

**Review for the final defense of the doctoral dissertation:**

**Titled “*Removal of Pharmaceutical Residue from Water and Real Wastewater Using Activated Carbon Derived from Agro-Industrial Wastes*”**

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Submitted to obtain a Ph.D. degree in Biochemical, Environmental and Chemical Engineering

**Environmental Engineering field**

University of Pannonia

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This doctoral dissertation presents an innovative and sustainable approach to the removal of pharmaceutical contaminants from various water matrices using activated carbon (AC) derived from agro-industrial waste. The study aligns with the field of environmental engineering, focusing on wastewater treatment, pollution control, and resource recovery. It addresses a pressing global issue: the persistence of pharmaceutical residues (such as diclofenac, ciprofloxacin, amoxicillin, and carbamazepine) in water bodies and their potential impact on ecosystems and human health. The dissertation explores the synthesis, characterization, and application of activated carbon produced from three types of agricultural waste: olive stone (OSAC), banana peel (BPAC), and pinewood (FPWAC). These bio-adsorbents were chemically activated and tested across a range of pH values and real-world conditions, including wastewater and lake water. Analytical techniques such as FTIR, SEM-EDS, XRD, Boehm titration, TGA, and surface area measurements were used to examine the physicochemical properties of the ACs.

This dissertation makes a significant contribution to environmental engineering by developing cost-effective and sustainable activated carbons from agro-industrial wastes (olive stones, banana peels, and pinewood) for the removal of pharmaceutical pollutants from water, wastewater, and lake water. The dissertation demonstrated that these bio-adsorbents achieve high removal efficiencies, maintain reusability over multiple cycles, and operate effectively across varying pH levels and environmental conditions. The synthesized activated carbons were up to 36 times more cost-efficient than commercial alternatives, with production costs ranging from \$3.77 to \$4.53 per kilogram. Through comprehensive analyses involving adsorption kinetics, isotherms, thermodynamics, and desorption behavior, the dissertation enhances our understanding of pollutant removal mechanisms. This work not only addresses critical water contamination issues but also promotes the valorization of agricultural waste, aligning with circular economy principles and offering scalable solutions for sustainable water treatment in both developed and resource-limited settings.

Osamah provides a well-structured and comprehensive review that spans a wide array of relevant topics, including the occurrence of pharmaceuticals in the environment, properties of selected

pharmaceuticals, current removal methods, and the potential of agro-industrial wastes as adsorbent precursors. The literature review is systematic, progressing logically from the identification of the problem to the exploration of possible solutions, effectively situating the study within the broader academic and practical context. The incorporation of up-to-date and high-impact publications, including references to European regulations and recent directives, showcases an engagement with current and authoritative sources. Moreover, the review not only summarizes existing knowledge but also critically highlights the gaps and challenges in current practices, providing a clear justification for the research. The use of figures, such as classification diagrams and process schematics, further enhances clarity and aids in synthesizing complex information. Moreover, the candidate critically evaluates existing methods by comparing their efficiencies, limitations, and practical applications, rather than merely summarizing prior work. This critical approach is evident in the clear justification for selecting agro-industrial wastes as precursors and the insightful discussion of their advantages and challenges. The literature synthesis not only contextualizes the research problem effectively but also identifies specific knowledge gaps that the study addresses. Overall, the candidate has excelled in critically analyzing the literature, making the dissertation a significant contribution to the field.

The experimental methods employed in this dissertation are meticulously designed and demonstrate a high degree of scientific rigor. The selection of three different agro-industrial wastes as precursors for activated carbon production aligns seamlessly with the thesis objective of developing sustainable, cost-effective adsorbents for pharmaceutical removal from water matrices. The use of varied chemical activation methods (KOH,  $H_3PO_4$ ,  $NH_4NO_3/NaOH$ ) is particularly commendable, as it allows comparative analysis of activation efficiency and material properties, enriching the scope of the study. Furthermore, the detailed characterization techniques (SEM-EDS, FTIR, XRD, Boehm titration, pH<sub>pzc</sub>, TGA, SBET) reflect a comprehensive approach to understanding the physicochemical properties of the synthesized adsorbents, which is critical to elucidating the adsorption mechanisms. The inclusion of adsorption kinetics, isotherms (Langmuir, Freundlich), thermodynamic analyses, and desorption studies ensures that the adsorption behavior is evaluated from multiple scientific perspectives, thereby providing robust, reproducible, and reliable data. Overall, the experimental methods are not only technically sound but also highly suitable for addressing the research objectives, offering an innovative and environmentally sustainable solution to pharmaceutical pollutant removal from complex water matrices.

Osamah demonstrates an exceptional standard in processing and analyzing research results, reflected in the methodical integration of mathematical and statistical models throughout the experimental sections. Osamah skillfully employs key adsorption isotherm models (Langmuir and Freundlich) as well as pseudo-first-order and pseudo-second-order kinetic models, and intraparticle diffusion models, enabling a comprehensive and comparative understanding of the adsorption mechanisms. The inclusion of thermodynamic evaluations further strengthens the scientific rigor of the study. Moreover, the dissertation's statistical treatment of experimental data, as evidenced by the use of standard deviations, model fitting parameters, and comparison tables, ensures reproducibility and reliability of the results. Figures presenting experimental data alongside isotherm and kinetic model fits illustrate a high level of clarity and precision. The meticulous approach in comparing the prepared activated carbons with other reported systems in the literature reflects a critical and analytical mindset. Overall, Osamah's quantitative and

statistical handling of the data not only meets but exceeds academic expectations, ensuring that the conclusions drawn are well-supported, robust, and scientifically credible.

