VALORIZATION OF BIOMASS WASTE FOR THE REMOVAL OF FLUOXETINE FROM AQUEOUS MATRICES

KIM NICOLAS MATTOS

Dissertation Presented to

Escola Superior de Tecnologia e Gestão - Instituto Politécnico de Bragança

To satisfy the requirements for the degree of Master's in **Chemical Engineering**

within the scope of the double degree program with the **Federal University of Technology of Paraná**

Supervisors

Prof. Doutora Ana Maria Alves Queiroz da Silva

Prof. Doutor António Manuel Esteves Ribeiro

Prof. Doutor Paulo Miguel Pereira Brito

Co-Supervisor

Prof. Doutor Vanderlei Aparecido de Lima

BRAGANÇA, 2026

AGRADECIMENTOS ESPECIAIS

Inicialmente, gostaria de agradecer ao Instituto Politécnico de Bragança e à Universidade Tecnológica Federal do Paraná. Ambas disponibilizaram infraestrutura, equipamentos e recursos financeiros que tornaram possível o desenvolvimento desta pesquisa.

Agradeço à Professora Ana Queiroz e ao Professor Paulo Brito pelo apoio e pelas contribuições ao longo deste percurso. Expresso um agradecimento especial ao Professor António Ribeiro, que dedicou generosamente grande parte do seu tempo para me orientar, compartilhar seus conhecimentos e auxiliar em todas as etapas deste trabalho. Agradeço também ao Professor Vanderlei, que, mesmo à distância, esteve sempre disponível para oferecer apoio e orientação.

Aos meus colegas de laboratório, Guilherme Schmidte e Lorena Tsubouchi, deixo minha sincera gratidão pelos conhecimentos, experiências e momentos compartilhados, que contribuíram significativamente para o meu crescimento acadêmico e profissional.

Sou profundamente grato ao meu pai, à minha irmã e a toda a minha família, que sempre acreditaram em mim e me incentivaram a perseguir meus objetivos. O apoio, a confiança e o carinho de vocês foram fundamentais durante toda essa jornada. Sem vocês, eu não seria a pessoa que sou hoje.

Por fim, gostaria de agradecer à minha terapeuta, Fernanda Apollo, pelo suporte prestado ao longo deste período. Aproveito também para destacar a importância da saúde mental no ambiente universitário. A universidade é um espaço de intenso aprendizado, mas também de desafios, marcado por elevados níveis de exigência, pressão e competitividade em busca de desempenho. Cuidar da saúde mental é essencial para que estudantes possam desenvolver seu potencial de forma saudável.

A todas as pessoas que, de alguma forma, contribuíram para a realização deste trabalho, expresso minha mais sincera gratidão. Sem o apoio de cada uma delas, este trabalho não teria sido possível.

Muito obrigado!

ABSTRACT

This research examines the use of olive pit waste to remove fluoxetine, a pharmaceutical drug classified as an emerging pollutant. It is an constituent that threatens the environment due to its bioaccumulation. Conventional wastewater treatment plants cannot remove it completely. The activated charcoal used was previously recovered from olive pits and chemically activated with NaOH.

Kinetic tests showed that as temperature increased, the amount of removal from the material increased as well, reaching up to 98.87% at 45 degrees Celsius. Overall the pseudo second-order kinetic model fit the experimental data the best with a χ^2 value equal to 0.003725 at 45 degrees Celsius. The Freundlich model fit the equilibrium study better than the Langmuir model, indicating that adsorbing compounds are done so on heterogeneous surfaces and continue through multiple layers. The maximum capacity of adsorption from Langmuir's model was identified to be 17.07 mg g^{-1} with regards to olive pit adsorbent.

For thermodynamic analysis, the activation energy was found to be 42.32 kJ mol^{-1}, and the Gibbs free energy indicated a value of -0.851 kJ mol^{-1} supporting the conclusion that the adsorption process is both spontaneous and favourable thermodynamically. Based on these results, it can be concluded that activated carbon from olive pits is a sustainable and effective alternative for treating water with pharmaceutical micropollutants.

Keywords: Activated carbon, Adsorption, Biomass valorization, Emerging pollutants, Fluoxetine

RESUMO

Esta pesquisa examina o uso de resíduos de caroço de azeitona para remover fluoxetina, um medicamento farmacêutico classificado como um poluente emergente, pois é um constituinte que ameaça o meio ambiente devido à sua bioacumulação. Estações convencionais de tratamento de esgoto não conseguem removê-lo completamente. O carvão ativado utilizado foi anteriormente recuperado do caroço de azeitona e ativado quimicamente com NaOH.

Testes cinéticos mostraram que, à medida que a temperatura aumentava, a quantidade de remoção do material também aumentava, chegando a 98,87% a 45 graus Celsius. No geral, o modelo cinético de pseudo-segunda ordem se ajustou melhor aos dados experimentais, com um valor χ^2 igual a 0,003725 a 45 ºC. O modelo de Freundlich se ajusta melhor ao estudo de equilíbrio do que o modelo de Langmuir, indicando que compostos adsorventes são feitos em superfícies heterogêneas e continuam por múltiplas camadas. A capacidade máxima de adsorção do modelo de Langmuir foi identificada como 17,07 mg g^{-1} no que diz respeito ao adsorvente preparado com caroço de azeitona.

Para análise termodinâmica, a energia de ativação foi encontrada em 42,32 kJ mol^{-1}, e a energia livre de Gibbs indicou um valor de -0,851 kJ mol^{-1}, apoiando a conclusão de que o processo de adsorção é tanto espontâneo quanto favorável termodinamicamente. Com base nesses resultados, pode-se concluir que o carvão ativado dos caroços de azeitona é uma alternativa sustentável e eficaz para tratar água com micropoluentes farmacêuticos.

Palavras-chave: Adsorção, Carvão ativado, Fluoxetina, Poluentes emergentes, Valorização de biomassa

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LIST OF ABBREVIATIONS AND ACRONYMS

CFR	Code of Federal Regulations
BET	Brunauer–Emmett–Teller
DEA	Drug Enforcement Administration
EDX	Energy-Dispersive X-Ray Spectroscopy
EPA	U.S. Environmental Protection Agency
FTIR	Fourier Transform Infrared Spectroscopy
HPLC	High-Performance Liquid Chromatography
NIR	Near Infra-Red
RCRA	Resource Conservation and Recovery Act
SEM	Scanning Electron Microscopy
WSER	Wastewater Systems Effluent Regulations
WWTP	Wastewater Treatment Plant

1 MOTIVATION AND OBJETIVES

1.1 INTRODUCTION

Due to an increase in climate change and strong political pressure to highlight the global impact of pollution on water supply, environmental concerns have risen tremendously within the last few years. As a result, many research efforts that have produced better knowledge of new types of pollutants which include but are not limited to pharmaceuticals, hormones, and microplastics have produced major challenges for monitoring and mitigating these types of compounds.

One major area of concern with respect to emerging pollutants is the continuous introduction of fluoxetine (Prozac) into the environment, as it has the potential to bioaccumulate in aquatic organisms until it can no longer be biologically degraded.

Conventional Wastewater Treatment Plants are unable to adequately remove these pharmaceutical compounds from wastewater. This limitation will require a search for an alternate, low-cost method of removal, such as adsorption via biochar. Adsorption is an ideal removal method due to its simplicity, high removal efficiency, and low operational costs. This study aims to evaluate the residual fluoxetine adsorbed by the activated carbon produced via the chemical activation of olive pit residue using NaOH in order minimizing the environmental impacts associated with these pharmaceutical micropollutants.

1.2 OBJECTIVES

To evaluate the study of fluoxetine removal from aqueous matrices through the adsorption process using low operating cost adsorbents.

2 LITERATURE REVIEW

In this work, the removal of fluoxetine from aqueous matrices will be carried out through the adsorption of biochars.

2.1 WATER POLLUTION

Water has a vital function in the environment and in human society (Rodell et al., 2018), all living beings depend on water to survive, rivers, lakes and oceans are natural habitats for a vast number of organisms.

The anthropogenic activities associated with the industrial act together with climate change have intensified the pollution of the environment across the planet. Putting human health and the living beings that depend on it at risk, thus becoming a challenge for global sustainable development bodies (Luo et al., 2024).

In North America, the excessive discharge of 12.000 tons of nitrogen irresponsibly dumped by chemical industries into rivers caused damage of more than 8.1 million square kilometers of drinking water, generating scarcity and making water unfit for consumption (Tang et al. 2021). In Asia, more than 62.000 cancer cases per year are attributed to exposure to contaminated water, with the majority of diagnoses related to areas affected by water scarcity (Jiang et al., 2024). Therefore, dealing with the management of anthropogenic pollution has become a necessary issue.

In this scenario, it becomes important to analyze emerging pollutants, such as pharmaceuticals, hormones, microplastics, and derivatives of hygiene products, whose widespread presence and sublethal effects pose new challenges for monitoring, regulation, and remediation.

