UNIVERSIDAD AUTÓNOMA DE SAN LUIS POTOSÍ

FACULTAD DE CIENCIAS QUÍMICAS

Posgrado en Ciencias en Ingeniería Química

Desarrollo de materiales avanzados mediante estrategias de transformación y funcionalización para la fotodegradación visible de colorantes

Tesis que para obtener el grado de:

Doctorado en Ciencias en Ingeniería Química

Presenta:

Jonathan Michel Sánchez Silva

Director de Tesis: **Dr. RAÚL OCAMPO PÉREZ**

Codirector de Tesis: **Dra. MA. ANGÉLICA AGUILAR AGUILAR**

SAN LUIS POTOSÍ, S. L. P. **FECHA: JULIO 2026**

Este proyecto se realizó en el Laboratorio de Procesos Avanzados de Oxidación, adscrito a la Facultad de Ciencias Químicas de la Universidad Autónoma de San Luis Potosí, durante el periodo comprendido entre agosto de 2022 y julio de 2026, bajo la dirección del Dr. Raúl Ocampo Pérez.

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Por este medio me permito informarle el porcentaje de similitud obtenido mediante Ithenticate para la tesis titulada Desarrollo de materiales avanzados mediante estrategias de transformación y funcionalización para la fotodegradación visible de colorantes presentada por el autor M.C. Jonathan Michel Sánchez Silva. La tesis es requisito para obtener el grado de Doctorado en el Posgrado en Ciencias en Ingeniería Química. El análisis reveló un porcentaje de similitud de 1% excluyendo referencias y metodología.

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"*Un profesor trabaja para la eternidad: nadie puede decir dónde acaba su influencia*"

Resumen

Esta tesis doctoral aborda el desarrollo de materiales avanzados mediante estrategias de transformación y funcionalización para la fotodegradación de colorantes bajo irradiación visible. La investigación se inició con la caracterización termoquímica de biomasa lignocelulósica proveniente de residuos agroindustriales, con el fin de evaluar su potencial como precursor de materiales carbonosos funcionales. Posteriormente, mediante carbonización hidrotermal, se sintetizaron hidrocarbones con propiedades fisicoquímicas favorables para aplicaciones fotocatalíticas. Los resultados demostraron que estos materiales presentan grupos funcionales oxigenados, dominios aromáticos conjugados y radicales libres persistentes, características que favorecen la generación de especies reactivas de oxígeno ($•OH$, $O_2•^-$ y $^1O_2$), lo que evidencia que los hidrocarbones pueden actuar como fotocatalizadores intrínsecamente activos y no únicamente como soportes carbonosos. Con el fin de mejorar su desempeño fotocatalítico, los hidrocarbones fueron funcionalizados mediante la incorporación de TiO_2 y CeO_2, obteniéndose materiales compuestos con efectos sinérgicos que favorecieron la separación de cargas, ampliaron la respuesta espectral hacia la región visible y aumentaron la eficiencia de degradación de colorantes. Finalmente, se desarrolló una estrategia de funcionalización basada en la inmovilización covalente de carbon quantum dots (CQDs) sobre sepiolita, lo que permitió obtener un fotocatalizador híbrido estable que supera las limitaciones asociadas a la recuperación y reutilización de estos nanomateriales. En conjunto, los resultados permitieron establecer relaciones entre la composición del precursor, las estrategias de transformación y de funcionalización, la estructura de los materiales y su desempeño fotocatalítico. Esta investigación aporta nuevo conocimiento sobre el papel activo de los hidrocarbones en la fotocatálisis heterogénea y propone una estrategia novedosa para la inmovilización de carbon quantum dots, lo que contribuye al diseño estratégico de materiales carbonosos sostenibles para procesos avanzados de oxidación.

Palabras clave: Biomasa lignocelulósica, hidrocarbones, carbonización hidrotermal, puntos cuánticos de carbono, fotocatálisis heterogénea, materiales avanzados, fotodegradación de colorantes.

Abstract

This doctoral dissertation addresses the development of advanced materials through transformation and functionalization strategies for the visible-light-driven photodegradation of dyes. The research began with the thermochemical characterization of lignocellulosic biomass derived from agro-industrial residues to evaluate its potential as a precursor for functional carbon-based materials. Subsequently, hydrothermal carbonization was employed to synthesize hydrochars with physicochemical properties suitable for photocatalytic applications. The results demonstrated that these materials possess oxygen-containing functional groups, conjugated aromatic domains, and persistent free radicals, features that promote the generation of reactive oxygen species (•OH, $O_2^{\bullet-}$, and $^1O_2$), thereby demonstrating that hydrochars can function as intrinsically active photocatalysts rather than merely as carbon supports. To further enhance their photocatalytic performance, the hydrochars were functionalized by incorporating TiO_2 and CeO_2, yielding composite materials with synergistic effects that promoted charge separation, extended the photoresponse into the visible-light region, and improved dye degradation efficiency. Finally, a functionalization strategy based on the covalent immobilization of carbon quantum dots (CQDs) onto sepiolite was developed, resulting in a stable hybrid photocatalyst that overcomes the limitations associated with the recovery and reuse of these nanomaterials. Collectively, the findings established structure–property–performance relationships by correlating precursor composition, transformation, and functionalization strategies, material structure, and photocatalytic performance. This research provides new insights into the active role of hydrochars in heterogeneous photocatalysis and proposes a novel strategy for the immobilization of carbon quantum dots, thereby contributing to the strategic design of sustainable carbon-based materials for advanced oxidation processes.

Keywords: Lignocellulosic biomass, hydrochar, hydrothermal carbonization, carbon quantum dots, heterogeneous photocatalysis, advanced materials, dye photodegradation

.

Abbreviations

AA	Ascorbic acid
AOPs	Advanced oxidation processes
APTES	3-Aminopropyltriethoxysilane
ASTM	American Society for Testing and Materials
BCS	*Byrsonima crassifolia* stones
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
BQ	Benzoquinone
CO_2	Carbon dioxide
CQDs	Carbon quantum dots
CV	Crystal violet
DLBP	4-(dimethylamino)benzophenone
DLPM	4,4'-bis(dimethyl-amino)benzophenone
DTG	Derivative Thermogravimetry
E_A	Apparent Activation Energy
EDS	Energy dispersive spectroscopy
EDTA-NA	Ethylenediaminetetraacetic acid sodium salt
EPR	Electron paramagnetic resonance
ESI	Electrospray ionization
FC	Fixed carbon
FTIR	Fourier transform infrared spectroscopy
FWO	Flynn–Wall–Ozawa
H_2O	Water
H_2O_2	Hydrogen peroxide
HC	Hydrochar
HHV	High heating value
HTC	Hydrothermal carbonization
ICTAC	International Confederation for Thermal Analysis and Calorimetry
IPA	Isopropyl alcohol
IUPAC	International Union of Pure and Applied Chemistry
JCPDS	Joint Committee on Powder Diffraction Standards
KAS	Kissinger–Akahira–Sunose
LED	Light-Emitting Diode

LHV	Lower heating value
LS	Light source
MB	Methylene Blue
MC	Moisture content
N_2	Nitrogen
NMR	Nuclear Magnetic Resonance
OFGs	Oxygenated functional groups
$O_2^{\bullet -}$	Superoxide radical
P25	Commercial TiO_2 P25 (Evonik Degussa)
PDS	Peroxydisulfate
PFRs	Persistent free radicals
PL	Photoluminescence
PMS	Peroxymonosulfate
PPB	*Prunus persica* biomass
PSD	Pore size distribution
RhB	Rhodamine-B
ROS	Reactive oxygen species
S_{BET}	BET surface area
SCV	Scavenger
SEM	Scanning Electron Microscopy
SEP	Sepiolite
SK	Starink
SSE	Sum of Squared Errors
TEM	Transmission Electron Microscopy
TEA	Techno-economic analysis
TGA	Thermogravimetric analysis
UPLC	Ultra-Performance Liquid Chromatography
UV	Ultraviolet
UV-vis DRS	Ultraviolet-Visible Diffuse Reflectance Spectroscopy
VM	Volatile matter
XPS	X-Ray photoelectron spectroscopy
XRD	X-Ray Diffraction
$^1O_2$	Singlet oxygen
•OH	Hydroxyl radical

Prologue

The increasing demand for sustainable technologies to address environmental challenges has spurred the development of advanced materials derived from renewable resources and their application in pollution control processes. Among the various strategies currently under investigation, the transformation and functionalization of biomass-derived materials have emerged as promising approaches for developing environmentally friendly technologies that operate under visible-light irradiation.

The research presented in this thesis originated from an interest in the valorization of agro-industrial residues as renewable feedstocks for the production of value-added materials. The initial stage of the work focused on understanding the physicochemical characteristics and thermochemical conversion behavior of lignocellulosic biomass, providing fundamental knowledge regarding its suitability as a precursor for carbonaceous materials. This stage established the scientific basis for the subsequent transformation of biomass via hydrothermal carbonization. Building upon this foundation, the research evolved toward synthesizing and evaluating hydrochars as functional materials for environmental remediation. The results demonstrated that biomass-derived hydrochars are not simply carbonaceous by-products but can act as intrinsically active photocatalytic materials under visible-light irradiation. These findings highlighted the importance of structural feat features, such as oxygen-containing functional groups and persistent free radicals, in promoting the generation of reactive oxygen species and the degradation of pollutants. To further enhance photocatalytic performance, subsequent studies explored the functionalization of hydrochars by incorporating semiconductor materials.

The development of hydrochar-based TiO_2 and CeO_2 composites demonstrated synergistic interactions between carbonaceous matrices and semiconductor phases, leading to improved light absorption, charge separation, and photocatalytic efficiency. These investigations contributed to a deeper understanding of the structure-property-performance relationships governing composite photocatalytic materials. As the research progressed, attention shifted toward more advanced functionalization strategies involving carbon-based nanomaterials. In particular, carbon quantum dots emerged as attractive candidates due to their unique optical and electronic properties. Their covalent immobilization onto mineral supports enabled the development of stable hybrid photocatalysts that efficiently degrade organic pollutants under visible light, further expanding the scope of material design strategies explored throughout this doctoral work. Beyond environmental remediation, the knowledge generated during this research enabled the exploration of new application areas. The incorporation of carbon-based nanomaterials into biopolymeric matrices has led to the development of bionanocomposite systems for postharvest disease management, demonstrating the versatility of transformation and functionalization approaches in addressing challenges in both the environmental and agro-food sectors. Furthermore, the research activities conducted during this doctoral program contributed to the training of undergraduate students and to the establishment of new research directions in carbon dots, hydrochars, hybrid photocatalysts, and sustainable postharvest technologies. Overall, this thesis demonstrates how the systematic transformation and functionalization of materials can generate innovative solutions for visible-light-driven environmental and agro-food applications. The work integrates concepts from biomass valorization, materials science, nanotechnology, and photocatalysis, contributing to the development of sustainable technologies based on advanced materials.

