distilled water                 |    | Aqua clear       |
| 5.  | Granular Activated Carbon (GAC) |    | Jacobi Carbons   |
| 6.  | Silica Sand                     |    | Pool Filter Sand |
| 7.  | Gravel                          |  | Fluorite         |
| 8.  | Cotton                          |  | Filter Foam      |
| 9.  | Syringe Column                  |  | Hamilton         |
| 10. | UV spectrophotometer            |  | UV4000           |

### 2.3 Preparation of Adsorption Column

A 50 mL plastic syringe was used as the column body. This choice was intentional to show that an effective water treatment system can be built from inexpensive materials that anyone can access. Before packing the column, all materials were washed and prepared carefully.

The column was designed using simple and locally available materials as a sustainable alternative to conventional water treatment methods. Unlike advanced treatment systems that require

electricity, specialized equipment, and technical expertise, this gravity-driven Granular Activated Carbon GAC column operates without any external energy source, making it a practically accessible option for water treatment in resource-limited settings (Rakić et al., 2015)

### **2.3.1 Washing of Materials**

Sand and gravel were washed with tap water several times until the water running off them looked completely clear. They were then rinsed once more with distilled water. GAC was washed multiple times with distilled water to remove black dust and fine particles. After washing, the GAC was soaked in distilled water for 30 minutes before use. Washing and soaking GAC before use is a standard preparation step that helps stabilize flow and removes loose particles that could interfere with results (Sotelo et al., 2012).

### **2.3.2 Column Packing**

The column was packed while wet to avoid trapping air bubbles inside the layers, which could disrupt the flow of water. Packing was done from the bottom outlet upward in this order. First, a 0.5 cm cotton plug was placed at the very bottom of the syringe outlet to hold everything in place. Above the cotton, 1 cm of gravel was added as a support base. A 4 cm of silica sand was placed above the gravel to trap fine particles. Finally, 4 cm of GAC was packed on top of the sand. This top layer is the most important part it is where the actual drug removal happens. The total bed height was approximately 9.5 cm.

The Experimental setup shown in fig 2.1

Pack the syringe from bottom to top in the following order (bottom = outlet end):

0.5 cm of cotton (at the very bottom)

1 cm gravel

4 cm of silica sand

4 cm of GAC (at the very top) Granular Activated Carbon was packed on top of the sand as the primary adsorption layer.



**Figure 2.2** *Packed fixed-bed adsorption column showing layers of cotton, 1 cm gravel, 4 cm silica sand, and 4 cm granular activated carbon (GAC) from bottom to top.*

**Table 2.2** *Experimental Conditions and Parameters Used in the Study*

| Parameters    | Values                 |
|---------------|------------------------|
| Concentration | 5,25,50mg/L            |
| pH            | 5, 7, 10               |
| Contact time  | 15, 30, 45, 60 minutes |
| Water type    | Distilled, Tap         |
| Adsorbent     | GAC                    |

### 2.3.3 Column Conditioning

After packing was complete, distilled water was passed through the column for 20 to 30 minutes before starting any experiment. This step was done to settle the layers, flush out remaining fine particles, and make sure the flow was stable and consistent (Sotelo et al., 2012). Skipping this step could lead to inconsistent results between experiments.

### 2.3.4 Flow Rate

The column ran on gravity only no pump or pressure was used. Water was poured into the top and flowed downward through the layers on its own. The flow rate was kept between 1 and 3 mL per minute in all experiments. To check the flow rate, water coming out of the bottom was collected for 5 minutes and the total volume was divided by 5 to get mL per minute.

## 2.4 Preparation of Synthetic Drug Solutions

A stock solution of 1000 mg/L was prepared for each drug. One tablet of 500 mg was crushed into fine powder using a mortar and pestle. The powder was dissolved in a small amount of methanol first because both mefenamic acid and naproxen do not dissolve well in water alone and need methanol to dissolve completely (Lach and Szymonik, 2019). The dissolved mixture was then poured into a 500 mL flask and topped up to the 500 mL mark with distilled water. This gave a final concentration of 1000 mg/L. The same steps were followed for both drugs. All stock solutions were made fresh on the day of each experiment to prevent the drug from breaking down during storage. Before use, all solutions were passed through a 0.45  $\mu\text{m}$  syringe filter to remove any undissolved tablet excipients that could affect UV readings



**Figure 2.1 Preparation of stock solutions: (a) grinding tablets to fine powder, (b) dissolving powder in methanol, (c) transferring to volumetric flask, (d) filtration through 0.45  $\mu\text{m}$  syringe filter.**

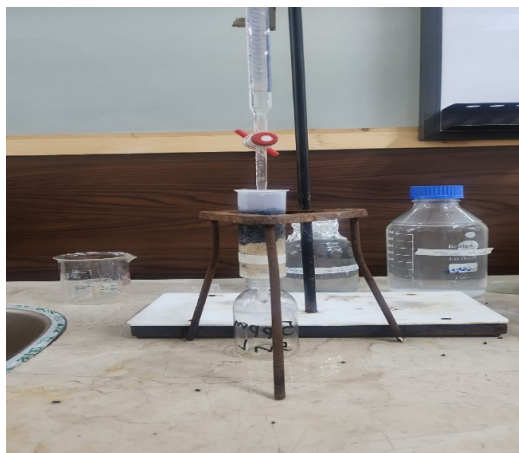
### 2.4.1 Control Run (Baseline)

Before using the drugs solution, run a control experiment with distilled water.

Pass distilled water through the stabilized column and measure the flow rate.

**2.4.2 Observed flow rate:** Approximately 5 mL of distilled water passed through the column in 3 minutes.

Collect the control sample (filtered distilled water) for UV analysis. This provides the baseline absorbance value.



***Figure 2.2 Serial dilution apparatus used to prepare working concentrations of 50 mg/L, 25 mg/L, and 10 mg/L from stock solution.***

## **2.5 Preparation of Working Concentrations**

Three working concentrations were prepared from the stock solution — 50 mg/L, 25 mg/L, and 5 mg/L. These concentrations were chosen to represent a range from high to low contamination levels and to test how concentration affected removal (Rakić et al., 2015). Each concentration was made separately for both drugs in a total volume of 50 mL using the dilution formula  $C_1V_1 = C_2V_2$ , where  $C_1$  is the stock concentration,  $V_1$  is the volume taken from the stock,  $C_2$  is the desired concentration, and  $V_2$  is the final volume.

