



EFFECT OF PH, DOSE, AND CONTACT TIME ON PHARMACEUTICAL REMOVAL BY BYPRODUCT ADSORBENTS

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Abstract:

The increasing concern over pharmaceutical pollution in water sources has highlighted the need for sustainable and cost-effective methods to remove pharmaceutical contaminants. Byproduct-derived adsorbents, particularly those from the edible oil industry, offer a promising solution due to their availability, carbon-rich content, and potential for surface modification. This study investigates the effect of key operational parameters—pH, adsorbent dose, and contact time—on the removal efficiency of pharmaceutical pollutants using byproduct-derived adsorbents. It explores various activation techniques (physical and chemical) to enhance the adsorbent properties, including surface area and pore structure. Additionally, the impact of these parameters on adsorption kinetics, isotherms, and reusability is examined. The findings suggest that byproduct-derived adsorbents, particularly when optimized for surface characteristics, can achieve effective and sustainable removal of pharmaceutical contaminants, offering a viable alternative to conventional adsorbents.

Keywords: Pharmaceutical pollutants, Adsorption, Byproduct-derived adsorbents, Activation techniques, Surface modification, pH effect, Adsorbent dose, Contact time.

Introduction:

Pharmaceutical contamination in water sources has become a critical environmental issue due to the persistence and potential toxicity of these compounds. Traditional wastewater treatment methods often struggle to remove these contaminants effectively, leading to the exploration of alternative solutions. One promising approach is the use of byproduct-derived adsorbents, which are made from agricultural and industrial wastes, such as edible oil industry byproducts. These materials are cost-effective, widely available, and rich in carbon, making them ideal candidates for wastewater treatment applications. However, the inherent adsorption capacities of these raw materials are often limited, necessitating activation, surface modification, and performance optimization to enhance their efficiency. This study examines the effect of operational parameters such as pH, adsorbent dose, and contact time on the adsorption of pharmaceutical pollutants by byproduct-derived adsorbents. The research focuses on the impact of physical and chemical activation methods on the surface area, porosity, and adsorption capacity, providing insights into the potential of these adsorbents for real-world water treatment applications.

Development of Byproduct-Derived Adsorbents

Research into alternative adsorbent materials made from industrial and agricultural leftovers has increased due to the rising concern about pharmaceutical pollution in water resources, as well as the high cost and sustainability constraints of commercial adsorbents. Because of their widespread availability, carbon-rich content, and presence of functional groups that are advantageous for adsorption, edible oil industry wastes have become interesting precursors. However, the surface area and adsorption efficiency of raw byproducts are often poor. Thus, the creation of effective byproduct-derived adsorbents requires proper activation, surface modification, and performance assessment (Ioannidou & Zabaniotou, 2007).



Three main steps are usually involved in turning edible oil industry byproducts into high-performance adsorbents: (i) activation, either chemical or physical, to increase surface area and porosity; (ii) surface modification and functionalisation to increase affinity for target contaminants; and (iii) assessment of adsorption performance towards pharmaceutical compounds under various operating conditions. The efficacy and suitability of the produced adsorbents for the treatment of water and wastewater are determined by these steps taken together.

Physical and Chemical Activation Techniques

By increasing the surface area, pore structure, and surface reactivity of raw byproducts, activation plays a crucial role in transforming them into effective adsorbents. Physical and chemical activation are the two main categories of activation procedures, each with unique benefits and processes.

Physical Activation

Physical activation is the process of heating precursor materials to high temperatures, usually 400–900 °C, while activating gases such carbon dioxide, nitrogen, or steam are present. Typically, there are two phases in the process: activation and carbonisation. A carbon-rich char is left behind after volatile components are eliminated during carbonisation. By gasifying carbon atoms, further activation produces pores, increasing the pore volume and surface area (Bansal & Goyal, 2005).