2.2 EMERGING POLLUTANTS

Emerging pollutants are chemical compounds of synthetic or natural origin, with a rapid effect on the environment, especially on water resources, which cause negative effects on nature. These substances can degrade, reacting and transform into unknown by-products (Hejna et al., 2022; Zhang et al., 2025).

Emerging pollutants are diverse, their groups include pharmaceuticals (Sardar et al., 2025), polymers (Ma et al., 2024), pesticides (Shaji et al., 2024), hygiene and personal care products (Xu et al., 2025), among other fields of the chemical industry (Bell et al., 2025).

Some of these substances are found in low concentrations but present potential ecological risk. The relationship between emerging pollutants and micropollutants is associated, but while the concept of emerging pollutants emphasizes the scientific and regulatory concern around these substances, the concept of micropollutants is associated with the quantities present in the environment.

2.3 MICROPOLLUTANTS

Emerging micropollutants are substances present in the environment in very small amounts, in the range of micrograms or nanograms (Lim et al., 2024). However, perpetual use and consistent disposal can have harmful effects on human and ecosystem health (Helbling et al., 2010).

Despite technological advances, these pollutants are at levels close to detection limits, which depend on expensive and professional analyses, such as gas or liquid chromatography (Gopal et al., 2020). The use of this equipment requires a high operational cost to comply with the legislative standards of each country.

2.4 FLUOXETINE

In the early seventies, the first evidence of serotonin's role in the treatment of depression emerged, one of the hypotheses of studies considered that the increase in the neurotransmitter serotonin would be a plausible response to depression. Based on this hypothesis, possible agents were pointed out that could endanger the reuptake of serotonin by proteins of the neuron itself, causing an increase in the concentration of the molecule in the synaptic cleft, where the improvement effects arise. These studies

led to the discovery of fluoxetine as we know it today, which was subsequently approved for the treatment of depression by the U.S. Food and Drug Administration in 1987 (Wong et al., 2005).

Fluoxetine, known by the trade name Prozac, in the IUPAC form N-methyl-3-phenyl-3-[4-(trifluoromethyl)phenoxy]propane-1-amine has a molecular weight of 309.33 g mol^{-1} (Lima & Vaz, 2025), representation in Figure 1.

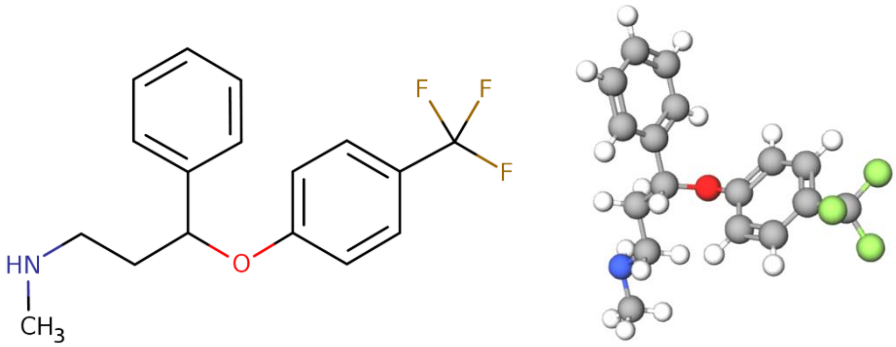

Figure 1 – Structural formula of fluoxetine and 3D visualization of the molecule.
Adapted from: Herman Bergwerf, 2025.

Fluoxetine is almost as effective as other tricyclic antidepressants, but it is not indicated for conditions where the patient has symptoms of severe melancholic depression, this medication is indicated for cases of moderate depression. Once treatment is initiated, when there is little improvement within the period of four to six weeks, in these situations other treatments should be sought. Overdoses do not cause serious effects, common effects include nausea, loss of appetite, insomnia and nervousness are the most notable symptoms. All these effects can be controlled according to the dose ingested by the patient.

The dramatic pace of lifestyle in modern society, where loneliness is increasingly widespread, has caused an increase in various psychiatric disorders and the number of diagnoses for mental disorders continues to grow worldwide, generating a relevant impact with great social and economic consequences, which in themselves also reflect on the environment (Gili et al., 2014).

2.5 EUROPEAN LEGISLATION

Several directives and regulations have been carried out by European Union countries related to emerging pollutants since the 2000s (Hawash et al., 2023).

Recently in Europe, the Urban Wastewater Treatment Directive, established by the European Union in 1991 (Liberti et al., 2024), imposes the implementation of regular periodic treatments with targets to be met by the year 2045.

Currently, the organization is targeting the removal of twelve emerging micropollutants, subclassified into two groups according to the difficulty of treating the waste. The first group includes amisulpride, carbamazepine, clarithromycin, citalopram, diclofenac, hydrochlorothiazide, metoprolol, venlafaxine. In the second category we have the "common" conventional disposal products: candesartan, benzotriazo, 4-methyl-benzitriazole and Ibersatan. The entity establishes a limit of 80% as the minimum removal efficiency to be guaranteed in the annual average. If the concentrations of more than six substances are below the established detection limits, regulators are allowed to select additional substances for the calculation of the removal efficiency of the materials (Domínguez et al., 2025).

2.6 U.S. LEGISLATION

Within the U.S. territory, the agency with the highest environmental authority responsible for waste treatment is the EPA (Environment Protection Agency) (US EPA, 2013), which determines the rules for the treatment and disposal of wastewater with the support of the RCRA (Resource Conservation and Recovery Act), in addition there are other institutions with specific powers that do not overlap with the EPA, but they have complementary relevance in specialized areas such as the DEA (Drug Enforcement Administration, 2024).

The Code of Federal Regulations (CFR) informs the official administrative standards for the protection of the environment. This code provides specifications on

water quality standards which are defined in Title Forty, Chapter I, Subchapter D, Part 141 under Water Programs. While there is regulation for metals, pesticides, and by-products, there are no direct citations about "emerging pollutants" (United States Environmental Protection Agency, 2024).

In countries like Canada, each territory has its own ministry of the environment that regulates, licenses and inspects the ETE's (Sewage Treatment Plants). Despite this localized control, the main regulation is made official by the Wastewater Systems Effluent Regulations (WSER) which establishes the parameters of the legal limits for water treatment in article SOR/2012-139, but there are no mentions or regulations citing emerging poles (WSER, 2012).

2.7 BRAZILIAN LEGISLATION

In Brazil, CONAMA (National Council for the Environment and Climate Change), collegiate of the Ministry of the Environment, guides environmental guidelines for municipal and state entities in the country.

CONAMA Resolution No. 357/2005 establishes conditions and standards for the discharge of effluents into water bodies such as total coliforms, pH, turbidity, among others. However, there are no citations about emerging compounds in any regulation (CONAMA, 2005).

2.8 IMPACTS OF FLUOXETINE ON THE ENVIRONMENT

The impacts of fluoxetine on the environment are low for the exposure levels that currently occur; however this substance can bioaccumulate in tissues and in the aquatic environment, causing interference in the endocrine system of aquatic beings (Gould et al., 2021).

As a result of the high consumption, the substance studied can be observed in different regions and concentrations (Souza et al., 2022) on the planet. 7.6 – 20 ng

L^{-1} have been reported in a Wastewater Treatment Plant (WWTP) in Canada (Lajeunesse et al., 2012); Between 100-600 ng L^{-1} in Costa Rican WWTP's (Ramírez-Morales et al., 2020); From 7.5–750 ng L^{-1} in Belgium (Boogaerts et al., 2019); Values of 50 until 58 ng L^{-1} have been reported in a WWTP London, United Kingdom (Ng et al., 2020); 4.7-9.4 ng L^{-1} in the Dal River located in Sweden (Lindim et al., 2016); 2.1–19.5 ng L^{-1} Rio Lis in Portugal (Paíga et al., 2016) and 0.58 ng L^{-1} in the sea of Baiá de Santos in Brazil (Cortez et al., 2019). The concentrations described are found in Table 1.

Table 1 – Fluoxetine concentrations found in different aqueous media in different countries.

Country	Local	Concentration (ng L^{-1})	Author
Canada	WWTP	7.6 – 20	(Lajeunesse et al. 2012)
Costa Rica	WWTP	100 – 600	(Ramírez-Morales et al. 2020)
Belgium	WWTP	7.5 - 750	(Boogaerts et al. 2019)
United Kingdom	WWTP	50 – 58	(Ng et al. 2020)
Sweden	River	4.7–9.4	(Lindim et al. 2016)
Portugal	River	2.1–19.5	(Paíga et al. 2016)
Brazil	Sea	0.58	(Cortez et al. 2019)

In North Carolina, USA, fluoxetine levels between 104-119 ng L^{-1} were measured by samples from a local river. In the laboratory, the effects of the medication diluted in samples from 300 µg L^{-1} to 3000 µg L^{-1} in mussels were evaluated. The effects on mussels include disruptions in the reproductive cycle, responsible for a difference in natural behavior, which can compromise reproduction and the survival of the species. When subjected to concentrations of 300 µg L^{-1}, females of *E. complanata* showed non-viable larval participation and changes in increased bait display behavior, which impacts the reproductive cycle. When subjected to concentrations of 3000 µg L^{-1}, males of *E. complanata* significantly induced the release of spermatozeugmata during a 48h exposure. Although these tests have lower concentrations than those measured in nature, additional tests are warranted to evaluate the effects of fluoxetine exposure in the long term (Bringolf et al., 2010).