The required volume of stock solution was measured using a graduated cylinder, poured into a clean flask, and topped up to 50 mL with distilled water. Before passing each concentration through the column, a 5 mL control sample was taken directly from the solution without any treatment. This control sample was kept as the reference value for calculating removal. The column was rinsed with distilled water between each concentration to prevent any leftover drug.

## 2.6 Column Operation and Sample Collection

Before passing any drug solution through the column, a control sample of 5 mL was collected directly from each prepared concentration. This control sample was not passed through the column. It represented the original concentration of the drug before any treatment and was used later to calculate how much drug was removed by the column.

After collecting the control sample, the drug solution was poured into the top of the column. The solution moved downward through the bed layers on its own due to gravity. No pump or pressure was used at any point. As the treated water came out from the bottom of the column, it was collected as the effluent sample.

Every time the filter column is washed with distilled water before passing new concentrations.

Chapter 1 Samples were collected in volumes of 5 mL at different time intervals. Each sample was clearly labeled so it could be identified during analysis. All samples including the control and the effluent were then taken to the UV-Visible spectrophotometer for absorbance measurement.

## 2.7 Tap Water Experiment

Most laboratory studies use distilled water to prepare drug solutions. However, in real life, water sources such as tap water contain naturally occurring minerals, salts, and chlorine. To make this study more practical and relevant to real world conditions, an additional experiment was carried out using tap water instead of distilled water. (Rakić et al., 2015).

The study was carried out using tap water instead of distilled water to simulate environmental conditions (Figure 2.4).



***Figure 2.4 Simulation of real-world conditions using tap water as the aqueous matrix for drug solution preparation***

The same three concentrations of 50 mg/L, 25 mg/L, and 5 mg/L were prepared using tap water. The drug was dissolved and diluted in tap water following the same steps used for distilled water experiments. Before passing each concentration through the column, a 5 mL control sample was taken directly from the prepared solution. Each concentration was then passed through the GAC column one by one under gravity flow. The treated water coming out from the bottom of the column was collected as an effluent sample. All samples were sent for absorbance measurement using the UV-Visible spectrophotometer and removal efficiency was calculated using the same formula as previous experiments.

## **2.8 Effect of Contact Time (Retention Time)**

Contact time is how long the drug solution stays in contact with the GAC inside the column. The longer this time, the more opportunity drug molecules have to attach to the carbon surface (Lach and Szymonik, 2019). To test this, four identical columns were set up at the same time, each packed the same way as described in Section 3.3.

The 25 mg/L solution was used for this experiment. A 5 mL control sample was taken before starting. Equal amounts of the 25 mg/L solution were poured into all four columns at the same time and a timer was started immediately. All four columns were left running continuously without stopping. At 15 minutes the first column outlet was opened, and 5 mL of effluent was collected and labeled 15 min. At 30 minutes the second column was sampled and labeled 30 min. The same was done at 45 minutes and 60 minutes for the third and fourth columns. By the end, five samples were ready, one control and four effluent samples. All were analyzed using the UV spectrophotometer.

## **2.9 Effect of pH**

The pH of a solution affects whether drug molecules carry an electric charge or not, which in turn affects how strongly they stick to the GAC surface (Lach and Szymonik, 2019). To study this, the 25 mg/L drug solution was tested at three pH levels — pH 5 (acidic), pH 7 (neutral), and pH 10 (basic).

For pH 5, a few drops of 0.1N hydrochloric acid were added slowly to the solution while watching the pH meter until it read exactly 5. For pH 7, the solution was used as it was since its natural pH was already around 7. For pH 10, a few drops of 0.1N sodium hydroxide were added dropwise until the meter reach 10. Acid and base were added slowly drop by drop to avoid going past the target

pH. A 5 mL control sample was collected from each pH-adjusted solution before column treatment. Each solution was then passed through the column separately and the effluent collected. The column was rinsed with distilled water between each pH test. All six samples, three controls and three effluents were analyzed on the UV spectrophotometer.



*Figure 2.5. Digital pH meter used for adjustment and monitoring of solution pH during experiments.*

## 2.10 UV-Visible Spectrophotometric Analysis

All samples from all experiments were measured using a UV-Visible spectrophotometer at 270 nm. This wavelength was selected because it represents the maximum absorbance point for both mefenamic acid and naproxen, consistent with values reported in published studies (Lach and Szymonik, 2019).

Before measuring any sample, the spectrophotometer was calibrated with distilled water as a blank. The cuvette was rinsed with distilled water, filled with distilled water only, and placed in the machine to set the baseline to zero. This removed any background interference so that all readings reflected only the drug in the sample. After setting the blank, the cuvette was rinsed again before each sample.

For every experiment, the untreated control sample was measured first to get  $C_0$ . Then the treated effluent was measured to get  $C_t$ . Removal efficiency was then calculated using this formula:

Removal % =  $[(C_0 - C_t) \div C_0] \times 100$  A higher removal percentage means more drug was removed by the column. The normalized ratio  $C_t/C_0$  was also calculated for all experiments values close to 0 mean excellent removal and values close to 1 mean poor removal (Sotelo et al., 2012).

## 2.11 Data Analysis

The experimental data gathered from the fixed-bed column operations were systematically evaluated to quantify and compare the removal efficiencies of Mefenamic Acid and Naproxen. Raw data generated via the digital pH meter and the UV-Visible Spectrophotometer were recorded and managed using Microsoft Excel.

To determine the extent of pharmaceutical contaminant removal, the percentage removal efficiency ( $R\%$ ) was calculated for each experimental run using the following mass balance equation:

$$\text{Removal \%} = [(C_0 - C_t) \div C_0] \times 100$$

Where:

$C_0$  represents the initial control baseline concentration of the target pharmaceutical compound (mg/L) prior to column treatment.

$C_t$  represents the final residual concentration of the pharmaceutical compound (mg/L) in the column effluent collected at a given time interval ( $t$ ).

The target concentrations ( $C_0$  and  $C_t$ ) were calculated by relating the measured optical absorbance values obtained at  $\lambda = 270\text{ nm}$  against standard calibration curves generated via serial dilutions.