This thesis is organized into seven chapters. Chapter 1 presents the thermochemical characterization and valorization potential of *Byrsonima crassifolia* stone as an agro-industrial residue. Chapter 2 investigates biomass-derived hydrochars as metal-free photocatalysts for the visible-light-driven degradation of pollutants. Chapter 3 explores the functionalization of hydrochars with semiconductor materials to obtain hybrid photocatalysts with enhanced performance. Chapter 4 provides a comprehensive review of hydrochar synthesis, properties, and applications in water decontamination. Chapter 5 focuses on the development of hybrid photocatalysts by immobilizing carbon quantum dots onto sepiolite clay. Finally, Chapter 6 discusses the expansion of the research toward agro-food applications, human resource training, and future research directions, drawing on the work presented throughout this thesis.

General Index

1. Introduction

The growing need for sustainable technological solutions to address environmental challenges has driven the development of materials with specifically physicochemical properties (Rehman et al., 2024). In particular, the transformation and functionalization of carbon-based materials derived from renewable resources have proven effective in generating high-value products while promoting the sustainable management of agro-industrial residues (Bachs-Herrera et al., 2023). These strategies align with the principles of the circular economy, in which waste is redefined as a valuable feedstock for synthesizing advanced materials, thereby mitigating its contribution to environmental pollution. Lignocellulosic biomass is among the most abundant and versatile renewable resources due to its high carbon content, widespread availability, and inherent renewability (Vázquez et al., 2020). Every year, substantial quantities of agricultural and agro-industrial residues are generated worldwide, and inadequate management or disposal practices often result in environmental pollution. Consequently, considerable research efforts have been devoted to developing technologies that convert these residues into value-added products. Beyond their conventional use in energy production, lignocellulosic materials have emerged as promising precursors for synthesizing carbon-based materials for applications in catalysis, adsorption, energy storage, sensing, and environmental remediation (Mendoza-Martinez et al., 2021).

The development of biomass-derived materials requires a comprehensive understanding of precursor characteristics. The chemical composition, thermal stability, and decomposition behavior of lignocellulosic biomass significantly influence conversion pathways and ultimately determine the physicochemical properties of the resulting carbon-based materials (Cavali et al., 2023). Accordingly, thermochemical characterization and kinetic analysis are essential for selecting suitable feedstocks and designing efficient conversion processes. These analytical approaches provide fundamental insights into biomass reactivity during thermochemical conversion and establish the scientific basis for the subsequent development of advanced carbon-based materials.

Hydrothermal carbonization (HTC) has emerged as a sustainable technology for converting biomass into hydrochars under relatively mild operating conditions (Mendoza-Martinez et al., 2021). Unlike conventional thermal treatments, HTC enables the direct conversion of wet biomass into carbonaceous solids without extensive drying, making it particularly advantageous for processing agro-industrial residues (Ighalo et al., 2022). Hydrochars produced through HTC exhibit a complex surface chemistry characterized by oxygen-containing functional groups, partially conjugated aromatic domains, structural defects, and persistent free radicals (Qin et al., 2018; Chen et al., 2017). These characteristics can be tailored by adjusting synthesis parameters, thereby endowing hydrochars with distinctive physicochemical properties that extend their utility beyond conventional solid-fuel and adsorption applications. Recent studies have demonstrated that hydrochars can serve as active materials for environmental remediation (Ighalo et al., 2022; Yu et al., 2024). Beyond their inherent adsorption capacity, biomass-derived hydrochars can generate reactive oxygen species under visible-light irradiation, highlighting their potential for photocatalytic and advanced oxidation processes (Zhang et al., 2019; Gao et al., 2018). These findings challenge the conventional perception of hydrochars as passive carbon supports and underscore new opportunities for developing sustainable photocatalytic materials derived from renewable biomass.

The performance of carbon-based materials can be significantly enhanced through functionalization strategies that modify their surface chemistry, electronic structure, and interfacial properties (Jin et al., 2018). Such functionalization enables the incorporation of active components that improve light absorption, facilitate charge separation, and promote the generation of reactive oxygen species (Afza et al., 2022; Zhang et al., 2024). A common approach involves developing hybrid materials that integrate carbonaceous matrices with semiconductor phases. Titanium dioxide (TiO_2) and cerium dioxide (CeO_2) are widely employed owing to their photocatalytic activity, chemical stability, and favorable redox properties (Bayan et al., 2021; Channei et al., 2021). When combined with carbon-based supports, these semiconductors often exhibit synergistic effects, resulting in enhanced photocatalytic efficiency through improved charge transfer and increased photoresponse to visible light (Chen et al., 2017; Hu et al., 2016).

Carbon quantum dots (CQDs) have recently emerged as a new class of carbon nanomaterials distinguished by their exceptional optical and electronic properties (Long et al., 2021). Their tunable photoluminescence, remarkable electron-transfer capability, and responsiveness to visible light have attracted considerable interest in photocatalytic applications (Yu et al., 2024; Sadjadi et al., 2021). However, the nanoscale dimensions and colloidal nature of CQDs pose challenges for their recovery and reuse following treatment. Consequently, immobilization strategies based on covalent attachment to solid supports have gained increasing attention as an effective approach for preserving photocatalytic activity, enhancing operational stability, and facilitating recyclability. These advances highlight the role of sequential transformation and functionalization strategies in enabling the strategic design of advanced carbon-based materials for environmental applications.

Water pollution caused by synthetic organic dyes remains a major environmental challenge. Large volumes of dye-containing effluents are routinely discharged into aquatic systems from the textile, pharmaceutical, printing, leather, and related industries (Sewnet et al., 2022; Wang and Cheng, 2023). Many dye molecules exhibit high chemical stability, resistance to biodegradation, and potential toxicity to aquatic organisms and humans. Conventional physicochemical and biological treatment methods are often insufficient to achieve the complete removal of these contaminants, particularly in complex industrial wastewater matrices (Foroutan et al., 2022; Mondol et al., 2021). Consequently, considerable research efforts have been devoted to developing more efficient and environmentally sustainable treatment technologies.

Advanced oxidation processes (AOPs) have emerged as highly effective alternatives for the degradation of persistent organic contaminants through the generation of reactive oxygen species, including hydroxyl radicals (•OH), superoxide radicals ($O_2^{\bullet-}$), and singlet oxygen ($^1O_2$) (Hübner et al., 2024; Ganiyu et al., 2022). Among these processes, heterogeneous photocatalysis has attracted considerable attention due to its ability to harness light energy to activate photocatalytic materials, thereby enabling oxidation reactions under relatively mild conditions (Yu et al., 2024). However, conventional photocatalysts, particularly TiO_2 and CeO_2, are limited by their wide band gap and the rapid recombination of photogenerated electron–hole pairs, which together result in low efficiency under visible-light irradiation (Baca et al., 2020). These limitations have driven the development of alternative photocatalytic materials and hybrid systems to improve visible-light utilization and enhance overall photocatalytic performance (Alalm et al., 2021; Mishra and Sundaram, 2023). Despite significant advances in the development of biomass-derived carbon materials and hybrid photocatalysts, substantial scientific challenges remain.

The interrelationships among biomass precursor characteristics, transformation processes, material structure, surface chemistry, and photocatalytic performance remain incompletely understood. Furthermore, the specific contributions of oxygen-containing functional groups, persistent free radicals, semiconductor interfaces, and carbon nanostructures to the generation of reactive oxygen species require further investigation. Therefore, establishing clear structure-property-performance relationships is essential for the strategic design of efficient, stable, and sustainable photocatalytic materials. In this context, the present doctoral research investigates advanced materials developed through sequential transformation and functionalization strategies for the visible-light photodegradation of organic dyes. The study encompasses the thermochemical characterization of agro-industrial biomass; its valorization as a precursor for carbon-based materials; the synthesis of hydrochars exhibiting intrinsic photocatalytic activity; their subsequent functionalization by incorporating semiconductors; and the fabrication of hybrid photocatalyst incorporating carbon quantum dots and sepiolite clay. Particular emphasis is placed on elucidating the effects of transformation and functionalization on material properties and photocatalytic performance. Collectively, the studies presented in this dissertation advance the development of sustainable carbon-based materials for environmental remediation and provide a scientific foundation for the strategic design of advanced visible-light-activated photocatalysts.

2. Background

The development of advanced photocatalytic materials increasingly relies on integrating transformation and functionalization strategies to control their physicochemical, structural, and electronic properties (Rehman et al., 2024). In this context, carbon-based materials derived from renewable resources have attracted considerable attention, offering sustainable alternatives to conventional photocatalysts while contributing to the valorization of agro-industrial residues (Mendoza-Martinez et al., 2021). Among the various conversion pathways, thermochemical processes enable the transformation of lignocellulosic biomass into functional carbon-based materials whose composition and properties can be tailored according to precursor characteristics and synthesis conditions (Cavali et al., 2023). Therefore, understanding biomass composition, its thermal behavior, and the underlying conversion mechanisms is essential for designing materials with specific functionalities. Building upon these conversion pathways, hydrothermal carbonization has emerged as one of the most attractive transformation technologies for producing hydrochars from lignocellulosic biomass (Ighalo et al., 2022).

The resulting materials possess a complex carbonaceous structure composed of oxygen-containing functional groups, partially conjugated aromatic domains, structural defects, and persistent free radicals (PFRs), all of which influence their adsorption capacity,, and chemical reactivity (Chen et al., 2017; Zhu et al., 2020; Luo et al., 2023). Although hydrochars were initially investigated primarily as adsorbents and solid fuels, growing evidence suggests that they can also function as intrinsically active photocatalysts (Chen et al., 2017; Hu et al., 2017; Ruan et al., 2019). Their activity under visible-light irradiation has been associated with the generation of reactive oxygen species (ROS), including hydroxyl radicals (•OH), superoxide radicals ($O_2^{\bullet-}$), and singlet oxygen ($^1O_2$), which participate in the degradation of organic pollutants through advanced oxidation processes (Hübner et al., 2024; Su et al., 2024; Yu et al., 2024).