Fish and other aquatic organisms are exposed to a constant load of contaminants from sewage treatment plants. Fish are good indicators of water quality due to their ability to absorb pollutants present in the water, although studies point to the presence of different drugs in low concentrations, they still do not represent an immediate risk to exposed organisms (Huerta et al., 2018).

In view of the various environmental impacts caused by fluoxetine, it is important to develop efficient methods for its removal, so different approaches have been studied to mitigate the effects caused in nature.

2.9 FLUOXETINE REMOVAL

Conventional WWTP's (Sewage Treatment Plants) were not designed and are not prepared to eliminate a large part of pharmaceutical products, making it a questionable problem for environmental contamination (Silva et al., 2021).

2.9.1 Alternative removal methods

Several methods of fluoxetine removal are employed for the aqueous matrices of aquatic environments: Adsorption, advanced oxidative processes (Aghaeinejad-Meybodi et al., 2019), reverse osmosis (Dalbosco et al., 2021), coagulation, among others.

In hydrolysis and photolysis studies, the persistence of fluoxetine (selective serotonin reuptake inhibitor) (Chighine et al., 2025) in aqueous solutions were investigated in exposed environments with variations of ultraviolet abutment, showing high stability in the face of the analyzed processes. The phase dissipation rate was similar between the illuminated and dark atmospheres, proving resistant to the incidence of ultraviolet light. Fluoxetine has also been shown to be highly stable in aqueous solutions and at microbial degradations (Kwon & Armbrust, 2006).

2.9.2 Adsorption Methods

Adsorption plays an important role in processes related to water treatment, due to its simplicity and low operating cost with good removal efficiency (Thakur et al., 2021).

Different adsorbents have been studied in the literature aiming at the removal of contaminants from aqueous matrices. Table 2 shows a comparative summary containing information on the type of adsorbent, removal capacity, kinetic constant, experimental conditions and adsorption mechanisms. It is noted that biochar adsorbents with different origins present variations in the result. The raw material, obtained by different pyrolysis processes, shows variations in porosity and availability of active sites that are directly related to the adsorption capacity of the material. On the other hand, materials such as clay and microporous gels tend to have a higher adsorption capacity, but with lower kinetics, associated by the physical adsorption mechanism.

Table 2 – Capacity to remove different adsorbents for fluoxetine.

Adsorbent	Removal Capacity ($mg\ g^{-1}$)	Kinetic constant ($g\ mg^{-1}\ min$)	Temperature (Cº)	Chemisorption/ Fissorption	Author
Biochar (Commercial)	8.14	0.242	25	Chemisorption	Escudero-Curiel et al., 2021
Biochar (Eucalyptus)	6.41	-	25	Chemisorption	Fernandes et al., 2019
Biochar (Rice)	20.49	0.181	25	Chemisorption	Escudero-Curiel et al., 2021
Purified Tunisian clay	390.28	0.033	25	Chemisorption and Fissorption	Atawa et al., 2025
Printed Magnetic Polymer	38.54	7.79×10^{-3}	25	Chemisorption	Tian et al., 2025
Macroporous Cryogels	80.6	$0.81x10^{-2}$	25	Fissorption	Nkana, Chabot, et al., 2024b
Activated biochar (Olive pit)	4.82 - 146.45	$4x10^{-3}$	25	Chemisorption and Fissorption	(Pratishtha Khuranet al.a, 2025)

2.10 CHARACTERIZATION METHODS

For the characterization of adsorptions is constantly performed mainly through the Fourier Transform Infrared Spectroscopy (FTIR) technique that measures light absorption and converts the raw data to an improved version (Wang et al., 2025) (Giraldo & Marin, 2025) when compared to traditional Near Infrared Red (NIR). For complementary characterization, X-ray diffraction (EDX) (Bhattacharjee, 2025; Leek, 2023) is also recurrently used. Table 3 shows the most recurrent methods of characterization of adsorbent materials.

Table 3 – Current characterization methods for activated carbons.

Characterization method	Author
(EDX)	(Nkana, Lajeunesse, et al., 2024)
(EDX)	(Rad et al., 2024)
(FTIR)	(Narayanan et al., 2021a)
(EDX)	(Escudero-Curiel et al., 2022)

In a complementary way, Scanning Electron Microscopy (SEM) (Shahabi & Tavakol, 2017) can be used to measure material morphology and the Brunauer–Emmett–Teller (BET) methods (Moura et al., 2024) are used to measure the surface area and volume of the activated carbons.

3 MATERIALS AND METHODS

3.1 REAGENTS

Ultra-pure Water, Fluoxetine hydrochloride (≥ 98%) from Sigma-Aldrich and acetonitrile (≥ 99.9%) from Honeywell.

3.2 MATERIALS AND EQUIPMENT

A Jasco high-performance liquid chromatography (HPLC) system (Extreme series) coupled with a DAD detector and a Nucleosil 100-5 C18 chromatography column with 5 µm particle diameter, 250 mm x 4.6 mm from Macherey-Nagel, was used for the identification and quantification of fluoxetine. Photos of the equipment are shown in Figure 2.

Figure 2 – Jasco HPLC and incubator.

The pH of the samples was measured using a digital pH meter from Hanna Instruments, Edge line. The assays were conducted in an incubator with orbital shake model ES-20/60 Orbital Shaker-Incubator. Ultra Pure Water was used in all experiments.

3.3 ACTIVETED CARBON EMPLOYED IN THE ADSORPTION EXPERIMENTS

An important attribute of activated carbon is a high specific surface area; having this characteristic means the ability to capture many different types of materials on the activated carbon through adsorption is high. Furthermore, not only is it important that an activated carbon product has a large specific surface area, but it must also have a microporous structure, as this provides for the ability to adsorb even very small-sized materials such as volatile organic chemicals, and pharmaceuticals (Wu et al., 2026).

Based on previous research made by colleagues, chemical activation presented more efficient results than physical activation. For this reason, the adsorbent chosen was chemically activated carbon with olive pit (Milani, 2023).

The activation of olive stones generated by chemical activation using sodium hydroxide with a 1:0.1:2 ratio of adsorbent, base, and water at 500 ºC for 1.5 hours produced activated carbon from olive stones. Carbonization yield was 33.86% ±1.71%. The activated carbon produced from olive stone showed the highest alkalinity and the activated carbon produced contained the highest concentration of basic functional groups.

3.3.1 pH point of zero charge

Knowing how to establish the zero point of charge pH_{PZC} provides a means by which to determine at what pH the surface charge of the particulate becomes neutral. This is vital information when determining how the surface of the adsorbent will interact with the species in the liquid. pH_{PZC} and functional groups are shown in Table 4 below.

Table 4 – pH_{PZC} and functional groups (adapted from Milani, 2023).

pH_{PZC}	**Basic functional groups (mmol g^{-1})**	**Acidic functional groups (mmol g^{-1})**
8.92 ± 0.04	0.72 ± 0.01	0.32 ± 0.01

The pH_{PZC} was found to be 8.92 ± 0.04. When the pH is less than 8.92 the adsorbent surface has a predominance of positive surface charge and when the pH is greater than 8.92 the adsorbent surface has a predominance of negative surface charge. The number of more basic groups 0.72 ± 0.01 m $molg^{-1}$ is greater than the number of more acid groups 0.32 ± 0.01 m $molg^{-1}$. The basic nature of the adsorbent surface is indicative of the surface charge type. The properties of coal are listed in Table 5.

Table 5 – Textural Properties of activated carbon (adapted from Milani, 2023).

S_{BET} (m^2 g^{-1})	S_{mic} (m^2 g^{-1})	V_{tot} (cm^3 g^{-1})	D_p (nm)
27 ± 0.4	15 ± 0.3	0.039 ± 0.002	1.54 ± 0.02

S_{BET} (superficial area); S_{mic} (surface area of micropores); V_{tot} (total pore volume); D_p (average pore diameter).

Approximate elemental composition of activated carbon produced from olive stone is: 80.73% Carbon; 2.65% Hydrogen; 5.53% Ash. The C/H molar ratio of 2.54 is indicative of successful carbonization and chain aromatization. In the thermogravimetric analysis between 350 and 547 °C, a mass loss of 93.70% occurred. Image of the charcoal used in Figure 3.