Descriptive statistics, including averages, percentage differences, and trend visualizations, were applied to assess the individual and interactive impacts of the independent operational variables—namely, initial drug concentrations (5 mg/L, 25 mg/L, and 50 mg/L), adjustment of solution pH (acidic condition at pH 5 and basic condition at pH 10), varying contact/retention times (15, 30, 45, and 60 minutes), and the nature of the background aqueous matrix (distilled water versus municipal tap water). Any atypical fluctuations or experimental anomalies observed during continuous running—such as sudden shifts in effluent concentration due to temporary desorption or saturation profiles were explicitly identified and treated as outliers in the final data rendering. The processed values were organized into comparative tables and plotted as breakthrough and trend curves to illustrate the comparative affinity of Granular Activated Carbon (GAC) toward both pharmaceutical compounds.

## **CHAPTER 3**

### **RESULTS AND DISCUSSION**

#### **3.1 Overview**

This chapter presents and discusses the results obtained from four sets of laboratory experiments designed to evaluate the removal efficiency of mefenamic acid and naproxen from aqueous solutions using a gravity-driven GAC fixed-bed adsorption column.

Three working concentrations of 5 mg/L, 25 mg/L, and 50 mg/L were prepared from a 1000 mg/L stock solution for both drugs separately. Before passing each concentration through the column, a control sample of distilled water was first analyzed to establish the baseline absorbance reading. This control run confirmed that the column and distilled water produced no background absorbance, ensuring that all subsequent readings reflected only the drug present in the solution.

The experiments examined four key factors initial drug concentration, solution pH, contact time, and water matrix type to understand how each one affects the adsorption performance of the column. For the pH experiment, the 25 mg/L solution was tested at pH 5 and pH 10. For the contact time experiment, samples were collected at 15, 30, 45, and 60 minutes using four separate columns running simultaneously. An additional experiment was carried out using tap water instead of distilled water to evaluate performance under more realistic conditions.

All collected samples were analyzed using a UV-Visible spectrophotometer at a wavelength of 270 nm. The untreated control sample was measured first for each experiment to get the initial absorbance value, and then the treated effluent sample was measured. The difference between these two readings was used to calculate how much drug was removed by the column. The results for both drugs are presented and discussed in relation to published literature on pharmaceutical adsorption using activated carbon.

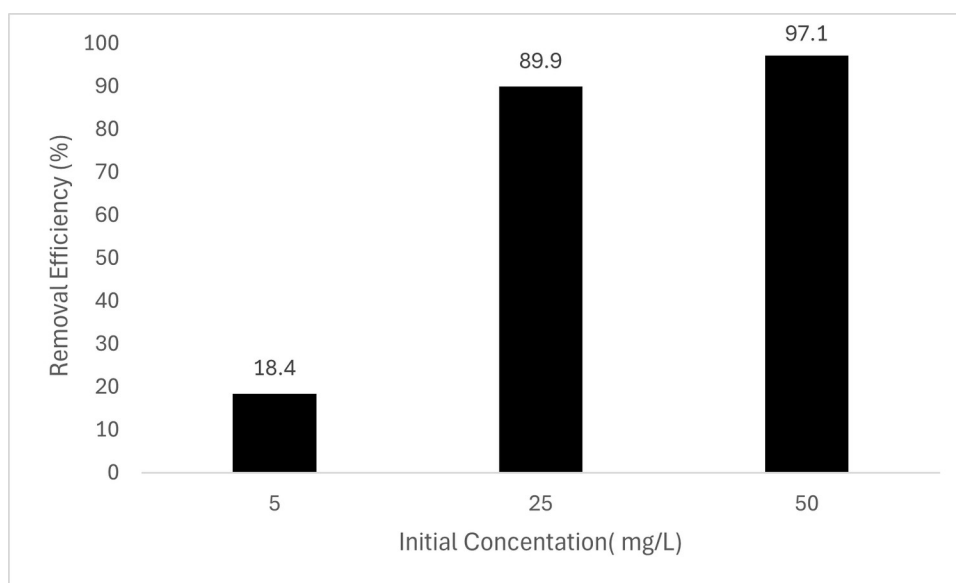
#### **3.2 Effect of Initial Concentration in Distilled Water**

##### **3.2.1 Mefenamic Acid**

The first set of experiments evaluated the adsorption of mefenamic acid onto GAC at three initial concentrations 50 mg/L, 25 mg/L, and 5 mg/L using distilled water as the solvent matrix. The absorbance values of the control (C<sub>0</sub>) and treated samples (C<sub>t</sub>), along with the calculated removal efficiencies, are summarized in Table 4.1.

**Table 3.1 UV-Visible absorbance values and removal efficiency of mefenamic acid at different initial concentrations.**

| Concentration (mg/L) | Control ( $C_0$ ) | Sample ( $C_t$ ) | Removal % |
|----------------------|-------------------|------------------|-----------|
| 50                   | 1.605             | 0.046            | 97.1%     |
| 25                   | 0.761             | 0.077            | 89.9%     |
| 5                    | 0.261             | 0.213            | 18.4%     |



**Figure 3.1. Removal efficiency of mefenamic acid at different initial concentrations using**

The results showed a strong and clear concentration-dependent adsorption trend. At 50 mg/L, the GAC column achieved an exceptional removal efficiency of 97.1%, with a normalized concentration ratio ( $C_t/C_0$ ) of just 0.029, indicating that almost all drug molecules were successfully adsorbed. At 25 mg/L, removal remained high at 89.9%. However, at the lowest concentration of 5 mg/L, removal dropped sharply to only 18.4%, with a  $C_t/C_0$  ratio of 0.816, meaning that most of the drug passed through the column without being captured.

This concentration-dependent behavior is explained by the driving force theory of adsorption. At higher initial concentrations, a steeper concentration gradient exists between the bulk solution and the GAC surface. This gradient acts as the driving force that pushes drug molecules toward the adsorbent surface and into its pore structure where they bind to available active sites. At low

concentrations such as 5 mg/L, this driving force is significantly weaker, resulting in fewer successful molecular collisions with the carbon surface and consequently lower removal efficiency (Sotelo et al., 2012).