Heterogeneous photocatalysis involves the excitation of photoactive materials under irradiation, generating electron–hole pairs that initiate oxidation–reduction reactions through the formation of reactive oxygen species (Sacco et al., 2021; Ali et al., 2023). Conventional semiconductor photocatalysts, such as titanium dioxide (TiO_2) and cerium dioxide (CeO_2), exhibit excellent chemical stability, low toxicity, and high photocatalytic activity (Bayan et al., 2021; Channeri et al., 2021). However, their practical application is limited by their relatively wide band gap and the rapid recombination of photogenerated charge carriers, which restricts their efficiency under visible-light irradiation (Alalm et al., 2021; Eddy et al., 2023; Baca et al., 2020). To address these limitations, extensive research has focused on functionalization strategies that integrate carbon-based materials with semiconductor phases (Bayan et al., 2021; Dalmaschio et al., 2020; Benz et al., 2022).

Hydrochar-based composites incorporating semiconductors such as TiO_2 and CeO_2 have demonstrated synergistic effects, including improved charge separation, enhanced visible-light absorption, and more efficient interfacial electron transfer, resulting in superior photocatalytic performance compared with their individual components (Changotra et al., 2023; Zhang et al., 2024). More recently, the development of carbon nanomaterials has further expanded opportunities for the design of advanced photocatalysts (Wang et al., 2023; Cavali et al., 2023). Carbon quantum dots (CQDs) exhibit remarkable optical and electronic properties, including tunable photoluminescence, efficient charge transfer capability, and responsiveness to visible light, making them attractive candidates for photocatalytic applications (Yu et al., 2024; Sadjadi et al., 2021). However, their nanoscale dimensions and colloidal behavior complicate their separation and reuse after treatment. Consequently, immobilization strategies based on the covalent attachment of CQDs to mineral or carbon-based supports have emerged as effective approaches to enhance operational stability while preserving photocatalytic functionality. These approaches illustrate the on going evolution of material functionalization strategies toward increasingly sophisticated hybrid systems designed to maximize photocatalytic efficiency.

Despite these advances, several important scientific challenges remain unresolved. A key challenge is to elucidate how precursor characteristics and transformation processes determine the physicochemical properties of the resulting carbon-based materials. Likewise, the specific contributions of oxygen-containing functional groups, persistent free radicals, and conjugated carbon domains to visible-light activity remain poorly understood. In hybrid photocatalysts, the interfacial mechanisms governing charge transfer between carbon-based materials and semiconductor phases remain an active area of research. At the same time, the influence of carbon nanomaterial immobilization on photocatalytic performance and operational stability continues to be investigated. Addressing these knowledge gaps requires establishing structure–property–performance relationships that link biomass transformation, material functionalization, and photocatalytic activity. Such understanding is essential for the strategic design of advanced materials that promote the efficient visible-light-driven degradation of organic pollutants.

3. Justification

The development of sustainable materials to address contemporary environmental challenges has become a central focus of research. Among these challenges, water contamination by persistent organic pollutants, especially synthetic dyes, presents significant environmental and public health risks due to their chemical stability, resistance to conventional treatment methods, and potential toxicity. While heterogeneous photocatalysis has proven effective in degrading these pollutants by generating reactive oxygen species, the practical implementation of conventional photocatalytic materials is hindered by limited visible-light absorption, rapid recombination of photogenerated charge carriers, and, in some cases, high costs or limited sustainability of the materials used. Simultaneously, the increasing production of agro-industrial residues has prompted the development of strategies to valorize them within the circular economy framework.

Lignocellulosic biomass, an abundant renewable resource, can be converted into functional carbonaceous materials via thermochemical and hydrothermal processes. These transformation strategies not only reduce waste disposal but also enable the production of materials with tailored structural, chemical, and electronic properties for advanced environmental applications. Furthermore, subsequent functionalization with semiconductors or nanomaterials offers additional opportunities to enhance photocatalytic performance by improving visible-light harvesting, charge separation, and the generation of reactive oxygen species. Despite significant advances in the development of biomass-derived carbon materials and composite photocatalysts, several critical scientific questions remain unresolved. Specifically, the influence of precursor characteristics on the properties of the resulting materials, the roles of oxygen-containing functional groups and persistent free radicals in photocatalytic activity, and the interfacial interactions that govern charge-transfer processes in composite systems remain incompletely elucidated.

Additionally, although various functionalization strategies have been proposed, a comparative understanding of how these sequential modifications affect material performance remains limited. Within this context, the present doctoral research addresses the need to develop and systematically evaluate advanced materials produced through sequential transformation and functionalization strategies, and to elucidate the relationships among precursor characteristics, synthesis processes, material structure, and photocatalytic performance. By integrating biomass thermochemical characterization, hydrochar production, semiconductor functionalization, and carbon quantum dot immobilization within a unified research framework, this study offers a comprehensive understanding of the evolution of carbon-based materials toward more efficient photocatalytic systems. Consequently, this research advances both the fundamental understanding of transformation and functionalization processes in carbon-based materials and the development of sustainable technologies for visible-light-driven environmental remediation. Simultaneously, it promotes the valorization of agro-industrial residues as renewable resources for producing advanced functional materials, thereby reinforcing the principles of sustainability, resource efficiency, and circular economy that underpin the development of next-generation photocatalytic systems.

4. Hypothesis

The sequential transformation of renewable precursors into carbonaceous materials, followed by their functionalization with semiconductor phases and carbon nanostructures, enables the development of advanced materials with tailored structural, and physicochemical properties. These modifications enhance visible-light absorption, improve charge separation and transfer, promote the generation of reactive oxygen species, and consequently increase the efficiency of photocatalytic degradation of organic dyes. Furthermore, establishing structure–property–performance relationships provides the basis for the strategic design of sustainable photocatalytic materials for environmental remediation.

5. Objectives

5.1 General objective

To develop advanced photocatalytic materials through transformation and functionalization strategies, and to establish relationships among precursor characteristics, synthesis processes, material structure, and photocatalytic performance for the visible-light-driven degradation of organic dyes.

5.2 Specific objectives

1. To characterize lignocellulosic agro-industrial biomass and evaluate its thermochemical behavior to determine its suitability as a precursor for the production of functional carbonaceous materials.
2. To synthesize hydrochars through hydrothermal carbonization and investigate the influence of transformation conditions on their structural, physicochemical, and photocatalytic properties under visible-light irradiation.
3. To develop hydrochar-based composite photocatalysts through functionalization with semiconductor materials and evaluate the influence of interfacial interactions on light absorption, charge separation, reactive oxygen species generation, and photocatalytic performance.
4. To design advanced hybrid materials based on the immobilization of carbon quantum dots onto sepiolite clay and assess their potential for visible-light-driven photocatalytic degradation of organic dyes.
5. To establish structure-property-performance relationships that explain how transformation and functionalization strategies govern the photocatalytic behavior of carbon-based materials and contribute to the strategic design of sustainable materials for environmental remediation.

Chapter 1. Thermochemical Characterization and Valorization Potential of Lignocellulosic Biomass

Chapter overview

This chapter introduces the initial stage of the doctoral research, focusing on the thermochemical characterization and valorization potential of *Byrsonima crassifolia* stone, an agro-industrial residue produced during nanche fruit processing. The study aims to establish a scientific foundation for converting lignocellulosic biomass into value-added products by examining its physicochemical properties and thermal decomposition behavior. To address this aim, the biomass underwent proximate, elemental, and compositional analysis, followed by non-isothermal thermogravimetric experiments. Special emphasis was placed on determining the kinetic triplet, which includes apparent activation energy, pre-exponential factor, and reaction model, through various isoconversional methods. Additionally, thermodynamic parameters were assessed to evaluate the energetic feasibility of the pyrolysis process and to guide the design and optimization of thermochemical conversion systems.

The findings indicate that *Byrsonima crassifolia* stone exhibits favorable properties for thermochemical valorization, including high volatile matter content, low ash content, and a relatively low activation energy compared to other lignocellulosic biomasses. These results support its potential as a precursor for producing carbonaceous materials and energy-related products. In addition to advancing biomass pyrolysis research, this chapter establishes a foundation for the subsequent investigations detailed in this thesis. Comprehensive knowledge of biomass composition, thermal behavior, and transformation mechanisms underpins the development of hydrochars and advanced functional materials through transformation and functionalization strategies for visible-light-driven environmental remediation applications.

Article 1. Pyrolysis Kinetics of *Byrsonima crassifolia* Stone as Agro-Industrial Waste Through Isoconversional Models

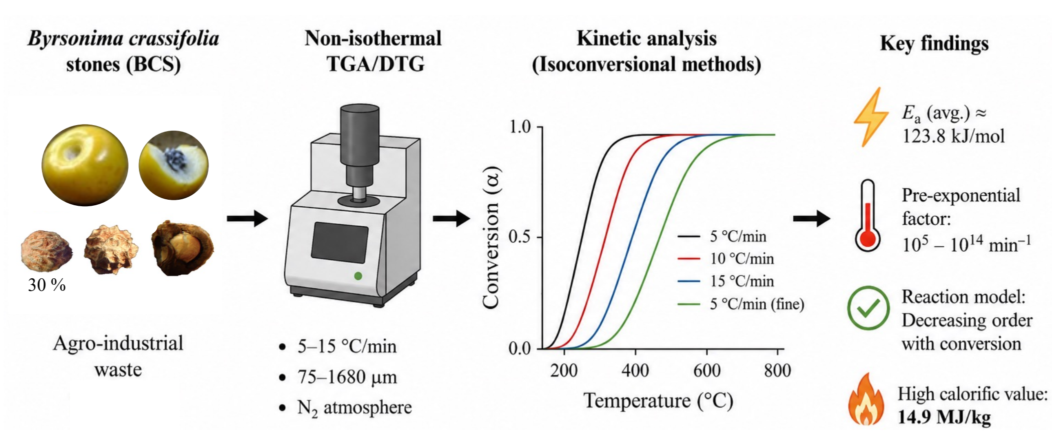
This work was published in the journal *Molecules* under the following reference:

Sanchez-Silva, J. M., Ocampo-Pérez, R., Padilla-Ortega, E., Sangaré, D., Escobedo-Bretado, M. A., Domínguez-Arvizu, J. L., Hernández-Majalca, B. C., Salinas-Gutiérrez, J. M., López-Ortiz, A., & Collins-Martínez, V. (2023). Pyrolysis kinetics of Byrsonima crassifolia stone as agro-industrial waste through isoconversional models. Molecules, 28(2), 544. https://doi.org/10.3390/molecules28020544.