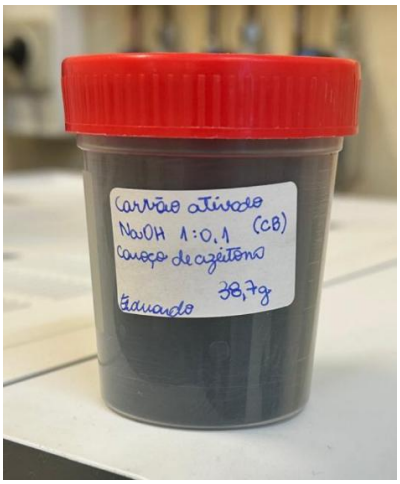

Figure 2 – Activated carbon pre-prepared from olive stones and activated with NaOH.

After the selection of activated carbon at optimum conditions, the calibration curve was plotted to assess its adsorbing capacity. The calibration curve was made from the standard solutions of the adsorbate. Through this, it is possible to calculate the amount of adsorbate left when it comes into contact with activated carbon.

3.4 FLUOXETINE CALIBRATION CURVE

Quantification of fluoxetine was performed using Jasco Extreme HPLC. In the mobile phase 60% acetonitrile, 40% ultrapure water with 0.05% trifluoroacetic acid. The detection was performed using λ= 227 nm and a flow rate of 1.0 mL min^{-1}.

The calibration standards were prepared from fluoxetine hydrochloride that was diluted in ultrapure water with concentrations ranging from 1 ppm to 100 ppm. The linear coefficient of determination R^2 was obtained with Equation 1.

The stock solution with 100 ppm was prepared using ultrapure water, the calibration points were prepared ranging from 1, 2, 4, 6, 8 and 10 ppm.

3.5 KINETICS

The kinetics study was carried out at 15, 30, 45, 60, 120, 240, and 480 minutes using 25mL of fluoxetine at 3 ppm and 25 mg of adsorbent, stirring for 150 rpm at 25, 35, and 45 ºC.

The coefficient of determination, standard deviation (SD) and Relative Standard Deviation (RSD) that expresses the relative dispersion of the data compared to the mean were calculated with Equations 1, 2 and 3 respectively.

$$R^2 = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \quad (1)$$

R² or coefficient of determination is obtained using the formula given below. The calculation of R² involves dividing the sum of the squared difference between the predicted value $\hat{y}_i$ and the mean of the experimental value $\bar{y}$ by the sum of the square of the difference between y_i and the mean of the experimental value. n represents the number of points on the graph.

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n}} \quad (2)$$

$$CV = \frac{\sigma}{\bar{x}} \cdot 100 \quad (3)$$

The standard deviation σ or statistical measure that shows the estimation of variability from the mean $\bar{x}$ is explained in Equation 2. The computation of this measure comes from the square root of the mean values divided by the total number of members in the sample.

CV (coefficient of variation) is defined in Equation 3 as the relative measure that shows how any particular member varies relative to other members in the sample. This is a measure that determines the relative variability in relation to the mean value as a percentage. The reason percentages are used is because their calculations do not have units of measurement, which means we can make comparisons between variabilities in the same way even though they may be from different units or different magnitudes of data.

To calculate the percentage efficiency of fluoxetine removal, Equation 4 was used (Salehian et al., 2026a).

$$R.E\ (\%) = \frac{c_i - c_f}{c_i} \cdot 100 \quad (4)$$

Where c_i is the initial concentration, c_f is the final concentration, both expressed in mg L^{-1}.

The amount adsorbed as a function of time was calculated with Equation 5 (Narayanan et al., 2021b).

$$q_t = \frac{(c_0 - c_f) \cdot V}{m_{ads}} \quad (5)$$

Equation 5 calculates the amount of adsorbed substance per unit mass of adsorbent, as a function of time and is denoted by q_t, which is defined by the difference

between the initial concentration c_0 of the solution and the final concentration c_f, divided by the mass of m_{ads} adsorbent used in the experiment in $mg_{FLX}\ g_{Ads}^{-1}$.

In kinetics, the pseudo-first order, pseudo-second order and Elovich models were adjusted according to the experimental data, in the Equations 5, 6 and 7 q_t represent the amount adsorbed over time in $mg_{FLX}\ g_{Ads}^{-1}$ and t time in minutes (K, 1898).

In accordance with this theory, the Equation 6 shows that the speed of adsorption depends directly on the number of sites where anything can be adsorbed. This theory is most appropriate when analyzing adsorption processes at the early stages, when adsorption takes place under the influence of diffusion from outside and movement of the substance. The kinetic constant k_1 min^{-1} denotes pseudo-first-order adsorption, while q_e is the equilibrium adsorption capacity in mg g^{-1} (Korol et al., 2026).

$$q_t = q_e[1 - e^{k_1 t}] \quad (6)$$

In this model of Equation 7, the adsorption mechanism considered is attributed to the interaction force between adsorbate and adsorbent (chemisorption). Moreover, the model considers the site-occupancy rate to be proportional to the square of the number of availabilities of active sites. It should be noted that this model has been widely accepted since it fits adequately to the experimental data for all contact times and also calculates both q_e mg g^{-1} and pseudo-second order rate constant k_2 g mg^{-1} min^{-1}.

$$q_t = \frac{q_e^2 k_2 t}{(1 + k_2 q_e t)} \quad (7)$$

Empirical model is helpful to us in explaining the chemisorption of heterogeneous surfaces in other words, when we activate the surface using a chemical reaction, and hence this empirical model would prove to be very helpful in cases of chemisorption involving heterogeneous surfaces. The parameter α mg g^{-1} min^{-1} is

indicative of the adsorption rate at the beginning of the process while β g mg^{-1} represents the surface coverage and amount adsorbed over time q_t. What must be noted here is that, unlike the other empirical models, the Elovich empirical model assumes no order of reaction for a particular system (Khamis et al., 2024). Elovich's model is described in Equation 8.

$$q_{t} = \frac{1}{\beta}\ln(1+\alpha\beta t) \tag{8}$$

3.6 ERROR ANALYSIS

To evaluate which model had the best fit, the reduced chi-square described by Equation 9 was used. To achieve the best optimized value for the Freundlich and Langmuir functions, the functions were minimized using the chi-quadrature method.

$$X^2 = \frac{(O_i - E_i)^2}{\rho_i^2} \tag{9}$$

X^2 is the abbreviated form of what is known as the chi-square statistic. O_i is the abbreviation for experimental observations. On the other hand, E_i stands for the predicted values in the model, while ρ_i indicates the standard deviation of all the observations. This statistical requirement helps compare quantitatively different models, that is, it enables us to see the extent to which the model differs from the experimental observation through the distribution of the data (J. Wang & Guo, 2020).

Optimization was accomplished through the use of the Solver function in Excel. Solver was defined to minimise the chi-square function shown in Equation 9, by changing the model parameters iteratively and obtaining the lowest possible value of chi-square through each iteration. This allowed for the identification of the parameter set that produced the best fit between the experimental data and the predicted values of the adsorption model.

3.7 THERMODYNAMIC STUDIES

For the thermodynamic studies, the pseudo-second order rate constants k_2 obtained and calculated in kinetics are necessary. Applying natural logarithm to the constant and doing the inverse transformation with the Temperature in Kelvin, we obtain a linear regression with data that allows us to calculate E_a with Arrhenius Equation. Data for the computation of E_a corresponding to the adsorption process were based only on the kinetic experiments conducted at three different temperatures (25, 35, and 45 ºC).

Bellow Equation 10 relates standard free energy change and equilibrium constant of the system using the relation between ΔG^0 and standard free energy change or spontaneity of reaction, where R is the universal gas constant, T is the absolute temperature in Kelvin, and ln is the natural logarithm and $\ln k_L$ is equilibrium constant related to Langmuir model of adsorption (Zaheer et al., 2019).

$$\Delta G^0 = -RT \ln k_L \tag{10}$$

The value of ΔGº determines if and how easy it is for a particular reaction to happen or has happened since it determines if there was any difference in the Gibbs free energy of the system.

Equation 11 shows the Arrhenius Equation used to calculate the activation energy. In Equation 12, its linearized form.

$$k_2 = Ae^{-\frac{E_a}{RT}} \tag{11}$$

$$\ln k_2 = \ln A - \frac{E_a}{RT} \tag{12}$$

A is the pre-exponential factor that represents the frequency of effective collisions, whereas E_a in J mol^{-1} is the activation energy required for the reaction. The symbol R with the value of 8.314 J mol^{-1} K^{-1} stands for universal gas constant, while

T denotes absolute temperature measured on the Kelvin scale, whereas e is the base of the natural logarithm.

3.8 ADJUSTMENT MODELS FOR ADSORPTION BALANCE

The study of the adsorption equilibrium was carried out by varying the mass of activated carbon between 2 and 30 mg with 25 mL of fluoxetine at 3 ppm diluted in ultrapure water with pH 6.5 with agitation of 150 rpm at 45 ºC for 24 h, and adjusted to the models. The experiments were performed in duplicate and the readings in triplicate.