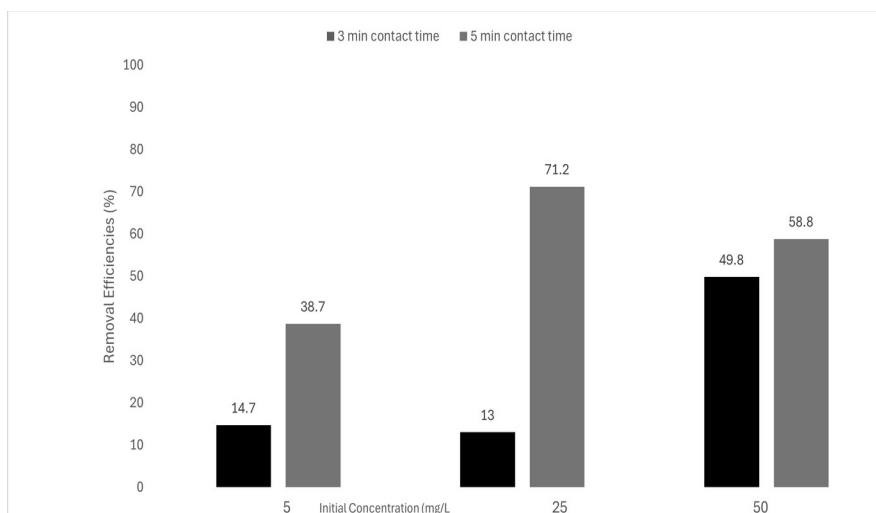
The results of this study compare favorably with findings reported in published literature. (Sotelo et al. (2012) investigated the adsorption of diclofenac, a non-steroidal anti-inflammatory drug belonging to the same pharmacological class as mefenamic acid on GAC fixed-bed columns and reported high removal efficiencies at elevated initial concentrations, consistent with the concentration-dependent trend observed in the present (Rakić et al. (2015) confirmed that initial concentration is a key determinant of adsorption performance for pharmaceutical compounds on activated carbon, with higher concentrations yielding stronger adsorption driving forces a pattern consistent with the results obtained for mefenamic acid and naproxen in this and confirmed that initial concentration is a key determinant of adsorption performance, with higher concentrations yielding stronger adsorption driving forces. The 97.1% removal achieved in the present study at 50 mg/L is consistent with the high removal efficiencies reported for structurally similar NSAIDs under comparable concentration conditions, validating the effectiveness of the GAC column constructed in this study.

### 3.2.2 Naproxen

Naproxen was tested at the same three concentrations but at two different contact times 3 minutes and 5 minutes. Table 4.2 shows the results.

***Table 3.2 UV-Visible spectrophotometric data and removal efficiency of naproxen at different initial concentrations and contact times.***

| <b>Concentration<br/>(mg/L)</b> | <b>Control</b> | <b>S1 time 3<br/>minutes</b> | <b>Removal %</b> | <b>S2 time 5<br/>minutes</b> | <b>Removal %</b> |
|---------------------------------|----------------|------------------------------|------------------|------------------------------|------------------|
| 50                              | 0.255          | 0.128                        | 49.8%            | 0.105                        | 58.8%            |
| 25                              | 0.146          | 0.127                        | 13.0%            | 0.042                        | 71.2%            |
| 5                               | 0.075          | 0.064                        | 14.7%            | 0.046                        | 38.7%            |



***Figure 3.3. Comparison of naproxen removal efficiencies (%) at 3 min and 5 min contact times for different initial concentrations using GAC column.***

Two significant observations emerge from these results. First, extending the contact time from 3 to 5 minutes produced a substantial improvement in removal efficiency across all concentrations. The most dramatic improvement was observed at 25 mg/L where removal increased from 13.0% to 71.2% and an improvement of 58.2 percentage points simply by allowing two additional minutes of contact between the drug solution and the GAC bed. This confirms that contact time is one of the most critical parameters in fixed-bed adsorption systems. Lach and Szymonik (2019), in their study of naproxen sodium adsorption on commercial activated carbons, confirmed that adsorption kinetics play a decisive role in removal efficiency, with longer contact times allowing more complete diffusion of drug molecules into the micropore structure of the carbon.

Second, comparing naproxen results with mefenamic acid results at equivalent concentrations reveals that mefenamic acid consistently achieved higher removal. At 50 mg/L for example, mefenamic acid achieved 97.1% removal compared to only 58.8% for naproxen at the longer 5-minute contact time, and a  $C_t/C_0$  of 0.029 versus 0.412 for naproxen. This marked difference in adsorption performance is directly related to the physicochemical properties of both molecules. Mefenamic acid has a higher octanol-water partition coefficient ( $\log K_{ow} \approx 5.12$ ) compared to naproxen ( $\log K_{ow} \approx 3.18$ ), indicating greater hydrophobicity. Since GAC adsorption is primarily driven by hydrophobic interactions between the non-polar carbon surface and the drug molecule,

compounds with higher log Kow values are expected to adsorb more strongly and achieve higher removal efficiencies under identical conditions (Sotelo et al., 2012).

### 3.3 Effect of pH

The pH of a solution plays an important role in adsorption because it determines the ionization state of the drug molecule and the surface charge of the GAC. When the pH changes, the drug molecule can become either neutral or charged, which directly affects how strongly it sticks to the carbon surface. In this study, the effect of pH was investigated using the 25 mg/L working concentration for both mefenamic acid and naproxen. The solution pH was adjusted to pH 5 representing acidic conditions and pH 10 representing basic conditions using hydrochloric acid and sodium hydroxide respectively. A control sample was collected from each pH adjusted solution before column treatment and the results are presented below.