Article overview

This article serves as the starting point for the research presented in this thesis and focuses on the thermochemical characterization of *Byrsonima crassifolia* stone, an agro-industrial residue. Through physicochemical characterization and pyrolysis kinetic analysis, the thermal behavior, reactivity, and energetic potential of this lignocellulosic biomass were investigated using isoconversional models. This study establishes the scientific basis for the valorization of agro-industrial waste through thermochemical transformation processes by identifying the key parameters governing biomass decomposition and conversion. Furthermore, it provides fundamental knowledge of the relationships among biomass composition, thermal degradation pathways, and energy conversion potential, which is essential for the subsequent development of carbonaceous materials. The findings presented in this work support the selection of lignocellulosic residues as suitable precursors for the synthesis of functional carbon-based materials and constitute the foundation for the hydrothermal transformation and material functionalization strategies explored in the following chapters of this thesis.

Graphical Abstract



Chapter 2. Biomass-Derived Hydrochars as Metal-Free Photocatalysts for Dye Removal

Chapter overview

This chapter examines the development of biomass-derived hydrochars as photocatalysts for environmental remediation under visible-light irradiation. Building on the thermochemical valorization concepts introduced previously, this research stage focuses on the hydrothermal transformation of agro-industrial residues into functional carbonaceous materials with intrinsic photocatalytic activity. Hydrochars derived from *Prunus persica* stone were synthesized under varying hydrothermal carbonization conditions to assess the effects of reaction temperature and residence time on their structural and photocatalytic properties. The materials were characterized to elucidate relationships among synthesis conditions, surface chemistry, and photocatalytic performance. Special emphasis was placed on identifying oxygen-containing functional groups and persistent free radicals, which are recognized as key determinants of the visible-light activity of hydrochar materials. The photocatalytic performance of the synthesized hydrochars was assessed by the photodegradation of methylene blue under visible-light irradiation. The results indicate that hydrochars function as efficient, metal-free photocatalysts, achieving high degradation efficiencies without the need for conventional semiconductor materials. Mechanistic studies identified the involvement of reactive oxygen species, such as hydroxyl radicals, superoxide radicals, and singlet oxygen, underscoring the critical role of the hydrochar surface in facilitating photocatalytic reactions.

This chapter presents experimental evidence that hydrochars are not simply carbonaceous by-products of biomass conversion but can serve as active photocatalytic materials. The findings advance understanding of the relationships among hydrochar structure, persistent free radicals, and visible-light-driven photocatalytic activity. Additionally, these results lay the foundation for subsequent chapters, in which hydrochars are further modified through functionalizing them with semiconductor materials to improve charge separation, light absorption, and photocatalytic efficiency. Overall, this work constitutes a significant step toward the development of advanced materials for sustainable environmental remediation.

Article 2. Hydrochar of *Prunus persica*: green promoter of radical species to degrade methylene blue with visible irradiation

This work was published in the journal *Environmental Science and Pollution Research* under the following reference:

Sánchez-Silva, J. M., Sangaré, D., Belmonte-Vázquez, J. L., Aguilar-Aguilar, A., Padilla-Ortega, E., González-Chávez, R., & Ocampo-Pérez, R. (2025). Hydrochar of Prunus persica: Green promoter of radical species to degrade methylene blue with visible irradiation. Environmental Science and Pollution Research, 32, 8481–8497. https://doi.org/10.1007/s11356-025-36214-9

Article overview

This article reports the first demonstration of biomass-derived hydrochars as intrinsically active photocatalytic materials for visible-light-driven environmental remediation. Hydrochars synthesized from Prunus persica stone were evaluated for their ability to degrade organic pollutants without conventional semiconductor materials. The study examined the relationship between hydrothermal synthesis conditions, hydrochar properties, and photocatalytic performance. The results showed significant visible-light activity, attributed to oxygen-containing functional groups and persistent free radicals that promote the generation of reactive oxygen species responsible for pollutant degradation. These findings establish hydrochar as a metal-free photocatalyst capable of driving advanced oxidation processes under visible-light irradiation and demonstrate that biomass-derived carbonaceous materials can actively participate in photocatalytic reactions through their intrinsic structural and electronic properties. Furthermore, this work provides the foundation for the functionalization strategies presented in subsequent chapters, where hydrochars are combined with semiconductor materials to enhance photocatalytic performance.

Graphical Abstract

Chapter 3. Hydrochar-Based Semiconductor Composites for Enhanced Visible-Light Photocatalysis

Chapter overview

This chapter examines the functionalization of biomass-derived hydrochars by incorporating semiconductor materials to enhance photocatalytic performance under visible-light irradiation. Building on previous findings that established hydrochars as metal-free photocatalysts, this research investigates synergistic interactions between carbonaceous matrices and semiconductor phases to enhance light harvesting, charge separation, and overall photocatalytic efficiency. The focus is on the synthesis and evaluation of hydrochar-based composites containing TiO_2 and CeO_2, prepared using hydrothermal methods. Emphasis is placed on understanding how integrating semiconductor particles into the hydrochar structure alters the physicochemical properties of the resulting materials and their photocatalytic activity. Structural, morphological, and optical characterizations were conducted to assess semiconductor dispersion, surface properties, and light absorption behavior.

Photocatalytic experiments were performed using various irradiation sources and model dye pollutants to evaluate the composites under environmentally relevant conditions. The results indicate that incorporating semiconductor phases significantly enhances photocatalytic activity compared with hydrochar alone, demonstrating synergistic effects between the carbonaceous matrix and the semiconductor components. These interactions enable more efficient utilization of visible light and facilitate the generation of reactive species that degrade pollutants. This chapter marks a critical stage in the development of advanced photocatalytic materials through functionalization strategies. The findings establish that hydrochars can serve not only as active photocatalysts but also as effective platforms for semiconductor integration, yielding composite materials with superior photocatalytic performance. Additionally, the knowledge generated forms the foundation for the development of more sophisticated functionalized systems discussed in subsequent chapters.

Article 3. Hydrothermal synthesis of a photocatalyst based on *Byrsonima crassifolia* and TiO_2 for the degradation of crystal violet by UV and visible radiation

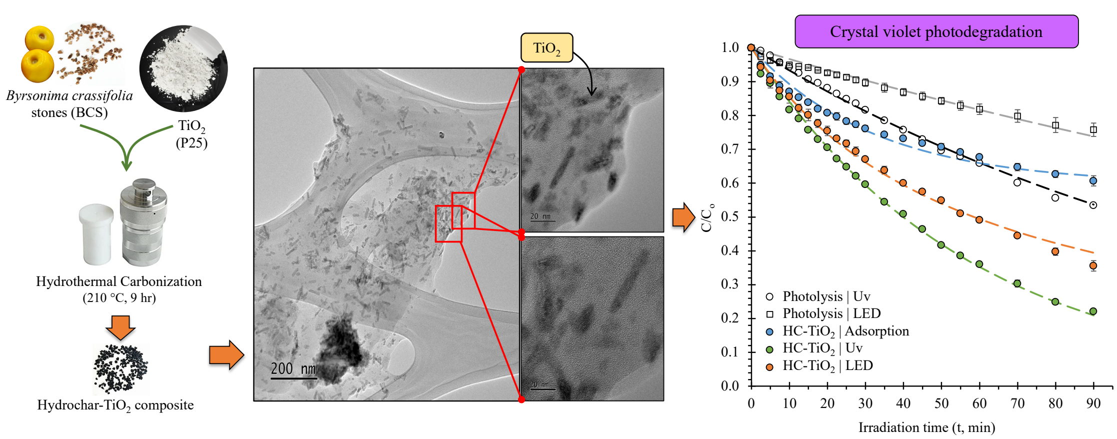
This work was published in the journal *Environmental Research* under the following reference:

Sánchez-Silva, J. M., Aguilar-Aguilar, A., Labrada-Delgado, G. J., Villabona-Leal, E. G., Ojeda-Galván, H. J., Sánchez-García, J. L., Collins-Martínez, H., López-Ramón, M. V., & Ocampo-Pérez, R. (2023). Hydrothermal synthesis of a photocatalyst based on Byrsonima crassifolia and TiO_2 for degradation of crystal violet by UV and visible radiation. Environmental Research, 231(3). https://doi.org/10.1016/j.envres.2023.116280

Article overview

This article presents the first functionalization strategy explored in this thesis, which involves incorporating TiO_2 into a biomass-derived hydrochar matrix via a one-step hydrothermal synthesis process. The study evaluates the influence of the carbonaceous support on the photocatalytic behavior of TiO_2 under both UV and visible-light irradiation. The results demonstrate that the hydrochar matrix promotes effective dispersion of the semiconductor and enhances visible-light absorption, thereby improving the photocatalytic degradation of crystal violet. This work establishes the role of hydrochar as an active structural component that participates in light absorption and charge-transfer processes, providing the first evidence of synergistic interactions between biomass-derived carbonaceous materials and semiconductor photocatalysts. Furthermore, this study introduces a functionalization approach that serves as the foundation for developing more efficient hydrochar-based hybrid photocatalysts, which are explored in the subsequent stages of this thesis.

Graphical Abstract



Article 4. Synergistic photocatalysis of a hydrochar/CeO_2 composite for dye degradation under visible light

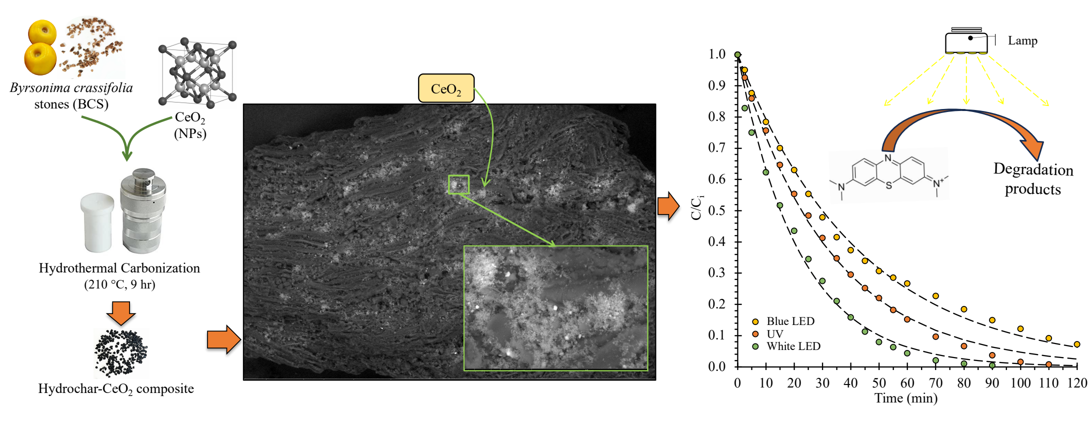
This work was published in the journal *Environmental Science and Pollution Research* under the following reference:

Sánchez-Silva, J. M., Ojeda-Galván, H. J., Villabona-Leal, E. G., Labrada-Delgado, G. J., Aguilar-Maruri, S. A., Fuentes-Ramírez, R., González-Ortega, O., López-Ramón, M. V., & Ocampo-Pérez, R. (2024). Synergistic photocatalysis of a hydrochar/CeO_2 composite for dye degradation under visible light. Environmental Science and Pollution Research, 31, 16453–16472. https://doi.org/10.1007/s11356-024-32281-6

Article overview

This article expands the functionalization strategy introduced in the previous study by investigating the incorporation of CeO_2 into a hydrochar matrix to develop a visible-light-responsive hybrid photocatalyst. The work focuses on understanding the synergistic effects between the semiconductor phase and the carbonaceous structure and their impact on photocatalytic performance. The synthesized hydrochar/CeO_2 composite exhibited high photocatalytic efficiency under various irradiation sources, demonstrating that the interaction between hydrochar and CeO_2 enhances the utilization of visible light and the efficient degradation of organic pollutants. The results confirmed that hydrochar can serve as an effective support for semiconductor materials while simultaneously enhancing photocatalytic activity through its carbonaceous structure. This study consolidates the concept of hydrochar-based semiconductor composites as an effective functionalization strategy for developing advanced photocatalytic materials. It provides further evidence supporting the structure-property-performance relationships proposed throughout this thesis.