Langmuir and Freundlich isotherm models have an important role in understanding and describing how adsorbents interact with adsorbates in terms of equilibrium isotherms. With these models is possible to compare, interpret, or generalize experimentally generated equilibrium.

Equation 13 is appointed as Freundlich model, assumes that the surface is heterogeneous and allows the possibility of multilayer behavior, is given by K_F mg g^{-1} (L $mg^{-1})^{1/n}$; the constant of equilibrium or adsorption, which represents the ability of the adsorbent and n is the constant which represents the heterogeneity of the surface adsorbed as well as the preference for the adsorption process. The higher the value of n, the more favorable the adsorption (J. Wang & Guo, 2020).

$$q_e = K_F\, c_e^{1/n} \tag{13}$$

The Langmuir model describes the situation when you have a homogeneous adsorbent with a limited supply of sites, all identical in nature. It also predicts the formation of only one layer of adsorbate molecule. The Langmuir equation is given by Equation 14, the maximum adsorption capacity for the adsorbent is given by Q_m mg g^{-1} and the Langmuir constant K_L L mg^{-1} that measure the strength of the adsorbent for the adsorbate, the larger K_L, the stronger affinity between the adsorbate and the adsorbent (Demiti et al., 2025a).

$$q_e = \frac{Q_m K_L c_e}{1 + K_L c_e} \quad (14)$$

4 RESULTS AND DISCUSSION

4.1 IDENTIFICATION AND QUANTIFICATION OF FLUOXETINE

Fluoxetine chromatogram presented in Figure 4 shows retention time at approximately 2.475 minutes.

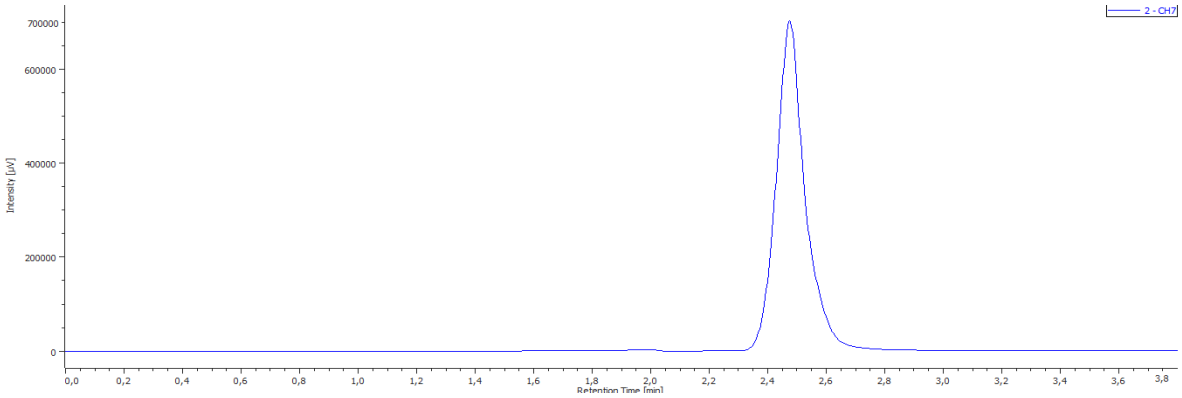

Figure 3 – Example of a fluoxetine HPLC-DAD chromatogram.

The chromatogram area is proportional to the amount of the analyte.

With the fluoxetine calibration curve, it is possible to interpret the concentration as a function of the signal received from the equipment. The constructed curve has a total of 6 calibration points ranging from 1 to 10 ppm. The trend line is described by the equation y = 61047x – 3061.4. The coefficient of determination (R^2) is 0.9993. Graph shown in Figure 5 below.

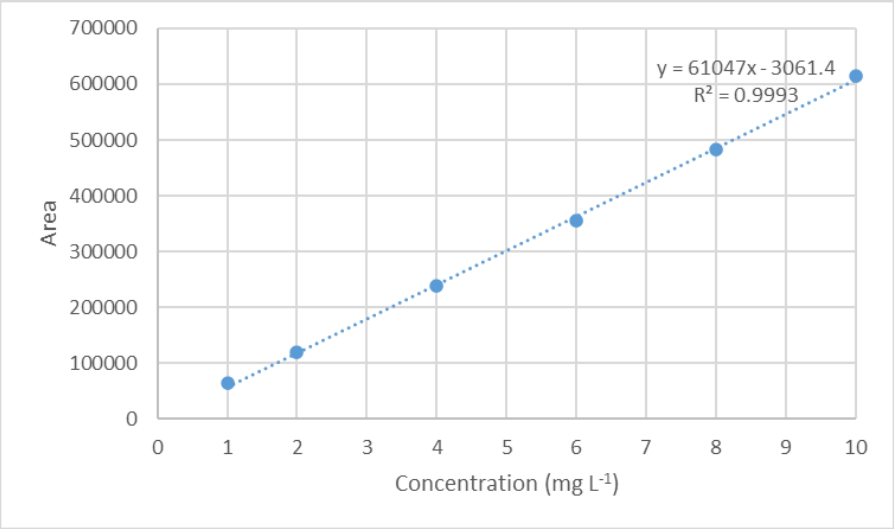

Figure 4 – Fluoxetine calibration curve.

In Table 6, at nearly all levels examined, the coefficient of variation is higher than 10%, ranging from 9.25 to 34.12%; however, at 1 ppm, the coefficient of variation is 34.12 and thus, more variable than the other levels assessed.

Table 6 – Statistical parameters SD and RSD of the calibration curve.

C (ppm)	Area 1	Area 2	Area 3	Average	SD	RSD (%)
1	52342	49683	88470	63498	21667	34.12
2	133811	114521	111340	119891	12160	10.14
4	210103	248462	255226	237930	24335	10.23
6	323996	352097	389511	355201	32868	9.25
8	460042	541770	446332	482715	51601	10.69
10	605154	688691	550740	614862	69486	11.30

4.2 KINETICS OF FLUOXETINE ADSORPTION

The initial concentration of fluoxetine was 3 ppm, the experiment was carried out at temperatures of 25, 35 and 45 ºC. Figures 6 and 7 show the removal and amount of fluoxetine as a function of time.

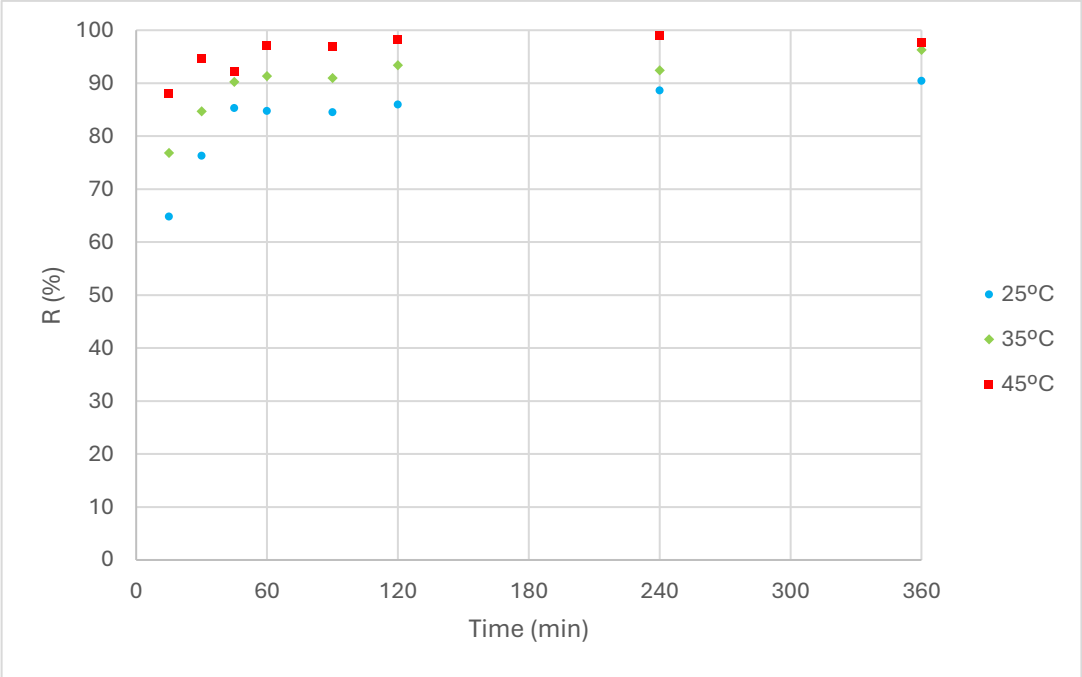

Figure 5 – Removal of fluoxetine as a function of time at 25, 35 and 45 ºC.