#### 3.3.1 Mefenamic Acid

***Table 3.2 UV-Visible spectrophotometric data and removal efficiency of naproxen at different initial concentrations and contact times.***

| <b>pH</b> | <b>Control (C<sub>0</sub>)</b> | <b>Sample (C<sub>t</sub>)</b> | <b>Removal %</b> |
|-----------|--------------------------------|-------------------------------|------------------|
| <b>5</b>  | 0.489                          | 0.115                         | 76.5%            |
| <b>10</b> | 0.604                          | 0.195                         | 67.7%            |

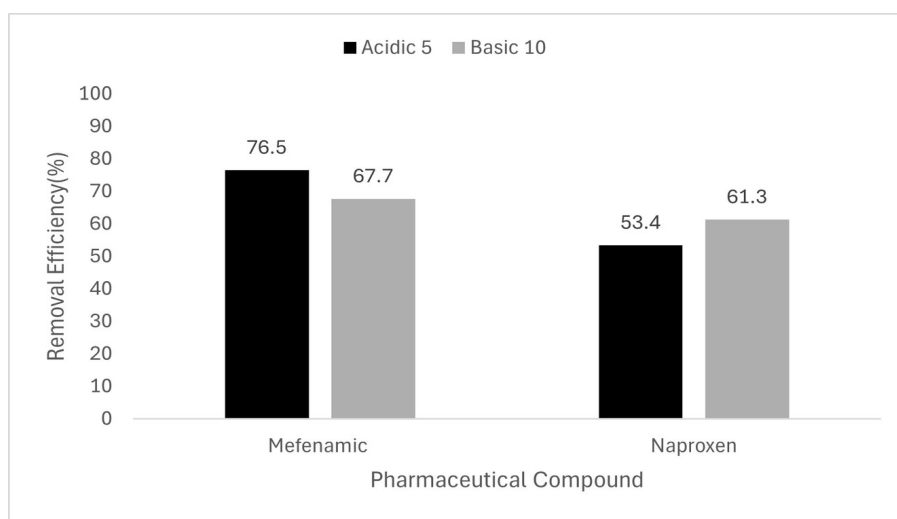
Mefenamic acid was removed better in acidic conditions. At pH 5 the removal was 76.5% and at pH 10 it dropped to 67.7%. This makes sense when you consider the chemistry of the molecule. Mefenamic acid is a weak acid with a pKa around 4.2. At pH 5 the molecule is mostly in its neutral uncharged form. Neutral molecules are more hydrophobic and stick more easily to the carbon surface. At pH 10 the molecule loses a hydrogen ion and becomes negatively charged. The GAC surface also becomes negatively charged at high pH. Since both are now negative, they repel each other like two magnets facing the same way which reduces how well the drug adsorbs (Rakić et al.,

2015). Despite this the removal at pH 10 was still close to 68% which shows the column works reasonably well even under basic conditions.

### 3.3.2 Naproxen

**Table.3. 4 Effect of pH on naproxen removal efficiency using GAC at 25 mg/L initial concentration.**

| pH Value | Control (C <sub>0</sub> ) | Sample (C <sub>t</sub> ) | Removal % |
|----------|---------------------------|--------------------------|-----------|
| 5        | 0.410                     | 0.191                    | 53.4%     |
| 10       | 0.398                     | 0.154                    | 61.3%     |



**Figure 3.3. Effect of pH on removal efficiency of mefenamic acid and naproxen using GAC at 25 mg/L initial concentration.**

Mefenamic acid was removed better in acidic conditions, as shown in Figure 4.3. At pH 5 the removal was 76.5% and at pH 10 it dropped to 67.7%. This makes sense when you consider the chemistry of the molecule. Mefenamic acid is a weak acid with a *pKa* around 4.2. At pH 5 the molecule is mostly in its neutral uncharged form. Neutral molecules are more hydrophobic and stick more easily to the carbon surface. At pH 10 the molecule loses a hydrogen ion and becomes negatively charged. The GAC surface also becomes negatively charged at high pH. Since both are

now negative, they repel each other like two magnets facing the same way which reduces how well the drug adsorbs (Rakić et al., 2015). Despite this the removal at pH 10 was still close to 68% which shows the column works reasonably well even under basic conditions.

Interestingly, naproxen showed the opposite pattern slightly better removal at pH 10 (61.3%) than at pH 5 (53.4%). Lach and Szymonik (2019) studied pH effects on naproxen adsorption and found that pH influences naproxen adsorption through changes in both molecular charge and carbon surface chemistry. The marginally higher removal at pH 10 in this study may be related to  $\pi$ - $\pi$  interactions between the aromatic naphthalene ring of naproxen and the carbon surface becoming stronger at higher pH, partially compensating for the electrostatic repulsion effect. Both drugs maintained removal above 50% at both pH values which confirms the GAC column performs adequately across a range of water pH conditions.

### 3.4 Effect of Contact Time

Contact time refers to the duration for which the drug solution remains in contact with the GAC bed inside the column. It is one of the most important parameters in fixed-bed adsorption because it directly determines how much time drug molecules have to diffuse from the bulk solution into the pores of the activated carbon and attach to available adsorption sites (Lach and Szymonik, 2019). In this study, the effect of contact time was evaluated using the 25 mg/L working concentration for both mefenamic acid and naproxen. Four identical columns were operated simultaneously, and effluent samples were collected at four-time intervals 15 minutes, 30 minutes, 45 minutes, and 60 minutes. A control sample was taken before starting the experiment. The results for mefenamic acid and naproxen are presented separately in the tables below

#### 3.4.1 Mefenamic Acid

*Table 3.5 Effect of contact time on mefenamic acid removal at 25 mg/L*

| Contact Time | Control (C <sub>0</sub> ) | Sample (C <sub>t</sub> ) | Removal % |
|--------------|---------------------------|--------------------------|-----------|
| 15 minutes   | 0.672                     | 0.344                    | 48.8%     |
| 30 minutes   | 0.672                     | 0.261                    | 61.2%     |
| 45 minutes   | 0.672                     | 0.220                    | 67.3%     |
| 60 minutes   | 0.672                     | 0.180                    | 73.2%     |

The results show a steady and consistent improvement in removal every 15 minutes. From 48.8% at 15 minutes the removal climbed to 61.2%, then 67.3%, and finally 73.2% at 60 minutes. The  $C_t/C_0$  ratio kept dropping which confirms the column was actively removing drug throughout the whole hour. The column had not yet reached saturation at 60 minutes, which means even higher removal would likely be achieved with longer contact times.