Graphical Abstract



Chapter 4. Hydrochars for Water Decontamination: From Biomass Transformation to Environmental Applications

Chapter overview

This chapter provides a comprehensive analysis of the current state of knowledge regarding hydrochars derived from lignocellulosic biomass and their application in water decontamination processes. Building upon the experimental studies presented in the previous chapters, this review integrates the fundamental concepts governing hydrochar synthesis, physicochemical properties, and environmental performance, with particular emphasis on their role as sustainable materials for water treatment. The chapter examines the influence of biomass composition and hydrothermal carbonization conditions on the structural and surface characteristics of hydrochars, highlighting the formation of oxygen-containing functional groups, persistent free radicals, and other features that determine their reactivity. Special attention is given to the mechanisms by which hydrochars participate in water decontamination, including adsorption, heterogeneous photocatalysis, and advanced oxidation processes.

In addition, the chapter discusses recent advances in the development of hydrochar-based materials for environmental remediation, emphasizing the relationship between synthesis conditions, material properties, and treatment performance. The advantages and limitations of hydrochars are critically evaluated, along with the challenges that must be addressed to facilitate their implementation in practical applications. This chapter serves as a conceptual framework for the research presented throughout this thesis. By integrating current knowledge with the experimental findings obtained during this doctoral work, it establishes the scientific basis for the continued development of advanced carbonaceous materials through transformation and functionalization strategies for visible-light-driven environmental remediation.

Article 5. Lignocellulosic-based hydrochars: Synthesis, characterization and application in water decontamination

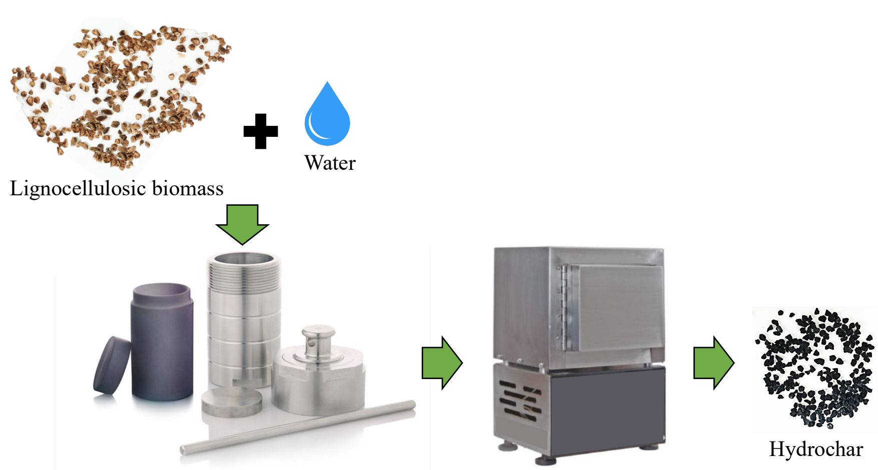
This work was published in the journal *Next Sustainability* under the following reference:

Sánchez-Silva, J. M., Aguilar-Aguilar, A., Sangaré, D., & Ocampo-Pérez, R. (2025). Lignocellulosic-based hydrochars: Synthesis, characterization and application in water decontamination. Next Sustainability, 6. https://doi.org/10.1016/j.nxsust.2025.100150

Article overview

This review article consolidates the scientific knowledge surrounding the production and environmental applications of hydrochars derived from lignocellulosic biomass. The work analyzes the relationships between biomass composition, hydrothermal carbonization conditions, material properties, and water decontamination performance. Particular emphasis is placed on the role of oxygen-containing functional groups and persistent free radicals as key features governing hydrochar reactivity in adsorption and advanced oxidation processes. Furthermore, the article discusses the use of hydrochars in heterogeneous photocatalysis and persulfate activation, highlighting their potential as sustainable materials for environmental remediation. This work provides the theoretical framework for the experimental studies presented in this thesis. It establishes the foundations for the design and functionalization of advanced hydrochar-based materials for visible-light-driven water-treatment applications.

Graphical Abstract



Chapter 5. Functionalization of Clay Supports with Carbon Quantum Dots for Visible-Light Photocatalysis

Chapter overview

This chapter explores advanced functionalization strategies for covalently immobilizing carbon quantum dots (CQDs) onto mineral supports for visible-light-driven environmental remediation. Building on the transformation and functionalization approaches presented in previous chapters, this stage of the research focuses on developing hybrid photocatalytic materials that combine the unique photophysical properties of CQDs with the structural stability and recoverability of naturally abundant clay minerals. The chapter investigates the synthesis of a sepiolite-based hybrid material through surface modification and covalent grafting of CQDs. Particular attention is given to overcoming one of the main limitations of carbon quantum dots: their nanoscale size and colloidal nature, which often hinder recovery and reuse in heterogeneous photocatalytic systems. By immobilizing CQDs onto a solid support, their advantages can be retained while improving material stability and operational applicability.

Comprehensive characterization was performed to assess the successful incorporation of CQDs and their influence on the optical and structural properties of the hybrid material. The photocatalytic performance was assessed by measuring the degradation of various organic dyes under visible-light irradiation, thereby establishing the relationship between material design and photocatalytic activity. This chapter demonstrates that covalent functionalization is an effective strategy for developing advanced hybrid photocatalysts with enhanced visible-light responsiveness. Furthermore, it extends the thesis's scope beyond biomass-derived materials and highlights the versatility of carbon-based nanomaterials for environmental remediation. The findings presented here represent the culmination of the transformation and functionalization approaches explored throughout this doctoral research.

Article 6. Sepiolite-Based Hybrid Photocatalyst via Covalent Immobilization of Carbon Quantum Dots for Visible-Light Water Remediation

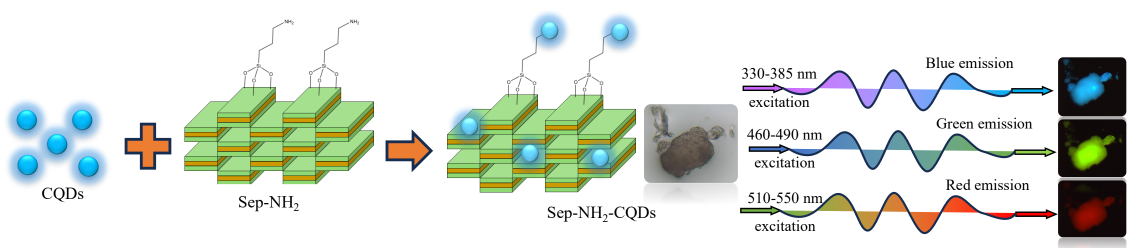
This work was published in the journal *Applied Clay Science* under the following reference:

Sánchez-Silva, J. M., Belmonte-Vázquez, J. L., Carrasco-Marín, F., Ocampo-Pérez, R., Padilla-Ortega, E., & Aguilar-Aguilar, A. (2025). Sepiolite-based hybrid photocatalyst via covalent immobilization of carbon quantum dots for visible-light water remediation. Applied Clay Science, 278, 108019. https://doi.org/10.1016/j.clay.2025.108019

Article overview

This article presents an advanced functionalization strategy that covalently immobilizes carbon quantum dots onto sepiolite, thereby developing a stable hybrid photocatalyst for visible-light-driven water remediation. The study addresses the limitations of CQD recovery and reuse by integrating them into a solid mineral support while preserving their photocatalytic properties. The results demonstrated that the immobilized CQDs enhanced visible-light absorption and promoted the generation of reactive oxygen species, leading to efficient degradation of various organic dyes. Furthermore, the hybrid material exhibited good stability and performance in both single and multicomponent pollutant systems. This work demonstrates the potential of covalent functionalization as an effective approach to designing advanced hybrid photocatalysts. It extends the material development strategies explored throughout this thesis beyond biomass-derived carbonaceous materials to nanostructured carbon-based systems for environmental remediation.

Graphical Abstract



Chapter 6. Expansion of Functional Materials Research: Current Developments and Future Directions

Chapter Overview

This chapter expands the research line established throughout this doctoral work and presents current developments and future research directions arising from the transformation and functionalization strategies explored in previous chapters. While the main body of this thesis focuses on the development of advanced materials for visible-light-driven pollutant degradation, the knowledge generated has enabled the extension of these concepts toward new environmental and agro-food applications. Recent research has explored the integration of carbon-based nanomaterials into biopolymeric systems for postharvest disease management. In particular, studies on bionanocomposite coating technologies and chitosan/carbon dot composite films have demonstrated the potential of functional nanomaterials to improve antimicrobial performance, barrier properties, and ultraviolet protection in food preservation systems. These investigations broaden the scope of application for material functionalization strategies beyond water remediation and highlight the versatility of carbon nanomaterials in sustainable technologies.