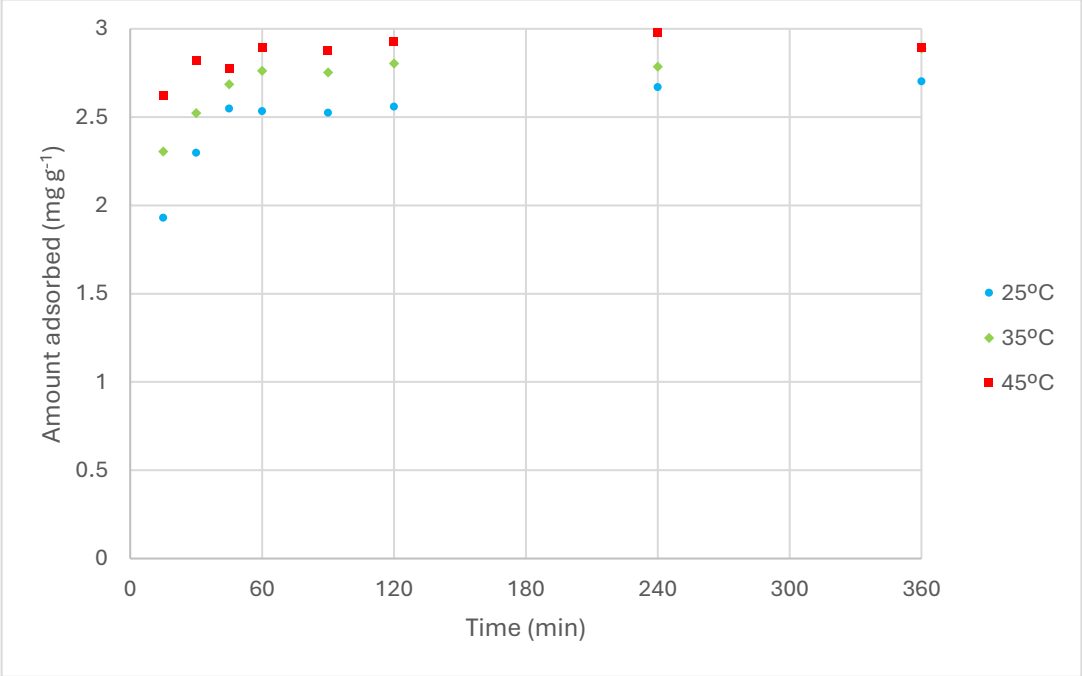

Figure 6 – Amount of fluoxetine adsorbed as a function of time at 25, 35 and 45 °C.

Table 7 shows the highest percentage of fluoxetine removal at temperatures of 25, 35 and 45 ºC.

Table 7 – Best removal efficiency results as a function of temperature.

R. E (%)	T (ºC)
90.41	25
96.00	35
98.87	45

Examining the graph, there is possible see a relationship between as removal increases, so does the temperature. For instance, for a temperature of 25 °C, we have R = 90.4%, for 35 °C we get R = 96.0%, and at 45 °C the value of removal becomes 98.8%. Based on this, there seems to be some relationship between R(%) and temperature. This is true as long as the temperature does not have a negative impact on liquid diffusion.

At very high temperatures, the desorption rate could increase. This is true as long as the temperature does not have a negative impact on the break of the dipole-dipole interaction. At very high temperatures, the desorption rate could increase due to the excess energy overcoming.

For purposes of establishing the ideal method of fitting experimental results into the kinetics models, pseudo-first order, pseudo-second order, and Elovich/intraparticle diffusion. Models were used and evaluated based on the lowest value of the reduced Chi-square value (χ^2) using Equation 8 (Largitte et al., 2016).

With kinetic modeling we can determine which adsorption mechanism is involved, facilitating a better understanding of the equilibrium process and the predominant interactions in the active sites.

The kinetic models, containing information about the graphs in Figures 8, 9, and 10, followed by Table 8 with the main information including temperature, model type, optimized parameters, and finally the calculated error with reduced chi-square.

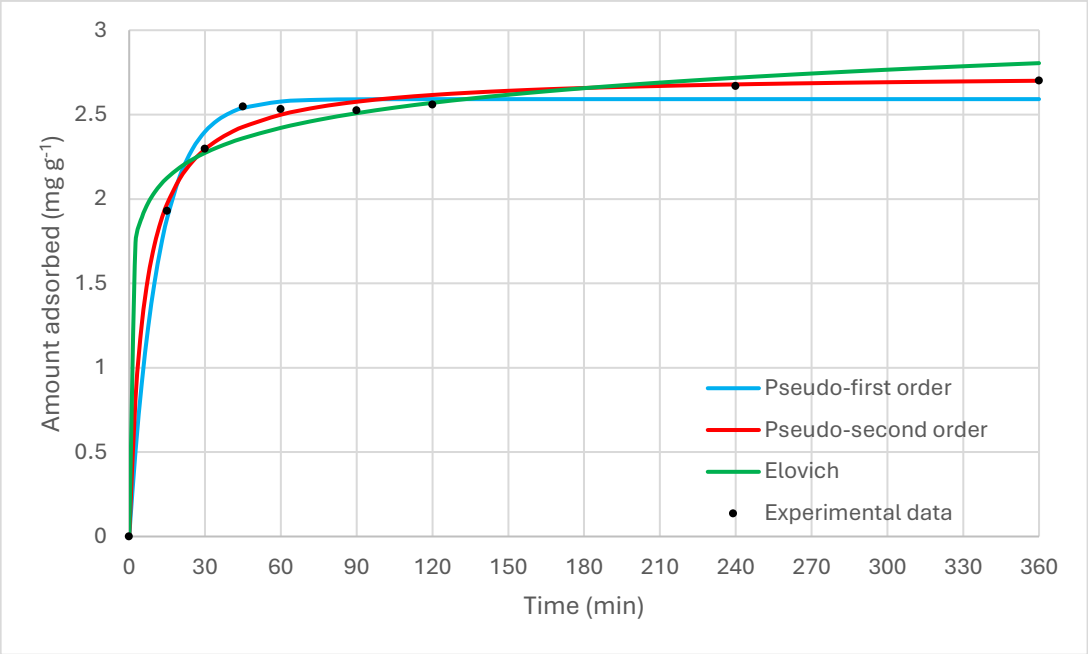

Figure 7 – Comparison between experimental results (points) and predicted adsorption behavior predicted by the kinetic models at 25 ºC.

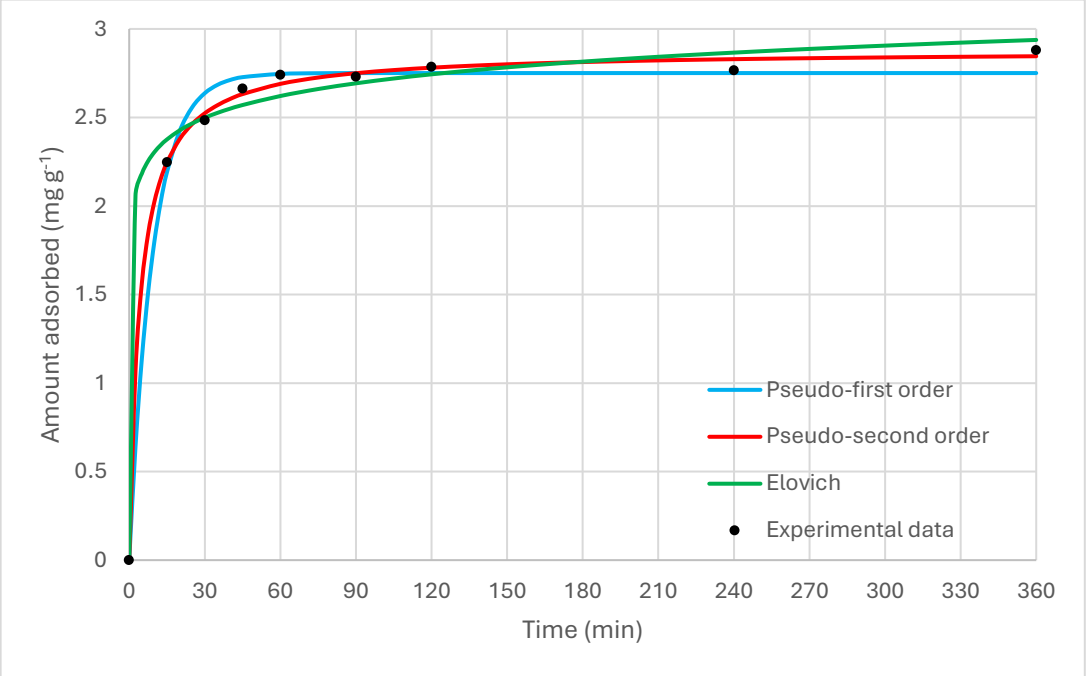

Figure 8 – Comparison between experimental results (points) and predicted adsorption behavior predicted by the kinetic models at 35 ºC.