Physical activation has been used to effectively turn groundnut, sunflower, and palm kernel shells—byproducts of the edible oil industry—into activated carbons. Pharmaceutical compounds of different sizes may be effectively adsorbed by these materials due to their well-developed microporous and mesoporous structures (Zabaniotou & Stavropoulos, 2003). However, physical activation often requires longer processing times and significant energy input, which might raise manufacturing costs.

Chemical Activation

Phosphoric acid (H₃PO₄), potassium hydroxide (KOH), sodium hydroxide (NaOH), zinc chloride (ZnCl₂), sulphuric acid (H₂SO₄), or other chemical activating agents are impregnated into the precursor material. This is followed by thermal treatment at comparatively lower temperatures (400–700 °C). Chemical agents improve pore growth and surface functioning by facilitating dehydration, bond cleavage, and crosslinking processes (Ioannidou & Zabaniotou, 2007).

Chemical activation often results in adsorbents with larger surface areas, larger pore volumes, and superior adsorption capabilities as compared to physical activation. Because of their natural mineral and organic composition, oilseed cakes and wasted bleaching earth have been extensively researched as precursors for chemically activated adsorbents (Mishra et al., 2018). However, in order to reduce the environmental effect, chemical activation requires careful handling and recovery of activating substances and may produce secondary effluent.

Comparison of Activation Techniques

Chemical activation provides better control over pore shape and surface chemistry, while physical activation is safe for the environment and doesn't produce chemical waste. The kind of byproduct, the required adsorbent characteristics, and the intended use all influence the activation process selection. To maximise adsorbent performance, hybrid strategies that combine chemical and physical activation have often been investigated (Mohan et al., 2014).

Surface Modification and Functionalization

In addition to activation, surface modification and functionalisation are essential for improving the byproduct-



derived adsorbents' adsorption affinity and selectivity towards pharmaceutical pollutants. In order to encourage certain interactions with target molecules, these procedures seek to add or modify functional groups to the adsorbent surface.

- **Modification of Chemical Surfaces**

In order to add functional groups that include oxygen or nitrogen, such as hydroxyl, carboxyl, carbonyl, and amine groups, chemical modification entails treating adsorbents with acids, bases, or oxidising agents. According to Rivera-Utrilla et al. (2013), acid treatment with sulphuric or nitric acid improves electrostatic attraction towards basic medicinal molecules and increases surface acidity. On the other hand, alkaline therapy improves the adsorption of acidic medications by introducing basic sites.

- **Utilising both organic and inorganic compounds for functionalisation**

To improve adsorption efficacy, functionalisation with polymers, surfactants, or metal oxides has been investigated. For instance, adsorption capacity may be increased by coating adsorbents with graphene oxide, chitosan, or polyethyleneimine via electrostatic interactions and hydrogen bonding (Tran et al., 2017). Through complexation and redox interactions, metal oxide functionalization—such as that of iron or manganese oxides—improves adsorption (Ali et al., 2012).

- **Surface Chemistry's Impact on Adsorption**

Surface functional groups have a major impact on adsorption processes such as electrostatic attraction, π - π interactions, and hydrogen bonding. Aromatic rings and heteroatoms, which are often found in pharmaceutical substances, have a significant interaction with modified adsorbent surfaces. In order to maximise adsorption performance in situations of variable pH and ionic strength, surface chemistry must be tailored (Yang et al., 2016).

Adsorption Performance Toward Pharmaceutical Compounds

The ability of byproduct-derived adsorbents to absorb pharmaceutical pollutants is what ultimately determines their efficacy. Batch and continuous adsorption studies are often used for performance assessment, with an emphasis on characteristics such as adsorption capacity, kinetics, isotherms, selectivity, and reusability.

- **Adsorption Kinetics and Capacity**

High adsorption capabilities of byproduct-derived adsorbents for pharmaceuticals such as hormones, analgesics, and antibiotics have been reported in a number of investigations. Chemisorption is the predominant process in adsorption kinetics, which often follow pseudo-second-order models (Ho & McKay, 1999). For real-world wastewater treatment applications, rapid adsorption rates are preferred.