Throughout the Results and Discussion, Osamah compares the performance of the developed activated carbons (OSAC, BPAC, and FPWAC) with those reported in previous studies. This comparative approach is particularly evident in sections dedicated to the evaluation of adsorption efficiency, isotherm modeling, and kinetic parameters, where benchmark data from the literature are juxtaposed with the study's results. These comparisons not only validate the novel materials' effectiveness but also position the work within the broader research context, reflecting a critical and comprehensive understanding of the field.

This dissertation presents several new scientific results that make a significant contribution to the field of environmental engineering, particularly in the area of pharmaceutical removal from water. Among the novel contributions, the two-stage chemical activation process applied to pinewood using  $\text{NH}_4\text{NO}_3$  and  $\text{NaOH}$  is particularly innovative, yielding high surface area materials with excellent adsorption performance. Furthermore, the dissertation provides comprehensive comparative studies of these activated carbons in various water matrices (Milli-Q, lake water, and real wastewater), supported by rigorous characterization and adsorption-desorption cycle analysis. It also establishes cost-effectiveness by comparing production costs with commercial activated carbon, showing the viability of these materials for large-scale application. The long-term reusability of the developed adsorbents and their ability to effectively remove multiple pharmaceuticals simultaneously further demonstrate the novelty and practical relevance of the work.

The structural organization of the dissertation is highly commendable, reflecting a meticulous approach to academic writing and research presentation. The work is systematically divided into well-defined chapters, beginning with a comprehensive introduction that establishes the research context and significance, followed by an extensive literature review and a detailed methodology section. Each subsequent chapter, including the results, discussion, and conclusion, flows logically and cohesively, reinforcing the central thesis of the research.

The charts and graphs presented throughout the dissertation are exceptionally clear, well-structured, and enhance the overall readability of the scientific content. Each figure is accompanied by appropriately labeled axes, legends, and units, allowing readers to easily interpret complex data trends and experimental outcomes. For example, Figures depict variations in pharmaceutical removal efficiencies across different pH levels, concentrations, and contact times, demonstrating a strong alignment between visual representation and textual explanation. Schematic illustrations clearly outline the preparation processes of the activated carbons, aiding in the reader's conceptual understanding of the experimental setup. Moreover, the use of comparative plots (such as those for Langmuir and Freundlich isotherms or kinetic models) not only demonstrates methodological rigor but also facilitates the evaluation of model fit and adsorbent performance. Overall, the graphical content in this dissertation significantly supports the scientific narrative and contributes to the document's academic quality.

The dissertation presents a commendable integration of literature references, cited from recent and high-impact scientific publications. Osamah consistently supports theoretical and methodological

discussions with credible sources. In terms of style, the dissertation maintains a formal academic tone, with clear structure and coherent progression across chapters, making complex technical content accessible to a broad scientific audience. The use of precise language and detailed procedural explanations further reflects a disciplined and methodical approach to scholarly writing. Additionally, the external appearance of the dissertation is highly professional; the formatting adheres to standard academic conventions, with a well-organized table of contents, clearly labeled figures and tables, and consistent typography, all of which contribute to the readability and visual clarity of the work. Overall, the dissertation reflects both scholarly depth and high editorial quality.

At the end, Osamah has also published his research findings in prestigious, high-impact international journals (D1 category), demonstrating the academic rigor and significance of his contributions. Additionally, he has an impressive research output, with around 53 publications and an H-index of 13, reflecting the substantial impact and recognition of his work within the scientific community.

#### Questions:

- 1- Given the scope and outcomes of your study, what specific treatment system would you advocate for implementation in industrial-scale applications within the field of Environmental Engineering, particularly those targeting pharmaceutical removal from wastewater streams?
- 2- Can you elaborate on the scientific implications of pharmaceutical pollutants adhering to the Langmuir isotherm model? Specifically, what does this suggest about the nature of the adsorption process?

## **Final Conclusion**

The doctoral dissertation titled "*Removal of Pharmaceutical Residue from Water and Real Wastewater Using Activated Carbon Derived from Agro-Industrial Wastes*", authored by Osamah Jaber Oudah Al-Sareji and submitted to the Doctoral School of Chemical Engineering and Material Science at the University of Pannonia, clearly fulfills all academic and scientific requirements for the award of a PhD in the field of Environmental Engineering. The work demonstrates a high level of scientific rigor, originality, and relevance, particularly in its innovative approach to addressing pressing environmental challenges through sustainable wastewater treatment technologies. The methodological soundness, thorough data analysis, and practical implications for water resource management reflect the scholarly standards expected at the doctoral level. Therefore, the dissertation is **strongly recommended for acceptance**.

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