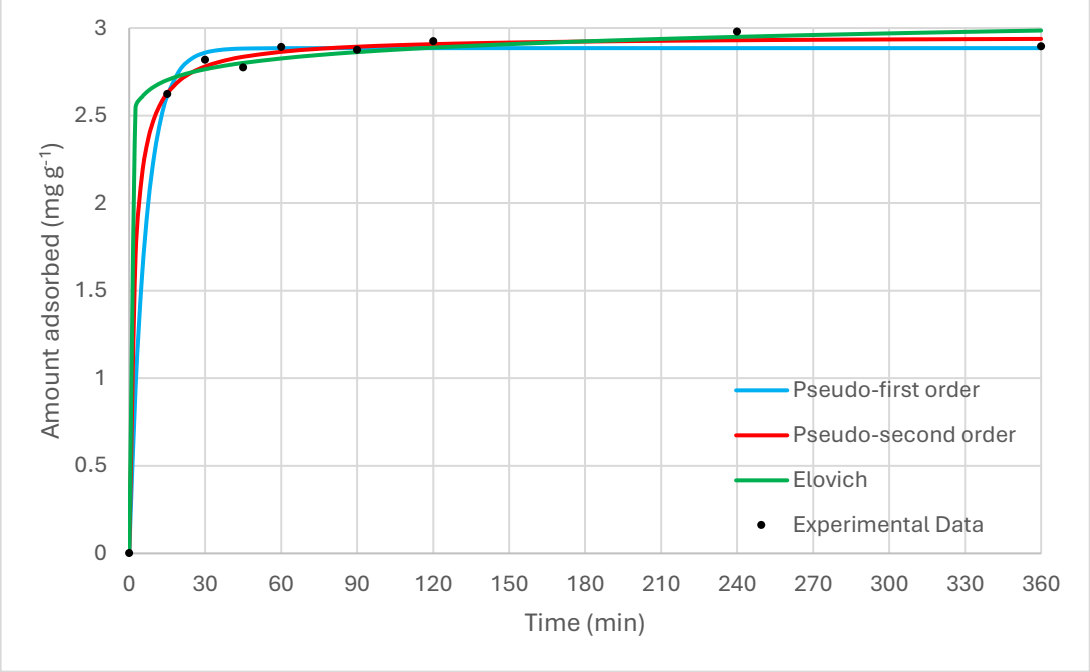

Figure 9 – Comparison between experimental results (points) and predicted adsorption behavior predicted by the kinetic models at 45 ºC.

The adjustments obtained for adsorption kinetics in the pseudo-first-order, pseudo-order, and Elovich models are shown in Table 8.

Table 8 – Estimated kinetic parameters for different models and temperatures.

Tempeture	Modelo	Parameter 1	Parameter 2	Error (X^2)
25 ºC	Pseudo 1^{st} Order	q_e = 2.60	k_1 = 0.0867	0.015197
	Pseudo 2^{st} Order	q_e = 2.75	k_2 = 0.0619	0.009669
	Elovich	α = 292.4307	β = 4.6724	0.043120
35 ºC	Pseudo 1^{st} Order	q_e = 2.75	k_1 = 0.1062	0.018621
	Pseudo 2^{st} Order	q_e = 2.88	k_2 = 0.0822	0.003915
	Elovich	α = 8104.5295	β = 5.6568	0.021550
45 ºC	Pseudo 1^{st} Order	q_e = 2.88	k_1 = 0.1572	0.008250
	Pseudo 2^{st} Order	q_e = 2.96	k_2 = 0.1821	0.003725
	Elovich	α = 9222108575	β = 11.2377	0.008770

q_e (mg g^{-1}); α (mg g^{-1} min^{-1}); k_1 (min^{-1}); k_2 (g mg^{-1} min^{-1}); β (g mg^{-1}).

The pseudo-second kinetic models resulted in the best fit to the experimental data in all fluoxetine adsorption with the best result at 45 °C (χ^2 = 0.003725), suggesting that the limiting step of adsorption possibly consists of chemisorption chemical interactions between the adsorbate and the adsorbent. The pseudo-first-order model and the Elovich model showed weaker fit, suggesting their inadequacy to describe the process at this temperature. Compared to 25 and 35 °C, the adsorption capacity at equilibrium (q_e = 2.96 mg g^{-1}) and the pseudo-second-order velocity constant (k_2 = 0.1821 g mg^{-1} min^{-1}) increased by temperature are the most appropriate, due to the effect of temperature on molecular mobility and adsorption energy. No desorption or decreased efficiency occurs if adsorption occurs at the highest temperature experienced. Therefore, it appears to be a suitable temperature for the fluoxetine adsorption process under the conditions considered.

The process of chemisorption can be represented by the pseudo second order kinetic model. An adsorption process that takes place when a chemical reaction happens at active sites on the surface of the adsorbent in the rate-limiting step is indicated. The rate of adsorption according to this model will be influenced by the availability of sites, and the strength of the bond between the adsorbate and the adsorbent (Ezzati et al., 2024; Ezzati, 2025).

Thus, the rate of adsorption will be predicted using the general or specific adsorption isotherms to obtain the parameters of k_2 (adsorption velocity constant) and q_e (equilibrium adsorption capacity). According to the Arrhenius equation, the steady state velocity constant k_2 depends on the activation energy, for adsorption. When the rate of adsorption is determined by the pseudo second order mechanism, high E_a values mean that chemisorption processes occur with strong chemical bond strengths and need more energy. With temperature rises, higher k_2 values will result, leading to faster adsorption rates for particular pairs of adsorbate-sorbent systems. This finding confirms the conclusion that chemisorption is the major type of adsorption process since, in general, lower activation energy values are needed for physisorption compared to chemisorption.

4.3 ADSORTION EQUILIBRIUM ISOTHERMS

Concentration of fluoxetine was 3 ppm. The adsorption equilibrium experiment was carried out at a temperature of 45 ºC with the mass varying between 2 and 30 mg of activated carbon for a period of 24 hours. Figure 11 shows the adsorption of isotherms for the Freundlich and Langmuir models. The experiments were performed in duplicate and the readings in triplicate.

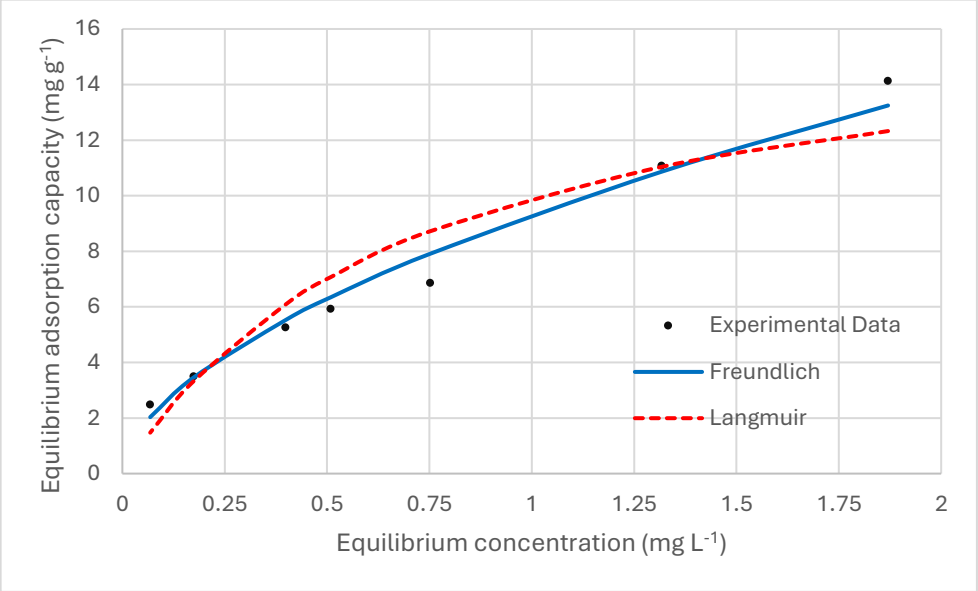

Figure 10 – Adsorption equilibrium isotherms of fluoxetine on the studied adsorbent at 45 ºC.

According to the data presented in Figure 11, there is a positive correlation between equilibrium concentration and equilibrium adsorption capacity, indicating a positive interaction between the adsorbent and adsorbate. Both Freundlich and Langmuir type adsorption models approximated the experimental data, but the Freundlich model was a better fit throughout the entire test range, more at higher concentrations. This supports the idea that adsorption occurs on surfaces with a variety of energy levels and supports multiple layers (Umpleby et al., 2001), the Langmuir model suggests there may be an upper limit to the amount of adsorption that occurs to a single surface, thus allowing for the formation of a monolayer by using the Langmuir method to determine the maximum adsorption amount for the tested adsorption system (Salehian et al., 2026b). Therefore, from the analyses performed, the results would indicate that the Freundlich model would provide the best description of the system

being studied. This indicates that the active sites have a heterogeneous structure (Fernandes et al., 2019b).

Table 9 contains the name of the model, its parameters obtained and its deviation from the experimental data.

Table 9 – Estimated equilibrium adsorption isotherm parameters for fluoxetine.