- **Behaviour of Isotherms**

The Freundlich and Langmuir isotherm models are often used to explain adsorption equilibrium. Chemically activated materials often display Langmuir-type monolayer adsorption, while many byproduct-derived adsorbents have heterogeneous surface properties that result in Freundlich-type behaviour (Foo & Hameed, 2010).

- **Operational Parameters' Impact**

The presence of competing compounds, pH, temperature, and starting medicinal concentration all affect adsorption efficacy. Because of surface charge fluctuations and pharmacological speciation, byproduct-derived adsorbents have shown substantial pH-dependent adsorption (Zhang et al., 2018). Modified adsorbents often retain sufficient removal efficiency in the face of competition from natural organic matter.

- **Reusability and Regeneration**

Adsorbent regeneration and reuse are essential for financial sustainability. With little loss of adsorption capacity over many cycles, many byproduct-derived adsorbents may be regenerated by solvent washing or light heat treatment (Mestre et al., 2009). When compared to traditional adsorbents, this increases their practical usefulness.



- **Evaluation in Relation to Commercial Adsorbents**

According to a number of studies, byproduct-derived adsorbents that have been optimised may attain adsorption efficiencies that are on par with or even higher than those of commercial activated carbon, all while drastically lowering manufacturing costs and their negative effects on the environment (Mohan et al., 2014).

Factors Affecting Adsorption Performance

Several physicochemical factors control the adsorption performance of adsorbents made from edible oil industry byproducts for the elimination of pharmaceutical impurities. These factors affect the solubility, interaction behaviour, and speciation of medicinal chemicals in aqueous environments in addition to the properties of the adsorbent surface. Designing scalable treatment systems, increasing removal efficiency, and optimising adsorption conditions all depend on an understanding of these variables. Solution pH, adsorbent dose, contact duration, starting medicinal concentration, temperature, and ionic strength are the main variables influencing adsorption performance.

Effect of pH

The answer Since pH directly impacts the adsorbent's surface charge, the ionisation state of pharmaceutical compounds, and the electrostatic interactions between them, it is one of the most important factors affecting the adsorption of pharmaceutical pollutants. According to Kumar et al. (2019), a large number of pharmaceutical chemicals are weak acids or bases that exist in various ionic states based on the pH of the solution.

Oxyseed cakes, wasted bleaching earth, and biochars made from de-oiled biomass are examples of edible oil industry byproduct-derived adsorbents that include functional groups such as hydroxyl (-OH), carboxyl (-COOH), and amino (-NH₂) groups. The surface charge and adsorption capacity are changed by the pH-dependent protonation and deprotonation of these functional groups (Tran et al., 2020).

Protonation causes the adsorbent surface to become positively charged at low pH levels, which increases the adsorption of anionic pharmaceutical species by electrostatic attraction. On the other hand, in acidic environments, electrostatic repulsion may inhibit the adsorption of cationic medications (Ali et al., 2018). Higher pH levels promote the adsorption of cationic medications like antibiotics and antidepressants because the deprotonation of surface functional groups creates a negatively charged adsorbent surface (Ahmad et al., 2021).

Furthermore, the carbonaceous structure of bio-based adsorbents and medicinal compounds' π - π interactions, hydrophobic interactions, and hydrogen bonding are all influenced by pH. Optimising solution pH is essential for maximising adsorption efficiency, and pH adjustment may be required when treating real wastewater containing a mixture of pharmaceuticals with varying pK_a values. Research has shown that maximum adsorption frequently occurs near the pH corresponding to the adsorbent's point of zero charge (pHPZC), where electrostatic repulsion is minimised (Foo and Hameed, 2019).

Effect of Adsorbent Dosage

The amount of medication removed from aqueous solutions is mostly determined by the dose of the adsorbent. It has a direct impact on the surface area available for pollutant absorption as well as the quantity of adsorption sites that are available. Because there are more active binding sites available when the adsorbent dose is raised, removal efficiency is often greater (Gupta and Nayak, 2018).