Additionally, research conducted within this framework has led to the development of undergraduate projects focused on photocatalytic materials, including carbon-dot-functionalized clays and hydrochar-based photocatalysts for visible-light-driven degradation. These studies have contributed to human resource training and expanded the range of materials and reactor configurations investigated within the research group. Future research efforts will focus on the synthesis of hydrochars derived from tropical wood residues for adsorption and heterogeneous photocatalysis applications, as well as the development of advanced semiconductor–carbon dot composites designed to improve visible-light harvesting, charge separation, and photocatalytic efficiency. These directions represent the natural continuation of the scientific contributions presented throughout this thesis and reinforce the potential of transformation and functionalization strategies for developing advanced materials with environmental applications.

6.1 Expansion toward Agro-Food Applications

The research presented throughout this thesis has primarily focused on the development of advanced materials through transformation and functionalization strategies for environmental remediation. However, the knowledge acquired regarding the synthesis, characterization, and application of carbon-based materials has also enabled the exploration of new research directions beyond water treatment technologies. One of these emerging areas involves the application of functional nanomaterials in postharvest disease management. As with environmental remediation processes, postharvest preservation requires the development of sustainable technologies that control biological degradation while minimizing reliance on conventional chemical agents. In this context, nanomaterials offer unique opportunities due to their tunable physicochemical properties, antimicrobial activity, and compatibility with biopolymeric matrices.

Building on expertise in the synthesis and functionalization of carbon-based nanomaterials, recent research efforts have focused on incorporating carbon dots and other nanostructured materials into biodegradable coatings and films to protect fruits and vegetables. These studies aim to exploit the multifunctional properties of nanomaterials, including ultraviolet shielding, antioxidant activity, antimicrobial activity, and controlled interactions with biological systems.

As part of this research expansion, a comprehensive review titled "*Bionanocomposite Coating Film Technologies for Disease Management in Fruits and Vegetables*" was developed to critically evaluate the current state of the art In nanomaterial-based coatings and films for postharvest applications. The review highlights the potential of combining biopolymers with nanomaterials to create multifunctional systems that can improve fruit quality, extend shelf life, and reduce postharvest losses.

Furthermore, experimental studies were conducted to investigate the incorporation of carbon dots into biopolymeric matrices, resulting in the development of functional bionanocomposite films. In particular, the study entitled "*Physicochemical and In Vitro Antifungal Evaluation of Chitosan/Carbon Dots Bionanocomposite Films*" examined the influence of carbon dots on the physicochemical, optical, barrier, and antifungal properties of chitosan-based films.

The results demonstrated that carbon dot incorporation improved mechanical strength, thermal stability, water-vapor barrier performance, and ultraviolet protection, while also providing significant antifungal activity against *Botrytis cinerea*. These findings provide valuable insights into the behavior of carbon dot-based bionanocomposites and demonstrate their potential as sustainable materials for postharvest disease management.

Collectively, these studies demonstrate the versatility of carbon-based nanomaterials and reveal new opportunities for applying transformation and functionalization concepts in sustainable agro-food technologies. Moreover, they establish a new research direction that complements the environmental remediation focus of this thesis and will continue to be explored through the development of nanomaterial-based systems for postharvest preservation and disease control.

6.2 Human Resource Training and Research Consolidation

The research presented in this thesis has not only advanced scientific knowledge in the fields of advanced materials and environmental remediation but has also facilitated the training of undergraduate students and the consolidation of new research topics within the group.

The methodologies, synthesis routes, and characterization strategies established during this doctoral work have served as the foundation for several undergraduate research projects focused on developing visible-light-responsive materials for environmental applications. These projects have expanded the scope of the original research by exploring alternative supports, functionalization approaches, and photocatalytic reactor configurations. Through these activities, students have gained experience in material synthesis, physicochemical characterization, photocatalytic evaluation, and data analysis, contributing to the dissemination and continuity of the research line.

Among the undergraduate projects developed under this framework, the thesis titled "*Synthesis and Application of Bentonite-NH_2-CDs for Visible Photodegradation of Methylene Blue*" investigated the functionalization of clay minerals with carbon dots to obtain hybrid photocatalytic materials that operate under visible-light irradiation. This work extended the concepts explored in Chapter 6 by evaluating alternative clay supports and further examining the role of carbon dots in photocatalytic processes. A second project, entitled "*Hybrid Hydrochar-TiO_2 Photocatalysts: Design, Immobilization and Performance in Continuous-Flow Visible Photodegradation*", focused on the immobilization of hydrochar-based photocatalysts for continuous-flow operation. This study represents an important step toward the practical implementation and scale-up of hydrochar-semiconductor composites developed in Chapters 3 and 4.

Finally, the thesis entitled "*Functionalization of Sepiolite with Carbon Dots and Its Application in Visible Photodegradation of Dyes*" further investigated the synthesis and performance of sepiolite-based CDs-Chitosan composites, contributing to the optimization and understanding of the hybrid materials introduced in Chapter 6.

Collectively, these projects demonstrate the continuity and growth of the research line established during this doctoral work. Moreover, they highlight the potential of transformation and functionalization strategies to generate new scientific questions, support student training, and foster the development of advanced materials for environmental applications.

6.3 Future Research Directions

The research presented in this thesis has demonstrated the potential of transformation and functionalization strategies for developing advanced materials for environmental remediation and, more recently, for postharvest disease management. Nevertheless, several scientific and technological challenges remain to be addressed, providing opportunities for future research and innovation. One of the primary research directions involves the continued development of hydrochars derived from lignocellulosic biomass, particularly those obtained from tropical wood residues. Future studies will focus on understanding the relationships among biomass composition, hydrothermal carbonization conditions, and material properties to optimize performance in adsorption and heterogeneous photocatalysis. Particular attention will be given to identifying active surface sites, charge-transfer mechanisms, and the generation of reactive oxygen species under visible-light irradiation.

Another promising direction concerns the design of hybrid semiconductor–carbon dot composites. Carbon dots have demonstrated significant potential as photosensitizers and electron-transfer mediators, making them attractive components for the development of next-generation photocatalysts. Future work will investigate the integration of carbon dots with various semiconductor materials to improve visible-light harvesting, charge-separation efficiency, and photocatalytic performance for the degradation of emerging contaminants. In parallel, future studies will continue exploring the application of carbon-based nanomaterials in postharvest disease management.

The incorporation of carbon dots and other functional nanomaterials into biodegradable coatings and films offers opportunities to develop multifunctional systems that combine antimicrobial activity, ultraviolet protection, controlled release of active agents, and shelf-life extension. These efforts aim to provide sustainable alternatives to conventional postharvest treatments while expanding the scope of application for the materials developed in this research. Overall, these future research directions represent the natural continuation of the scientific contributions presented in this thesis and reinforce the versatility of advanced carbon-based materials for addressing environmental and agro-food challenges through sustainable technological solutions.

6. General conclusions

This doctoral research successfully achieved its objective of developing advanced materials through sequential transformation and functionalization strategies for the visible-light-driven photodegradation of organic dyes. By integrating thermochemical biomass characterization, hydrothermal conversion, semiconductor functionalization, and carbon nanomaterial engineering into a single research framework, this work establishes a comprehensive approach for the strategic design of sustainable photocatalytic materials derived from renewable resources. The results presented throughout the thesis demonstrate that material performance is governed not only by the intrinsic properties of individual components but also by the structural and electronic modifications introduced during successive transformation and functionalization processes.

The first scientific contribution of this thesis is the establishment of lignocellulosic biomass as a suitable precursor for the development of advanced carbonaceous materials. Through detailed thermochemical characterization and kinetic analysis, the relationship between biomass composition, thermal decomposition behavior, and its potential for conversion into functional carbon materials was elucidated. These findings provide a scientific basis for precursor selection and process optimization, reinforcing the importance of biomass characterization as the first step in the strategic development of carbon-based materials.

A second and particularly significant contribution of this work is the demonstration that hydrochars produced by hydrothermal carbonization are intrinsically active photocatalytic materials rather than passive carbon supports. The results provide experimental evidence that hydrochars can generate reactive oxygen species under visible-light irradiation via structural features inherent to their carbonaceous framework, including oxygen-containing functional groups, conjugated carbon domains, and persistent free radicals. This finding advances the current understanding of hydrochar chemistry by demonstrating that photocatalytic activity can arise directly from biomass-derived carbonaceous structures, without the need for conventional semiconductor materials. Consequently, this work expands the role of hydrochars in heterogeneous photocatalysis and establishes them as a new class of sustainable, metal-free photocatalysts for advanced oxidation processes.

Beyond demonstrating photocatalytic activity, this thesis contributes to the mechanistic understanding of hydrochar-based systems by establishing structure-property-performance relationships that connect synthesis conditions, physicochemical characteristics, and photocatalytic behavior. The results reveal that hydrothermal carbonization conditions directly influence the formation of active structural features responsible for visible-light activity, providing valuable insights into tailoring hydrochar properties for specific environmental applications. This mechanistic framework constitutes an important scientific contribution because it provides design criteria for developing biomass-derived photocatalytic materials based on their intrinsic structural characteristics rather than through empirical optimization alone.

The functionalization strategies investigated throughout this work further demonstrate that the photocatalytic performance of carbonaceous materials can be significantly enhanced through rational interface engineering. The incorporation of TiO_2 and CeO_2 into hydrochar matrices generated hybrid photocatalysts in which synergistic interactions promoted improved visible-light harvesting, more efficient charge separation, reduced electron–hole recombination, and enhanced reactive oxygen species generation. These findings confirm that hydrochar functions not only as a structural support but also as an active component capable of participating in interfacial charge-transfer processes.

Consequently, this work contributes to the understanding of hydrochar–semiconductor interactions and offers new perspectives on the design of high-performance hybrid photocatalysts. An additional and original contribution of this doctoral research Is the development of a covalent immobilization strategy for carbon quantum dots on an inorganic mineral support. By successfully integrating CQDs onto sepiolite via covalent functionalization, this work addresses one of the principal limitations of carbon quantum dots, namely their difficulty in recovery and reuse in heterogeneous systems. The resulting hybrid material preserves the advantageous optical and photocatalytic properties of CQDs while providing improved operational stability and recyclability.

This strategy extends the scope of the thesis beyond biomass-derived materials. It demonstrates that rational functionalization can also be applied to nanostructured carbon materials, opening new possibilities for the development of robust heterogeneous photocatalysts.

Collectively, the studies presented in this thesis demonstrate that transformation and functionalization are complementary strategies for engineering advanced carbon-based materials with progressively enhanced functionality. Starting with agro-industrial biomass, the research advances through the production of hydrochars, the development of hydrochar–semiconductor composites, and, finally, the design of carbon quantum dot-based hybrid systems, illustrating a coherent evolution toward increasingly sophisticated photocatalytic materials. This progression reflects not only technological development but also a systematic effort to understand how structural modifications at different length scales influence photocatalytic behavior.