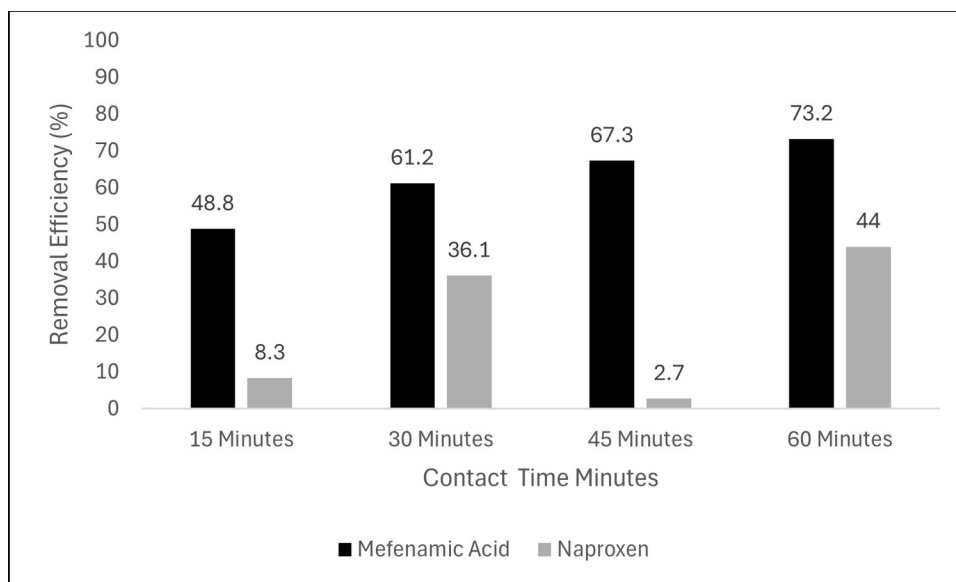
This steady improvement happens because adsorption is a two-step process. Drug molecules first attach to sites on the outer surface of GAC particles quickly. Then they slowly diffuse deeper into the internal pore structure where more adsorption sites exist. This second slower step is why removal keeps improving with time (Sotelo et al., 2012).

### 3.4.2 Naproxen

***Table 3.6 Effect of contact time on mefenamic acid removal at 25mg/L at 45 minutes 2.7% removal Anomalous result attributed to temporary desorption effect.***

| Contact Time | Control ( $C_0$ ) | Sample ( $C_t$ ) | Removal % |
|--------------|-------------------|------------------|-----------|
| 15 minutes   | 0.675             | 0.619            | 8.3%      |
| 30 minutes   | 0.675             | 0.431            | 36.1%     |
| 45 minutes   | 0.675             | 0.657            | 2.7%      |
| 60 minutes   | 0.675             | 0.378            | 44.0%     |

Table 3.6 presents the effect of contact time on naproxen removal at 25 mg/L initial concentration. The removal efficiency increased from 8.3% at 15 minutes to 36.1% at 30 minutes and reached 44.0% at 1 hour. An unexpected drop to 2.7% was observed at 45 minutes. This anomalous result is attributed to a temporary desorption effect, where accessible adsorption sites on the GAC surface became temporarily overloaded, causing previously adsorbed naproxen molecules to detach back into solution before the system re-stabilized and intraparticle diffusion resumed. Similar transient behavior has been documented for weakly adsorbing pharmaceutical compounds (Lach and Szymonik, 2019). Despite this anomaly, the overall trend confirms that longer contact time improves naproxen removal, though the improvement is less consistent and substantially lower than that observed for mefenamic acid.



***Figure 3.4: Effect of contact time on removal efficiency of mefenamic acid and naproxen at 25 mg/L initial concentration. The data point for naproxen at 45 minutes (2.7%) represents an experimental outlier and was excluded from interpretation of the over***

Figure 3.4 shows how contact time affects removal for both drugs when we started with 25 mg/L. The difference between them is clear. Mefenamic acid performed much better at every time point. It went up steadily from 48.8% at just 15 minutes to 73.2% after an hour. There were no signs of the GAC column getting saturated. This tells us mefenamic acid really likes sticking to the GAC surface, and the longer we give it, the more it gets removed. Naproxen was weaker overall. It did increase from 8.3% in 15 minutes to 36.1% in 30 minutes and then 44.0% at one hour. But at 45 minutes we got a strange result of only 2.7%. This is probably because the outer sites on the GAC got full temporarily, so some naproxen that had already stuck on came off again before it could move deeper into the pores. This kind of thing has been seen before with drugs that don't adsorb very strongly (Lach and Szymonik, 2019). If we ignore that one odd point, naproxen still shows that more time helps, just not as reliably as mefenamic acid. So basically, mefenamic acid is much easier to remove with GAC than naproxen. This matches what we saw in the concentration tests too. The reason is probably their different chemical structures. For both drugs though, giving them more contact time helps, which means the slow step is the drug moving into the tiny pores inside the GAC particles (Sotelo et al., 2012). For designing a real treatment plant, this means we need

longer contact times, especially if we want to remove strongly adsorbing drugs like mefenamic acid effectively.

### 3.5 Tap Water Experiment

In most laboratory studies distilled water is used to prepare solutions because it is free from any background substances. However, real water sources such as tap water contain dissolved minerals, calcium, magnesium, chlorine, and natural organic matter which can compete with drug molecules for adsorption sites on the GAC surface. To evaluate the performance of the column under more realistic conditions, this experiment was repeated using tap water instead of distilled water for both mefenamic acid and naproxen at the same three concentrations of 5 mg/L, 25 mg/L, and 50 mg/L. The results are presented in the tables below.

#### Mefenamic Acid

*Table 3.7 Effect of initial concentration on mefenamic acid removal efficiency in tap water.*

| Concentration (mg/L) | Control ( $C_0$ ) | Sample ( $C_t$ ) | Removal % |
|----------------------|-------------------|------------------|-----------|
| 50                   | 1.499             | 0.838            | 44.1%     |
| 25                   | 0.727             | 0.243            | 66.6%     |
| 5                    | 0.159             | 0.099            | 37.7%     |