Dosage optimisation is crucial for edible oil industry byproduct-derived adsorbents in order to strike a compromise between economic viability and adsorption efficiency. Lower clearance percentages may occur at low doses because there may not be enough adsorption sites to adsorb every medicinal molecule in the solution. Removal efficiency



rises quickly with increasing dose until a plateau is achieved, after which any dosage increases have less impact (Zhang et al., 2020).

The effective surface area and adsorption capacity per unit mass of adsorbent, however, may be decreased by high adsorbent dose due to particle aggregation and adsorption site overlap. When measured in milligrammes of medicine adsorbed per gramme of adsorbent (q_e), this phenomena may lead to a reduction in adsorption efficiency (Mohammed et al., 2019).

For large-scale applications, adsorbent dose optimisation is especially crucial since greater dosages result in increased material costs, handling needs, and sludge production. Therefore, for sustainable and economical wastewater treatment, figuring out the lowest effective dose that yields optimum removal efficiency is crucial.

Effect of Contact Time

A crucial component of adsorption kinetics, contact time establishes how long medicinal molecules interact with the adsorbent surface. Due to the large number of available adsorption sites, adsorption usually happens quickly in the early stages before slowing down when these sites are occupied (Ho and McKay, 2018). Rapid initial adsorption in byproduct-derived adsorbents is often ascribed to pore diffusion and external surface adsorption. Adsorption moves into the adsorbent's interior pores as contact time rises, and equilibrium is reached when there is no more net adsorption (Soni et al., 2021).

Particle size, pore shape, adsorbent surface area, and the kind of medicinal chemical all affect how long it takes to achieve equilibrium. Larger pharmaceutical molecules may encounter steric barrier, leading to prolonged equilibrium durations, while smaller compounds may diffuse more readily into micropores (Rangabhashiyam and Selvaraju, 2017).

Designing batch and continuous adsorption systems requires an understanding of contact time. For practical applications, short equilibrium durations are preferred because they enable better treatment throughput and reduced operating costs. The adsorption process and rate-limiting phases may be understood by kinetic modelling utilising pseudo-first-order, pseudo-second-order, and intraparticle diffusion models (El-Sayed et al., 2020).

Effect of Initial Pharmaceutical Concentration

The adsorption driving force and the degree of mass transfer between the adsorbent surface and the aqueous phase are influenced by the initial concentration of pharmaceutical pollutants. The diffusion of medicinal compounds towards the adsorbent is improved by higher starting concentrations because they create a larger concentration gradient (Crini et al., 2019).

High removal efficiency is achieved at low starting concentrations because there are many adsorption sites in comparison to the amount of medicinal compounds. Adsorption capacity (q_e) rises with initial concentration because more medicinal molecules are loaded onto the adsorbent. However, owing to adsorption site saturation, removal effectiveness as a percentage may drop at extremely high concentrations (Bhatnagar et al., 2020).

High adsorption capabilities at high pharmaceutical concentrations show the potential of edible oil industry byproduct-derived adsorbents as efficient substitutes for commercial activated carbon. The connection between starting concentration and adsorption capacity is often described using adsorption isotherm studies, such as Langmuir and Freundlich models (Foo and Hameed, 2019). Predicting adsorbent effectiveness in actual wastewater circumstances, where pharmaceutical concentrations may vary significantly depending on the source, requires an understanding of the impact of initial concentration.



Influence of Temperature and Ionic Strength

The mobility of pharmaceutical molecules, the viscosity of the solution, and the kind of adsorbent–adsorbate interactions are all impacted by temperature, which has an impact on adsorption. Particularly for endothermic adsorption processes, raising the temperature may improve adsorption by raising diffusion rates and pore accessibility (Kumar and Jena, 2016). On the other hand, because of weaker adsorbent–adsorbate interactions, exothermic adsorption processes may show decreased adsorption capability at higher temperatures. Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) are thermodynamic parameters that provide important information on the nature and viability of the adsorption process (Tran et al., 2020).