From an application perspective, the materials developed exhibited high photocatalytic activity under visible-light irradiation, efficient degradation of representative organic dyes, good structural stability, and the potential for reuse under heterogeneous operating conditions. Moreover, the use of renewable agro-industrial residues as precursors contributes to waste valorization and supports the implementation of circular economy principles by generating high-value functional materials for environmental remediation.

Overall, this doctoral thesis provides a significant scientific contribution to the fields of biomass valorization, hydrothermal carbonization, heterogeneous photocatalysis, and carbon-based functional materials. The knowledge generated advances the understanding of hydrochars as intrinsically active photocatalytic materials, establishes fundamental structure-property-performance relationships governing visible-light activity, and introduces a novel covalent immobilization strategy for carbon quantum dots on inorganic supports. Together, these contributions provide a solid scientific foundation for the strategic development of next-generation sustainable photocatalytic materials and open new research opportunities in advanced oxidation processes, environmental remediation, and the broader field of functional carbon materials.

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8. Appendix

8.1 Published articles

8.1.1 First author publications

Article 1. Sanchez-Silva, J. M., Ocampo-Pérez, R., Padilla-Ortega, E., Sangaré, D., Escobedo-Bretado, M. A., Domínguez-Arvizu, J. L., Hernández-Majalca, B. C., Salinas-Gutiérrez, J. M., López-Ortiz, A., & Collins-Martínez, V. (2023). Pyrolysis kinetics of Byrsonima crassifolia stone as agro-industrial waste through isoconversional models. Molecules, 28(2), 544. https://doi.org/10.3390/molecules28020544.

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Article

Pyrolysis Kinetics of *Byrsonima crassifolia* Stone as Agro-Industrial Waste through Isoconversional Models

Jonathan M. Sanchez-Silva [1], Raúl Ocampo-Pérez [1], Erika Padilla-Ortega [1], Diakaridia Sangaré [2], Miguel A. Escobedo-Bretado [3], Jorge L. Domínguez-Arvizu [4], Blanca C. Hernández-Majalca [4], Jesús M. Salinas-Gutiérrez [4], Alejandro López-Ortiz [4,*] and Virginia Collins-Martínez [4]

Article 2. Sánchez-Silva, J. M., Sangaré, D., Belmonte-Vázquez, J. L., Aguilar-Aguilar, A., Padilla-Ortega, E., González-Chávez, R., & Ocampo-Pérez, R. (2025). Hydrochar of Prunus persica: Green promoter of radical species to degrade methylene blue with visible irradiation. Environmental Science and Pollution Research, 32, 8481–8497. https://doi.org/10.1007/s11356-025-36214-9

Environmental Science and Pollution Research
https://doi.org/10.1007/s11356-025-36214-9

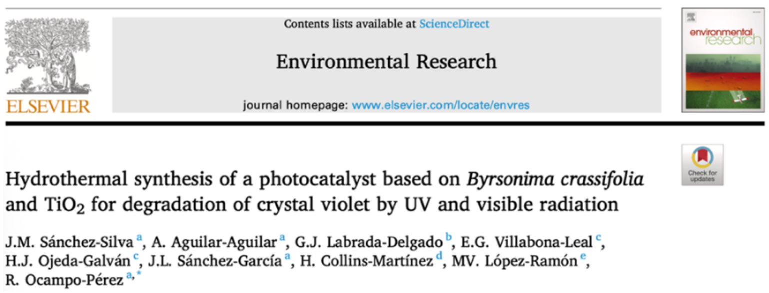
RESEARCH ARTICLE

Check for updates

Hydrochar of *Prunus persica:* green promoter of radical species to degrade methylene blue with visible irradiation

Jonathan Michel Sánchez-Silva [1] · Diakaridia Sangaré [2] · José Luis Belmonte-Vázquez [3] · Angelica Aguilar-Aguilar [1] · Erika Padilla-Ortega [1] · Rodolfo González-Chávez [1] · Raúl Ocampo-Pérez [1]

Article 3. Sánchez-Silva, J. M., Aguilar-Aguilar, A., Labrada-Delgado, G. J., Villabona-Leal, E. G., Ojeda-Galván, H. J., Sánchez-García, J. L., Collins-Martínez, H., López-Ramón, M. V., & Ocampo-Pérez, R. (2023). Hydrothermal synthesis of a photocatalyst based on Byrsonima crassifolia and TiO_2 for degradation of crystal violet by UV and visible radiation. Environmental Research, 231(3). https://doi.org/10.1016/j.envres.2023.116280


Contents lists available at ScienceDirect

Environmental Research

journal homepage: www.elsevier.com/locate/envres

Hydrothermal synthesis of a photocatalyst based on *Byrsonima crassifolia* and TiO_2 for degradation of crystal violet by UV and visible radiation

J.M. Sánchez-Silva[a], A. Aguilar-Aguilar[a], G.J. Labrada-Delgado[b], E.G. Villabona-Leal[c], H.J. Ojeda-Galván[c], J.L. Sánchez-García[a], H. Collins-Martínez[d], MV. López-Ramón[e], R. Ocampo-Pérez[a,*]

Article 4. Sánchez-Silva, J. M., Ojeda-Galván, H. J., Villabona-Leal, E. G., Labrada-Delgado, G. J., Aguilar-Maruri, S. A., Fuentes-Ramírez, R., González-Ortega, O., López-Ramón, M. V., & Ocampo-Pérez, R. (2024). Synergistic photocatalysis of a hydrochar/CeO_2 composite for dye degradation under visible light. Environmental Science and Pollution Research, 31, 16453–16472. https://doi.org/10.1007/s11356-024-32281-6


Environmental Science and Pollution Research
https://doi.org/10.1007/s11356-024-32281-6

RESEARCH ARTICLE

Synergistic photocatalysis of a hydrochar/CeO_2 composite for dye degradation under visible light

Jonathan Michel Sánchez-Silva[1] · Hiram Joazet Ojeda-Galván[2] · Edgar Giovanny Villabona-Leal[2] · Gladis Judith Labrada-Delgado[3] · Saul Alejandro Aguilar-Maruri[1] · Rosalba Fuentes-Ramírez[4] · Omar González-Ortega[1,2] · María Victoria López-Ramón[5] · Raúl Ocampo-Pérez[1]

Article 5. Sánchez-Silva, J. M., Aguilar-Aguilar, A., Sangaré, D., & Ocampo-Pérez, R. (2025). Lignocellulosic-based hydrochars: Synthesis, characterization and application in water decontamination. Next Sustainability, 6. https://doi.org/10.1016/j.nxsust.2025.100150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Next Sustainability

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Lignocellulosic-based hydrochars: Synthesis, characterization and application in water decontamination

Jonathan Michel Sánchez-Silva [a,*], Angélica Aguilar-Aguilar [a], Diakaridia Sangaré [b], Raúl Ocampo-Pérez [a]

Article 6. Sánchez-Silva, J. M., Belmonte-Vázquez, J. L., Carrasco-Marín, F., Ocampo-Pérez, R., Padilla-Ortega, E., & Aguilar-Aguilar, A. (2025). Sepiolite-based hybrid photocatalyst via covalent immobilization of carbon quantum dots for visible-light water remediation. Applied Clay Science, 278, 108019. https://doi.org/10.1016/j.clay.2025.108019


Contents lists available at ScienceDirect

Applied Clay Science

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Research Paper

Sepiolite-based hybrid photocatalyst via covalent immobilization of carbon quantum dots for visible-light water remediation

J.M. Sánchez-Silva [a], J.L. Belmonte-Vázquez [b], F. Carrasco-Marín [c], R. Ocampo-Pérez [a], E. Padilla-Ortega [a,*], A. Aguilar-Aguilar [a,*]

Article 7. Sánchez-Silva, J. M., López-García, U. M., Gutierrez-Martinez, P., Flores-Ramírez, A. Y., Ramos-Bell, S., Moreno-Hernández, C., Rivas-García, T., & González-Estrada, R. R. (2025). Bionanocomposite coating film technologies for disease management in fruits and vegetables. Horticulturae, 11(7), 832. https://doi.org/10.3390/horticulturae11070832

horticulturae MDPI

Review

Bionanocomposite Coating Film Technologies for Disease Management in Fruits and Vegetables

Jonathan M. Sánchez-Silva [1], Ulises M. López-García [2], Porfirio Gutierrez-Martinez [2], Ana Yareli Flores-Ramírez [2], Surelys Ramos-Bell [2], Cristina Moreno-Hernández [2], Tomás Rivas-García [3] and Ramsés Ramón González-Estrada [2,*]

Article 8. Physicochemical and In Vitro Antifungal Evaluation of chitosan/carbon dots Bionanocomposite Films. *BioNanoScience*, 2026 “En revisión”

8.1.2 Collaborative articles

Article 1. Mota-Resendiz, K., **Sánchez-Silva, J. M.**, Forgionny, A., Medellín-Castillo, N. A., Labrada-Delgado, G. J., & Ocampo-Pérez, R. (2025). Valorization of waste cigarette butts into high-performance activated carbons for water remediation. Journal of Water Process Engineering, 75, 107998. https://doi.org/10.1016/j.jwpe.2025.107998

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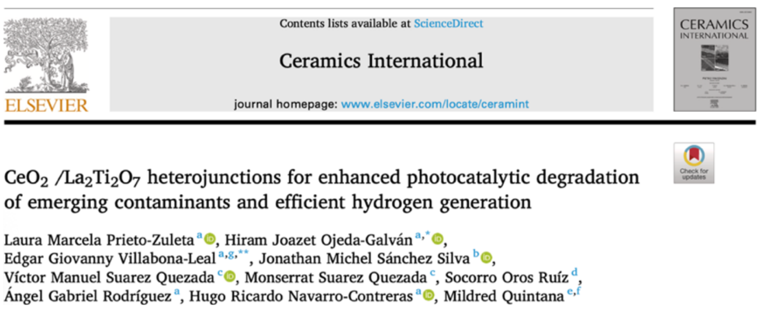
Journal of Water Process Engineering

journal homepage: www.elsevier.com/locate/jwpe

Valorization of waste cigarette butts into high-performance activated carbons for water remediation

Karina Mota-Resendiz [a], Jonathan Michel Sánchez-Silva [a], Angélica Forgionny [b,*], Nahum Andrés Medellín-Castillo [c,d], Gladis Judith Labrada-Delgado [e], Raúl Ocampo-Pérez [a,*]