Model	Parameter 1	Parameter 2	Error
Freundlich	K_F = 9.30	n = 1.77	0.3454
Langmuir	K_L = 1.39	Q_m = 17.07	1.6627

K_F (mg g^{-1} (L $mg^{-1})^{1/n}$); K_L (L mg^{-1}); Q_m (mg g^{-1})

To explain how well adsorbed materials behave when adsorbed onto adsorbent surfaces, both the Freundlich and Langmuir adsorption isotherms were used in the analysis of the samples. In comparing the two models, the Freundlich isotherm was more reliable than the Langmuir isotherm because of its lower error: 0.3454 against 1.6627.

Parameter 2 (n = 1.77) from the Freundlich isotherm, implies that substantial interaction occurs between the adsorbate and the adsorbent, and thus, adsorption was favored. Values of n > 1 indicates a favorable adsorption process (Ebrahimian Pirbazari et al., 2016).

The value for the Freundlich coefficient of K_F = 9.30 mg g^{-1} (L $mg^{-1})^{1/n}$ confirms that the material tested has a very good ability to be adsorbed. Therefore, it can be assumed that the interaction between the adsorbate and adsorbent was achieved through the heterogeneous nature of the adsorbent's surface and varied levels of activity (Arunkumar et al., 2024).

In contrast, while the Langmuir isotherm provides an estimate of the maximum possible value of Q_m =17.07 mg g^{-1} the fitting quality determined by the ability of the model to predict real results, is lower when compared with other methods. Thus, one could suggest that the assumptions of the Langmuir isotherm concerning a monolayer

of adsorbate on a homogeneous surface are not applicable to describe all the interactions that take place.

The behavior represented in the isotherms depends on the activation energy of the adsorption. The Freundlich model is deemed more adequate than the other models for describing adsorption; thus, the process is heterogeneous and involves surface reactions at a mixture of active sites with different energies. Because of this, the adsorption process would need different activation energies, based on where it occurred on the surface.

4.4 THERMODYNAMIC STUDIES

Activation energy is defined as the minimum energy required to go from an initial state to a final state. From a theoretical point of view, in this context, activation energy can be understood as the minimum energy of the adsorbent required to interact with the active site of activated carbon (Hussain et al., 2021a). Graph shown in Figure 12 below.

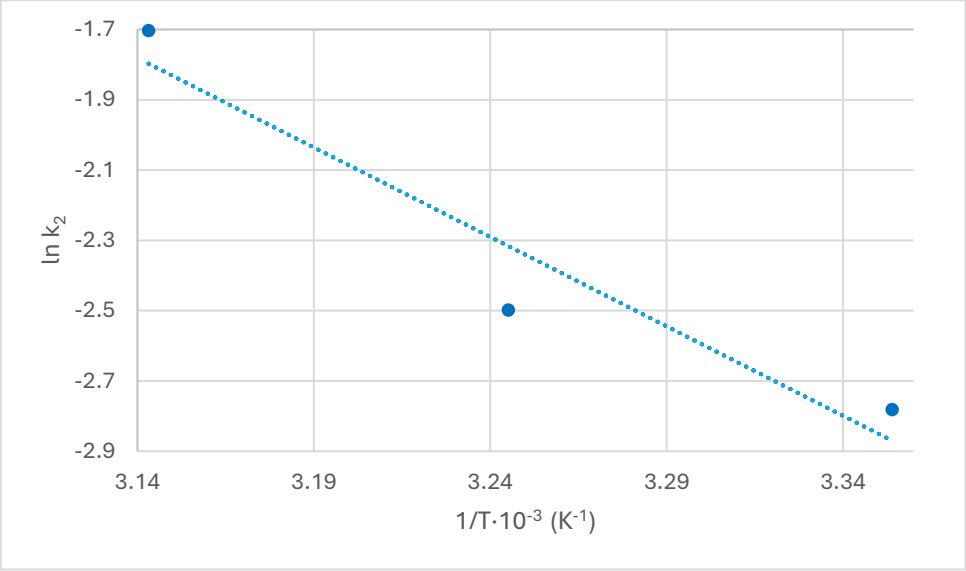

Figure 11 – Arrhenius plot for determination of activation energy in fluoxetine adsorption.

To determine the value of the activation energy E_a, the kinetic constants of the temperatures of 25, 35 and 45 ºC were used, Table 10 shows the results obtained.

Table 10 – Estimated thermodynamic parameters of for fluoxetine adsorption.

Temperature (C°)	2nd Order constant k_2 (g·mg^{-1}·min^{-1})	Activation Energy E_a (kJ·mol^{-1})	R squared (R^2)
25	0.0619	42.32	0.9204
35	0.0822		
45	0.1821		

The activation energy obtained was 42.32 kJ mol^{-1}, in addition, the coefficient of determination was 0.9204.

Two key thermodynamic properties that describe adsorption are activation energy and Gibbs free energy. Activation energy indicates the amount of energy required to overcome the barrier for the adsorption process to occur and helps to provide information on the nature of the mechanism and type of interaction between adsorbent and adsorbate. Conversely, Gibbs free energy ΔG indicates whether the adsorption process will occur spontaneously. Negative Gibbs free energy indicates that the adsorption process will occur spontaneously under the experimental conditions, and the extent of negativity indicates the extent of favorable thermodynamics. Thus, both energy activation and Gibbs free energy provide complementary information regarding the overall adsorption process.

The Gibbs free energy value obtained was −0.851 kJ mol^{-1}. If the process has a value between 0 and -20 kJ mol^{-1} it is considered a physical adsorption, when between -80 and -400 kJ mol^{-1} is considered as chemical adsorption, confirming another indication of physical interaction occurring in the system (Hussain et al., 2021b).

The relationship between Gibbs free energy within the system and the influence of pH on the adsorption of fluoxetine also exists because changes in pH will influence both the amount that the adsorbate will ionize and the amount of positive and negative charges on the surface of the adsorbent. In general, when the pH of the solution is favorable with respect to the active sites and their interaction with the adsorbent, the fluoxetine adsorbing to the active sites of the adsorbate have greater strength in terms of the attraction between the two molecules, will therefore, have greater amounts of binding of the fluoxetine to the adsorbent and will ultimately result in negative values

of ΔG, indicating that processes will occur spontaneously. In addition, pH changes can modify electrostatic forces, Van der Waals forces and hydrogen bonding, thus directly affecting the thermodynamic stability of the adsorption process. Thus, the behavior of Gibbs free energy shows that pH is an extremely important factor in determining the equilibrium and efficiency of the retention of fluoxetine by the adsorbent.

4.5 INFLUENCE OF PH IN FLUOXETINE REMOVAL

The adsorptive behavior of fluoxetine varies consistently with pH, depending on the charge of the adsorbent and fluoxetine itself. Thus, for the activated carbon surface, fluoxetine adsorption would be most favorable when it occurs between the pH_{PZC} of activated carbon 8.92 and the pKa of fluoxetine 10.05 (Formagini et al., 2026; Do et al., 2017), due to ionic interactions.

5 CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

5.1 CONCLUSIONS

The work reported here investigates the valorization of biomass waste (olive pits) to remove the drug fluoxetine (an anti-depressant) from aqueous matrices. Fluoxetine and other emerging and micropollutants can be found in water, making environmental and public health risks, and existing conventional wastewater treatment plants do not effectively remove them from discharged effluent.

Activated carbon produced from olive pits and chemically activated with NaOH was used in this study. Characterization of the material showed that the material has a point of zero charge of 8.92; indicating when the pH is less than 8.92 the adsorbent surface has a predominance of positive surface charge. Kinetic tests demonstrated that the process is rapid, with the removal curve showing significant stabilization between 120 and 240 minutes, the rate of removal was highest at elevated temperatures (maximum of 98.87% at 45 ºC) . The pseudo-second-order kinetic model provided a good fit to the experimental data and indicated is most likely due to physical adsorption.

In conducting the equilibrium study, the Freundlich model (versus the Langmuir model) better described the nature of the adsorption process. Specifically, the results suggest that adsorption occurs on heterogeneous surfaces and occurs in multiple layers. The maximum adsorption capacity calculated, obtained through fitting to the Langmuir model, was 17.07 mg g^{-1}.Thermodynamic variables suggest activation energy for this process is 42.32 kJ mol^{-1} and the Gibbs free energy is = -0.851 kJ mol^{-1}; thus, the process is spontaneous and thermodynamically favorable.

It is concluded that olive pit-derived from the activated carbon is a sustainable and efficient alternative for treating water contaminated with fluoxetine.

5.2 SUGGESTIONS FOR FUTURE WORK

Future work could include evaluating the effect of pH to determine whether the experimental results agree with the theoretical predictions. FTIR analyses before and after adsorption should also be performed to identify chemical changes on the charcoal surface and provide insights into the adsorption mechanism. In addition, adsorption kinetics should be investigated at temperatures above 45 °C to determine the point at which the excess thermal energy begins to favor desorption. Further studies should also assess the regeneration and reuse of the adsorbent, as well as evaluate its performance using real wastewater matrices.

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