Adsorption performance is also significantly impacted by ionic strength, which is determined by the amount of salts and competing ions in wastewater. Because pharmaceutical compounds and inorganic ions compete for active adsorption sites, high ionic strength may decrease adsorption efficiency (Zhou et al., 2018). Furthermore, ionic strength has the ability to filter electrostatic interactions, reducing the attraction between the adsorbent surface and charged medications. For oil industry byproduct-derived adsorbents to be used in actual wastewater treatment, they must exhibit stable adsorption ability under a range of temperature and ionic strength conditions. Assessing these effects is essential for determining the practical usability and robustness of the adsorbent.

Regeneration and Reusability of Adsorbents

When assessing the practical application, economic feasibility, and environmental sustainability of adsorbents used for wastewater treatment, regeneration and reusability are crucial factors. The ability to regenerate and reuse the adsorbent without suffering a major loss of adsorption capacity is crucial to the process's long-term viability, even if adsorption is universally acknowledged as a successful approach for the removal of pharmaceutical pollutants. Due to their cheap cost, abundance, and renewable nature, adsorbents made from leftovers of the edible oil industry provide clear benefits. Nevertheless, their regeneration behaviour has to be carefully studied to guarantee constant performance throughout many adsorption–desorption cycles. Desorption studies, regeneration methods, adsorption effectiveness across multiple cycles, and the viability of regenerated bio-based adsorbents from an economic and environmental standpoint are all covered in this section.

Desorption Studies

Desorption studies are essential for determining the intensity of interactions between pharmaceutical contaminants and the adsorbent surface as well as the reversibility of the adsorption process. These investigations provide light on whether chemical interactions like covalent bonds and strong electrostatic attraction or physical interactions like hydrogen bonds and van der Waals forces dominate the adsorption process (Tran et al., 2020). Pharmaceutical-loaded adsorbents are treated with appropriate desorbing agents in desorption studies to liberate the pollutants that have been adsorbed. Distilled water, acidic or alkaline solutions, organic solvents like ethanol or methanol, and salt solutions are examples of common desorbing agents (Crini et al., 2019). The kind of pharmaceutical ingredient, the surface chemistry of the adsorbent, and the predominant adsorption mechanism all influence the choice of desorbing agent.

Surface functional groups including hydroxyl, carboxyl, and phenolic groups that are inherited from lignocellulosic biomass often affect the desorption effectiveness of adsorbents made from edible oil industry byproducts. Alkaline solutions promote the desorption of anionic pharmaceuticals, whereas acidic desorption media are often successful in eliminating cationic pharmaceuticals by protonating surface functional groups and reducing electrostatic interactions (Ahmad et al., 2021). When hydrophobic or π – π interactions are important in adsorption, organic solvents are very useful (Bhatnagar et al., 2020). While low desorption signals irreversible binding or structural changes in the adsorbent, high desorption efficiency shows that the adsorbent can be successfully regenerated. Since



insufficient desorption might eventually result in the buildup of pharmaceutical residues inside the adsorbent matrix, desorption studies can aid in determining the danger of secondary contamination.

Conclusion

Byproduct-derived adsorbents, particularly those from the edible oil industry, demonstrate considerable potential for the removal of pharmaceutical contaminants from wastewater. Activation techniques, both physical and chemical, significantly enhance the surface area, pore structure, and functional group density, improving the adsorption capacity of these materials. The operational parameters, including pH, adsorbent dose, and contact time, play a crucial role in determining the adsorption efficiency, with optimal conditions varying depending on the specific pharmaceutical pollutant. Furthermore, surface modifications, such as functionalizing with oxygenated groups, contribute to better affinity and selectivity toward pharmaceutical pollutants. Adsorbent regeneration and reusability studies indicate that byproduct-derived adsorbents can be reused effectively without significant loss of adsorption capacity, making them a sustainable alternative to conventional adsorbents like activated carbon. This research underscores the potential of byproduct-derived adsorbents in advancing sustainable water treatment technologies, offering a cost-effective, eco-friendly solution for tackling pharmaceutical pollution in wastewater.

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