Article 2. Prieto-Zuleta, L. M., Ojeda-Galván, H. J., Villabona-Leal, E. G., **Sánchez Silva, J. M.**, Suarez Quezada, V. M., Suarez Quezada, M., Oros Ruíz, S., Rodríguez, Á. G., Navarro-Contreras, H. R., & Quintana, M. (2025). $CeO_2/La_2Ti_2O_7$ heterojunctions for enhanced photocatalytic degradation of emerging contaminants and efficient hydrogen generation. Ceramics International, 51(23, Part A), 39029–39045. https://doi.org/10.1016/j.ceramint.2025.06.143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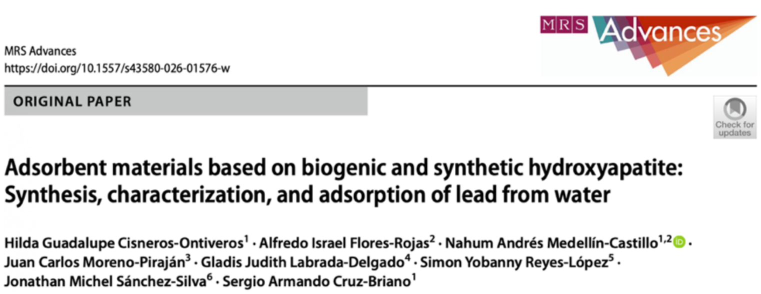
CeO_2 /$La_2Ti_2O_7$ heterojunctions for enhanced photocatalytic degradation of emerging contaminants and efficient hydrogen generation

Laura Marcela Prieto-Zuleta [a], Hiram Joazet Ojeda-Galván [a,*], Edgar Giovanny Villabona-Leal [a,g,**], Jonathan Michel Sánchez Silva [b], Víctor Manuel Suarez Quezada [c], Monserrat Suarez Quezada [c], Socorro Oros Ruíz [d], Ángel Gabriel Rodríguez [a], Hugo Ricardo Navarro-Contreras [a], Mildred Quintana [e,f]

Article 3. Cisneros-Ontiveros, H. G., Flores-Rojas, A. I., Medellín-Castillo, N. A., Moreno-Piraján, J. C., Labrada-Delgado, G. J., Reyes-López, S. Y., **Sánchez-Silva, J. M.**, & Cruz-Briano, S. A. (2026). Adsorbent materials based on biogenic and synthetic hydroxyapatite: Synthesis, characterization, and adsorption of lead from water. MRS Advances, 11, 187–194. https://doi.org/10.1557/s43580-026-01576-w

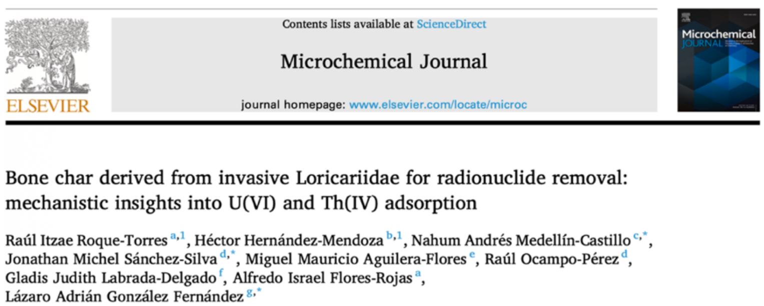

MRS Advances
https://doi.org/10.1557/s43580-026-01576-w

ORIGINAL PAPER

Adsorbent materials based on biogenic and synthetic hydroxyapatite: Synthesis, characterization, and adsorption of lead from water

Hilda Guadalupe Cisneros-Ontiveros[1] · Alfredo Israel Flores-Rojas[2] · Nahum Andrés Medellín-Castillo[1,2] · Juan Carlos Moreno-Piraján[3] · Gladis Judith Labrada-Delgado[4] · Simon Yobanny Reyes-López[5] · Jonathan Michel Sánchez-Silva[6] · Sergio Armando Cruz-Briano[1]

Article 4. Roque-Torres, R. I., Hernández-Mendoza, H., Medellín-Castillo, N. A., **Sánchez-Silva, J. M.**, Aguilera-Flores, M. M., Ocampo-Pérez, R., Labrada-Delgado, G. J., Flores-Rojas, A. I., & González Fernández, L. A. (2026). Bone char derived from invasive Loricariidae for radionuclide removal: Mechanistic insights into U(VI) and Th(IV) adsorption. Microchemical Journal, 227, 118690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Microchemical Journal

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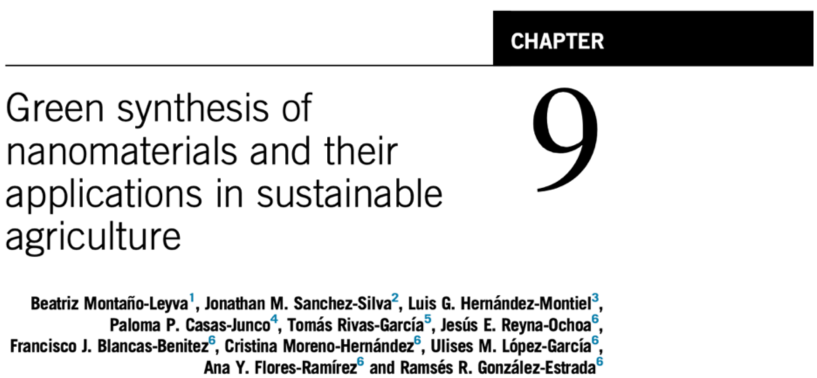
Bone char derived from invasive Loricariidae for radionuclide removal: mechanistic insights into U(VI) and Th(IV) adsorption

Raúl Itzae Roque-Torres [a,1], Héctor Hernández-Mendoza [b,1], Nahum Andrés Medellín-Castillo [c,*], Jonathan Michel Sánchez-Silva [d,*], Miguel Mauricio Aguilera-Flores [e], Raúl Ocampo-Pérez [d], Gladis Judith Labrada-Delgado [f], Alfredo Israel Flores-Rojas [a], Lázaro Adrián González Fernández [g,*]

8.1.3 Co-authored book chapters

Book-Chapter 1: Green synthesis of nanomaterials and their applications in sustainable agriculture. *Sustainable Agricultural Practices Plant and Soil Microbiome*, Academic Press, 2024, 185-208. https://doi.org/10.1016/B978-0-443-19150-3.00009-6

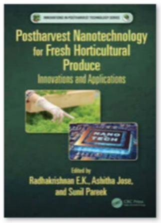

CHAPTER

9

Green synthesis of nanomaterials and their applications in sustainable agriculture

Beatriz Montaño-Leyva[1], Jonathan M. Sanchez-Silva[2], Luis G. Hernández-Montiel[3], Paloma P. Casas-Junco[4], Tomás Rivas-García[5], Jesús E. Reyna-Ochoa[6], Francisco J. Blancas-Benitez[6], Cristina Moreno-Hernández[6], Ulises M. López-García[6], Ana Y. Flores-Ramírez[6] and Ramsés R. González-Estrada[6]

Book-Chapter 2: Advancements in the Essential Oil-Based Packaging Materials. *Postharvest Nanotechnology for Fresh Horticultural Produce Innovations and Applications*, CRC Press, 2023, https://doi.org/10.1201/9781003142287-10



Chapter

Advancements in the Essential Oil-Based Packaging Materials

By Francisco J. Blancas-Benitez, Luis Guillermo Hernández-Montiel, Jonathan Michel Sanchez-Silva, Cristina Moreno-Hernández, P. Gutiérrez-Martinez, Héctor J. Cortés-Rivera, Lizet Aguirre-Güitrón, Surelys Ramos-Bell, Ramsés R. González-Estrada

Book-Chapter 3: Properties of biopolymer films and coatings in Natural Polymer-based Films and Coatings in Food Technology, Postharvest Nanotechnology for Fresh Horticultural Produce Innovations and Applications, 2026.

Natural Polymer-Based Films and Coatings in Food Technology

1st Edition - September 1, 2026 • Latest edition • Imprint: Elsevier

Editors: Ewelina Jamróz, Swarup Roy • Language: English • Paperback ISBN: 9780443342448

eBook ISBN: 9780443342455

Book-Chapter 4: Current research on Biochar and its impact on plant-microbe interactions. Plant-Microbe Synergies for Climate-Smart Agriculture. 2026, “En revisión”.

8.2 Scientific Dissemination and Outreach Activities

8.2.1 Oral presentations

1. 2.º Congreso Internacional de Estudiantes de Posgrado en Ingeniería Química, Benemérita Universidad Autónoma de Puebla, 2022.
2. Desarrollo de materiales verdes a partir de biomasas y su aplicación en la remoción de contaminantes del agua, Instituto Tecnológico de Tepic, 2023.
3. 2.ª Jornada Académica del Día del Químico, Facultad de Ciencias Químicas, UASLP, 2023.
4. "6ta Jornada Académica de Ingeniería Química y Bioquímica, Panel de egresados, Instituto Tecnológico de Tepic, 2023.
5. 4to. Coloquio Nacional Tendencias en el desarrollo de los materiales: hacia los procesos sostenibles, Universidad Juárez Autónoma de Tabasco, 2024.
6. XLI Semana del Químico, Universidad de Colima, 2024.
7. Reunión Anual de la AMEPAL 2025, UASLP, 2025.
8. 4.º Congreso de la Asociación Mexicana de Carbono, IPICYT, 2025.
9. XXIII Congreso Nacional de Ciencias Químico-Biológicas "Arcana", Universidad de las Américas Puebla, 2026.
10.

8.2.2 Poster presentations

1. Latin American Conference on Environmental and Chemical Process Systems Engineering, 2023.
2. Congreso Internacional sobre Investigación en STEM y Ciencias Sociales: Desafíos Transdisciplinarios Actuales, UASLP, 2024.
3. Primer Simposio Nacional de Investigación e Innovación en Nanociencias, UASLP, 2024.
4. Latin American Conference on Environmental and Chemical Process Systems Engineering, 2024.
5. LatinXChem, 2024.
6. Semana de Posgrado UASLP, 2025.
7. Tercer Congreso Nacional de Nanotecnología y Energías Renovables, CIACYT-UASLP, 2025.
8. Latin American Conference on Environmental and Chemical Process Systems Engineering, 2025.

8.2.3 Awards

1. Best poster award in the category #PSEBiomassOEI, 5—Latin American Conference on Environmental and Chemical Process Systems Engineering, 2024.
2. 2.º Lugar en cartel científico, Tercer Congreso Nacional de Nanotecnología y Energías Renovables, CIACYT-UASLP, 2025.
3.

8.2.4 Reviewer activity