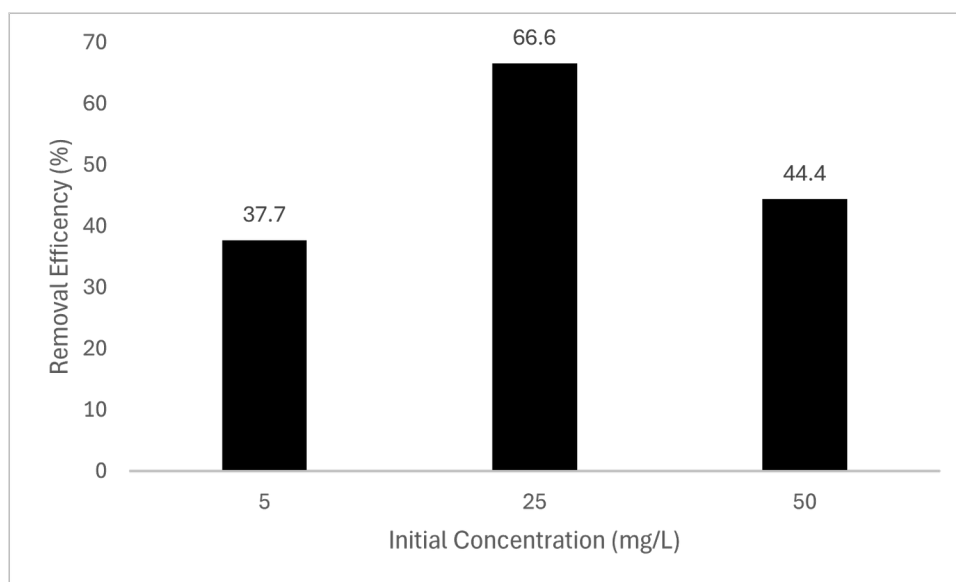


Figure 3.5: Effect of initial concentration on mefenamic acid removal efficiency in tap water.

Switching from distilled water to tap water caused a noticeable drop in removal for mefenamic acid. At 50 mg/L removal fell from 97.1% in distilled water to 44.1% in tap water. The reason is that tap water contains dissolved minerals, calcium, magnesium, chlorine, and natural organic matter. These substances compete with the drug molecules for the same adsorption sites on the carbon surface. When these competing substances fill up available sites fewer spots remain for the drug to attach to, so less drug gets removed (Rakić et al., 2015). This is an important finding for real-world applications because it shows the column will face more competition in real water and would need a deeper GAC bed or longer contact time to match the performance seen with distilled water.

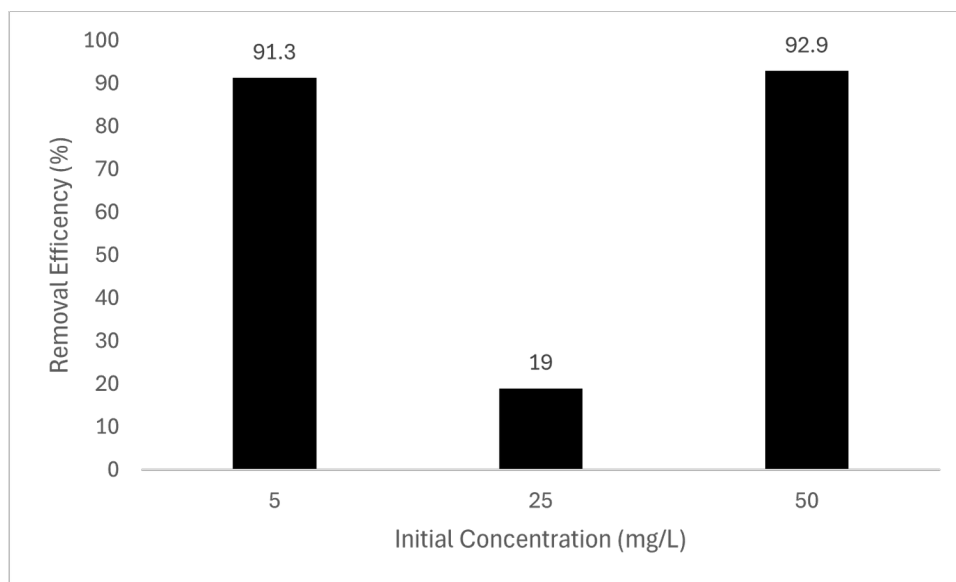
One interesting exception was at 5 mg/L where tap water removal of 37.7% was higher than distilled water removal of 18.4%. This could be due to the salting-out effect where dissolved ions in tap water reduce the solubility of the hydrophobic mefenamic acid molecule in water, pushing it more strongly toward the carbon surface. This effect is more noticeable at low drug concentrations.

### 3.5.2 Naproxen

*Table 3.8: Naproxen removal efficiency in tap water at different initial concentrations.*

| Concentration<br>(mg/L) | Control (C <sub>0</sub> ) | Sample (C <sub>t</sub> ) | Removal % |
|-------------------------|---------------------------|--------------------------|-----------|
| 50                      | 2.52                      | 0.180                    | 92.9%     |
| 25                      | 0.181                     | 0.145                    | 19%       |
| 5                       | 0.172                     | 0.015                    | 91.3%     |

Suspected experimental outlier due to inconsistent control absorbance value for 25 mg/L.



***Figure 3.6: Effect of initial concentration on naproxen removal efficiency in tap water.***

The naproxen tap water results showed an unusual pattern. The very high control absorbance of 2.52 at 50 mg/L suggests that tap water itself was contributing to the absorbance reading at 270 nm due to organic compounds naturally present in tap water. This means the apparent high removal of 92.9% at 50 mg/L may not fully reflect true drug removal and may be partially a result of this measurement interference. The 25 mg/L result of 19.9% is likely the most accurate result for naproxen in tap water and is consistent with the reduced performance seen for mefenamic acid in tap water.

### **3.6 Adsorption Mechanism**

The adsorption of both drugs onto GAC was driven mainly by two types of interactions. The first is hydrophobic interaction both mefenamic acid and naproxen have hydrophobic regions that are energetically unfavorable in water and are attracted to the non-polar carbon surface. The second is  $\pi$ - $\pi$  interaction both drugs have aromatic ring structures that interact with the  $\pi$ -electron system on the graphene-like surface of activated carbon. Mefenamic acid adsorbed more strongly throughout all experiments because of its higher log Kow value meaning it is more hydrophobic and has a stronger natural tendency to leave the water phase and attach to carbon (Sotelo et al., 2012). The pH experiments confirmed this mechanism at acidic pH where mefenamic acid is uncharged its hydrophobic character is strongest and removal is highest. For naproxen the slight improvement at

basic pH may reflect stronger  $\pi$ - $\pi$  contributions at higher pH when the molecule is fully ionized (Lach and Szymonik, 2019).

### 3.7 Overall Comparison Summary

***Table 3.9 Summary of best removal results for both drugs under different experimental conditions.***

| <b>Experiment</b>          | <b>Best Condition</b> | <b>Mefenamic Acid</b> | <b>Naproxen</b> |
|----------------------------|-----------------------|-----------------------|-----------------|
| Concentration<br>Distilled | 50 mg/L               | 97.1%                 | 58.8%           |
| pH Effect                  | pH 5                  | 76.5%                 | 61.3% (pH 10)   |
| Contact Time               | 60 minutes            | 73.2%                 | 44.0%           |
| Tap Water                  | 25mg/L                | 66.6%                 | 19.9%           |

Table 3.9 compares the best results from all experiments. In distilled water, mefenamic acid showed much higher removal than naproxen, 97.1% vs 58.8% at 50 mg/L. This is because mefenamic acid is more hydrophobic and sticks better to the carbon. In tap water the removal dropped for both drugs. Mefenamic acid went down to 66.6% and naproxen to 19.9%. The main reasons are competition from other things in tap water and interference during measurement at 270 nm. The pH and time tests also showed mefenamic acid always performed better. Overall, GAC works for removing these drugs, but real water gives lower results than lab water. This needs to be considered for actual treatment plants.

The results of this study demonstrate that the Granular Activated Carbon (GAC) fixed-bed column developed here compares favorably with removal efficiencies reported in published literature for GAC-based pharmaceutical treatment systems (Sotelo et al., 2012). Conventional treatment infrastructure such as advanced oxidation processes, nanofiltration, and reverse osmosis, while highly effective, require substantial capital investment and reliable electricity supply, making them inaccessible in many low-resource settings. The gravity-driven column evaluated in this study represents a simpler and more accessible alternative suitable for small-scale or decentralized water treatment applications (Rakić et al., 2015).

## CONCLUSION

This study evaluated the removal of mefenamic acid and naproxen from aqueous solutions using a gravity-driven GAC fixed-bed adsorption column under controlled laboratory conditions.

- Regarding the first objective, the GAC column successfully removed both pharmaceutical compounds. Mefenamic acid achieved a maximum removal of 97.1% at 50 mg/L while naproxen achieved 58.8% under the same conditions. Mefenamic acid consistently outperformed naproxen across all experiments due to its higher hydrophobicity and stronger affinity for the activated carbon surface.
- Regarding the second objective, removal efficiency increased with higher initial concentration for both drugs confirming concentration-dependent adsorption behavior. pH experiments showed mefenamic acid was better removed at pH 5 while Naproxen showed slightly better removal at pH 10. Contact time experiments confirmed that longer contact time improved removal for both drugs with mefenamic acid reaching 73.2% and naproxen reaching 44.0% at 60 minutes. Tap water experiments showed reduced removal compared to distilled water due to competing dissolved substances.
- Overall, the results confirm that GAC adsorption is an effective method for pharmaceutical removal from water and the findings contribute to SDG 6 and SDG 3.

## RECOMMENDATIONS

This study evaluated the removal of mefenamic acid and naproxen using a GAC fixed-bed column at laboratory scale with synthetic solutions. Based on the findings, the following recommendations are proposed for future research.

- First, future studies should test the column using real wastewater samples from hospital effluents or surface water sources instead of synthetic solutions. This would provide a more accurate assessment of column performance under actual environmental conditions where multiple competing substances are present.
- Second, adsorption isotherm and kinetic studies using Langmuir and Freundlich models should be conducted to determine the maximum adsorption capacity of GAC for both drugs. This would help better understand the adsorption mechanism and support the design of larger scale treatment systems.
- Third, the simultaneous removal of both mefenamic acid and naproxen together in a single mixed solution should be investigated. Since real pharmaceutical wastewater contains multiple compounds at the same time, this would give more realistic and practically relevant results compared to testing each drug separately as done in this study.

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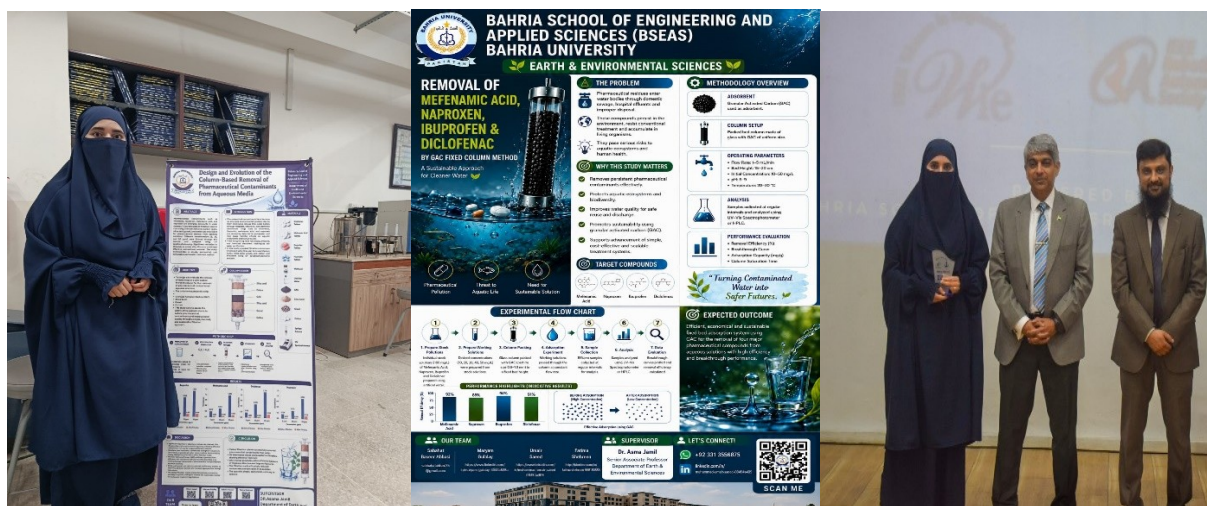
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## **APPENDIX**

**BAHRIA SCHOOL OF ENGINEERING & APPLIED SCIENCES (BSEAS) OPEN HOUSE  
2026**

## Participation in Open House Exhibition: Received Best Project Award

The following section presents the exhibition materials developed for the university Open House to communicate the findings of this research to a general audience. The standee poster and tri-fold brochure were designed to present the research objectives, methodology, results, and conclusions in a visually accessible format suitable for public display.



These exhibition materials were displayed at the university Open House 2026 alongside the physical filter demonstration column, allowing visitors to interact directly with the research and understand its practical implications for water treatment and public health.

This research was recognized through the receipt of two awards, the Best Project Award and the Best Research Award reflecting the academic quality and practical relevance of the work as assessed by the judging panel.

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